MECHANICAL BEHAVIOR OF LOW ALLOY STEELS WITH BAINITIC AND BAINITIC-MARTENSITIC STRUCTURE

A THESIS SUBMITTED TO THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES OF MIDDLE EAST TECHNICAL UNIVERSITY

BY

BAŞAK TEZGÖR

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE IN METALLURGICAL AND MATERIALS ENGINEERING

DECEMBER 2019

Approval of the thesis:

MECHANICAL BEHAVIOR OF LOW ALLOY STEELS WITH BAINITIC AND BAINITIC-MARTENSITIC STRUCTURE

submitted by **BAŞAK TEZGÖR** in partial fulfillment of the requirements for the degree of **Master of Science** in **Metallurgical and Materials Engineering Department, Middle East Technical University** by,

Prof. Dr. Halil KALIPÇILAR Dean, Graduate School of Natural and Applied Sciences	
Prof. Dr. Hakan GÜR Head of Department, Metallurgical and Materials Engineering	
Prof. Dr. Bilgehan ÖGEL Supervisor, Metallurgical and Materials Engineering, METU	
Examining Committee Members:	
Prof. Dr. Abdullah ÖZTÜRK Metallurgical and Materials Engineering, METU	
Prof. Dr. Bilgehan ÖGEL Metallurgical and Materials Engineering, METU	
Prof. Dr. Cevdet KAYNAK Metallurgical and Materials Engineering, METU	
Prof. Dr. Rıza GÜRBÜZ Metallurgical and Materials Engineering, METU	

Assist. Prof. Dr. Erkan KONCA Metallurgical and Materials Engineering, Atılım University

Date:

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Name, Surname: Başak Tezgör

Signature :

ABSTRACT

MECHANICAL BEHAVIOR OF LOW ALLOY STEELS WITH BAINITIC AND BAINITIC-MARTENSITIC STRUCTURE

Tezgör, Başak M.S., Department of Metallurgical and Materials Engineering Supervisor: Prof. Dr. Bilgehan ÖGEL

December 2019, 77 pages

This study aims to investigate the mechanical behavior of bainitic structures and bainitic-martensitic mixed structures in low alloyed steels. In this study, 51CrV4 and 60SiMn5 steels were used with different heat treatment cycles to get bainitic and bainitic-martensitic mixed structures. The specimens were austenitized at 880°C for 51CrV4 and at 950°C for 60SiMn5. It is followed by quenching in salt bath and isothermal holding at various temperatures above and below M_s temperature. Hardness, tensile and charpy impact testing were carried out. Both in 51CrV4 and 60SiMn5 steels, the specimens with bainite-martensite mixed structures show higher UTS values than 100% bainitic ones. The %elongation of 51CrV4 steel specimens were higher than that of 60SiMn5 specimens. The charpy impact toughnesses of 51CrV4 steel specimens varied in the range 12-25 J whereas that of 60SiMn5 specimens were below 10 J.

Keywords: bainite, bainitic - martensitic mixed steels, heat treatment, 51CrV4, 60SiMn5

BEYNİTİK VE BEYNİTİK-MARTENSİTİK YAPILI DÜŞÜK ALAŞIMLI ÇELİKLERİN MEKANİK DAVRANIŞLARI

Tezgör, Başak Yüksek Lisans, Metalurji ve Malzeme Mühendisliği Bölümü Tez Yöneticisi: Prof. Dr. Bilgehan ÖGEL

Aralık 2019, 77 sayfa

Bu çalışma ile beynitik ve beynitik-martensitik yapılı düşük alaşımlı çeliklerin mekanik davranışlarının incelenmesi amaçlanmaktadır. Bu çalışmada, beynitik ve beynitik - martensitik yapıları elde etmek için 51CrV4 ve 60SiMn5 çeliklerine farklı ısıl işlemler uygulanmıştır. 51CrV4 numuneleri 880°C, 60SiMn5 numuneleri 950°C'de östenitlenmiş; daha sonra numunelere M_s sıcaklığının üstünde ve altında olmak üzere farklı sıcaklıklardaki tuz banyosunda suverilmiş ve numuneler izotermal olarak bu sıcaklıklarda bekletilmiştir. Numunelere sertlik, çekme ve darbe testleri uygulanmıştır. Hem 51CrV4 hem de 60SiMn5 numunelerinde, beynitik - martensitik yapıya sahip numuneler 100% beynitik numunelerden daha yüksek çekme dayanımı göstermektedir. 51CrV4 numunelerinin %uzama miktarları 60SiMn5 numunelerine göre daha yüksek değerlerdedir. 51CrV4 numunelerinin darbe test sonuçları 12-25 J arasında iken 60SiMn5 numunelerinde bu değerin 10 J'un altında olduğu görülmüştür.

Anahtar Kelimeler: beynit, beynitik - martensitik çelikler, ısıl işlem, 51CrV4, 60SiMn5

To my precious family...

ACKNOWLEDGMENTS

First and foremost I would like to express my sincerest gratitude and thanks to my supervisor Prof. Dr. Bilgehan ÖGEL who guided me throughout this work with his valuable comments, understanding, encouragement and patience.

I gratefully acknowledge the support of ASIL ÇELİK A.Ş. in supplying the 60SiMn5 and 51CrV4 steels used in the experiments.

My very great appreciation goes to Dr. Süha Tirkeş for his understanding, generous support and advice. I am more than grateful for his on time helps whenever I go through the trouble.

My special thanks go to Serkan Yılmaz for his help in SEM and also for his friendship, advice and conversations for the last three years.

I would like to thank Cemal Yanardağ and Yusuf Yıldırım for giving me their best helps to finish my experiments at times.

I would also like to express my thanks to my lab mates Zeynep Öztürk, Ece Naz Yurtsever and Simge Bakır for their friendship and support.

My sincere thanks go to Kayacan Kestel for patiently answering all my questions about LaTex program and helping during thesis writing process.

I thank my dear friend Osman Emre Uzer for everything during the last eight years. He was the person that make the biggest contribution during undergraduate program and with his support and friendship during master program, I am able to finish this research.

Special thanks to my lifelong friends Damla Yıldız, Ecem Cansu Asan, Esra Kaderli, Göksun Güven and Özge Tuncer for always there for me.

I owe deepest gratitude to my teammate and my best friend, Oğuzhan Yeşil for his endless support, encouragement and inspiration during this research. I have been blessed with his unconditional friendship.

Finally, the greatest gratitude is to my parents Pınar Tezgör and Ertan Tezgör; and my grand-parents Yüksel Tezgör, Perihan Gür and Oğuz Gür for their endless patience, love and support throughout my life. I am so lucky to have them.

