NANOSCALE PRECIPITATION IN HOT ROLLED SHEET STEEL

by

Jun Sun
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Golden, CO
Date ____________

Signed: ________________
Jun Sun

Signed: ________________
Dr. John G. Speer
Advisor

Golden, CO
Date ____________

Signed: ________________
Dr. Michael J. Kaufman
Professor and Head
Department of Metallurgical and Materials Engineering
Some newer hot rolled high strength low alloy (HSLA) steels with a single phase ferrite matrix have obtained substantial strengthening from nanoscale precipitation. These HSLA are reported to have a good combination of strength, ductility and hole-expansion ability. In the current work, Gleeble® 3500 torsion testing was employed to simulate the hot rolling process with varying run-out table cooling rates and coiling temperatures on five microalloyed steels with additions of Ti, Nb, Mo, Cr and V, to investigate the effects of microalloy additions and processing conditions on microstructures as well as mechanical properties. Subsized tensile specimens obtained from as-twisted torsion samples were used to evaluate mechanical properties. The precipitation states of the five steels with different processing conditions were characterized using extraction replica TEM. Comparison of microstructures and mechanical properties was discussed.

Characterization of the microstructure via light optical microscopy showed the matrix microstructure was mainly influenced by coiling temperature, which indicates that the transformation from austenite to ferrite occurred during the coiling period. A higher Ti content was shown to reduce the second constituent fractions. Investigation of carbon extraction replica specimens via TEM revealed the presence of nanoscale precipitation. Extensive nanoscale precipitation was observed in most of the specimens having a polygonal ferrite matrix, while in the granular bainite/ferrite microstructure at lower temperatures, fewer microalloy carbides were present. The specimens with polygonal ferrite had similar or higher yield strength than the specimens with granular bainite microstructure, which suggests the effectiveness of precipitation strengthening from extensive nanoscale precipitates. In the Nb-Mo steel, more significant strengthening due to grain refinement was evident. Yield strength values were less than reported for JFE’s “NANOHITEN®” steel in specimens with similar microstructure (polygonal ferrite matrix with extensive nanoscale precipitation).
# TABLE OF CONTENTS

ABSTRACT .............................................................................................................................................. iii

LIST OF FIGURES ........................................................................................................................................ vi

LIST OF TABLES .......................................................................................................................................... xii

ACKNOWLEDGEMENTS ............................................................................................................................ xiii

CHAPTER 1 : INTRODUCTION ......................................................................................................................... 1

CHAPTER 2 : BACKGROUND .......................................................................................................................... 3

2.1 Review of Nanoscale Precipitation Strengthened Steels ........................................................................... 3

2.1.1 Microstructure Characterization of “NANOHITEN®” ............................................................................. 3

2.1.2 Properties of NANOHITEN® .................................................................................................................. 4

2.1.3 Alloying Effects in NANOHITEN® ......................................................................................................... 6

2.2 Precipitation in Steels ............................................................................................................................... 6

2.2.1 Precipitation in Austenite ....................................................................................................................... 7

2.2.2 Precipitation During Decomposition of the Austenite .......................................................................... 7

2.2.3 Precipitation from Supersaturated Ferrite ........................................................................................... 12

2.3 Precipitation Strengthening Mechanism .................................................................................................. 13

2.4 Thermomechanical Processing and Laboratory Simulation of HSLA Steels ............................................ 14

2.4.1 Slab Reheating Temperature .................................................................................................................. 14

2.4.2 Rolling Schedule .................................................................................................................................. 16

2.4.3 Cooling Rate and Coiling Temperature ................................................................................................... 17

2.4.4 Laboratory Simulation of Thermomechanical Processing ................................................................... 18

CHAPTER 3 : EXPERIMENTAL DESIGN ......................................................................................................... 21

3.1 Objectives .............................................................................................................................................. 21

3.2 Gleeble® 3500 Torsion Testing .............................................................................................................. 21

3.2.1 Hot Torsion Test Sample Development ............................................................................................... 22

3.2.2 Modification of Torsion Specimen Geometry ....................................................................................... 22

3.2.3 Characterization of Thermal Gradients ............................................................................................... 24

3.2.4 Temperature Monitoring and Control During Hot Torsion Testing ..................................................... 27

3.3 Validation of Subsized Tensile Sample Geometry for Mechanical Property Evaluation .................... 28

CHAPTER 4 : EXPERIMENTAL METHODS ..................................................................................................... 31

4.1 Materials ............................................................................................................................................... 31
4.2 Gleeble® 3500 Torsion Testing ................................................................. 31
4.3 Microstructure Characterization ................................................................. 34
4.4 Mechanical Evaluation ............................................................................... 35

CHAPTER 5 : RESULTS AND DISCUSSION ........................................................................... 36
5.1 Gleeble® 3500 Torsion Testing Evaluation ..................................................... 36
  5.1.1 Simulation of Hot Rolling ........................................................................ 36
  5.1.2 Effects of Strain Variation ...................................................................... 38
  5.1.3 Mechanical Properties of Subsized Tensile Specimens .......................... 39
5.2 Matrix Microstructure Characterization via LOM .......................................... 44
  5.2.1 Effects of Processing Conditions on the Matrix Microstructure .......... 44
  5.2.2 Effects of Microalloy Additions on Matrix Microstructure ................. 54
5.3 Precipitate Characterization ....................................................................... 57
5.4 General Discussion ..................................................................................... 66

CHAPTER 6 : CONCLUSIONS .................................................................................. 72

CHAPTER 7 : FUTURE WORK ................................................................................. 74

REFERENCES ........................................................................................................ 75

APPENDIX A. GLEEBLE® 3500 TORSION TESTING ................................................ 81
A.1 Modified Gleeble Torsion Specimen Geometry .......................................... 81
A.2 Gleeble® 3500 Torsion Testing Program Setup ........................................... 81

APPENDIX B. MECHANICAL PROPERTIES .......................................................... 84
B.1 Microhardness Testing Results .................................................................... 84
B.2 Engineering Stress-Strain Curves for Subsized Tensile Specimens ............ 84
LIST OF FIGURES

Figure 1.1 Influence of microstructure of steel matrix on the balance of hole-expansion ratio and elongation [4].................................................................2

Figure 2.1 SEM image of ferrite matrix (a) and TEM image of uniformly distributed precipitates (b) in NANOHITEN® [4]. .................................................................4

Figure 2.2 Calculated amount of solid solution strengthening, precipitation strengthening and grain refinement strengthening in NANOHITEN® [6]. ..5

Figure 2.3 TEM micrograph showing interphase precipitation in a solution treated niobium steel [24]. ....................................................................................8

Figure 2.4 TEM images of thin foil specimens showing (a) planar interphase precipitation, (b) curved interphase precipitation with uniform sheet spacing, (c) curved interphase precipitation with random sheet spacing and (d) fibrous interphase precipitation [33, 35]. ............................................9

Figure 2.5 Schematic representation of the mechanism of nucleation and growth of carbides on the austenite/ferrite interface. (a) regular ledge height, (b) irregular ledge height. Black dots in the figure indicate the precipitate positions [37]. .........................................................................................10

Figure 2.6 Generic CCT diagram for microalloyed steel indicating approximate regions of interphase and random precipitation in matrix [42]......................12

Figure 2.7 A TEM bright field image shows the randomly distributed precipitates in ferritic matrix of a Ti-microalloyed ferritic HSLA steel [44].........................13

Figure 2.8 Layout of a semi-continuous hot strip mill [46]. .................................................14

Figure 2.9 Calculated carbide/nitride solubility in austenite for (a) TiC, (b) VC, (c) NbC, (d) TiN, (e) VN and (f) NbN at 950, 1100 and 1250 °C. ..................15

Figure 2.10 Schematic temperature-time diagram showing three rolling types: (a) recrystallization controlled rolling, (b) conventional controlled rolling and (c) dynamic recrystallization controlled rolling [48]. ..............................17

Figure 2.11 CCT diagram for an HSLA steel containing 0.06C, 1.45Mn, 1.25Cu, 0.97Ni, 0.72Cr, and 0.42Mo, in wt pct. PF, polygonal ferrite; WF, Widmanstätten ferrite; AF, acicular ferrite; GF, granular ferrite [51]. ..........19
Figure 3.1 Stress-strain curves for comparison of tensile sample geometries. (a) low carbon steel (0.056C-1.16Mn-0.01Si in wt pct) and (b) TRIP steel (0.19 C-1.56 Mn-1.61 Si-0.051Al in wt pct) [71].

Figure 3.2 Modified torsion specimens. (a): Standard torsion specimen and (b): Modified torsion specimen, changing part of the hollow grip to solid to allow for tensile specimen grip ends; The dashed lines indicate holes drilled into the specimen.

Figure 3.3 Preparation of subsized samples and microstructure analysis piece from modified torsion specimen, with the top and bottom drawing showing the plan view and elevation view, respectively.

Figure 3.4 Schematic illustration of thermal gradient test setup. Thermocouples TC1, TC2, TC3 were welded at positions 0, 5, 10 mm away from the center of gage length, respectively.

Figure 3.5 Thermal gradient measurements for (a) standard (Figure 3.2 a) and (b) modified torsion specimens (Figure 3.2 b).

Figure 3.6 Thermal gradient test results for the “0-thrust” setup on modified specimen. TC1 and TC2 were welded at the same position as shown in Figure 3.4.

Figure 3.7 Dimensions of specimen in mm used for Gleeble® 3500 torsion simulations.

Figure 3.8 Two color optical pyrometer installed on the Gleeble® 3500.

Figure 3.9 Thermocouple control along with pyrometer control of the temperature during hot torsion testing.

Figure 3.10 Engineering stress-strain curves for subsized tensile specimens and normal size specimens.

Figure 4.1 Schematic illustration of thermomechanical profile used for torsion testing.

Figure 4.2 Specimen preparation for microstructure analysis, using coupons positioned as shown in Figure 3.3.

Figure 5.1 Thermal profile for torsion testing of Ti-Cr steel, with (a) cooling rate of 40 °C/s, coiling temperature of 650 °C and (b) cooling rate of 40 °C/s, coiling temperature of 550 °C. Temperatures below 600 °C are recorded as 600 °C by the pyrometer.
Figure 5.2  Schematic showing of shear strain variation over the cross section of torsion specimen. (a) Schematic drawing of twisted specimen and (b) strain states over the cross section []. a: radius of torsion specimen; θ: twisted angle; γ: shear strain; r: radius at random position; MT: torque; τ: shear stress. .........................................................38

Figure 5.3  LOM images showing the effects of strain variation upon the cross section of gage section with (a) obtained from surface and (b) center of cross section. (High-Ti steel, with cooling rate of 40 °C/s, coiling temperature of 650 °C)..............................................................................................38

Figure 5.4  Stress-strain curves of duplicates subsized tensile specimens: (a) Ti-Mo specimens coiled at 650 °C with cooling rate of 40 °C/s and (b) Nb-Mo specimens coiled at 650 °C with cooling rate of 20 °C/s........................................41

Figure 5.5  Tested V-Mo steel subsized tensile specimen coiled at 650 °C with cooling rate of 20 °C/s. Fracture was initiated at the location indicated. ......41

Figure 5.6  Tested subsized tensile specimen of Nb-Mo steel coiled at 600 °C with cooling rate of 40 °C/s. A crack on the specimen surface is indicated............42

Figure 5.7  (a) SEM image showing fracture surface of V-Mo subsized tensile specimen (coiled at 650 °C with cooling rate of 20 °C/s) and (b) EDS analysis for chemical composition of the mixed oxide inclusion, which is pointed out in (a)... .................................................................42

Figure 5.8  Stress-strain curves of duplicate subsized tensile specimens from V-Mo specimen coiled at 650 °C with cooling rate of 20 °C/s. Curve 2 corresponds to the specimen shown in Figure 5.5. The specimen shown in curve 1 was detected with surface defects which affected ductility measurement. .........................................................................................43

Figure 5.9  Stress-strain curves of duplicate subsized tensile specimens from Nb-Mo specimen coiled at 600 °C with cooling rate of 40 °C/s. Curve 2 corresponds to the specimen shown in Figure 5.6. .............................................43

Figure 5.10 Light optical micrographs of Ti-Mo steel, etched with 3% nital for 15s......46

Figure 5.11 Light optical micrographs of Nb-Mo steel, etched with 3% nital for 15s. ....47

Figure 5.12 Light optical micrographs of V-Mo steel, etched with 3% nital for 15s. ......48

Figure 5.13 Light optical micrographs of Ti-Cr steel, etched with 3% nital for 15s. ......49

Figure 5.14 Light optical micrographs of High-Ti steel, etched with 3% nital for 15s. ...50
Figure 5.15  Light optical micrographs of V-Mo steel, (a) coiling temperature: 650 °C; cooling rate: 40°C/s, (b) coiling temperature: 650 °C; cooling rate: 20°C/s.

Figure 5.16  Higher magnification light optical micrographs for selected steels at 600 °C coiling temperature (a) V-Mo steel with cooling rate of 40°C/s, (b) V-Mo steel with cooling rate of 20°C/s, (c) Ti-Cr steel with cooling rate of 20°C/s and (d) High-Ti steel with cooling rate of 20°C/s.

Figure 5.17  Light optical micrographs at higher magnification for low coiling temperature (550 °C) and high cooling rate (40°C/s) conditions of (a) Ti-Mo steel, (b) Nb-Mo steel, (c) V-Mo steel, (d) Ti-Cr steel and (e) High-Ti steel.

Figure 5.18  Retardation of recrystallization due to presence of Nb, Ti and V for a 0.07C-1.4Mn-0.25Si steel [78].

Figure 5.19  Light optical micrographs of (a) Ti-Mo and (b) High-Ti. Both specimens are coiled at 600 °C with cooling rate of 20 °C/s.

Figure 5.20  Effects of alloying additions on eutectoid transformation temperature associated with its ferrite stabilizing character. [47].

Figure 5.21  Light optical micrograph of (a) Ti-Mo steel and (b) Ti-Cr steel. Both specimens were coiled at 600 °C with cooling rate of 20 °C/s. The Ti-Cr specimen exhibited more low temperature transformation products than Ti-Mo specimen.

Figure 5.22  TEM bright field images of carbon extraction replicas showing precipitates in Ti-Mo steel.

Figure 5.23  TEM bright field images of carbon extraction replicas showing precipitates in Nb-Mo steel.

Figure 5.24  TEM bright field images of carbon extraction replicas showing precipitates in V-Mo steel.

Figure 5.25  TEM bright field images of carbon extraction replicas showing precipitates in Ti-Cr steel.

Figure 5.26  TEM bright field images of carbon extraction replicas showing precipitates in High-Ti steel.

Figure 5.27  TEM bright filed images of precipitates in V-Mo steel coiled at 550 °C with cooling rate of 20 °C/s. (a) Small precipitates with presence of a large particle and (b) small precipitates in the region pointed out at (a).
Figure 5.28  TEM bright field images of (a) extraction replicas of Ti-Mo steel coiled at 600 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.29  TEM bright field images of (a) extraction replicas of Nb-Mo steel coiled at 650 °C with cooling rate of 40 °C/s and (b) corresponding EDS spectrum.