TABLE OF CONTENTS

ABSTRACT
ÖZ
ACKNOWLEDGMENTS
TABLE OF CONTENTS
LIST OF TABLES
LIST OF FIGURES
LIST OF ABBREVIATIONS
CHAPTERS
1 INTRODUCTION
2 LITERATURE REVIEW
2.1 Background Information
2.1.1 Bainitic Steels
2.1.1.1 Microstructure of Bainite
2.1.1.2 Kinetics of Bainite Formation
2.1.1.3 Heat Treatment of Bainite
2.1.1.4 Mechanical Properties of Bainite
2.1.2 Martensitic Steels
2.1.3 Bainitic - Martensitic Structured Steels

	2.2 51Cr	V4 Steel	15
	2.2.1	Effect of Carbon	17
	2.2.2	Effect of Chromium	19
	2.2.3	Effect of Vanadium	20
	2.2.4	Mechanical Properties of 51CrV4	21
	2.3 60Sil	Mn5 Steel	23
	2.3.1	Effect of Silicon	23
	2.3.2	Effect of Manganese	25
	2.3.3	Mechanical Properties of Si-Mn Steels	25
3	MATERIAI	LS AND METHOD	27
	3.1 Mate	rials	27
	3.2 Heat	Treatment Process	28
	3.2.1	Equipment	28
	3.2.2	Heat Treatment Parameters	28
	3.2.3	Microstructural Characterization	29
	3.2.4	Phase Area Fraction Measurement	29
	3.2.5	Mechanical Testing	29
	3.2.	5.1 Hardness Testing	29
	3.2.	5.2 Tensile Testing	29
	3.2.	5.3 Notched Bar Charpy Impact Test	30
4	RESULTS		31
	4.1 Isothe	ermal Transformation (TTT) Data	31
	4.1.1	Calculated TTT Diagrams of 51CrV4 and 60SiMn5	32

	4.1.2 Microstructural Characterization of 51CrV4 and 60SiMn5	36
	4.1.3 Mechanical Test Results of Heat Treated 51CrV4 and 60SiMn5	51
	4.1.3.1 Hardness Tests	52
	4.1.3.2 Tensile Tests	54
	4.1.3.3 Charpy Impact Tests	56
5	DISCUSSION	63
	5.1 Microstructural Characterization	63
	5.2 Mechanical Properties	65
6	CONCLUSION	71
RI	EFERENCES	73

LIST OF TABLES

TABLES

Table 2.1	Microstructure and tensile results of newly developed steel [29]	15
Table 2.2	Mechanical properties of quenched and tempered flat product 51CrV4	16
Table 2.3	Chemical composition of 51CrV4 steel	17
Table 2.4	Mechanical properties of SAE 6150 Steel [38]	22
Table 2.5	Mechanical properties of 60SiMn5	23
Table 2.6	Chemical composition of 60SiMn5 steel	23
Table 3.1	Chemical composition of 51CrV4 and 60SiMn5 steels	27
Table 4.1	Phase Transformation data of 51CrV4 and 60SiMn5	34
Table 4.2	Summary of heat treatment processes of 51 CrV4 and 60 SiMn5	35
Table 4.3	Mechanical test results of 51CrV4 and 60SiMn5 samples	51
Table 4.4	Hardness values of heat treated 51CrV4	52
Table 4.5	Hardness values of heat treated 60SiMn5	53

LIST OF FIGURES

FIGURES

Figure 2.1	Schematically representation of bainite transformation on TTT	
diagra	m of a) eutectoid steel, b) alloy steel [6]	4
Figure 2.2	Upper bainite formed by isothermal holding [13]	5
Figure 2.3	Types of microstructure of bainite.	6
Figure 2.4 volum	Change of yield strength with lower (a) and upper (b) bainite the fraction [10]	7
Figure 2.5 tempe	Schematic representation of heat treatment processes at different ratures [21]	10
Figure 2.6	Hardness versus quenched steel's carbon content [25]	12
Figure 2.7 [27]	Effect of alloying elements on hardness of tempered martensite	13
Figure 2.8 tion di	Isothermal transformation and continuous cooling transforma- iagrams of 51CrV4 [30]	16
Figure 2.9 tent [1	Change in transformation temperatures as varying carbon con- 7]	17
Figure 2.10 E and	Comparison of transformation driving forces $(\Delta G \gamma \alpha)$ for Alloy Alloy F [5]	18
Figure 2.11 Cr add	Time-temperature-transformation curves of low carbon base steel, led steel and Cr-Al added steel. [33]	19

Figure	2.12	Engineering strain-stress curves of low carbon Cr-free steel and	
	Cr-ado	led steel. [32]	20
г.	0.10		
Figure	2.13	Stress-strain curve of 51CrV4 in different microstructural states	
	[37].		22
Figure	2.14	Predicted time – temperature - precipitation diagram for cemen-	
0	tite pro	ecipitation with 1.2% wt carbon at different silicon content [5]	24
	···· P··		
Figure	3 1	Eurnaces used in this study a) muffle furnace b) salt bath c) dry	
riguie	boot of	rumaces used in this study a) munic furnace b) sait bath c) dry-	20
	neat st		20
Figure	3.2	Tensile Test Specimen.	30
-			
Figure	3.3	V-notched Charpy Impact Test Specimen.	30
Figure	4.1	TTT diagram of 51CrV4 constructed by using JMatPro	31
	1.0		22
Figure	4.2	TTT diagram of 60SiMn5 constructed by using JMatPro	32
Figure	4.3	Schematic cooling paths of 51CrV4	33
C			
Figure	4.4	Schematic cooling paths of 60SiMn5	34
Figure	45	The SEM micrograph of isothermally transformed 51CrV4 spec-	
Inguie	imen s	at 300° C for 30 minutes. Bainite sheaves	36
	inten e		50
Figure	4.6	The SEM micrograph of isothermally transformed 51CrV4 spec-	
	imen a	at 300°C for 1 hours. Bainite sheaves.	37
Figure	4.7	The SEM micrograph of isothermally transformed 51CrV4 spec-	
	imen a	at 300°C for 5 hours. Bainite sheaves.	37
Figure	4.8	a)Optical and b) SEM micrographs of isothermally transformed	
8	51CrV	V_4 specimen at 300°C for 5 minutes	39
			.,
Figure	4.9	a)Optical and $b)$ SEM micrographs of isothermally transformed	
	51CrV	74 specimen at 300°C for 10 minutes.	40

Figure 4.10	a)Optical and b) SEM micrographs of isothermally transformed	
51CrV	/4 specimen at 300°C for 15 minutes	41
Figure 4.11 imen a bainite	The SEM micrograph of isothermally transformed 51CrV4 spec- at 270°C for 5 hours. Martensite (dark blocky islands) with lower e sheaves	42
Figure 4.12	The SEM micrographs of isothermally transformed 51CrV4 spec-	
imen a bainite	at 240°C for 1 hour. Martensite (dark blocky islands) with lower e sheaves	43
Figure 4.13 imen a bainite	The SEM micrograph of isothermally transformed 51CrV4 spec- at 240°C for 5 hours. Martensite (dark blocky islands) with lower e sheaves.	43
Figure 4.14 calcul	Area fractions of 240°C/1h, 240°C/5h and 270°C/5h specimens ated by ImageJ. Dark area is bainite, white area is martensite	44
Figure 4.15 for <i>a</i>)2	SEM micrograph of 51CrV4, oil quenched tempered at 220°C 2 hours and b) 4 hours. Tempered martensite.	45
Figure 4.16 specin	The optical micrograph of isothermally transformed 60SiMn5 nen at 250°C for 4 hours. Lower bainite sheaves.	46
Figure 4.17 imen a	The SEM micrograph of isothermally transformed 60SiMn5 spec- at 250°C for 4 hours. Carbide-free lower bainite sheaves	47
Figure 4.18 men a	SEM micrograph of isothermally transformed 60SiMn5 speci- t 200°C for 1 hour.	48
Figure 4.19 imen a	The SEM micrograph of isothermally transformed 60SiMn5 spec- at 200°C for 4 hours.	48
Figure 4.20 specin trix.	The optical micrograph of isothermally transformed 60SiMn5 nen at 200°C for 4 hours. Bainite with austenite/martensite ma-	49

Figure	4.21	The SEM micrograph isothermally transformed 60SiMn5 spec-	
	imen a	t 180°C for 4 hours. \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots	49
Figure	4.22	SEM micrograph of isothermally transformed 60SiMn5 speci-	
	men at	250°C for 1 hours. The flat regions are most probably martensite	
	islands	3	50
Figure	4.23	Hardness of 51CrV4 and 60SiMn5 samples	54
Figure	4.24	Tensile test results of 51CrV4 samples	55
Figure	4.25	Tensile test results of 60SiMn5 samples	56
Figure	4.26	Charpy impact test results of 51CrV4 samples	57
Figure	4.27	The SEM micrographs of 300°C/10min sample's fractured sur-	
	faces.		57
Figure	4.28	The SEM micrographs of T450°C/2h sample's fractured surfaces.	58
Figure	4.29	The SEM micrographs of 300°C/5h sample's fractured surfaces.	58
Figure	4.30	The SEM micrographs of 240°C/5h sample's fractured surfaces.	59
Figure	4.31	Charpy impact test results of 60SiMn5 samples	59
Figure	4.32	The SEM micrographs of 200°C/4h sample's fractured surfaces.	60
Figure	4.33	The SEM micrographs of 250°C/1h sample's fractured surfaces.	60
Figure	4.34	The SEM micrographs of 250°C/12h sample's fractured surfaces.	61

LIST OF ABBREVIATIONS

M_s	Martensite Start Temperature
\mathbf{M}_{f}	Martensite Finish Temperature
\mathbf{B}_s	Bainite Start Temperature
γ	Austenite
α	Ferrite
T ₀	Temperature that bainitic ferrite and austenite have identical free energy at similar composition
HV	Vickers Pyramid Number
M/A	Martensite/Austenite
SEM	Scanning Electron Microscope
FCC	Face Centered Cubic
BCT	Body Centered Tetragonal
В	Bainite
B+M	Bainite + Martensite
ТМ	Tempered Martensite

CHAPTER 1

INTRODUCTION

Quality of a product can be defined as the fulfill of the customer's requirements while performing three basics: cost efficient, defect-free production in short time. These requirements also stand for steel industries. To provide these requirements at the same time, steel industries prefer to produce either high strength steels with low ductility or vice versa. However, for optimization of both strength and ductility, bainitic steels recieve attention in today's industry.

Bainite is a microstructure that forms by decomposition of austenite [1]. It was found in 1930s by Bain and Davenport who firstly referred it as acicular ferrite [1]. It showed excellent strength, toughness and ductility balance relative to conventional steels. Its mechanical properties mostly controlled by microstructure characteristics which can be altered by heat treatment processes [2].

To get the balance of strength, toughness and ductility in bainitic steels, the width of the bainitic ferrite sheaves have to be reduced. It is known that as the isothermal transformation temperature of bainite decreases, thickness of bainite sheaves decrease [3]. In addition to this, low temperature transformation limits the size of constituent which can be developed with uniform properties [4]. This is similar to formation of fine martensitic microstructures. However, bainitic formation differ from martensitic one in terms of controlled response to heat treatment process [4].

In most of the literature works, the focus is put on alloy added bainitic steels that are formed by transformation of high carbon steels at low temperature. As an example, in a study of Soliman et al., different alloying elements were added to high carbon steel to get low temperature bainite [5]. This study shows that silicon has an effect of suppressing the brittle cementite, while chromium and manganese increasing hardenability. Therefore, a carbide free bainite shows higher toughness even at 2000 GPa strength levels.

Also, there are studies about low carbon carbide-free bainitic transformations. Cabalerro et al. studied low carbon steel with addition of Si, Cr and Mn and obtained very fine bainite with high strength and toughness at low transformation temperatures without carbide precipitation [4].

Most of the studies try to get superior properties by obtaining fully lower bainitic microstructure. Despite all, there are very few studies about the behaviour of bainite in medium carbon low alloy steels. Moreover, the mechanical proerties of bainite-martensite mixtures are not studied to a high extent.

In this study, low alloyed spring steels 51CrV4 and 60SiMn5 were heat treated to obtain bainitic and bainitic – martensitic structures. The Si content of 60SiMn5 steel is expected to suppress the cementite formation. However, the bainite of 51CrV4 steel should contain cementite. Therefore, the properties of the two different carbide phase mixture can be compared. It is also aimed to obtain bainitic – martensitic structure at lower temperatures and compare their mechanical properties with fully bainitic and tempered martensitic steels.

CHAPTER 2

LITERATURE REVIEW

2.1 Background Information

Mechanical properties of a steel can vary due to its microstructure. There are three basic microstructures that a steel can have: pearlite, bainite and martensite. These microstructures are controlled by heat treatment process of steel. All of these microstructures form during cooling from austenite phase.

$\gamma \rightarrow \alpha + \mathrm{Fe}_3\mathrm{C}$

Austenite decomposes into ferrite and cementite phases. Depending on the cooling rate and temperature, shapes and the amount of ferrite and cementite modifies which alteres the microstructure of steel. Pearlite is the multiphase structure that form at relatively slow cooling rates and high temperatures. Because of this, it has a coarse microstructure which reduces its mechanical properties such as strength and hardness.

Bainite is the multiphase structure that forms by decomposition of austenite. It takes shape lower temperatures than pearlite. As a result, bainite has finer microstructure than pearlite. Finer microstructure results as better mechanical properties. Bainite formation needs an isothermal treatment most of the time.

On the other hand, martensite is a structure that has a non-equilibrium single phase. It transforms from austenite diffusionless by quenching to the temperatures under the martensite start temperature (M_s). It has very fine, needle-like microstructure that causes extremely high strength but brittleness.

2.1.1 Bainitic Steels

Bainite is a eutectoid transformation products which consists of plate shape ferrite and non - lamellar cementite (Fe₃C) phases under standard conditions [6]. It has been found 1930s as an unstratified microstructure obtained above martensite transformation temperature [7]. It forms by austenitic decomposition to get a shape depending on the temperature of transformation [1]. The highest temperature that bainite and martensite can be form in a given composition steel is consider as bainite start temperature (B_s) and martensite start temperature (M_s) which are the upper and lower limits of bainite generation at isothermal heat treatment [8].