Figure 5.30  TEM bright field images of (a) extraction replicas of V-Mo steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.31  TEM bright field images of (a) extraction replicas of Ti-Cr steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.32  TEM bright field images of (a) extraction replicas of High-Ti steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.33  TEM bright field images of large precipitate observed in (a) Ti-Mo steel coiled at 600 °C with cooling rate of 20 °C/s and (b) High-Ti steel coiled at 650 °C with cooling rate of 40 °C/s.

Figure 5.34  TEM bright field image of thin foil specimen from Nb-Mo steel, coiled at 650 °C with cooling rate of 40 °C/s.

Figure 5.35  Yield strength versus coiling temperature for 5 steels.

Figure 5.36  Yield strength versus coiling temperature for five steels at two cooling rates following the hot rolling simulation.

Figure 5.37  Engineering stress-strain curves of two Ti-Mo steel specimens, coiled at 600 °C and 550 °C with cooling rate of 40 °C/s.

Figure 5.38  Yield strength versus $d^{1/2}$ for specimens with a polygonal ferrite matrix (specimens coiled at 650 °C with cooling rate of 40 °C/s).

Figure 5.39  Engineering stress-strain curves of Nb-Mo steel specimen coiled at 650 °C with cooling rate of 40 °C/s.

Figure A. 1  Modified Gleeble torsion specimen geometry (dimensions in inch).

Figure A. 2  Torsion testing program employed to simulate hot rolling process on Gleeble® 3500. (processing conditions are: 600 °C coiling temperature; cooling rate: 40 °C/s)

Figure B. 1  Microhardness versus coiling temperatures for 5 steels.
Figure B. 2  Engineering stress-strain curve of Ti-Mo subsized specimens...............85
Figure B. 3  Engineering stress-strain curve of Nb-Mo subsized specimens. ...............86
Figure B. 4  Engineering stress-strain curve of V-Mo subsized specimens. .................87
Figure B. 5  Engineering stress-strain curve of Ti-Cr subsized specimens. .................88
Figure B. 6  Engineering stress-strain curve of High-Ti subsized specimens. ..............89
LIST OF TABLES

Table 2.1 Mechanical Properties of NANOHITEN® and Conventional HSLA Steel [4]..........................3
Table 2.2 Chemical composition of NANOHITEN® steel [6].................................4
Table 2.3 Alloys Examined in the Investigation of Interphase Precipitation [26, 27, 28, 29, 30, 31]........................8
Table 2.4 Solubility Relations for Microalloy Carbides/Nitrides in Austenite [47]........16
Table 2.6 Comparison of Various Testing Methods Used in Laboratory Simulation of Thermomechanical Processing of Steels [53, 54, 66]..........................20
Table 3.1 Dimension of Normal and Subsized Specimens..............................................29
Table 3.2 Tensile Properties Measured from the Engineering Stress-Strain Curves of Subsized and Normal Size Specimens...................................................29
Table 4.1 Chemical Composition of Experimental Steels.................................................32
Table 4.2 Torsion Experiment Schedule Simulating 8-pass Hot Rolling Process [75]..........................33
Table 4.3 Experimental Matrix..............................................................33
Table 5.1 Mechanical Properties from Subsized Tensile Specimens .........................39
Table 5.2 Summary of Matrix Microstructures Characterization...............................51
Table 5.3 Grain Size of Specimens with Polygonal Ferrite Microstructure of 5 Steels. (650 °C coiling and 40 °C/s cooling rate).............................................54
Table B. 1 Average Vickers Microhardness of the 5 Steels...........................................84
Table B. 2 Summary of Defects Detected in Subsized Tensile Specimens....................90
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CHAPTER 1 : INTRODUCTION

In the automotive industry, as manufacturers are challenged to improve safety and fuel economy, steel design for automotive applications has increasingly focused on the enhancement of strength without compromise in ductility, as well as weight reduction that reduces fuel consumption. High strength low alloy (HSLA) steels were among the first widely used high strength steels in the automotive industry [1]. Fine dispersed microalloy carbides/nitrides in the ferrite matrix enabled precipitation strengthening while acting as important factors in the rolling process to obtain a fine matrix grain size. While HSLA steel is usually designed to meet mechanical specifications and is typically with good toughness and weldability, its strength and workability have been exceeded with the advanced high strength steels (AHSS). However, some multiphase AHSS steels show deteriorated stretch-flangeability or hole expansion performance, due to stress concentration regions between soft ferrite and hard martensite phases [2]. In a recent study by C. Chiriac of dominant factors responsible for hole expansion performance on two commercial DP 780 MPa steels [3], microhardness data revealed that smaller differences in hardness between ferrite and martensite correlated with better hole expansion performance.

A schematic diagram of the influence of microstructure on the balance of elongation and hole expansion ratio is shown in Figure 1.1[4]. A single phase ferrite matrix exhibits the optimum combination of elongation and hole expansion ratio. However, the contribution from conventional strengthening mechanisms in HSLA steels is limited such that these steels are challenged to meet manufacturers’ current requirements for strength.

Increased precipitation strengthening was obtained by JFE Steel Corporation using a Ti-Mo microalloyed HSLA steel [4]. These steels, with extensive fine precipitates distributed in a polygonal ferrite matrix, reportedly exhibit excellent combinations of strength, ductility and hole-expansion ability.

The present work was initiated to develop a method to simulate the hot rolling process using torsion testing on Gleeble® 3500 and to evaluate mechanical properties and microstructures on as-twisted torsion specimens. By simulating variations in processing parameters on the Gleeble® 3500 with various experimental steels, the effects of different microalloying additions
and processing parameters on microstructure, particularly precipitation states and mechanical properties were evaluated.

![Diagram showing influence of microstructure on balance of hole-expansion ratio and elongation.](image)

**Figure 1.1** Influence of microstructure of steel matrix on the balance of hole-expansion ratio and elongation [4].
CHAPTER 2 : BACKGROUND

2.1 Review of Nanoscale Precipitation Strengthened Steels

Some newer hot rolled HSLA steels have obtained substantial strengthening from nanoscale precipitation of about 300 MPa, which is 2~3 times more than the precipitation strengthening (about 100~150 MPa) reported in conventional HSLA steels [4]. These nanoscale precipitation strengthened steels with single phase ferrite are promoted by JFE Steel Corporation under the commercial name “NANOHITEN®” [4]. A mechanical property comparison between NANOHITEN® and conventional HSLA steel is shown in Table 2.1. Details of microstructure and properties will be further reviewed in the next section, but the table illustrates the favorable combination of strength and hole expansion ratio ($\lambda$).

Table 2.1– Mechanical Properties of NANOHITEN® and Conventional HSLA Steel [4].

<table>
<thead>
<tr>
<th>Steel</th>
<th>Mechanical Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>YS(MPa)</td>
</tr>
<tr>
<td>NANOHITEN®</td>
<td>745</td>
</tr>
<tr>
<td>Conventional HSLA</td>
<td>696</td>
</tr>
<tr>
<td>steel</td>
<td></td>
</tr>
</tbody>
</table>

2.1.1 Microstructure Characterization of “NANOHITEN®”

The reported chemical composition of NANOHITEN® in a 780 MPa grade is shown in Table 2.2 [6]. In the NANOHITEN® product, the low carbon steel is microalloyed with titanium (Ti) and molybdenum (Mo), and through carefully controlled thermo-mechanical processing, the steel is with unique microstructure features, as follows [4, 5]:

1. The steel has a single phase microstructure of ferrite as the matrix with an average grain size of about 3 µm, as shown in Figure 2.1(a).
2. The steel is strengthened by uniformly distributed precipitates refined to less than 10 nm in size, as shown in Figure 2.1(b).
3. No pearlite or large cementite is present since as the steel has a low carbon level and contains strong carbide forming elements (Ti, Mo).

Table 2.2 – Chemical composition of NANOHITEN® steel [6].

<table>
<thead>
<tr>
<th>Elements</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>N</th>
<th>Ti</th>
<th>Mo</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt pct</td>
<td>0.043</td>
<td>0.19</td>
<td>1.62</td>
<td>0.001</td>
<td>0.0032</td>
<td>0.092</td>
<td>0.19</td>
<td>0.008</td>
</tr>
</tbody>
</table>

Figure 2.1 SEM image of ferrite matrix (a) and TEM image of uniformly distributed precipitates (b) in NANOHITEN® [4].

The aligned morphology of precipitates in Figure 2.1(b) is clear evidence of interphase precipitation formed during the austenite to ferrite transformation [7]. From Funakawa’s observation of the precipitates using TEM [8], the diameter of the precipitates was determined to be approximately 3 nm. The EDS spectrum of fine precipitates indicated the presence of carbon, oxygen and copper as well as titanium and molybdenum. X-ray diffraction showed that the crystal structure of the nanoscale precipitates is NaCl type, with a lattice parameter of 0.433 nm. The fine precipitates in NANOHITEN® were determined to be Ti-Mo composite carbide, with the composition Ti$_{0.54}$Mo$_{0.46}$C [9]. Timokhina et al. investigated another Ti-Mo alloyed low carbon steel (composition: Fe-0.1C-1.24Mn-0.03Si-0.11Cr-0.11Mo-0.09Ti-0.09Al wt pct) strengthened by nanometer-sized precipitates using atom probe tomography and reported the nanoscale carbides to be Ti$_{0.98}$Mo$_{0.02}$C$_{0.6}$ [10].

2.1.2 Properties of NANOHITEN®

The properties of NANOHITEN® corresponding to the characteristic microstructures can
be generalized as follows [2, 4, 5, 6, 11]:

1. The steel has an excellent combination of strength, ductility and hole expansion ability. With a single ferrite matrix, NANOHITEN® obtains its strength mainly from precipitation strengthening and grain refinement strengthening, whilst the contribution from solid solution strengthening is relatively small, as shown in Figure 2.2. Since NANOHITEN® is ferritic without substantial quantities of other constituents, its balance between elongation and hole expansion ratio is reportedly superior to multiphase steels [5].

2. The steel reportedly exhibits stable mechanical properties compared with conventional HSLA steels, as the nanoscale precipitates have excellent high thermal stability against coarsening during coiling.

3. Due to the large strength increment achieved by precipitation hardening, it is possible to reduce the amount of Si as a solid solution strengthening element. While Si is an important element used for solution strengthening in conventional HSLA steel, the use of Si would deteriorate zinc-galvanizing surface quality [12, 13]. Also, Si can cause internal oxidation, which results in reduced fatigue performance and corrosion resistance.

4. With a low carbon level, the steel is a good welding candidate. Also, the initial presence of nanoscale precipitates might inhibit ferrite grain growth during welding [14].

5. The low rolling load to produce NANOHITEN® in the finish rolling mill reportedly enables the manufacture of thinner and wider sheets [5, 15].

![Figure 2.2](image.png)

**Figure 2.2** Calculated amount of solid solution strengthening, precipitation strengthening and grain refinement strengthening in NANOHITEN® [6].
Excellent ductility, both global and local, allows the application of NANOHITEN® in the manufacture of complex-shape automotive components, such as body structure, suspension, chassis, wheels and ancillary parts. Moreover, since Si is not added as a strengthening element, NANOHITEN® is applicable for hot dipped galvanized sheet, for use as automotive panels [16].

2.1.3 Alloying Effects in NANOHITEN®

The microalloying additions chosen for NANOHITEN® are Ti and Mo, which play a significant role in forming nanoscale carbides and influencing the thermal stability of the precipitates. Funakawa and co-workers conducted experiments investigating the coarsening behavior of nanometer-sized carbides in hot-rolled high strength sheet steel with different microalloying additions [17]. Two alloying systems were used in the experiment, Ti-Nb and Ti-Mo, which form (Ti, Nb)C and (Ti, Mo)C precipitates, respectively. The results suggested that Ti-Mo alloy system exhibits precipitates with better thermal stability [17]. Another study of the influence of Mo on tempering precipitation in Nb-Mo-V microalloyed steel showed that the segregation of Mo at the outer layer of carbides could inhibit the diffusion of the Nb and V from the matrix into the carbides as well as reduce the diffusion rate of C and alloy elements in the matrix [18], hence, the growth rate of the precipitates is greatly impeded. It might be reasonable to expect Mo to play the same role in refining precipitates in the Nb and V systems as in the Ti system. Mo can also retard the formation of both pearlite and cementite at grain boundaries [4].

There is an addition of about 1.5 wt pct Mn added in NANOHITEN®, which is to suppress Fe₃C precipitation, by lowering the austenite-ferrite transformation temperature (Ar₃) [20]. The nitrogen content should be reduced, because nitrogen exhausts titanium by forming large TiN. The alloy system in NANOHITEN® provides a basic framework of the alloy design for the experimental materials in this project, with further details discussed in CHAPTER 4.

2.2 Precipitation in Steels

Microalloy precipitation reactions in steels can be classified into three types [19, 20, 21]:

1. Precipitation in austenite following temperature reduction, prior to the transformation from austenite to ferrite.
2. Precipitation during the austenite to ferrite transformation process where the solubility decreases discontinuously at the austenite-ferrite boundary, known as interphase precipitation.

3. Precipitation from supersaturated solid solution in ferrite.

2.2.1 Precipitation in Austenite

Precipitates formed in austenite are mostly due to a supersaturated austenite solid solution as the solubility of microalloy carbide/nitride reduces as temperature decreases. Precipitation often occurs through a strain induced precipitation mechanism. When hot deformation is applied in the austenite region, the introduction of dislocations, dislocations cells and substructures during the hot working process provide nucleation sites for the precipitation of carbides/nitrides [20]. A common example is strain induced precipitation of Nb(C, N), which is employed in controlled rolling to suppress recrystallization in order to obtain “pancaked” austenite. However, precipitates formed in austenite are relatively coarse and therefore have only a very limited contribution to strengthening. Also, precipitation in austenite can consume a substantial portion of the microalloy additions (Ti, V, Nb), which would reduce the amount available for precipitation at lower temperature in the ferrite region.

2.2.2 Precipitation During Decomposition of the Austenite

A non-random dispersion of precipitates aligned in rows can form during transformation, known as interphase precipitation, which takes place in association with the austenite to ferrite transformation interface during austenite decomposition. Interphase precipitation strengthening is reported to be the main strengthening factor in NANOHITEN®, as mentioned above. In this section, the literature of interphase precipitation will be reviewed regarding the morphology, mechanism and strengthening contribution.

Interphase precipitation was first reported in a Nb microalloyed low carbon steel (0.15 C-1.11Mn-0.02Nb in wt pct) by W.B. Morrison, J.H. Woodhead and others [22, 23, 24, 25]. A significant strengthening was discovered in some Nb-microalloyed steels when a higher austenizing temperature was employed and isothermally held at austenite to ferrite transformation temperature. In these steels, precipitation with a banded distribution was first detected under TEM (Figure 2.3) [24].
Since then, interphase precipitation has been detected in many microalloyed steels, and due to its potentially significant strengthening contribution to HSLA steels, significant research has been done regarding the mechanism of interphase precipitation formation. R.W.K. Honeycombe and co-workers conducted a fundamental investigation of interphase precipitation in various alloy systems by applying isothermal transformation. Table 2.3 summarizes the alloy systems they examined [26, 27, 28, 29, 30, 31], and within all of them interphase precipitation occurred.

Table 2.3 – Alloys Examined in the Investigation of Interphase Precipitation [26, 27, 28, 29, 30, 31].

<table>
<thead>
<tr>
<th>Alloy system</th>
<th>Composition (wt pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-C-Nb</td>
<td>0.07C-0.031Nb</td>
</tr>
<tr>
<td>Fe-C-Nb-B</td>
<td>0.09C-0.036Nb-0.003B</td>
</tr>
<tr>
<td>Fe-C-Cr</td>
<td>0.2C-5Cr/12Cr</td>
</tr>
<tr>
<td>Fe-Mo-C</td>
<td>0.2<del>1.0C-0.5</del>4Mo</td>
</tr>
<tr>
<td>Fe-C-Nb-Mn</td>
<td>0.07C-0.33Nb-1.07Mn</td>
</tr>
<tr>
<td>Fe-C-V-Mn</td>
<td>0.82C-11.9Mn-0.48V</td>
</tr>
</tbody>
</table>

Various morphologies of interphase precipitation were observed, which can be summarized into four types and listed below [32, 33, 34], with TEM images shown in Figure 2.4. Different morphologies may form under different thermal-processing conditions of the same alloy [20].
Figure 2.4 TEM images of thin foil specimens showing (a) planar interphase precipitation, (b) curved interphase precipitation with uniform sheet spacing, (c) curved interphase precipitation with random sheet spacing and (d) fibrous interphase precipitation [33, 35].

The four types of precipitates are summarized as follows:

1. Planar interphase precipitation with uniform sheet spacing (Figure 2.4 a). This is the most well characterized interphase precipitation.

2. Curved interphase precipitation with uniform (Figure 2.4 b) or random (Figure 2.4 c) sheet spacing, which is often observed at high transformation temperatures.

3. Fibrous interphase precipitation (Figure 2.4 d), which is commonly observed in high carbon steels.

The characteristic morphologies of interphase precipitation are closely related to the thermomechanical processing conditions of the steel, and dependent on the transformation
Different mechanisms have been proposed to explain interphase precipitation, and the commonly adopted one was the “ledge mechanism”, by Davenport and Honeycombe [36]. The ledge mechanism is schematically shown in Figure 2.5. The figure illustrates the nucleation and growth of precipitates at the austenite/ferrite interface during transformation. The horizontal austenite/ferrite boundaries in Figure 2.5 are lower energy immobile interface segments formed by the passage of high energy ledges [37], which are indicated as short, vertical segments of the boundaries in the figure. The high energy ledges are moving too fast to be nucleation sites, while the immobile interfaces are available for precipitates to nucleate and grow, until the next ferrite ledge sweeps over and repeat the process [20]. Hence, the precipitates on the low energy interfaces should be in sheets with spacing which is the same as the ledge height, as revealed in Figure 2.5 (b) with irregular ledge height resulting in various distances between precipitate rows. The uniformity of the particle distribution on each step is believed to be related to the diffusion distance of the microalloy elements and associated nucleation frequency [20].

![Figure 2.5 Schematic representation of the mechanism of nucleation and growth of carbides on the austenite/ferrite interface. (a) regular ledge height, (b) irregular ledge height. Black dots in the figure indicate the precipitate positions [37].](image)

The orientation relationships between the carbides and ferrite matrix have confirmed that the precipitates nucleate with low energy interfaces [21]. It is reported in many papers that the Kurdjumov-Sachs (K-S) relationship is adapted between interphase precipitates and the ferrite matrix [31, 36, 38], which is,
\[(111)_{\text{MC}}//(110)_{\alpha}\]
\[[110]_{\text{MC}}//[111]_{\alpha}\]

where MC means microalloy carbides.

The Nishiyama-Wasserman (N-W) relationship has also been reported between interphase carbides and the ferrite matrix [31, 33, 21, 39, 40]. K-S, N-W and Baker-Nutting (B-N) are three orientation relationships commonly observed for fcc precipitates in a bcc matrix [21]. Interphase precipitates form in a K-S (or N-W) relationship with the ferrite due to a three-phase (austenite, ferrite and microalloy carbide) relationship at the ferrite growth interface.

The ledge mechanism explains the typical structure of interphase precipitation, while some detailed aspects of the morphology such as the intersheet spacing variation with temperature are not as clearly illustrated [41]. The solute-depletion model, proposed by Roberts and developed by R. Lagneborg and S. Zajac, are based on the ledge mechanism and solute diffusion control models [41].

The solute-depletion model assumes that the growth of ferrite into austenite is controlled by carbon diffusion in austenite while maintaining local equilibrium at the interface [41, 42, 43]. During the ferrite growth, several correlated processes: nucleation of microalloy carbides/nitrides particles in the austenite-ferrite interface, extension of microalloy element depletion zones around the precipitates and the migration of the interphase boundary away from the formed precipitates altogether result in a repeated precipitation behavior [41]. The microalloy element depletion zone expands more quickly immediately after nucleation, obeying the parabolic growth law, \(\text{radius} \propto (\text{time})^{1/2}\). The same parabolic growth law also applies to the growth of ferrite but on a large scale, so the ferrite growth rate can be regarded as essentially constant for the short distance corresponding to the interphase precipitation reaction. The relative growth velocity between the microalloy element depleted zone and ferrite changes during precipitate growth so that the austenite-ferrite interface is first behind the alloy element depleted zone when nucleation occurs, and then catches up and re-enters the region with normal microalloy content, so nucleation of a new sheet of precipitates then repeats itself. This model is capable of predicting the sheet spacing by incorporating the diffusivity of microalloy elements and ferrite growth velocity, and was validated for V-microalloyed steel [42].

The spatial dispersion of the interphase precipitation is such that TEM may only reveal the distinct rows of particles when observed from specific orientations, and the linear arrays are “lost”
if the same region is tilted, with the precipitates showing an apparent random distribution [20],
which makes it challenging to detect interphase precipitation using TEM.

2.2.3 Precipitation from Supersaturated Ferrite

Precipitation from supersaturated ferrite occurs following the austenite to ferrite transformation, and is commonly known as matrix precipitation. The process can occur after rapid cooling to the low temperature phase region either during coiling or upon reheating during a sub-critical annealing or normalizing treatment. Figure 2.6 shows approximate temperature regions for interphase precipitation and precipitation from supersaturated ferrite [42].

Unlike interphase precipitation, precipitates from supersaturated ferrite usually result in a random dispersion in the matrix. These random precipitates nucleate directly in ferrite and usually obey the Baker-Nutting (B-N) relationship with the ferrite grains [21]. That is,

\[(001)_{\text{MC}}//(001)_{\alpha}\]
\[[110]_{\text{MC}}//[010]_{\alpha}\]

Significant strengthening can also be obtained from matrix precipitation. As reported in a recent publication [44], matrix precipitates with average diameter of 10 nm contribute about 450 MPa to the yield strength of a 0.04C HSLA steel, having 0.08Ti, 0.18Mo, 0.011Nb, and 0.015V (in weight percent). These fine precipitates are homogeneously dispersed in the matrix, as shown in Figure 2.7 [44].

Figure 2.6 Generic CCT diagram for microalloyed steel indicating approximate regions of interphase and random precipitation in matrix [42].
2.3 **Precipitation Strengthening Mechanism**

The precipitation strengthening due to nanoscale precipitation can be explained by the Ashby-Orowan mechanism [20, 45]. As fine carbides/nitrides are hard and undeformable, the dislocations would bow around particles and then pass through, forming “Orowan loops” [45]. The increase in applied stress from the interaction between the particle and dislocation loop can be explained using the Orowan equation:

$$\Delta\sigma_{ppn} = \frac{Gb}{L}$$  \[Equation 2.1\]

where $\Delta\sigma_{ppn}$ is the increase in yield strength due to precipitation strengthening, $G$ is the shear modulus of the matrix, $b$ is the Burgers’ vector and $L$ the particle spacing. In the Orowan equation, $L$ is the factor relating to the state of precipitation, which indicates that smaller spacing between particles would contribute a higher strengthening amount. So to obtain the largest amount of strengthening, a high volume fraction of nanoscale precipitates should be achieved. As discussed above, both interphase precipitation and matrix precipitation can have a significant strengthening effect if their size and volume fraction are suitably controlled through thermomechanical processing.
2.4 Thermomechanical Processing and Laboratory Simulation of HSLA Steels

In order to obtain the optimum precipitation state as described in the previous section, careful control of thermomechanical processing is necessary. Figure 2.8 schematically shows the main processes in a hot strip mill [46]. In this section, several thermomechanical processing factors which are critical in obtaining final microstructures are reviewed to assist in planning experimental parameters for simulating hot strip mill processes.

![Figure 2.8 Layout of a semi-continuous hot strip mill [46].](image)

2.4.1 Slab Reheating Temperature

The initial stage of any hot rolling process is the reheating stage, and the slab reheating temperature has a strong influence on the strength, toughness and microstructure of microalloyed steels [43]. A low slab reheating temperature results in finer austenite grains, which refines the final microstructure of the material and hence improves the low temperature toughness. Refinement is partly attributed to the precipitates remaining after low temperature reheating, which helps suppress the grain growth of austenite. However, low slab reheating temperatures may also decrease yield strength and tensile strength, as a reduction of dissolved microalloying elements results in a decrease of precipitation hardening after cooling, and may influence the austenite conditioning behavior during rolling.

As the current study focuses on obtaining a high fraction of fine precipitates, a slab reheating temperature high enough to dissolve nearly all of the microalloyed carbides/nitrides would be preferred, in order to achieve the maximum precipitation during later processes. A commonly adopted slab reheating temperature in the hot strip mill is somewhat above 1250 °C. The solubility curves for Ti, Nb and V carbides/nitrides at several temperatures are shown in Figure 2.9, using data in Table 2.4. TiN has relatively smaller solubility compared with other microalloy carbide/nitride. These calculations will be used for determining the austenizing temperatures in the current study.
Calculated carbide/nitride solubility in austenite for (a) TiC, (b) VC, (c) NbC, (d) TiN, (e) VN and (f) NbN at 950, 1100 and 1250 °C.
Table 2.4 – Solubility Relations for Microalloy Carbides/Nitrides in Austenite [47].

<table>
<thead>
<tr>
<th>Carbide/Nitride</th>
<th>Solubility in Austenite (element concentrations in wt pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiC</td>
<td>( \log[Ti] [C] = - \frac{7480}{T} + 3.21 )</td>
</tr>
<tr>
<td>NbC</td>
<td>( \log[Nb] [C] = - \frac{6770}{T} + 2.26 )</td>
</tr>
<tr>
<td>VC</td>
<td>( \log[V] [C] = - \frac{9500}{T} + 6.72 )</td>
</tr>
<tr>
<td>TiN</td>
<td>( \log[Ti] [N] = - \frac{15790}{T} + 5.40 )</td>
</tr>
<tr>
<td>NbN</td>
<td>( \log[Nb] [N] = - \frac{10150}{T} + 3.79 )</td>
</tr>
<tr>
<td>VN</td>
<td>( \log[V] [N] = - \frac{7700}{T} + 2.86 )</td>
</tr>
</tbody>
</table>

2.4.2 Rolling Schedule

In the hot strip mill, a multi-pass rolling schedule including rough rolling and finish rolling is usually employed. Based on various factors including rolling temperature, strain, strain rate, interpass time, finish rolling temperature, etc., the rolling schedules can be separated into several types. Conventional controlled rolling, recrystallization controlled rolling and dynamic recrystallization rolling are three common ways to roll HSLA steels. Figure 2.10 schematically shows deformations over different temperature ranges for three rolling types [48].

In recrystallization controlled rolling, shown in Figure 2.10 (a), finish rolling is carried out above the \( T_{nr} \), which allows the deformed austenite to achieve complete recrystallization if the interpass time is long enough and no strain is accumulated. In conventional controlled rolling (Figure 2.10 b), the particle pinning resulting from strain-induced precipitates, mainly Nb(C, N), retards or even prevents austenite recrystallization and hence creates “pancaked” or flattened austenite. Conventional controlled rolling is commonly adopted in plate rolling where “pancaked” austenite would result in a fine microstructure. Strain-induced precipitation consumes a considerable amount of microalloy elements, however, which would reduce precipitation strengthening afterwards in the low temperature range. Dynamic recrystallization (Figure 2.10 c) occurs between passes during rolling when total strain exceeds the critical strain [49, 50]. The strain at which dynamic recrystallization is initiated is named the critical strain, which may be achieved below \( T_{nr} \).
via strain accumulation [48]. Dynamic recrystallization rolling usually creates a finer final microstructure than recrystallization controlled rolling. Also, the short interpass time of dynamic recrystallization controlled rolling barely allows precipitation, so most of the microalloying elements are kept in solid solution in the matrix. Grain size is influenced by the specific rolling schedule. Variations in deformation amount and interpass time influence the austenite to ferrite transformation temperature and transformation kinetics, which would further affect the precipitation states (interphase precipitation and precipitation in ferrite) in later cooling and coiling periods.

Figure 2.10  Schematic temperature-time diagram showing three rolling types: (a) recrystallization controlled rolling, (b) conventional controlled rolling and (c) dynamic recrystallization controlled rolling [48].

### 2.4.3 Cooling Rate and Coiling Temperature

The run-out table cooling rate and coiling temperature will determine the types of transformation products produced in the final microstructure. Also, most of the precipitates that account for precipitation strengthening form during the cooling and coiling process. So the cooling and coiling conditions exert a major effect on the final microstructure and hence the mechanical properties. It is known that during interphase precipitation, the sheet spacing and inter-particle spacing are greatly influenced by the isothermal transformation temperature. Yen et al.
investigated the sheet spacing and inter-particle spacing for interphase precipitation under different isothermal holding temperatures in three microalloyed steels [39], with results shown in Table 2.5. It is evident that sheet spacing and inter-particle spacing decrease with reduced temperatures.

Table 2.5 – Sheet Spacing and Inter-Particle Spacing for Three Steels Isothermally Treated at 650, 680 and 700°C [39].