The diagrams that illusturate isothermal transformation referred as time-temperaturetransformation (TTT) diagrams [6]. Figure 2.1 illustrates two TTT diagrams of a eutectoid iron-carbon steel and an alloyed steel respectively. Presence of an alloying elements change the shapes of the curves in TTT diagrams such as shifting the austenite-to-pearlite transformation nose and forming a separate bainite nose [6].



Figure 2.1: Schematically representation of bainite transformation on TTT diagram of a) eutectoid steel, b) alloy steel [6]

In Figure 2.1, "A" represents austenite, "B" is bainite, "P" is pearlite and "M" is martensite. In Figure 2.1a above the nose and in Figure 2.1b the upper C-curve stands for ferritic and pearlitic transformations starts, whereas the lower parts stands for bainitic ones [4].

2.1.1.1 Microstructure of Bainite

Bainitic microstructure is classified with ferrite morphology and carbide distribution [9] whose two morphologies hinge on transformation temperatures [10]. Bainite obtained at lower and higher temperature ranges is named as lower bainite and upper bainite respectively [7]. Lower bainite shapes at temperatures that near to martensite transformation temperature while the upper one does at temperature close to the pearlite transformation temperature [10].

In 1947, Hultgren find out upper bainite that firstly called it troostite. As his investigation, it could form by initial precipitation of ferrite with Widmanstatten morphology followed by cementite precipitation on its sides [11]. It is shown in Figure 2.2.

Upper bainite is formed by ferrite lath aggregates which also named as sheaves [12]. These sheaves have almost same orientation which cause the same oriented-sheaves named as packets [12]. During bainitic transformation of austenite, carbides precipitates between the ferritic sheaves that resulted by separating the ferritic laths with cementite [11].



Figure 2.2: Upper bainite formed by isothermal holding [13].

As the transformation temperatures decreases, carbon starts to precipitate inside the ferritic sheaves forms fine needle like plates with the ferrite laths containing carbide in [11]. This structure is named as lower bainite. They are formed relatively low temperatures than upper bainite which resulted finer microstructures. Garcia-Mateo et al explained how microstructure of bainite become finer with decrement of transformation temperature. According to his study, thickness of bainitic ferrite relies on dislocation density of austenite and austenite strength at transformation temperature which increase as the temperature of transformation decreases [8].



Figure 2.3: Types of microstructure of bainite.

The major difference of upper and lower bainite is cementite particles which precipitates inside the ferrite plates in lower bainite. Also, due to higher amount of dissolved carbon in ferrite laths and more uniformly distribution of carbides, lower bainite shows better mechanical properties [12].

Due to the fact of lower bainite have fine and more uniform distributed carbides, more stresses required for dislocations to move on this carbides [10]. Besides, density of dislocation and the probability of dislocation pile-up in lower bainite is greater than the upper one [10]. These higher dislocation density and finer microstructure causes superior the properties of lower bainite, whereas high transformation temperatures that results to form upper bainite, causes weak mechanical properties [7].

Abbaszadeh et al. studied mechanical properties of upper and lower bainite which are seen at Figure 2.4. The results of comparison of upper and lower bainite's yield strengths support the previous explanations. According to Abbaszadeh's study, as the volume fraction of upper bainite increases in a mixture of upper bainite-martensite, the strength decreases without peaking in a point. On the contrary, a peak in yield strength is seen as the volume fraction of lower bainite increases in a mixture of upper bainite-martensite [10].



Figure 2.4: Change of yield strength with lower (a) and upper (b) bainite volume fraction [10]

2.1.1.2 Kinetics of Bainite Formation

Formation of ferritic and pearlitic microstructure occurs by diffusional mechanism while martensitic one's are by displacive, shear mechanism [14]. On the other hand, bainitic transformation takes place temperatures between these two mechanisms which makes unclear this phenomenon since it is discovered.

Mechanism of bainite formation is discussed by two different theories due to its two types; upper and lower bainite.

Diffusive Theory

After the first suggestion of formation of upper bainite by Hultgren, the concept was further investigated by Hillert who does not differ the Widmanstatten ferrite and bainitic ferrite since there is no discontuinity of kinetic [9]. He explains that nucleation of bainitic ferrite occurs at austenite grain boundaries and growth rate is driven by the carbon diffusivity. In other words, both take places via diffusion.

Oblak and Hehemann observed in their study that bainite has a slower growth rate than martensite which has an displacive formation mechanism. According to their study, bainitic growth of 'sub– units' which aggregate to form sheaves or packets. Since the transformation mechanism is controlled by the nucleation rate of the subunits, the growth rate is slower [15]. Lengthening of sub-units gains dominance over thickening due to difference of growth rate via directions. As a consequence of this, ledges are formed and start to propagate while growth of cementite and bainitic ferrite happens at the same time because of the fact that segmentation of carbon causes increment of cementite formation driving force. For validation of the diffusive theory, growth limits of both Widmanstatten ferrite and bainitic ferrite can be measured in terms of austenite carbon content. There are also some predictions and founding of bainite and other structures form by diffusion from the point of metallographic similarities, growth rates and surface relief characteristics [16].

Although there are supporting studies, the diffusive theory is limited since it is unable to count quantitatively for the effects of carbide forming alloy elements [16].

Diffusionless Theory

Other transformation mechanism that discussed is diffusionless at which growth occurs by displacive transformation [4]. Carbon supersaturated bainite forms from austenite during isothermal transformation where carbon content increases due to rejection of excess carbon by diffusion into austenite. As a result, the sub-unit of bainite nucleate and growth with smaller driving force [16]. The growth of these bainitic sub-units are instant and displacive until stopped due to adjacent austenite's plastic deformation [14]. After the formation of bainitic sub-unit, the formation mechanism goes on by autocatalytic nucleation and displacive growth [9].

In 1946, Zener analyzed austenitic decomposition with different alloying elements to find their effect on retarding bainitic transformation which was, in his words, "closely related" to martensitic transformation. His conclusion was that the effect of elements which pile up at nucleation sites to make carbides, were not associated to nucleation but to free energy for transformation on bainite and martensite. At lower temperatures, these substitutions are immobile; therefore, segregation to form carbides cannot take place. As a result, transformation driving force is changed due to this effect [16]. As a consequence of his work, Zener explained the changes in microstructure between upper an lower bainites by a diffusionless mechanism of bainite formation [16].

In recent research of Samanta et al., isothermal transformation below M_s temperature was studied and resulted by obtained bainite under M_s . According the research, the transformation mechanism is based on displacive, shear transformation where bainite have similar characteristic with martensite [14].

Cabalerro and Bhadesia tried to find the lowest temperature that can be produce bainite. According to their research, bainite cannot be formed above T'_0 where T_0 is the point of the chemical composition of austenite and ferrite have same free energy and T'_0 is similiarly but addition of ferrite's stored energy due to displacive mechanism. The chemical free energy change ($\Delta G\gamma\alpha$) accompanying the transformation of austenite without chemical composition change when neglecting all the stored energy terms is written;

 $G\gamma\alpha = G\alpha - G\gamma$

where G Gibbs free energy, α and γ are product and parent phases, respectively. As a result they can be achieved to get bainite under Ms temperature [4, 17].