<table>
<thead>
<tr>
<th>Steel (wt pct)</th>
<th>Sheet Spacing (nm)</th>
<th>Inter-particle Spacing (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>650 °C</td>
<td>680 °C</td>
</tr>
<tr>
<td>0.1C-0.2Ti</td>
<td>12.1</td>
<td>15.4</td>
</tr>
<tr>
<td>0.1C-0.2Ti-0.2Mo</td>
<td>11.3</td>
<td>14.2</td>
</tr>
<tr>
<td>0.1C-0.2Ti-0.04Nb</td>
<td>12.5</td>
<td>17.6</td>
</tr>
</tbody>
</table>

The ferrite microstructure under different cooling rate and coiling temperature conditions should also be taken into consideration, as certain ferrite microstructures might deteriorate some properties of the steel. For example, low temperature (<600 °C) Widmanstätten ferrite formation may not be desired in some steels because of toughness considerations [21]. In the current study, polygonal ferrite with a fine grain size is the desired matrix microstructure considering the requirements for elongation properties. Different ferrite microstructures on a typical CCT diagram of a HSLA steel are shown in Figure 2.11 [51]. The desired type of ferrite should be obtained under proper cooling rate (coiling temperature) conditions. Microalloying elements also affect ferrite microstructure in several ways, such as altering transformation temperature [20].

Because of the sensitivity of properties to processing conditions, an investigation into various cooling rates and coiling temperatures is key to identify processing parameters that provide the desired microstructures and optimized mechanical properties.

2.4.4 Laboratory Simulation of Thermomechanical Processing

Physical simulation is a reproduction of the mechanical and thermal parameters of a real-world manufacturing process on a laboratory scale [52]. With careful selection and control of parameters such as temperature, heating and cooling rates, strain and stress states, reliable data can be generated about the material and process investigated.

The purposes of laboratory hot rolling simulation can be generalized into two categories. The first one is to optimize the rolling schedule by investigation into thermomechanical
parameters such as cooling rates, rolling load, interpass time and deformation [53, 54, 55, 56, 57]. The other main purpose is to study material behaviors during the hot rolling process, such as recrystallization (static, dynamic, metadynamic recrystallization or non-recrystallization temperatures), phase transformation states, alloying effects and precipitation states [58, 59, 60, 61, 62, 63]. However, there is less information from literature regarding mechanical property evaluation methods after compression or torsion testing. Methods to generate reliable mechanical property data are essential in order to characterize a material and evaluate the effects of thermomechanical parameters.

Uniaxial tension, torsion and compression of axisymmetric or planar samples have all been utilized for simulating material behavior in the actual process [64, 65], with compression and torsion tests being the two most common testing methods employed by researchers to simulate hot rolling conditions. The advantages and disadvantages of these testing methods are summarized in Table 2.6. Torsion testing shows its advantages in simulating multi-pass rolling process, compared with tension and compression testing, as the specimen geometry remaining unchanged during torsion testing and allows for desired strain amount.

In this project, hot torsion testing is employed to simulate the hot strip rolling process and a method to evaluate mechanical properties after hot torsion test is evaluated, which will be further explained in the next chapter.

![CCT diagram](image)

Figure 2.11  CCT diagram for an HSLA steel containing 0.06C, 1.45Mn, 1.25Cu, 0.97Ni, 0.72Cr, and 0.42Mo, in wt pct. PF, polygonal ferrite; WF, Widmanstätten ferrite; AF, acicular ferrite; GF, granular ferrite [51].
<table>
<thead>
<tr>
<th>Testing Method</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension testing</td>
<td>• Easiest to perform</td>
<td>• Only low strains are possible</td>
</tr>
<tr>
<td></td>
<td>• Governed by ASTM standards to minimize laboratory variability</td>
<td>• Uniaxial stress distribution is lost when necking occurs</td>
</tr>
<tr>
<td>Compression testing</td>
<td>• Large strains are possible</td>
<td>• Friction problem exists, which might cause barreling</td>
</tr>
<tr>
<td></td>
<td>• Compressive stress state, as in rolling</td>
<td>• Non-uniform strain distribution in stress-applied direction</td>
</tr>
<tr>
<td>Torsion testing</td>
<td>• High total strain is achievable</td>
<td>• Torsional stress and strain vary over the cross section, which causes variations in microstructure</td>
</tr>
<tr>
<td></td>
<td>• Specimen geometry does not change, allowing a large number of passes</td>
<td>• Application of torsion ductility data to workability prediction remains unsatisfactory</td>
</tr>
<tr>
<td></td>
<td>• Easy to achieve constant rate of strain</td>
<td></td>
</tr>
</tbody>
</table>
3.1 Objectives

The purpose of this project was to investigate the effects of thermomechanical processing parameters on mechanical properties and precipitation states of formable high strength hot rolled steels with different microalloy additions, which involves several parts:

1. Simulation of hot strip multi-pass rolling process by torsion testing on the Gleeble® 3500 and establishment of a method to evaluate mechanical properties on as-twisted torsion specimens;
2. Investigation of the effects of various thermomechanical parameters as well as microalloying additions on mechanical properties and microstructures;
3. Initial characterization of precipitation states in materials with different microalloyed elements and thermomechanical processing parameters.

3.2 Gleeble® 3500 Torsion Testing

The Gleeble® 3500 is an integrated digital closed loop control thermal and mechanical testing system which is capable of various types of thermal-mechanical materials testing and physical simulations [67]. Hot torsion testing on the Gleeble® 3500 was employed to simulate the hot rolling process. The most important advantage of using torsion testing in this project, comparing with tension or compression testing, is the capability to simulate the entire multi-pass rolling schedule: slab reheating, rough rolling, finish rolling, run-out table cooling and coiling, so multiple thermomechanical parameters could be investigated with careful control based on the experimental plan.

During torsion testing, the shear stress and strain vary over the cross section, so careful selection of radial positions within the specimen for microstructure analysis and mechanical property evaluation is necessary. Richardson outlined a method to calculate equivalent stress-strain from torque-twist data [68, 69]. Equivalent strain, $\bar{\varepsilon}$, is given by,

$$\bar{\varepsilon} = \frac{0.724a\theta}{\sqrt{3}l}$$  \hspace{1cm} [Equation 3.1]
where $a$ is the radius of the gauge section, $\theta$ is the twisted angle and $l$ is the length of the gauge section. $\bar{r} = 0.724a$ represents the “equivalent radius”, which indicates the position on the cross section of the sample where the strain equals the equivalent strain. Microstructure analysis and mechanical property evaluation were conducted at the “equivalent radius” position in the present work.

3.2.1 Hot Torsion Test Sample Development

Hot torsion testing on the Gleeble® 3500 provides essential data for the hot rolling simulation. However, in providing information regarding room temperature mechanical properties, torsion tests have shown unsatisfactory results. Hall and Worobec first proposed a method to prepare tensile samples from torsion samples after thermomechanical processing: the center of the torsion sample was drilled out to remove the non-uniform structure and the resulting cylindrical tube was tested in uniaxial tension. In tension, the tube samples tended to fail in a corkscrew manner with very limited elongation [70]. A similar problem was noticed in D. Futch’s tests with different tensile sample geometries [71], as shown in Figure 3.1. The hollow tube tensile specimens obtained from as-twisted torsion specimens had unrepresentative elongation values and sometimes failed before necking.

In order to obtain reasonable tensile mechanical properties in samples thermomechanically processed using hot torsion simulation, a subsized tensile specimen geometry was explored. To prepare subsized tensile samples from torsion samples, the torsion specimen geometry needed to be modified from the customary specimen geometry recommended by the manufacturer.

3.2.2 Modification of Torsion Specimen Geometry

The standard torsion specimen has a hollow grip section on both sides of the gauge section, in order to maintain a more homogeneous thermal profile in the longitudinal direction. The hollow grips made it impossible to prepare subsized tensile samples that can be tested using an 8 mm extensometer, so a modified torsion specimen geometry was considered. There were two main concerns regarding the torsion specimen: 1) the strain exerted on the sample must be predictable based on the twist angle, and 2) the heating rate along the sample and temperature gradient in the gage length must be suitable. Information from the technical personnel of Dynamic Systems Inc. (DSI) confirmed that as long as the gage section is kept to the original length, the strain state will
not be affected. The modified torsion specimen was then designed by changing a portion of the grip section closest to the gage length, as shown in Figure 3.2.

The modified torsion specimen increased the cross section area of the grip section closest to the gage region. Since the Gleeble® 3500 employs resistance heating with a current passing through the specimen, the enlarged cross section area results in lower local resistance, which theoretically should increase the thermal gradient along the longitudinal direction near the end of the gage section. Tests to examine the temperature gradient were conducted to validate the suitability of the specimen and the results are shown in the next section.

Figure 3.1  Stress-strain curves for comparison of tensile sample geometries. (a) low carbon steel (0.056C-1.16Mn-0.01Si in wt pct) and (b) TRIP steel (0.19 C-1.56 Mn-1.61 Si-0.051Al in wt pct ) [71].

Figure 3.2  Modified torsion specimens. (a): Standard torsion specimen and (b): Modified torsion specimen, changing part of the hollow grip to solid to allow for tensile specimen grip ends; The dashed lines indicate holes drilled into the specimen.
Details of the subsized tensile samples and microstructure analysis coupon taken from a torsion specimen are shown in Figure 3.3. Two subsized tensile samples can be prepared from one torsion specimen from the symmetric “equivalent radius” positions. One sample for microstructure analysis can also be prepared between the two subsized tensile specimens, from the same “equivalent radius” position.

![Figure 3.3](image)

**Figure 3.3**  Preparation of subsized samples and microstructure analysis piece from modified torsion specimen, with the top and bottom drawing showing the plan view and elevation view, respectively.

### 3.2.3 Characterization of Thermal Gradients

The Gleeble® 3500 system uses electric resistance to heat the specimen based on the thermal profile programmed by the user. A thermocouple is percussion welded to the surface of the gage section at mid-length to monitor the temperature and control the current passing through so as to adjust the temperature. A longitudinal thermal gradient is inherent in this type of heating configuration [72]. For simulation of the thermomechanical process, it is necessary to make sure that the gage section of the torsion specimen subject to deformation experiences uniform temperatures within an acceptable range. Thermal gradient tests were conducted on both the standard and modified specimens.

Three thermocouples TC1, TC2 and TC3 were percussion welded at positions 0 mm, 5 mm, and 10 mm away from the center of the torsion specimen, respectively, in order to obtain
the thermal profile along the gage section, as schematically shown in Figure 3.4. TC1 is the control thermocouple.

![Figure 3.4](image)

Figure 3.4  Schematic illustration of thermal gradient test setup. Thermocouples TC1, TC2, TC3 were welded at positions 0, 5, 10 mm away from the center of gage length, respectively.

The thermal gradient tests concentrated on the slab reheating (slab reheating temperature at 1250 °C) and hot rolling periods (cooling rate at 20 °C/s), as the thermal gradient particularly affects the uniformity of the deformation state along the gage section at high temperature. No actual deformation was applied during these thermal tests. The temperature test results are shown in Figure 3.5.

In the standard specimen, the reheating temperature at position TC2 was the highest as shown in Figure 3.4 a, about 25 °C higher than TC1 and TC3. Since TC1 is the control thermocouple, welded to the center of the gage section, it should have experienced the highest temperature as the grip ends are expected to be colder. One explanation of why TC2 was higher than TC1 is that the specimen tends to expand under compression when heated, particularly in the center of the gage section (where TC1 is welded) which undergoes the highest heating rate. With a fixed grip on both sides, the whole specimen is in a state of compression after heating, which can be noticed from the “thrust” reading recorded during the test, which measures the load on grips where the specimen is fixed. The center of gage section bulges, resulting in increased cross section, which leads to a reduced local resistance. Reduced resistance would result in a subsequently lower heat input under a given current input compared with TC2 where there is hardly a noticeable change of cross section. This explanation was confirmed by another thermal gradient test where a “0-thrust” control mode was employed, which would leave the specimen “free” for expansion during the heating period. No obvious bulging was observed in this setup, comparing with a sample from the previous test in which a “0-stoke” model, or fixed grips, was used. The temperature profile is shown in Figure 3.6, and it can be observed that TC2 remains at
the same temperature as TC1 for most of the isothermal holding period when bulging is absent.

Figure 3.5  Thermal gradient measurements for (a) standard (Figure 3.2 a) and (b) modified torsion specimens (Figure 3.2 b).

Figure 3.6  Thermal gradient test results for the “0-thrust” setup on modified specimen. TC1 and TC2 were welded at the same position as shown in Figure 3.4.

The thermal gradient test for the modified specimen geometry also included the coiling period. As shown in Figure 3.5, the temperature distribution during the slab reheating period of modified specimen is similar to the standard specimen, with TC2 being about 20 °C higher than TC1, and about 25 °C higher than TC3. For the “rolling” and coiling periods, however, there is hardly any observable temperature difference.

For the modified specimen, the thermal gradient is quite limited in both the slab reheating period and the “hot rolling” period of the simulations, which is similar to the standard specimen. So the modified specimen was considered valid to be used in hot torsion simulation experiments.
Detailed dimensions of the modified torsion specimen are shown in Figure 3.7. Preferred specimen dimensions are shown in the appendix.

![Figure 3.7 Dimensions of specimen in mm used for Gleeble® 3500 torsion simulations.](image)

3.2.4 Temperature Monitoring and Control During Hot Torsion Testing

Before the optical pyrometer was installed on the Gleeble® 3500 system in May, 2012 at Colorado School of Mines, spot-welded K-type thermocouples were used to measure and control the temperature during a test. However, during a hot torsion test, the thermocouples welded in the center position of the gage section easily detached when the specimen was twisted, and failed to provide temperature control during the entire test. Employment of the pyrometer (Figure 3.8) has solved the problem of controlling the temperature during hot rolling.

![Figure 3.8 Two color optical pyrometer installed on the Gleeble® 3500.](image)
The temperature that the pyrometer can measure is limited to a range between 600 and 1300 °C; between 600 and 650 °C, the pyrometer loses accurate control of the temperature. For this project, accurate control of temperature below 650 °C is necessary for the coiling simulation. So a thermocouple is used along with the pyrometer to obtain the complete processing schedule. As schematically shown in Figure 3.9, one thermocouple is welded on the end of the (non-rotating) grip section very close to the gage section, designated as “shoulder thermocouple”. During the slab reheating and hot rolling periods, pyrometer control of temperature is used, while for the run-out table cooling and coiling periods, temperature is switched to be under thermocouple control.

![Figure 3.9 Thermocouple control along with pyrometer control of the temperature during hot torsion testing.](image)

3.3 Validation of Subsized Tensile Sample Geometry for Mechanical Property Evaluation

In order to validate the mechanical properties measured by the subsized tensile specimens designed to be removed from the Gleeble specimen, tensile tests were conducted on subsized tensile samples and normal size tensile samples taken from the same bulk material. The dimensions of the samples are shown in Table 3.1. The normal sized specimen was machined with a 6.35 mm thickness to maintain a similar shape as the 1.5 mm thickness subsized specimen. The subsized specimens were designed to be machined from Gleeble torsion samples as shown previously in Figure 3.3.

All specimens were machined from the same material, a “Medium Nb Steel”, in the “as-received” condition from prior work of Nowill [73]. The tensile tests were conducted consistent with Thomas’ work [72], using an MTS screw driven frame at a constant crosshead speed of 2.54 mm/min (0.1 inches/min). The displacement of the subsized tensile specimen was measured using an 8 mm extensometer. The displacement of the normal sized tensile specimen
was measured using a 50.8 mm extensometer. Engineering stress-strain curves were generated for duplicate specimens from the load-displacement data and were compared to evaluate the validity of subsized tensile specimen. The engineering stress-strain curves are shown in Figure 3.10. The measured tensile properties are shown in Table 3.2.

Table 3.1 – Dimension of Normal and Subsized Specimens

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Normal Sized Specimen(mm)</th>
<th>Subsized Specimen(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G, gage length</td>
<td>50.8</td>
<td>12</td>
</tr>
<tr>
<td>W, width</td>
<td>12.7</td>
<td>3</td>
</tr>
<tr>
<td>T, thickness</td>
<td>6.35</td>
<td>1.5</td>
</tr>
<tr>
<td>R, radius of fillet</td>
<td>12.7</td>
<td>1.875</td>
</tr>
<tr>
<td>L, overall length</td>
<td>203.2</td>
<td>48</td>
</tr>
<tr>
<td>A, length of reduced section</td>
<td>57.15</td>
<td>15</td>
</tr>
<tr>
<td>B, length of grip section</td>
<td>50.8</td>
<td>15</td>
</tr>
<tr>
<td>C, width of grip section</td>
<td>19.05</td>
<td>4.5</td>
</tr>
</tbody>
</table>

Table 3.2 – Tensile Properties Measured from the Engineering Stress-Strain Curves of Subsized and Normal Size Specimens.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>0.2% Yield strength (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Uniform elongation (pct)</th>
<th>Total elongation (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Subsized</td>
<td>430</td>
<td>616</td>
<td>9.9</td>
<td>31.9</td>
</tr>
<tr>
<td></td>
<td>434</td>
<td>616</td>
<td>10.9</td>
<td>34.3</td>
</tr>
<tr>
<td>Normal Size</td>
<td>429</td>
<td>621</td>
<td>10.9</td>
<td>21.6</td>
</tr>
<tr>
<td></td>
<td>421</td>
<td>610</td>
<td>9.8</td>
<td>23.1</td>
</tr>
</tbody>
</table>

As seen in Figure 3.10, the engineering stress-strain data for the two geometries essentially match each other up to the point of necking. After necking, the subsized specimens show more total elongation than the normal size samples. The results are similar to Thomas’ observation [72]; the reason behind the larger total elongation value from subsized tensile specimen is that the region of localized necking is a greater fraction of the gage length for the 8 mm extensometer than for the
50.8 mm extensometer.

The results showed that subsized tensile specimens could be used to predict some of the tensile properties, mainly those prior to necking, which include yield strength (YS), ultimate tensile strength (UTS), uniform elongation and strain hardening behavior.

![Engineering stress-strain curves for subsized tensile specimens and normal size specimens.](image)

**Figure 3.10** Engineering stress-strain curves for subsized tensile specimens and normal size specimens.
CHAPTER 4: EXPERIMENTAL METHODS

In this Chapter, details of the experimental procedures are presented, including material selection and design principles, thermomechanical profiles employed in torsion testing for hot strip rolling simulations, and characterization methods for microstructure and mechanical properties after torsion testing.

4.1 Materials

Five microalloyed steels with additions of Ti, Nb, Mo, Cr and V were designed with ASPPRC sponsor input and the compositions are shown in Table 4.1. The atomic ratio of microalloying elements (Ti+Nb+V) to carbon and nitrogen is also shown. Steel Ti-Mo is the base steel essentially identical to the composition of JFE’s NANOHITEN® [6], while Steel V-Mo and Steel Nb-Mo use V-Mo and Nb-Mo microalloy additions, respectively, to compare the effects of alternative alloy systems with respect to microstructure evolution and mechanical properties. A higher amount of Ti is used in Steel High-Ti to explore the influence of greater additions on precipitation states and to achieve higher strength. Steel Ti-Cr is a Mo-free steel in which Cr is used as a replacement for Mo. Mo, as reported in JFE’s NANOHITEN® [6], was included to retard the formation of pearlite or coarse cementite at grain boundaries and to achieve increased resistance to precipitate coarsening. As an addition that suppresses ferrite formation [74], it is expected that Cr in Steel Ti-Cr would exhibit similar effects as Mo. Steel V-Mo and steel Nb-Mo were designed to maintain a constant atom fraction of microalloy additions, as in the Ti-Mo base steel composition.

Each material was received as 5 sections removed from plates with thickness of about 20 mm. The as-received plates were rolled from ingots of about 63.5 mm in thickness without any specific control of thermomechanical processing, or any subsequent heat treatment.

4.2 Gleeble® 3500 Torsion Testing

Figure 4.1 schematically shows the thermomechanical profile used for torsion testing on the Gleeble® 3500, which was designed to simulate the hot strip rolling process.
Table 4.1—Chemical Composition of Experimental Steels (in wt pct).

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Nb</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>M/C*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Mo</td>
<td>0.045</td>
<td>1.59</td>
<td>0.21</td>
<td>-</td>
<td>0.2</td>
<td>0.090</td>
<td>-</td>
<td>-</td>
<td>0.029</td>
<td>0.004</td>
<td>0.005</td>
<td>0.01</td>
<td>0.46</td>
</tr>
<tr>
<td>V-Mo</td>
<td>0.043</td>
<td>1.59</td>
<td>0.21</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>0.093</td>
<td>0.016</td>
<td>0.013</td>
<td>0.006</td>
<td>0.01</td>
<td>0.40</td>
</tr>
<tr>
<td>Nb-Mo</td>
<td>0.045</td>
<td>1.58</td>
<td>0.21</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
<td>0.18</td>
<td>-</td>
<td>0.031</td>
<td>0.004</td>
<td>0.005</td>
<td>0.01</td>
<td>0.48</td>
</tr>
<tr>
<td>Ti-Cr</td>
<td>0.045</td>
<td>1.58</td>
<td>0.21</td>
<td>0.351</td>
<td>-</td>
<td>0.096</td>
<td>-</td>
<td>-</td>
<td>0.032</td>
<td>0.004</td>
<td>0.004</td>
<td>0.01</td>
<td>0.50</td>
</tr>
<tr>
<td>High-Ti</td>
<td>0.048</td>
<td>1.59</td>
<td>0.21</td>
<td>-</td>
<td>0.2</td>
<td>0.150</td>
<td>-</td>
<td>-</td>
<td>0.029</td>
<td>0.004</td>
<td>0.005</td>
<td>0.01</td>
<td>0.73</td>
</tr>
</tbody>
</table>

*M-microalloying elements Ti, Nb, V; C-carbon and nitrogen.

Figure 4.1  Schematic illustration of thermomechanical profile used for torsion testing.

The specimens were heated up to 1250 °C and isothermally held at 1250 °C for 10 min, which simulates the slab reheating period. The isothermal holding temperature of 1250 °C was adopted to ensure that all of the microalloy carbonitrides would dissolve in the austenite (except for TiN), based on the calculated results shown in Figure 2.9. An 8-pass twisting schedule was employed to simulate the multi-pass hot strip rolling process, modified from a rolling schedule proposed by N. Nakata and M. Militzer [75], as shown in Table 4.2. The torsion angle in Table 4.2 was calculated using Equation 3.1. The torsional strain rate is 1 s⁻¹ for all passes. The last twisting temperature was set at 950 °C, representing the finish rolling temperature, because the transformation temperature of austenite to ferrite is relatively high due to low carbon content, and to avoid extensive strain induced precipitation in the austenite. The parameters varied in the experimental matrix are the cooling rate after deformation and the isothermal holding temperature,
which simulate run-out table cooling rate and coiling temperatures, respectively. The experimental matrix is shown in Table 4.3. All of the five steels were tested using these six conditions. After isothermal holding, each specimen was quenched to room temperature by helium.

**Table 4.2 – Torsion Experiment Schedule Simulating 8-pass Hot Rolling Process [75].**

<table>
<thead>
<tr>
<th>Pass No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equivalent Strain</td>
<td>1.00</td>
<td>0.36</td>
<td>0.51</td>
<td>0.41</td>
<td>0.33</td>
<td>0.30</td>
<td>0.25</td>
<td>0.10</td>
</tr>
<tr>
<td>Torsion Angle (radian)</td>
<td>9.57</td>
<td>3.45</td>
<td>4.88</td>
<td>3.92</td>
<td>3.16</td>
<td>2.87</td>
<td>2.39</td>
<td>0.96</td>
</tr>
<tr>
<td>Temperature °C</td>
<td>1100</td>
<td>1050</td>
<td>1003</td>
<td>986</td>
<td>973</td>
<td>963</td>
<td>956</td>
<td>950</td>
</tr>
<tr>
<td>Interpass Time (s)</td>
<td>10</td>
<td>80</td>
<td>4.8</td>
<td>3.2</td>
<td>2.3</td>
<td>1.7</td>
<td>1.3</td>
<td>-</td>
</tr>
</tbody>
</table>

**Table 4.3 – Experimental Matrix.**

<table>
<thead>
<tr>
<th>Testing Condition</th>
<th>Cooling Rate (°C/s)</th>
<th>Isothermal Holding Temperature(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>650</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td>600</td>
</tr>
<tr>
<td>3</td>
<td>20</td>
<td>550</td>
</tr>
<tr>
<td>4</td>
<td>40</td>
<td>650</td>
</tr>
<tr>
<td>5</td>
<td>40</td>
<td>600</td>
</tr>
<tr>
<td>6</td>
<td>40</td>
<td>550</td>
</tr>
</tbody>
</table>

Torsion testing was conducted under high vacuum (<10^{-3} torr) to avoid oxidation of the specimen. Temperature was controlled and monitored using both a K-type thermocouple and optical pyrometer, as mentioned in previous chapter. A constant power angle was used to heat the specimen to 650 °C, at which temperature the optical pyrometer control was started, and the specimen was heated at a constant rate of 25 °C per second to the reheating temperature. Temperature control was switched to thermocouple control after the last twist at 950 °C, wherein the temperature during the cooling and coiling periods was controlled by the thermocouple welded on the shoulder of adjacent to the gage section (Figure 3.9).

During hot rolling, ambient cooling was used, which resulted in a higher rolling temperature than scheduled at each pass using the interpass times in Table 4.2. Gas quenching to achieve the scheduled temperature was avoided here during the short interpass time to avoid an
ever larger temperature deviation from the scheduled rolling temperature.

The cooling rate after deformation was achieved by helium quenching. Two independent quenching channels were employed, each with a ball valve to adjust the helium flow rate. Several trials were made to find the best flow rate to accurately simulate the desired cooling rates of 20 and 40 °C/s. Once the specimen cooled to the planned isothermal holding temperature, the quenching channel having a higher flow rate of helium was turned off, with the other one remaining open to stabilize the temperature at the beginning stage of coiling. After isothermal holding for 60 min, each specimen was quenched to room temperature using helium.

4.3 Microstructure Characterization

Light optical microscopy (LOM) and transmission electron microscopy (TEM) were used to characterize the microstructure of each specimen. As discussed in Section 3.3, due to strain variations in the torsion specimen, specimens for microstructure characterization needed to be prepared from the position where the applied strain equals the equivalent strain. As schematically shown in Figure 4.2, the coupon obtained from the as-twisted torsion specimens was sliced into three parts, and one thin piece was sliced off in order to examine the surface at the “equivalent radius” position (shown as darker plane in Figure 4.2). The thickness of the sliced thin piece varied as the coupons were not machined to exactly the same dimensions. It should be noted that the exact “equivalent radius” plane is impossible to obtain, as it is actually a curved surface within the coupon.

Specimens for LOM were prepared from all of the five steels with six different processing conditions. The specimens were diamond polished to 1 μm and etched using 3% nital solution for 15 seconds. Carbon extraction replicas for investigation of precipitates in a transmission electron microscope were also prepared from all the specimens, which were first polished to 1 μm and then etched with 2% nital solution. A carbon film was deposited and then scored on the specimens. The film was floated off using one of two different solutions: 10% nital solution and an aqueous solution with 3.3 volume percent nitric acid, 3.3 volume percent acetic acid and 0.1 volume percent hydrofluoric acid in water [76]. The lifted carbon films were collected on 200 mesh Cu grids for subsequent TEM investigation. A Philips CM 200 TEM was operated at 200 kV in bright filed illumination to characterize the precipitates. Energy dispersive spectroscopy (EDS) was employed to analyze the chemical compositions of selected regions of the replica containing
precipitates. EDS analyses were conducted using the 10 μm selected area aperture.

![Diagram of specimen preparation](image)

**Figure 4.2** Specimen preparation for microstructure analysis, using coupons positioned as shown in Figure 3.3.

### 4.4 Mechanical Evaluation

Duplicate subsized tensile specimens were machined from each as-twisted torsion sample and uniaxial tensile tests were conducted using a MTS screw driven frame. A constant crosshead speed of 2.54 mm/min (0.1 inches/min) was adopted and the displacement was measured using an 8 mm extensometer. As discussed in Section 3.3, the subsized tensile specimens were expected to provide useful mechanical properties prior to necking.

Vickers microhardness testing was conducted on all specimens, which were those after LOM analysis. A 1000 gf load was employed with 10 s hold time to make the indent. 7 indents of 0.05 inch apart were made and an average value was calculated. Results of microhardness testing are included in Appendix B.
Chapter 4 presented the experimental methods to simulate hot strip rolling using Gleeble® 3500 torsion testing and to characterize microstructures as well as mechanical properties. In this chapter, the experimental results are presented and discussed. Examples of thermal profiles from Gleeble® 3500 torsion testing are addressed to verify the accuracy of the simulation, followed by matrix microstructure and precipitate characterization. A general discussion about the relationship between microstructure and mechanical properties is also presented.

5.1 Gleeble® 3500 Torsion Testing Evaluation

Results of torsion testing as the method to simulate the hot rolling process were discussed in this section.

5.1.1 Simulation of Hot Rolling

Two examples of thermal profiles for torsion testing are shown in Figure 5.1, with processing conditions involving a 40 °C/s cooling rate, and 650 °C and 550 °C coiling temperatures, respectively. As mentioned in Section 3.2.4, both a thermocouple and pyrometer were employed to control and monitor the temperature, with pyrometer control covering the reheating and rolling periods, and shoulder thermocouple control covering the cooling and coiling periods.

As shown in Figure 5.1, small variations of temperature existed at the beginning stage of the coiling period. For different specimens, the temperature variation was slightly different. Because the isothermal holding treatment required a balance between heating and helium quenching, it is difficult for the Gleeble® 3500 system to obtain a stable temperature instantly after cooling. For all the specimens, the variations of temperature during isothermal holding were less than 30 °C.

The pyrometer started gathering valid data during heating when the temperature exceeded 600 °C. A small temperature gradient existed between the measurements of the thermocouple and pyrometer during the reheating period, but once cooling started, the temperature gradient was
hardly noticeable. During the coiling period at a temperature of 550 °C, the pyrometer reading remained at 600 °C as it cannot read below 600 °C, and the thermocouple showed a very steady value. Previous thermal gradient testing (Figure 3.6) showed that during the coiling period the temperature gradient was very small and can be neglected between the gage section and grip section very close to gage section during this stage of processing.