2.1.1.3 Heat Treatment of Bainite

Mechanical properties of steels can be altered by modifying the heat treatment processes. Heat treatment is the most effective way to vary the microstructure of steel that outcome with a desired property.

For bainitic steels, heat treatment occurs in three basic steps: Austenitization, cooling down to temperature range of bainitic transformation, and air cooling to room temperature [18].

As explained in 2.1.1; bainite is an austenite decomposition product [1]. Because of that bainitic transformation starts by heating the steel to the austenitic ranges and holding there sufficient time to get homogenous austenite [19] [20]. After austenitization, steel is cooled down to bainitic transformation temperature range. This cooling process takes place either as continuous cooling or isothermally holding at a temperature after quenching to B_s temperature. For continuous cooling, final microstructure of steel depends on the cooling rate and cooling environment. Although this cooling technique is more practical and easy to perform, there is a chance of pearlite formation due to crossing over pearlitic transformation range which is detrimental for mechanical properties [6].

On the other hand, isothermal cooling is not the most efficient technique to carry on due to its cost and energy consumption. In order to obtain fully bainitic microstructure, steel should be held long enough isothermally at a decided temperature. The completion of transformation can be take months depending on the temperature. Figure 2.5 shows an example of the schemetic representation of heat treatment processes at different temperatures.



Figure 2.5: Schematic representation of heat treatment processes at different temperatures [21]

2.1.1.4 Mechanical Properties of Bainite

As indicated previously, microstructural characteristics determine the mechanical properties. It also goes for the bainitic steels. The balance of toughness/strength and ductility properties comes from the reduction in bainitic ferrite sizes [3]. As the transformation temperature decreases, thickness of the bainitic ferrite plates reduces. The reason for this is explained by the increase in the yield strength of austenite at low temperatures with influencing the plastic deformation accompanying bainite growth [22]. It is well known that grain size is the key factor for varying yield strength. Decreasing grain size causes more grain boundaries per unit volume that boundaries act as an obstacle for dislocation motion [23]. As a result, high interface density provides higher strength.

On the other hand, low transformation temperature in isothermal bainite transformation needs longer time to complete the transformation which can limit mass production rate [3].

With decreasing transformation time, the amount of bainite formation decreases. The remaining structure may transform into martensite which can cause decrement of toughness while increasing hardness [24]. On the other side, increment in holding time increases bainite formation, therefore; more carbon is moved into austenite. As a result, austenite may become stabilized at lower temperatures which leads to form retained austenite which can turn into martensite as cooled down to room temperatures. This is associated with the main reason for poor ductility of bainite [3]. If the retained austenite remains at room temperature, it will transform into martensite under the load of stress which is also a reason of decrement in low impact toughness. Consequently, retained austenite had a negative effect on impact toughness [22].

2.1.2 Martensitic Steels

As like bainite, martensite is one of the austenitic decomposition products that formed by rapid cooling to low temperatures after austenitization. It has a structure of a non - equilibrium single phase which has a plate-like or needle-like morphology. Since martensitic transformation happens by rapid quenching, carbon does not have time for diffusing i.e diffusionless transformation occurs [6].

For martensitic formation, temperature that steel quenched in must be lower than M_s temperature. Transformation is completed when temperature reaches M_f . Above this temperature range, martensite cannot be formed even at the high cooling rates. Because of this, martensitic transformation is a time independent process. In other words, martensite forms by athermal transformation [25].

Instead of growth mechanism, a typical reaction of martensite progresses by nucleation. Transformation occurs almost immediately where martensitic plates have their needle like shape. Due to this instantaneous transformation, kinetics of martensite is controlled by nucleation [26]. Nucleation of a plate completed until reaches its limit. After that, a new plate is nucleate instead of growing of nucleated plates. This process occurs due to the constrained form of the matrix [25].

Face-centered cubic (FCC) austenite transforms into body-centered tetragonal (BCT) martensite. Volume difference of FCC and BCT cause a strain in the matrix which gives the steel high strength. Additionally, diffusionless transformation leads carbon atoms remain as interstitial impurities which create a supersaturated solid solution. As a result of this, hardness of as quenched martensitic steel increases with increasing carbon content [6].(Figure. 2.6)

Although high hardness and strength, as quenched martensite has very poor ductility. It shows brittle fracture due to internal stress. The needle-like microstructure act as crack initiation points under stress which cause detrimental brittleness. To prevent this harmful effect, tempering operation is applied to martensite after quenching [25].



Figure 2.6: Hardness versus quenched steel's carbon content [25]

Tempering is a heat treatment process where martensitic steel is heated to a temperature between 250°C and 650°C which is below the eutectoid transformation temperature. By this process, the internal stress that martensite had is relieved. Tempering is a diffusional process where unstable BCT martensite transform into tempered martensite consist of stable ferrite and cementite [6].

martensite (BCT, single phase) \rightarrow tempered martensite (α + Fe₃C phases)

As-quenched martensite transforms by a displacive, shear mechanism. On the other hand, tempered martensite transforms isothermally which occurs with a chemical driving force [14].

Although hardness and strength of steel decreases due to tempering, toughness increases. In addition to that, the study of Grange et al [27], reported that alloying elements in steel can retard softening above 204°C. Figure 2.7 illustrates the effects of alloying elements on hardness of tempered martensite at 204°C for 1 hour. It has to be noted that the longer the tempering time, the lower the hardness of tempered martensite.



Figure 2.7: Effect of alloying elements on hardness of tempered martensite [27]

2.1.3 Bainitic - Martensitic Structured Steels

In 1960s, it was come to the fore that bainite/martensite structure had better mechanical properties than single structure of martensite since bainite was shaped earlier and separate the prior austenite grains that limit the martensite packet sizes [28].

As explained earlier, formation of athermal martensite occurs between M_s and room temperature during cooling from austenitization. On the other hand, bainite can be

formed by isothermal treatments below M_s . It has been stated at several studies that during isothermal transformation under M_s , a product that neither fully bainitic nor martensitic was formed. That formed martensite identified as isothermal martensite which has similarities with athermal martensite [21].

Navarro-Lopez et al. explained in their research that, martensite has a shape of lath and bainitic ferrite has a shape of acicular units in isothermal treatment below M_s . Martensitic laths are wider and longer than acicular units which are parallelly aligned [21].

Mechanical properties of bainitic/martensitic structured steels are expected to be better than a single phase structured steels. Abbaszadeh et al. studied mechanical properties of mixed bainitic/martensitic structured steels. According to their results, strength increases in bainitic/martensitic microstructure because lower bainite partitions the prior austenite grains which restricted the martensite sizes. Reducing the martensite packet size increases the strength. Moreover, strength of lower bainite is enhanced due to plastic constrain effect [10].

According to Zhi-jun et al., toughness of bainitic/martensitic structure depend on the size of the packets. Decreasing the size of structure, decreases the toughness [28]. Abbaszadeh et al. clarify this by explaining the effect of volume fraction on toughness. It is found that by increasing the volume of the bainite in a mixed structure of bainite and martensite increases the ductility and toughness [10].