From the data obtained from torsion testing, the hot rolling thermal profile was accurately simulated on the gage section, though with some small temperature variations and a small thermal gradient between the center and end (Figure 3.5).

![Thermal profile for torsion testing of Ti-Cr steel, with (a) cooling rate of 40 °C/s, coiling temperature of 650 °C and (b) cooling rate of 40 °C/s, coiling temperature of 550 °C. Temperatures below 600 °C are recorded as 600 °C by the pyrometer.](image-url)
5.1.2 Effects of Strain Variation

As discussed in Section 3.2, the shear strain varies over the cross section, which results in a non-homogeneous microstructure over the cross section. Figure 5.2 schematically shows the strain states in a torsion test. The engineering shear strain $\gamma$ at the position with radius $r$ can be given as

$$\gamma = \frac{r \theta}{L}$$

[Equation 5.1]

where $\theta$ is the twisted angle and $L$ is the test length of the specimen. Over the cross section, the shear strain increases from 0 at the center to a maximum at the surface, as shown in Figure 5.2 (b).

The influences of strain variation are revealed from the matrix microstructure. The example in Figure 5.3 shows two LOM images taken from the surface and center position of the cross section of High-Ti steel specimen, which is composed of polygonal ferrite with fractions of second constituents. It is obvious that the microstructure at the center is much coarser than at the surface.

![Figure 5.2](image)

Figure 5.2 Schematic showing of shear strain variation over the cross section of torsion specimen. (a) Schematic drawing of twisted specimen and (b) strain states over the cross section [77]. $a$: radius of torsion specimen; $\theta$: twisted angle; $\gamma$: shear strain; $r$: radius at random position; $MT$: torque; $\tau$: shear stress.

![Figure 5.3](image)

Figure 5.3 LOM images showing the effects of strain variation upon the cross section of gage section with (a) obtained from surface and (b) center of cross section. (High-Ti steel, with cooling rate of 40 °C/s, coiling temperature of 650 °C)
5.1.3 **Mechanical Properties of Subsized Tensile Specimens**

Tensile properties obtained from uniaxial testing of subsized specimens are presented in Table 5.1.

<table>
<thead>
<tr>
<th>Steel/Conditions</th>
<th>YS (MPa)</th>
<th>TS (MPa)</th>
<th>UE</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Mo</td>
<td>650-40</td>
<td>482</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>650-20</td>
<td>537</td>
<td>684</td>
<td>11.4% Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>600-40</td>
<td>542</td>
<td>689</td>
<td>10.9% Discontinuous yielding/data from single specimen</td>
</tr>
<tr>
<td></td>
<td>600-20</td>
<td>620</td>
<td>749</td>
<td>- Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>550-40</td>
<td>539</td>
<td>734</td>
<td>9.5%</td>
</tr>
<tr>
<td></td>
<td>550-20</td>
<td>588</td>
<td>726</td>
<td>-</td>
</tr>
<tr>
<td>Nb-Mo</td>
<td>650-40</td>
<td>544</td>
<td>660</td>
<td>12.5% Discontinuous yielding/data from single specimen</td>
</tr>
<tr>
<td></td>
<td>650-20</td>
<td>604</td>
<td>696</td>
<td>14.2% Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>600-40</td>
<td>602</td>
<td>-</td>
<td>- Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>600-20</td>
<td>603</td>
<td>687</td>
<td>9.4% Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>550-40</td>
<td>605</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>550-20</td>
<td>572</td>
<td>710</td>
<td>9.2%</td>
</tr>
<tr>
<td>V-Mo</td>
<td>650-40</td>
<td>440</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>650-20</td>
<td>396</td>
<td>-</td>
<td>- YS data from single specimen</td>
</tr>
<tr>
<td></td>
<td>600-40</td>
<td>494</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>600-20</td>
<td>502</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>550-40</td>
<td>450</td>
<td>668</td>
<td>7.0% Data from single specimen</td>
</tr>
<tr>
<td></td>
<td>550-20</td>
<td>485</td>
<td>-</td>
<td>- YS data from single specimen</td>
</tr>
<tr>
<td>High-Ti</td>
<td>650-40</td>
<td>495</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>650-20</td>
<td>500</td>
<td>629</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>600-40</td>
<td>522</td>
<td>663</td>
<td>7.7%</td>
</tr>
<tr>
<td></td>
<td>600-20</td>
<td>625</td>
<td>-</td>
<td>- YS data from single specimen</td>
</tr>
<tr>
<td></td>
<td>550-40</td>
<td>605</td>
<td>-</td>
<td>- YS data from single specimen</td>
</tr>
<tr>
<td></td>
<td>550-20</td>
<td>630</td>
<td>784</td>
<td>- Data from single specimen</td>
</tr>
<tr>
<td>Ti-Cr</td>
<td>650-40</td>
<td>523</td>
<td>627</td>
<td>- Data from single specimen</td>
</tr>
<tr>
<td></td>
<td>650-20</td>
<td>500</td>
<td>665</td>
<td>10.2%</td>
</tr>
<tr>
<td></td>
<td>600-40</td>
<td>645</td>
<td>728</td>
<td>10.9% Discontinuous yielding</td>
</tr>
<tr>
<td></td>
<td>600-20</td>
<td>635</td>
<td>733</td>
<td>10.4%</td>
</tr>
<tr>
<td></td>
<td>550-40</td>
<td>551</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>550-20</td>
<td>561</td>
<td>724</td>
<td>8.7% Data from single specimen</td>
</tr>
</tbody>
</table>

*Processing conditions are designated according to the coiling temperature (°C) and cooling rate following hot rolling (°C/s).
For most of the 0.2% yield strengths (YS), the average value of duplicate specimens obtained from one torsion sample was used, except for the specimens whose yield strength was found to be affected by preexisting material defects. These single sample tests are identified in the “comment” column. Tensile strength (TS) and uniform elongation (UE) are provided only for samples where failure occurred after necking.

Examples of stress-strain curves obtained from duplicate subsized tensile specimens of one torsion sample are shown in Figure 5.4. The tensile behaviors obtained from the duplicate specimens were very similar, which indicated that the reproducibility of the subsized specimens was good and the data are suitable for evaluating mechanical properties with as-twisted torsion specimens. Individual stress-strain curves of all specimens are provided in Appendix B.

A portion of the specimens failed with limited elongation, and those specimens do not provide valid tensile strength and elongation data. Fracture analysis was conducted on some specimens that failed with limited elongation. Figure 5.5 to Figure 5.7 show example defects observed at the specimen surface or fracture surface. The defects shown in Figure 5.5 and Figure 5.6 appear as small sharp cracks and were not visible prior to tensile testing. During deformation the feature appeared and opened due to plastic flow. In some cases, as shown by the example in Figure 5.6, surface cracks developed in the flat portion of the tensile sample away from the neck. In other cases as illustrated by the sample in Figure 5.5, the defect was located at a corner within the reduced sample section, opened with deformation, and developed a notch which localized plastic flow leading to a low ductility measurement due to failure prior to achieving a true ultimate tensile strength. Figure 5.7 (a) shows the fracture surface of one defect associated with a neck where non-metallic material, interpreted as an aluminum-silicon oxide based on the EDS data shown in Figure 5.7 (b), existed on a flat facet interpreted to be similar to the sharp surface defect in Figure 5.6. It is apparent that some inclusions remaining from the laboratory steelmaking/casting process may have influenced the results for certain specimens.

The location of the cracks affect the stress-strain behavior, as shown in Figure 5.8, in which the stress-strain curves of duplicate specimens were presented. Curve 1 in Figure 5.8 shows the tensile behavior of the specimen with the non-metallic alumino-silicate defect shown in Figure 5.7, which resulted in a fracture before reaching the true ultimate tensile strength. Curve 2 in Figure 5.8 shows the tensile behavior of the specimen with a crack located inside the neck.
Figure 5.4  Stress-strain curves of duplicates subsized tensile specimens: (a) Ti-Mo specimens coiled at 650 °C with cooling rate of 40 °C/s and (b) Nb-Mo specimens coiled at 650 °C with cooling rate of 20 °C/s.

Figure 5.5  Tested V-Mo steel subsized tensile specimen coiled at 650 °C with cooling rate of 20 °C/s. Fracture was initiated at the location indicated.
Figure 5.6  Tested subsized tensile specimen of Nb-Mo steel coiled at 600 °C with cooling rate of 40 °C/s. A crack on the specimen surface is indicated.

Figure 5.7  (a) SEM image showing fracture surface of V-Mo subsized tensile specimen (coiled at 650 °C with cooling rate of 20 °C/s) and (b) EDS analysis for chemical composition of the mixed oxide inclusion, which is pointed out in (a).
Figure 5.8  Stress-strain curves of duplicate subsized tensile specimens from V-Mo specimen coiled at 650 °C with cooling rate of 20 °C/s. Curve 2 corresponds to the specimen shown in Figure 5.5. The specimen shown in curve 1 was detected with surface defects which affected ductility measurement.

Figure 5.9  Stress-strain curves of duplicate subsized tensile specimens from Nb-Mo specimen coiled at 600 °C with cooling rate of 40 °C/s. Curve 2 corresponds to the specimen shown in Figure 5.6.

Development of the crack during plastic deformation resulted in a reduced load-carrying ability. Surface defects developed away from the neck during plastic deformation did not necessarily affect the stress-strain behavior, as shown in Figure 5.9. Curve 2 in Figure 5.9 shows the tensile behavior when a sharp crack existed in the flat portion of the tensile sample away from the gage length of the specimen and is very similar to curve 1, for which no obvious flaw was
detected in the specimen. All of the subsized tensile specimens were examined using a stereo microscope and the detected defects are summarized in Appendix B.

5.2 Matrix Microstructure Characterization via LOM

Specimens for LOM were prepared from 5 steels using 6 different processing conditions for each. The specimens were examined at a position on the plane where the shear strain equals the equivalent strain (Figure 4.2). Microstructures for the 5 steels: Ti-Mo, Nb-Mo, V-Mo, Ti-Cr and High-Ti are shown in Figure 5.10 to Figure 5.14, respectively, and are discussed further in the next section.

Krauss discussed the various ferrite morphologies employing classification from the International Institute of Welding (IIW) [51], which are briefly reviewed here and are used to describe the matrix microstructure in the current study. Polygonal ferrite (PF) forms at high austenite transformation temperatures with slow cooling rates and is featured with equiaxed ferrite grains separated by continuous, linear boundaries. Quasi-polygonal ferrite (QF) usually forms when passing directly from the single-phase austenite region to the single-phase ferrite region with limited partitioning of carbon between austenite and ferrite, and features roughly equiaxed grains, but irregular grain boundaries. Acicular ferrite (AF) forms at high cooling rates in the intermediate temperature transformation range, and features aligned and elongated grains. Granular ferrite (GF), or granular bainite (GB) also forms at intermediate transformation temperature range, and consists of granular MA islands in a featureless matrix. Features observed in experimental specimens are indicated in the micrographs shown in Figure 5.10 to Figure 5.14, and summarized in Table 5.2. The dark-etched constituents observed in certain specimens were not further investigated at higher magnification, and are termed as secondary constituents (SC) in the micrographs.

The ferrite grain size was not quantitatively analyzed for all of the specimens in this study, as some of the boundaries were not clearly revealed using the current etching methods. EBSD techniques or further etching development would be needed to develop samples from which accurate grain size measurements could be obtained in some specimens.

5.2.1 Effects of Processing Conditions on the Matrix Microstructure

Figure 5.10a and 5.10b, Figure 5.11a and 5.11b, Figure 5.12a and 5.12b, Figure 5.13a and
5.13b and Figure 5.14a and 5.14b show the microstructures for the 5 steels coiled at 650 °C. Each specimen is primarily composed of polygonal ferrite, with varying fractions of fine (dark etching) C-rich constituents. Figure 5.15 shows micrographs of the Ti-Mo and V-Mo steels at lower magnification. It was revealed that at coiling temperature of 650 °C, slower cooling rate resulted in more fraction of equilibrium phases, such as the V-Mo specimens coiled at 650 °C, as shown in Figure 5.15, the one with a higher cooling rate of 20 °C/s showed a banded pearlite feature (dark etched), with a greater fraction of C-rich constituents than the specimen with a cooling rate of 40 °C/s.

Figure 5.10c and 5.10d, Figure 5.11c and 5.11d, Figure 5.12c and 5.12d, Figure 5.13c and 5.13d and Figure 5.14c and 5.14d show the microstructures of the 5 steels coiled at 600 °C. For steel Ti-Mo and Nb-Mo, the matrix was polygonal ferrite with small fractions of second phases, while low temperature transformation products appeared in steels V-Mo, Ti-Cr and High-Ti. These low temperature transformation products are better viewed at higher magnification in Figure 5.16. For V-Mo, acicular ferrite is the dominant microstructure at 600 °C, and for the specimen cooled at 40 °C/s, there is a greater fraction of martensite/austenite (MA) constituent. For steel Ti-Cr, the matrix is composed of quasi-polygonal ferrite, and for the specimen cooled at 20 °C/s, a small fraction of acicular ferrite was observed. For steel High-Ti, the matrix is mainly composed of quasi-polygonal ferrite without substantial MA constituent. Some etching evidence of substructure was also observed, which is known to be a feature of quasi-polygonal ferrite [51].

Figure 5.10e and 5.10f, Figure 5.11e and 5.11f, Figure 5.12e and 5.12f, Figure 5.13e and 5.13f and Figure 5.14e and 5.14f show the microstructures of the 5 steels coiled at 550 °C. Selected microstructures at higher magnification are shown in Figure 5.17. The matrix microstructures are primarily composed of granular ferrite/bainite, with MA islands.

The results show that the coiling temperature had a more significant influence on the base microstructure than cooling rate in this study. A transition from polygonal ferrite to low temperature transformation products occurred with decreasing coiling temperature for all of the steels, indicating that the transformation from austenite to ferrite occurred during the coiling period for the cooling rates of 20 °C/s or 40 °C/s considered in this work. A decrease of coiling temperature from 650 °C to 600 °C resulted in a finer base microstructure, particularly for steels Nb-Mo, V-Mo and Ti-Cr, as shown in Figure 5.11, Figure 5.12 and Figure 5.13.
Figure 5.10  Light optical micrographs of Ti-Mo steel, etched with 3% nital for 15s.
Figure 5.11 Light optical micrographs of Nb-Mo steel, etched with 3% nital for 15s.
Figure 5.12 Light optical micrographs of V-Mo steel, etched with 3% nital for 15s.
Figure 5.13  Light optical micrographs of Ti-Cr steel, etched with 3% nital for 15s.
Figure 5.14 Light optical micrographs of High-Ti steel, etched with 3% nital for 15s.
<table>
<thead>
<tr>
<th>Steel/Conditions*</th>
<th>Matrix Microstructures</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Ti-Mo</strong></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>650-20</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>600-40</td>
<td>polygonal ferrite</td>
</tr>
<tr>
<td>600-20</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>550-40</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td>550-20</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td><strong>Nb-Mo</strong></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>650-20</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>600-40</td>
<td>polygonal ferrite</td>
</tr>
<tr>
<td>600-20</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>550-40</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td>550-20</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td><strong>V-Mo</strong></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>650-20</td>
<td>polygonal ferrite+ secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>600-40</td>
<td>acicular ferrite+ MA</td>
</tr>
<tr>
<td>600-20</td>
<td>acicular ferrite+ MA</td>
</tr>
<tr>
<td>550-40</td>
<td>acicular ferrite+ MA</td>
</tr>
<tr>
<td>550-20</td>
<td>acicular ferrite+ MA</td>
</tr>
<tr>
<td><strong>High-Ti</strong></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>650-20</td>
<td>quasi-polygonal ferrite</td>
</tr>
<tr>
<td>600-40</td>
<td>quasi-polygonal ferrite</td>
</tr>
<tr>
<td>600-20</td>
<td>quasi-polygonal ferrite</td>
</tr>
<tr>
<td>550-40</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td>550-20</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td><strong>Ti-Cr</strong></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>polygonal ferrite+ small fraction of secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>650-20</td>
<td>polygonal ferrite+ secondary C-rich constituents(SC)</td>
</tr>
<tr>
<td>600-40</td>
<td>acicular ferrite+ quasi-polygonal ferrite</td>
</tr>
<tr>
<td>600-20</td>
<td>acicular ferrite+ quasi-polygonal ferrite</td>
</tr>
<tr>
<td>550-40</td>
<td>granular bainite/ferrite</td>
</tr>
<tr>
<td>550-20</td>
<td>granular bainite/ferrite</td>
</tr>
</tbody>
</table>

*Processing conditions are designated according to the coiling temperature (°C) and cooling rate following hot rolling (°C/s)
Figure 5.15 Light optical micrographs of V-Mo steel, (a) coiling temperature: 650 °C; cooling rate: 40°C/s, (b) coiling temperature: 650 °C; cooling rate: 20°C/s.

Figure 5.16 Higher magnification light optical micrographs for selected steels at 600 °C coiling temperature (a) V-Mo steel with cooling rate of 40°C/s, (b) V-Mo steel with cooling rate of 20°C/s, (c) Ti-Cr steel with cooling rate of 20°C/s and (d) High-Ti steel with cooling rate of 20°C/s.
Figure 5.17  Light optical micrographs at higher magnification for low coiling temperature (550 °C) and high cooling rate (40°C/s) conditions of (a) Ti-Mo steel, (b) Nb-Mo steel, (c) V-Mo steel, (d) Ti-Cr steel and (e) High-Ti steel.
5.2.2 Effects of Microalloy Additions on Matrix Microstructure

The average grain sizes for the specimens of the 5 steels with polygonal ferrite microstructure (specimens coiled at 650 °C with cooling rate of 40 °C/s) were measured using the linear intercept method and are shown in Table 5.3. Among the 5 steels where a polygonal ferrite microstructure is present, the Nb-Mo steel has the finest microstructure compared with the other 4 steels. The Nb-Mo microstructure was also finer at the other coiling temperatures. It is well known that the presence of microalloy additions such as Nb, Ti and V in steel can effectively retard austenite recrystallization and these elements are commonly used to obtain “pancaked” austenite to obtain finer microstructures. Previous research [78] has shown that among the microalloying elements employed for austenite conditioning, Nb is reported to be the most effective (e.g. Figure 5.18), which explains why Nb-Mo steel had the finest microstructure in the current study, as the same rolling schedule and cooling profile was applied to all 5 steels.

Table 5.3 – Grain Size of Specimens with Polygonal Ferrite Microstructure of 5 Steels. (650 °C coiling and 40 °C/s cooling rate)

<table>
<thead>
<tr>
<th>Steel</th>
<th>Ti-Mo</th>
<th>Nb-Mo</th>
<th>V-Mo</th>
<th>Ti-Cr</th>
<th>High-Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size (µm)</td>
<td>11.2</td>
<td>5.8</td>
<td>15.3</td>
<td>12.4</td>
<td>9.4</td>
</tr>
</tbody>
</table>

Figure 5.18 Retardation of recrystallization due to presence of Nb, Ti and V for a 0.07C-1.4Mn-0.25Si steel [78].
Figure 5.19 shows the Ti-Mo and High-Ti microstructures at higher magnification. Both specimens were coiled at 600 °C following cooling at 20 °C/s. Compared with the more conventional polygonal ferrite microstructure in the Ti-Mo specimen, the High-Ti specimen exhibited a ferrite matrix designated here as quasi-polygonal ferrite [51], which exhibits irregular grain boundaries. More importantly, both of these steels contain very low fractions of second constituent. It is reported that quasi-polygonal ferrite forms in low-C steels when passing from the single-phase austenite region to the single-phase ferrite region with rapid cooling, suppressing the partitioning of carbon between austenite and ferrite during the “massive” transformation [51]. As Ti is known to be a strong ferrite stabilizer (Figure 5.20), a higher Ti content in the steel would enlarge the ferrite region and increase the transformation temperature. In combination with reduced solute carbon associated with any TiC formation in austenite, the elevated transformation temperature apparently helps to promote completion of ferrite formation at higher temperature and reduced second constituent fractions.

A comparison between Figure 5.10 and Figure 5.13 shows that the Ti-Mo and Ti-Cr steels had similar microstructures at coiling temperature of 650 °C and 550 °C. For the specimens coiled at 600 °C, as shown in Figure 5.21 with higher magnification, the Ti-Cr specimens showed more low temperature products compared to the polygonal ferrite matrix of the Ti-Mo specimens.
Figure 5.20 Effects of alloying additions on eutectoid transformation temperature associated with its ferrite stabilizing character. [47].

Figure 5.21 Light optical micrograph of (a) Ti-Mo steel and (b) Ti-Cr steel. Both specimens were coiled at 600 °C with cooling rate of 20 °C/s. The Ti-Cr specimen exhibited more low temperature transformation products than Ti-Mo specimen.

It was mentioned in Section 2.2 that the NANOHITEN® steel is reported to be composed of polygonal ferrite with grain size of about 3 µm, without the presence of pearlite or coarse cementite. The matrix microstructures presented above for the 5 steels in current study revealed that the specimens with higher coiling temperature were mostly composed of polygonal ferrite with varying amounts of second C-rich constituents. However, the grain sizes of most of the specimens with polygonal ferrite are much coarser than reported for NANOHITEN®, which is possibly due to the rolling schedule and cooling profile adopted for the Gleeble® 3500 torsion test.
Finer microstructures might be obtained with lower finish rolling temperature or larger applied strains. Since rolling schedule was fixed in current study, experiments could be designed to obtained finer microstructures.

5.3 Precipitate Characterization

While in-depth precipitate characterization would help to achieve the desired understanding of precipitate morphology and precipitation behavior in these steels, advanced characterization was outside the scope of the MS thesis timeframe for this work. In this section, selected characterization of precipitates on TEM extraction replica specimens is provided. The current study on 5 microalloyed steels is implicitly focused on the presence of extensive nanoscale precipitation and on understanding their morphology.

Example TEM bright field images of the carbon extraction replica specimens of the 5 steels are shown in Figure 5.22 to Figure 5.27. Particles with varying sizes were observed, and a significant amount of fine particles were detected in most of the specimens, as indicated by the arrow in the figures. Due to the low contrast of these small precipitates and resolution capability, in some cases the nanoscale precipitates could not be detected with the presence of large particles or features with higher contrast, as shown in Figure 5.27. It is revealed in Figure 5.27 (a) that with the presence of a large particle, the small precipitates (shown in the figure) were hardly noticeable, while when the large particle was moved out of sight, the small particles were more visible, as shown in Figure 5.27 (b). So in the current investigation of nanoscale precipitates using TEM, there were several specimens in which no nanoscale particles were detected, and this does not necessarily indicate that no nanoscale precipitate exists in these specimens.

Chemical composition was analyzed using energy dispersive spectroscopy (EDS) on a region where several precipitates were detected, to characterize the type of precipitates present. With the limited capability of the Philips CM 200 TEM, it was difficult to obtain the composition of a single particle. Examples of nanoscale precipitates observed in the 5 steels and corresponding EDS spectra are shown in Figure 5.28 to Figure 5.32. The EDS spectra indicated that the microalloying additions were incorporated in the particles, such as in Figure 5.28 (b), both Ti and Mo peaks were detected. Except for V-Mo steel (Figure 5.30 b), in which only the V peak was detected, Ti-Mo, Nb-Mo, Ti-Cr and High-Ti specimens all showed peaks of added microalloying elements along with Mo or Cr. The types of precipitates present could be generalized as mixed
microalloyed carbides.

Figure 5.22 TEM bright field images of carbon extraction replicas showing precipitates in Ti-Mo steel.
Figure 5.23  TEM bright field images of carbon extraction replicas showing precipitates in Nb-Mo steel.
Figure 5.24  TEM bright field images of carbon extraction replicas showing precipitates in V-Mo steel.
Figure 5.25 TEM bright field images of carbon extraction replicas showing precipitates in Ti-Cr steel.
Figure 5.26 TEM bright field images of carbon extraction replicas showing precipitates in High-Ti steel.
Figure 5.27  TEM bright filed images of precipitates in V-Mo steel coiled at 550 °C with cooling rate of 20 °C/s. (a) Small precipitates with presence of a large particle and (b) small precipitates in the region pointed out at (a).

Figure 5.28  TEM bright field images of (a) extraction replicas of Ti-Mo steel coiled at 600 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.29  TEM bright field images of (a) extraction replicas of Nb-Mo steel coiled at 650 °C with cooling rate of 40 °C/s and (b) corresponding EDS spectrum.
Figure 5.30  TEM bright field images of (a) extraction replicas of V-Mo steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.31  TEM bright field images of (a) extraction replicas of Ti-Cr steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.

Figure 5.32  TEM bright field images of (a) extraction replicas of High-Ti steel coiled at 650 °C with cooling rate of 20 °C/s and (b) corresponding EDS spectrum.
Nanoscale precipitates (< 10 nm) were present in most of the specimens. These precipitates appeared to have a random distribution or sometimes located at matrix features appeared to be grain boundaries or substructures, with examples shown in Figure 5.22 (f).

The precipitation states in the specimens vary with different microalloying additions. Extensive nanoscale precipitates (< 10 nm) were detected in specimens coiled at 650 °C and 600 °C of Ti-Mo and High-Ti steels, as shown in Figure 5.22 (d) and Figure 5.26 (b). A few large precipitates (> 50 nm) were also detected, as indicated by the arrow in Figure 5.33, which perhaps formed in austenite. In the specimens coiled at 550 °C of Ti-Mo and High-Ti steels, extensive matrix precipitation was not evident, although some regions of nanoscale precipitates near matrix features (grain boundaries/substructure) were noted, as revealed in Figure 5.22 (f) and Figure 5.26 (e). In Ti-Cr steel (Figure 5.25), nanoscale precipitates were mostly found distributed near grain boundaries/substructures, and extensive matrix precipitation was only detected in the specimen coiled at 650 °C with a 40 °C cooling rate, as shown in Figure 5.25 (a). In the Nb-Mo specimens, nanoscale precipitates were detected for higher coiling temperatures of 650 °C and 600°C, while for coiling temperature of 550°C, nanoscale precipitates were more limited, with most precipitates being relatively larger (<20 nm), as shown in Figure 5.23. It was also revealed that in the Nb-Mo steel, more of the larger precipitates were present compared with the Ti-Mo, High-Ti and Ti-Cr steels, consistent with greater precipitation during rolling. In the V-Mo steel, fewer nanoscale precipitates were observed in comparison to the other steels. Figure 5.24 (b) shows the larger precipitates in the V-Mo specimen coiled at 650 °C with cooling rate of 20 °C/s.

It can be noticed that the extensive nanoscale precipitation tended to occur with a ferrite matrix microstructure, while the bainite matrix was less favorable, in terms of fine microalloy precipitation under these conditions.

It was impossible to distinguish interphase precipitation from supersaturated matrix precipitation using the carbon extraction replicas specimens, as the relative position of the precipitates is not evident using this technique. TEM thin foil specimens were prepared from some of the steels in which extensive nanoscale precipitation was detected in the carbon extraction replicas specimens. An example is provided in Figure 5.34 for the Nb-Mo steel. While nanoscale precipitates were also observed using thin-foil TEM, the distribution of particles appeared random but the interphase morphology could not be ruled out.
Figure 5.33 TEM bright field images of large precipitate observed in (a) Ti-Mo steel coiled at 600 °C with cooling rate of 20 °C/s and (b) High-Ti steel coiled at 650 °C with cooling rate of 40 °C/s.

Figure 5.34 TEM bright field image of thin foil specimen from Nb-Mo steel, coiled at 650 °C with cooling rate of 40 °C/s.

5.4 General Discussion

In this section, the mechanical properties are discussed with regard to alloying and processing, in the context of the matrix microstructures as well as precipitation states of the 5 steels. Discussion is focused on properties resulting from valid measurements, including yield strength for all conditions, and tensile strength for samples that necked prior to fracture.

The important strengthening mechanisms for the steels in the current study can be generalized as: grain refinement strengthening, solid solution strengthening, dispersion
(precipitation) strengthening and transformation strengthening in some instances. As the main solid solution strengthening elements such as Si and Mn are essentially the same for all steels, the effects of solid solution strengthening are similar for each condition. By comparing the mechanical properties with consideration of the matrix microstructure, favorable processing conditions for precipitation strengthening can be deduced.

The yield strength is plotted as a function of coiling temperature in Figure 5.35 and Figure 5.36, for comparison among the 6 processing conditions for each of 5 steels.

Figure 5.35  Yield strength versus coiling temperature for 5 steels.

Figure 5.36  Yield strength versus coiling temperature for five steels at two cooling rates following the hot rolling simulation.
As shown in Figure 5.35, for the Ti-Mo steel, the specimens with the slower cooling rate of 20 °C/s had slightly higher yield strength for all coiling temperatures. The specimens cooled at 600 °C and 550 °C had similar yield strength for a given cooling rate, higher than that of the specimens cooled at 650 °C. It was shown earlier that the specimens cooled at 600 °C had a polygonal ferrite matrix (Figure 5.10c and 5.10d) with extensive nanoscale precipitation (Figure 5.22c and 5.22d), and the specimens cooled at 550 °C had a finer granular bainite/ferrite microstructure with less precipitation apparent (Figure 5.22e and 5.22f). So it can be assumed that the greater strengthening in the specimens cooled at 600 °C involves nanoscale precipitation compared with the strengthening from the stronger bainitic constituent which contributes in the specimens cooled at 550 °C. It is apparent on the basis of yield strength that for Ti-Mo steel, 600-20 is the optimum processing condition for precipitation strengthening among all 6 processing conditions investigated in the current study.