Nie et al studied a newly developed steel that have a composition of 0.45%C $\neg 2.0\%$ Si $\neg 0.75\%$ Mn $\neg 0.9\%$ Cr. To investigate mechanical properties of different microstructures, heat treatment were applied on this steel [29]. The microstructure, heat treatment process and tensile strength of developed steel is given in Table 2.1.

Sampla	Quanching	Tomporing	Tensile
Sample	Quenching	Tempering	Strength
Bainite	875°C (30min) + 325°C (120s) +	$300^{\circ}C(120min)$	1075 MP ₂
Martensite1	oil quenching	300 C (1201111)	1975 Ivii a
Bainite	900°C (30min) + 350°C (120s) +	$300^{\circ}C$ (120min)	2065 MP ₂
Martensite2	oil queching	500 C (120mm)	2005 WII a
Martensite	925°C (30min) +	350°C (00min)	2025 MP ₂
	oil quenching	550 C (90mm)	2023 WII a

Table 2.1: Microstructure and tensile results of newly developed steel [29]

It is known that silicon can prevent carbide precipitation and leading thin retained austenite films between lower bainitic sheaves results carbide free bainite. According to the results of Nie et al., these thin films among the bainitic sheeves both eliminate the severe effect of carbide and may also decelerate the crack growth by making tip at the crack. As a result, strength of bainitic - martensitic steels can be better [29].

2.2 51CrV4 Steel

Spring steels are very wide range of steel with relatively high yield strength. High yield strength allows the steel withstand higher stresses without deformation permanently. 51CrV4 is one of the high strength spring steel used for heavy duty service condition like trains. In addition, heat treatment process is easily applied on 51CrV4 steels which is the reason for using them mostly as quenched and tempered conditions in industries.

51CrV4 is a grade of EN 10083 standard. It also graded as SAE 6150, SUP 10 and 50CrVA according to ASTM A29, JIS G4801 and GB/T 3077 standards, respectively. Isothermal cooling and continuous cooling diagrams of 51CrV4 is given in Figure 2.8.



Figure 2.8: Isothermal transformation and continuous cooling transformation diagrams of 51CrV4 [30]

Mechanical properties of quenched and tempered flat product 51CrV4 is given in Table 2.2. 51CrV4 is a medium carboned steel that main elements are chromium and vanadium. Chemical composition of 51CrV4 is given in Table 2.3.

Table 2.2: Mechanical properties of quenched and tempered flat product 51CrV4

Yield Strength (MPa)	>700							
Tensile Strength (MPa)	1000-1200							
Impact Strength (J)	>30							
	Wt%	%C	%Si	%Mn	%Cr	%V	%P	%S
---	--------	------	-----	-------------	-----	------	----------	----------
5	51CrWA	0.47	0.7	Max 0.025	0.9	0.1	Max 0.04	Max 0.04
	510174	0.55	1.1	IVIAN 0.023	1.2	0.25		

Table 2.3: Chemical composition of 51CrV4 steel

2.2.1 Effect of Carbon

Carbon is one of the main elements in steels. Depending on its concentration, properties of steel can be changed. The main effect of carbon in steels is retarding the transformation temperature. Increasing the carbon content cause suppression of M_s which let the bainitic transformation carrying out at lower temperatures [5].

Bhadeshia studied the effect of carbon on transformation temperatures and his results shows that as the carbon content increase, M_s temperature decreases significantly and the interval between B_s and M_s increases which enhances the chance of getting bainite at low temperatures [17]. The result of Bhadeshia is illusturated in Figure 2.9.



Figure 2.9: Change in transformation temperatures as varying carbon content [17]



Figure 2.10: Comparison of transformation driving forces ($\Delta G\gamma \alpha$) for Alloy E and Alloy F [5]

Soliman et al, studied two alloys with different carbon contents with Alloy E - 0.67%wt C and Alloy F - 0.55% wt C, respectively. According to results of study, decrement in the carbon content ends up in increment of bainite formation rate since transformation of Alloy F occurs higher temperatures than transformation of Alloy E [5]. Driving force comparison of Alloy E and Alloy F is illustrated in Figure 2.10.

Despite the decrement transformation temperature, high carbon concentration is limited in two points. On one hand, high carbon content makes steel poor for welding since untempered brittle martensite formed in heat affected zone. On the other hand, reduction of transformation temperature slows down the transformation rate results as extending time for fully bainitic transformation [8].

Considering the all, it is important to optimize the carbon content to get lower bainite with good mechanical properties. According to results of Soliman, bainitic microstructure of medium carbon content that heat treated in lower temperatures has optimized mechanical properties [5].

2.2.2 Effect of Chromium

Chromium as a substitutional alloying element in steel affect the bainitic formation in different ways. Goulas et al. [9] indicated that the presence of chromium delays the bainitic growth and restricts maximum bainitic fraction formed by isothermal treatment. The effect of Cr on continuous cooling diagram was studied by You et al. [31] who claimed that Cr addition split a single C-curve on CCT diagram which shows that austenite stability rises while bainitic transformation is retarded and M_s decreases. The study of Zhou et al. [32] explained these finding more detailed by investigating Cr effect on transformation and microstructure. The results showed that Cr has a restrainable effect on ferrite transformation.

As a study of Tian et al. [33], Cr addition improve austenite stability and hardenability. These results were obtained by the comparison of time-temperature-transformation curves of Cr+Al free base steel, a Cr added one and Cr-Al added steel. As seen in the Figure 2.11, Cr addition shifted the curve to bottom-right which eases to form bainite.

Eutectoid carbon content of steels can be decreased by Cr addition which make it possible to produce alike microstructures, i.e. properties, as hypereutectoid ones with lower carbon contents [9].



Figure 2.11: Time-temperature-transformation curves of low carbon base steel, Cr added steel and Cr-Al added steel. [33]

In the study of Zhou et al. [32], tensile properties of low carbon Cr-free and Cr-added steels were investigated. According to results, both tensile strength and yield strength of Cr-added steels were higher than Cr-free steel which is illustrated in Figure 2.12.

Moreover, Bracke and Xu [34] studied low carbon steel with differentiating the Cr content. Two steels with 0.3% wt and 0.9% wt was investigated to understand the Cr effect on mechanical properties of low carbon steel. As the result of Bracke and Xu, increment in Cr content improve the tensile strength, however the yield strength decreases. They explained this result as the increment of martensite-austenite islands presence which is dependent on Cr content. High Cr content induces higher fraction of secondary phases. These secondary phases improve strain hardening capacity which improve tensile strength but lowers the yield strength.



Figure 2.12: Engineering strain–stress curves of low carbon Cr-free steel and Cr-added steel. [32]

2.2.3 Effect of Vanadium

Vanadium is not commonly found in steels. However, it is known that vanadium mixed in solid solution during austenitization process and increases hardenability of steel. It is also mentioned in a study of Garcia-Mateo et al [35], V affects the bainitic transformation kinetics.

On contrary, Sourmai et al. [36] mentioned that bainite formation is not affected in lower temperature range except a minor acceleration explained by difference of prior austenite grain size. On the other hand, at higher temperature range, vanadium retards bainitic formation by segregate to the grain boundaries and limited driving force for nucleation of bainite.

2.2.4 Mechanical Properties of 51CrV4

As far as gathered from literature, quenched and tempered 51CrV4 was studied commonly, however there are few studies of isothermal bainitic transformation of 51CrV4.

Goulas et al. studied isothermal transformation of 51CrV4 at lower and higher transformation temperatures. Three samples of 51CrV4 isothermally treated at 300°C, 420°C and 510°C respectively after austenitization. According to the results of their research, bainitic ferrite's carbon content increases as decreasing transformation temperatures. When the morphology of lower bainite and upper one's compared, it is clarified that shapes of bainitic ferrite plates and carbides become coarser at higher temperature which is expected a decrement in mechanical properties [9].

Lambers et al. used 51CrV4 to investigate its mechanical properties and bainitic and martensitic phase transformation behavior. Tensile tests were performed in different microstructural states. Results can be seen in Figure 2.13, which indicates that fully martensitic specimen failed almost in elastic region while fully bainitic specimen has the highest strain level. It is also noticed that bainitic-martensitic mixed specimens have higher tensile stress than fully bainitic ones however there is not a systematic increment [37].



Figure 2.13: Stress-strain curve of 51CrV4 in different microstructural states [37].

Alp and Wazzan studied the effect microstructure on the mechanical behavior of SAE 6150 steel [38]. They compared tensile properties of martensitic, dual-phase and tempered martensitic SAE 6150 steels. Their conclusion was given in Table 2.4.

According to their results, tempered steel shows highest tensile strength with moderate ductility due to annealing out the resudial stress with tempering. On the other hand, highest yielding is seen in dual phase steel as a result of presence of pearlite. However, other mechanical properties are worse than tempered ones [38].

Processing Conditions	σ	UTS MPa	$\sigma_{\rm b}$	el, %	RA, %	BHN
AR		758	500	12.9	53.8	66
DP	459	590	342	25.0	69.0	56
М			146	1.9	1.5	90
Т	757	819	594	15.4	40.7	68

(a) AR: as received; DP: dual-phase steel with small amount of pearlite; M: martensitic; T: tempered; σ_y : yield stress; σ_b : breaking stress; BHN: Brinell hardness; UTS: ultimate tensile strength; el: elongation; RA: reduction in area.

Table 2.4: Mechanical properties of SAE 6150 Steel [38].

2.3 60SiMn5 Steel

Similar to 51CrV4, 60SiMn5 is a high strength spring steel used for heavy-duty vehicles.

Mechanical properties of 60SiMn5 is given in Table 2.5.

 Table 2.5: Mechanical properties of 60SiMn5

Tensile Strength (MPa)	1320-1520
Yield Strength (MPa)	>1300
Impact Strength (J)	>14

Basic additional elements of 60SiMn5 are silicon and manganese. These elements basicly affect the structure of steel by decreasing M_s and suppressing the carbide precipitation to form carbide free microstructure. Being a steel without carbide cause an expectation to increase in mechanical properties. Chemical composition of 51CrV4 is given in Table 2.6.

Table 2.6: Chemical composition of 60SiMn5 steel

Wt%	%C	%Si	%Mn	%P	%S
60SiMn5	0.55-0.65	1.0-1.3	0.9-1.3	Max 0.025	Max 0.05

2.3.1 Effect of Silicon

It is known that brittle cementite precipitate from austenite among ferrite plates that cause decrement of toughness during bainitic transformation [1]. In silicon alloyed steels, cementite precipitation from carbon enriched austenite is suppressed since silicon has low solubility in cementite [24] [4]. It plays a key role on dissolution and stability of carbide during phase transformation [39]. It has been indicated in many researches that silicon that about 2% wt bring front a carbide-free microstructure [4].



Figure 2.14: Predicted time – temperature - precipitation diagram for cementite precipitation with 1.2%wt carbon at different silicon content [5]

A study of Soliman and Palkowski was done a study with 1.2%wt carbon at different silicon content to get the predicted time – temperature - precipitation diagram for precipitation of cementite. The results are shown in Figure 2.14 that by the increasing of Si content the precipitation percentage of cementite decreases [5].

Silicon mobility at bainite formation temperatures is restricted. Cementite is trapped in the untransformed austenite leads to suppression of cementite that result as carbide free microstructure [5]. Carbide-free microstructure is mostly desired in bainitic steels since toughness increase in the absence of brittle cementite phase. As cementite plates cannot be developed, carbon enriches in austenite which lowers the M_s [5]. As a consequent of this, some amount of austenite stays untransformed after bainitic transformation. Therefore, carbide-free bainite composed of bainitic ferrite and untransformed austenite named as retained austenite [7]. As silicon concentration increases, percent of retained austenite increases which postpone the overall kinetics of bainitic transformation [39]. However, retained austenite has an undesired effect on mechanical properties of bainitic steels. Under the mechanical load, retained austenite transforms into brittle martensite phase which leads to decrement of toughness [22].

2.3.2 Effect of Manganese

Manganese has an effect of suppression M_s temperature with increasing austenite stability [23]. As a consequence of this effect, the bainite plates can be formed at low temperatures.

In a study of Soliman and Palkowski, Mn content of an alloyed steel altered. Decrement of the Mn content shows that Mn have a strong influence of enhancing the possibility of bainite formation by shifting T0 curve to lower carbon concentrations. Furthermore, addition of Mn lowers thermal stability of retained austenite which improves the rate of transformation [5].

2.3.3 Mechanical Properties of Si-Mn Steels

The usage of 60SiMn5 for the academical researches is quite rare. On the other hand, researches about Si-Mn containing steels can be helpful for the understanding of the mechanical behavior of 60SiMn5.

In a study of Santos et al., the effect of high carbon Si-Mn steels' microstructure on the mechanical properties had been investigated. The steel had 0.56%C, 1.43%Si, 0.58%Mn and 0.47%Cr. M_s and B_s temperatures of steel are 268° C and 594° C, respectively. Two samples austenitized at 900°C for 300s, one of them austempered at 400°C and the other one was 600°C for 300s. After that, both of them quenched to the water. 400°C austempered sample showed higher tensile strength, 1350 MPa, and ductility, 25% since it contains fine bainite with higher amount of retained austenite. On the other hand, it is seen that there is ferrite and pearlite in the microsturcture of 600° C austempered sample which reduces the strength to 1110 MPa [40].

Gao et al. studied a steel with 0.4%C–2.0%Mn–1.7%Si–0.4%Cr with different heat treatment processes. The mechanical properties of samples that obtained by bainite-based quenching and tempering (BQ&T), bainite austempering (BAT) treatments and bainite-based quenching with partitioning (BQ&P) were compared. The BQ&T sample has a bainitic - martensitic microstructure while BAT one has bainite with blocky martensite/austenite (M/A) islands and BQ&P has both bainite, martensite and

austenite in its microstructure. When their tensile strength were compared, it is seen that BAT and BQ&P samples have tensile strength of 1505 MPa and 1688 MPa with a % elongation of 21.2 and 25.2, respectively. On the other hand BQ&T sample has 1908 MPa strength with 10.9 % elongation which is quite lower than BAT and BQ&P samples. Since BAT and BQ&P samples have higher amount of retained austenite than BQ&T, they show better ductility. Besides, BQ&P sample has the highest impact toughness result of 82 J at 20°C due to the amount of retained austenite among samples which can be mainly seen nanosized thin film morhology. This morphology hinders the crack propogation due to its higher stability. However, BAT sample has M/A island that forms as blocky coarser morhology that has a low stability and can easily turns into the fresh martensite. These can cause secondary crack formation that results as the lowest toughness result, 28 J at 20°C [41].