The tensile properties of specimens Ti-Mo 600-40 and 550-40 were both valid through necking with stress-strain curves shown in Figure 5.37. It is revealed that the specimen cooled at 550 °C, in which the matrix microstructure was mainly composed of granular bainite/ferrite, had a higher tensile strength but with less ductility than the specimen cooled at 600 °C having a single-phase polygonal ferrite matrix. It is stated in literature review that a single-phase ferritic matrix may exhibit better elongation than a bainite/ferrite microstructure, as shown in Figure 1.1. The higher tensile strength exhibited in some specimens cooled at 550 °C is because a larger amount of work hardening occurred with larger amount of M/A constituent. It is confirmed that the steels with a polygonal ferrite matrix can achieve similar yield strengths as bainite/ferrite microstructures through precipitation strengthening while exhibiting better elongation properties.

Grain refinement also plays an important role in the strengthening of microalloyed steels. The Nb-Mo steel exhibited higher yield strengths than the other steels in most of the processing conditions, even though some of the precipitates observed in Nb-Mo specimens were larger than those in the Ti-containing steels. In JFE’s NANOHITEN®, it is reported that the contribution to strengthening from ferrite grains of about 3 µm diameter is approximately 300 MPa (Figure 2.2), which is similar to the amount of precipitation strengthening [6]. It is not apparent from the work here that Ti provides better strengthening than Nb, but differences in the ferrite matrix could perhaps influence hole-expansion behavior, which was not addressed in the present work.
Figure 5.37 Engineering stress-strain curves of two Ti-Mo steel specimens, coiled at 600 °C and 550 °C with cooling rate of 40 °C/s.

The yield strength was plotted versus grain size for the steels having a polygonal ferrite matrix in the current study (specimens coiled at 650 °C with cooling rate of 40 °C/s), for which the grain sizes were given in Table 5.3. The results are shown in Figure 5.38. Results for a steel similar to NANOHITEN® are also included for comparison using property and grain size (3µm) data reported in literature [6].

The amounts of strengthening from grain refinement and solid solution were calculated from the equation below [79]:

\[
Yield\ Strength(MPa) = 15.4 \left[ 3.5 + 2.1(\%Mn) + 5.4(\%Si) + 23(\%N_f) + 1.13(d^{-\frac{1}{2}}) \right]
\]

where d is grain diameter and \(N_f\) is free nitrogen in solution, which is neglected in the current calculation, presuming that all nitrogen formed nitrides with microalloy elements. The amounts of Si and Mn are from Table 4.1, which are essentially the same for all 5 steels used in current study. The solid line plots the relationship in Equation 5.2 for steels containing 0.21% Si and 1.59% Mn. Additional strengthening is presumed to result from precipitation.

The amount of precipitation strengthening can be estimated and compared among the various steels in Figure 5.38. For these different steels with the same processing condition, the
Ti-Cr steel appeared to provide the greatest amount of precipitation strengthening, about 200 MPa, and the precipitation strengthening increments for the Ti-Mo, High-Ti and Nb-Mo steels were essentially similar, slightly greater than in V-Mo steel. NANOHITEN® obtained about 300 MPa from precipitation strengthening based on the reported grain size and properties [6]. Processing parameters applied to the experimental steels thus far have not achieved the extent of strengthening reported for NANOHITEN® in literature.

It is found in Figure 5.36 that the V-Mo steel had the lowest yield strengths for most of the processing conditions compared with other steels, which is probably due to reduced precipitation strengthening, as acicular ferrite is commonly known to be a stronger microstructure than polygonal ferrite. The low carbon and modest nitrogen levels may also influence this behavior.

The yield strength of Ti-Cr specimens was not affected by cooling rate, as shown in Figure 5.35. The specimens coiled at 600 °C had higher yield strengths than the specimens coiled at 650 °C and 550 °C. The higher strength at 600 °C is probably due to the combined effects of grain refinement and precipitation strengthening. A comparison between the 650-20 and 550-20 specimens showed that the 550-20 specimen with granular bainite/ferrite microstructures exhibited less elongation than the 650-20 specimen, similar to the results observed for the Ti-Mo steel.
The higher Ti content in High-Ti steel resulted in higher yield strength than the Ti-Mo steel at a coiling temperature of 550 °C, with no obvious effects on yield strength for the other processing conditions.

Discontinuous yielding behavior was observed in some of the tensile specimens, as noted in the “comment” column in Table 3.2. An example of discontinuous yielding behavior from the Nb-Mo steel is shown in Figure 5.39. As summarized in Table 3.2, discontinuous yielding behavior occurred in some specimens with higher coiling temperatures of 650 and 600 °C; these specimens were composed of mainly polygonal ferrite with limited amounts of M/A constituents. Since these steels are predominantly low carbon ferrite, it can be assumed that the discontinuous yielding behavior resulted from the Cottrell pinning of dislocations by interstitial atoms during the coiling period [51], which indicated that the total carbon (or nitrogen) content was not all consumed to form microalloy carbides (nitrides). The High-Ti specimens did not exhibit discontinuous yielding. The low temperature transformation products exhibited continuous yielding, perhaps due to their higher dislocation content.

![Figure 5.39](image_url)  
**Figure 5.39**  Engineering stress-strain curves of Nb-Mo steel specimen coiled at 650 °C with cooling rate of 40 °C/s.
CHAPTER 6 : CONCLUSIONS

A method to simulate the entire hot rolling process by torsion testing on the Gleeble® 3500 was developed. A shoulder thermocouple was employed as well as optical pyrometer to monitor and control the temperature for the whole process. An optimum helium setup and flow rate was established to achieve accurate simulation of the thermal profile. Geometry of the torsion specimen was modified so that subsized tensile specimens could be obtained from as-twisted samples for mechanical property evaluation after torsion tests. Variations of microstructure were observed over the cross section of torsion specimens due to strain variation in torsion testing.

Characterization of the microstructure via light optical microscopy showed the matrix microstructure was mainly influenced by coiling temperature, which indicates that the transformation from austenite to ferrite occurred during the coiling period. Polygonal ferrite is the dominant microstructure for all specimens coiled at 650 °C with various amounts of C-rich second constituents among the different specimens. With decreasing coiling temperature, a transition of matrix microstructure from equiaxed ferrite to low temperature transformation products was observed. Granular bainite/ferrite is the dominant microstructure at a coiling temperature of 550 °C. The High-Ti steel exhibited quasi-polygonal ferrite at intermediate coiling temperature of 600 °C.

Investigation of carbon extraction replica specimens via TEM revealed the presence of nanoscale precipitation. Nanoscale precipitates (< 10 nm) are distributed either randomly in the matrix or at features appeared to be grain boundaries or substructures. Extensive nanoscale precipitation was observed in most of the specimens having a polygonal ferrite matrix. In the granular bainite/ferrite microstructure, fewer microalloy carbides were present. In the Nb-Mo specimens, nanoscale precipitates were present, along with some larger precipitates that would be less effective in precipitation strengthening but may enhance grain refinement.

The specimens with polygonal ferrite had similar or higher yield strengths than the specimens with granular bainite/ferrite microstructure, which indicates the effectiveness of precipitation strengthening from extensive nanoscale precipitates. The specimens with polygonal ferrite showed better ductility than the specimens with granular bainite/ferrite microstructure.
Significant strengthening due to grain refinement was evident in the Nb-Mo steel.

The specimens with similar microstructure (polygonal ferrite matrix, extensive nanoscale precipitation) exhibited lower yield strength values than reported for NANOHITEN®. Coarser grains for the specimens in current study resulted in less strengthening from grain refinement for the processing conditions examined, which indicated that the yield strength of these experimental steels can be increased with further grain refinement.
CHAPTER 7: FUTURE WORK

Mechanical property evaluation using subsized tensile specimens from as-twisted torsion samples should be further investigated, to obtain valid mechanical properties after torsion testing and to characterize possible effects of shear pre-strain on tensile behavior.

More detailed characterization such as chemical composition and volume fractions of the precipitates should be conducted to quantitatively evaluate the effects of processing conditions and alloying additions. Further examination of the precipitation behavior by TEM and atom probe would be helpful.

A wider range of processing conditions can be investigated to obtain detailed precipitation response and seek a finer ferrite microstructure. Quantitative analysis can be conducted to estimate the contribution to yield strength from grain refinement and precipitation.
REFERENCES


APPENDIX A.  GLEEBLE® 3500 TORSION TESTING

A.1 Modified Gleeble Torsion Specimen Geometry

Dimensions of the desired (planned) modified torsion specimen geometry are shown in Figure A.1. In the modified torsion specimen, a portion of the grip section closest to the gage length was changed compared with standard torsion specimen, for the purpose of preparing subsized tensile specimen after torsion testing.

![Modified Gleeble torsion specimen geometry](image)

Figure A. 1 Modified Gleeble torsion specimen geometry (dimensions in inch).

A.2 Gleeble® 3500 Torsion Testing Program Setup

An example program employed to simulate the hot rolling process on Gleeble® 3500 with coiling temperature of 600 °C and cooling rate of 40 °C/s is shown in Figure A.2.
Figure A.2  Torsion testing program employed to simulate hot rolling process on Gleeble® 3500. (processing conditions are: 600 °C coiling temperature; cooling rate: 40 °C/s)
Figure A.2 Torsion testing program employed to simulate hot rolling process on Gleeble® 3500. (processing conditions are: 600 °C coiling temperature; cooling rate: 40 °C/s) (continued)

A.3 Helium Flowing Rate

<table>
<thead>
<tr>
<th>Cooling rate</th>
<th>Quench 1</th>
<th>Quench 2</th>
<th>Valve</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 °C/s</td>
<td>4 turns</td>
<td>1 turn</td>
<td>40~45 psi</td>
</tr>
<tr>
<td>20 °C/s</td>
<td>2 turns</td>
<td>1 turn</td>
<td>40~45 psi</td>
</tr>
</tbody>
</table>
APPENDIX B. MECHANICAL PROPERTIES

B.1 Microhardness Testing Results

The microhardness data obtained from Vickers microhardness testing are summarized in Table B.1 (average of 7 measurements). A comparison was made among the 5 steels, as shown in Figure B.1.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>650-40</th>
<th>650-20</th>
<th>600-40</th>
<th>600-20</th>
<th>550-40</th>
<th>550-20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Mo</td>
<td>249.5</td>
<td>275.9</td>
<td>279.1</td>
<td>271.5</td>
<td>272.8</td>
<td>290.8</td>
</tr>
<tr>
<td>Nb-Mo</td>
<td>246.0</td>
<td>264.8</td>
<td>278.8</td>
<td>270.6</td>
<td>286.2</td>
<td>287.5</td>
</tr>
<tr>
<td>V-Mo</td>
<td>216.8</td>
<td>216.4</td>
<td>258.8</td>
<td>259.2</td>
<td>255.0</td>
<td>244.3</td>
</tr>
<tr>
<td>Ti-Cr</td>
<td>256.4</td>
<td>255.9</td>
<td>285.0</td>
<td>281.0</td>
<td>275.8</td>
<td>263.5</td>
</tr>
<tr>
<td>High-Ti</td>
<td>240.3</td>
<td>249.7</td>
<td>300.2</td>
<td>301.3</td>
<td>298.3</td>
<td>294.4</td>
</tr>
</tbody>
</table>

Figure B. 1 Microhardness versus coiling temperatures for 5 steels.

B.2 Engineering Stress-Strain Curves for Subsized Tensile Specimens

The engineering stress-strain curves for duplicate subsized tensile specimens of the 5 steels are shown in Figure B.2 to Figure B.6. The defects observed in the specimens are summarized in Table B.2.
Figure B. 2  Engineering stress-strain curve of Ti-Mo subsized specimens.
Figure B. 3  Engineering stress-strain curve of Nb-Mo subsized specimens.
Figure B. 4   Engineering stress-strain curve of V-Mo subsized specimens.
Figure B. 5  Engineering stress-strain curve of Ti-Cr subsized specimens.
Figure B. 6   Engineering stress-strain curve of High-Ti subsized specimens.
Table B. 2 – Summary of Defects Detected in Subsized Tensile Specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Defects</th>
<th>Fracture position</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-Mo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>1 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td>650-20</td>
<td>1 crack at necking position</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>600-40</td>
<td>1 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 localized necking</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>600-20</td>
<td>1 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td>550-40</td>
<td>1 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>550-20</td>
<td>1 extensometer knife defect</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>Nb-Mo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>1 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>650-20</td>
<td>1 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>600-40</td>
<td>1 no obvious flaws</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>600-20</td>
<td>1 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>550-40</td>
<td>1 surface cracks</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>550-20</td>
<td>1 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>V-Mo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>650-40</td>
<td>1 surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 brittle fracture</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>650-20</td>
<td>1 defects on fracture surface</td>
<td>within extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>600-40</td>
<td>1 brittle fracture/fraction at grip</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 brittle fracture</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td>600-20</td>
<td>1 brittle fracture</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 brittle fracture</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>550-40</td>
<td>1 surface cracks</td>
<td>on the edge of extensometer</td>
</tr>
<tr>
<td></td>
<td>2 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td>550-20</td>
<td>1 surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2 crack at fracture position</td>
<td>outside extensometer</td>
</tr>
</tbody>
</table>
Table B.2 – Summary of Defects Detected in Subsized Tensile Specimens (continued)

<table>
<thead>
<tr>
<th>Ti-Cr</th>
<th>650-40</th>
<th>1</th>
<th>no obvious flaws</th>
<th>within extensometer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>surface cracks</td>
<td>within extensometer</td>
</tr>
<tr>
<td>650-20</td>
<td>1</td>
<td>no obvious flaws</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>no obvious flaws</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>600-40</td>
<td>1</td>
<td>surface cracks</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>no obvious flaws</td>
<td>outside extensometer</td>
<td></td>
</tr>
<tr>
<td>600-20</td>
<td>1</td>
<td>surface cracks</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>surface flaws/localized necking</td>
<td>outside extensometer</td>
<td></td>
</tr>
<tr>
<td>550-40</td>
<td>1</td>
<td>surface cracks</td>
<td>outside extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>crack at necking position</td>
<td>outside extensometer</td>
<td></td>
</tr>
<tr>
<td>550-20</td>
<td>1</td>
<td>no obvious flaws</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>High-Ti</td>
<td>650-40</td>
<td>1</td>
<td>surface cracks</td>
<td>outside extensometer</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>650-20</td>
<td>1</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>surface cracks</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>600-40</td>
<td>1</td>
<td>no obvious flaws</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>no obvious flaws</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>600-20</td>
<td>1</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>brittle fracture</td>
<td>within extensometer</td>
<td></td>
</tr>
<tr>
<td>550-40</td>
<td>1</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
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<tr>
<td></td>
<td>2</td>
<td>brittle fracture</td>
<td>outside extensometer</td>
<td></td>
</tr>
<tr>
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<td>on the edge of extensometer</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>crack at necking position</td>
<td>within extensometer</td>
<td></td>
</tr>
</tbody>
</table>