CHAPTER 3

MATERIALS AND METHOD

Experimental part of this study consists of heat treatment processes, microstructural analysis by optical microscope and SEM, hardness measurements and tensile tests.

3.1 Materials

Two different spring steels were investigated in this study. First one was 51CrV4 which is a high strength spring steel that is used for heavy duty service condition like trains. Second one was 60SiMn5 which is a high strength, high silicon steel. The steels were supplied from Asil Çelik Sanayi ve Ticaret A.Ş. Chemical compositions of both 51CrV4 and 60SiMn5 are given in Table 3.1.

	Wt%	%C	%Si	%Mn	%P	%Cr	%V
51CrV4	Theorotical Composition	0.47-0.55	Max 0.4	0.7-1.1	Max 0.025	0.9-1.2	0.1-0.25
	Analyzed Composition	0.5	0.2	0.9	0.01	1.0	0.1
60SiMn5	Theorotical Composition	0.55-0.65	1.0-1.3	0.9-1.3	Max 0.025	-	-
	Analyzed Composition	0.6	1.2	1.0	0.01	_	_

Table 3.1: Chemical composition of 51CrV4 and 60SiMn5 steels

3.2 Heat Treatment Process

3.2.1 Equipment

For austenitization process, Protherm muffle furnace was used. On the other hand, isothermal bainitic transformation was carried out Protherm salt bath and Petrofer AS 135 salt is used for isothermal quenching. The specimens for microstructural study were, 20x20x40 mm³ in dimensions. They were cut by Metkon Metacut 251 Abrasive Cutter.



Figure 3.1: Furnaces used in this study a) muffle furnace b) salt bath c) dry-heat sterilizer.

3.2.2 Heat Treatment Parameters

The calculated TTT diagrams for 51CrV4 and 60SiMn5 were generated by JmatPro®. Austenitization temperatures of 51CrV4 and 60SiMn5 were selected as 880°C and 950°C, respectively. All samples were austenitized for 30 minutes and then directly quenched to the salt bath which was preheated and fixed to the desired isothermal holding temperatures. For tempering processes, samples were quenched to oil at room temperature after austenitization and then put in the preheated dry heat sterilizer for determined times. Heat treatment procedure was same for the mechanical test samples.

3.2.3 Microstructural Characterization

Samples for metallographic investigation were first cut with dimensions 20x20x30mm using Metkon Metacut 251 Abrasive Cutter to eliminate decarburized region. Then, they were mounted into bakalite by Metkon Ecopress 100. The samples were mechanically ground by using grit sand paper from 120 to 1200 on Metkon Grip0 2V Grinder – Polisher, respectively. Mecapol P230 Polisher and 6μ and 1μ of Metkon Diapat-P water-based polycrystalline diamond suspensions were used for fine polishing after grinding. For etching, 2% nital solution was used.

Microstructures were examined using an optical microscope, Huvitz Digital Microscope HDS-5800, and scanning electron microscopes, Jeol 6400 Scanning Electron Microscope and FEI 430 Nano Scanning Electron Microscope.

3.2.4 Phase Area Fraction Measurement

ImageJ software was used for digital measurement of area fraction. Since optical micrographs of phases have different colors, area fraction can be calculated by color contrast. Optical micrographs of samples were used for digital quantification.

3.2.5 Mechanical Testing

3.2.5.1 Hardness Testing

Hardness test were carried out by Emco Universal Digital Hardness Testing Machine using Vickers indenter under the load of 30 kgf. The average of five indents were taken as indicated values.

3.2.5.2 Tensile Testing

For tensile specimen preparation, ASTM E8/E8M – 16a standard was used. Specimens were round having 7 mm diameter and 40 mm gage length (Figure 3.2). IN-STRON 5582 Universal Testing Machine was used for tension tests. Three specimens

were tested for 51CrV4 and two specimens were tested for 60SiMn5 at each heat treatment process.



Figure 3.2: Tensile Test Specimen.

3.2.5.3 Notched Bar Charpy Impact Test

Specimens having dimensions $10x10x55 \text{ mm}^3$ and a V shape notch were prepared according to ASTM E23 – 16b (Figure 3.3). For each determined heat treatment processes, 3 specimens for either 51CrV4 and 60SiMn5 were tested. TINIUS-OLSEN pendulum type Charpy impact tester was used for tests. Fracture surfaces were analyzed.



Figure 3.3: V-notched Charpy Impact Test Specimen.

CHAPTER 4

RESULTS

4.1 Isothermal Transformation (TTT) Data

The calculated TTT diagrams for 51CrV4 and 60SiMn5 were generated by JmatPro® software and given in Figure 4.1 and Figure 4.2. Austenitization temperatures of 51CrV4 and 60SiMn5 were selected as 880°C and 950°C, respectively.



Figure 4.1: TTT diagram of 51CrV4 constructed by using JMatPro.



Figure 4.2: TTT diagram of 60SiMn5 constructed by using JMatPro

4.1.1 Calculated TTT Diagrams of 51CrV4 and 60SiMn5

<u>51CrV4</u>

The calculated TTT diagrams of 51CrV4 given in Figure 4.1. Martensite start (M_s) temperature is 300°C in the TTT diagram. Using the TTT diagram it is found that the bainitic transfomation starts at 50 second and 100% bainite is obtained in 30 minutes at around M_s temperature. Considering these, in this study; all samples of 51CrV4 were austenitised at 880°C for 30 minutes and M_s is taken as 300°C.

Table 4.1 shows phase transformation data of 51Crv4 steel. The schematic cooling paths of 51CrV4 are superimposed to the TTT diagram in Figure 4.3.

After austenitization at 880°C for 30 minutes, the samples were quenched in salt bath for isothermal holding process. For martensitic transformations, the samples were oil quenched and then tempered at different temperatures (Table.4.2).



Figure 4.3: Schematic cooling paths of 51CrV4

<u>60SiMn5</u>

The heat treatment paths of 60SiMn5 are superimposed on the calculated TTT diagram in Figure 4.4.

Austenitization temperature of 60SiMn5 steel is kept higher, i.e. at 950°C to minimize the amount of widmanstatten ferrite formation. The martensite start temperature is read as 250°C from the diagram. The bainitic transformation start and finish times at 250°C are interpolated as 6 minutes and 4 hours respectively. In the light of these calculations; in this study, all samples of 60SiMn5 were austenitised at 950°C for 30 minutes and M_s is taken as 250°C.

		Phase	Phase	Required Time	
Steel	Phase	Transformation	Transformation	for Phase	
		Status	Temperature (°C)	Transformation	
	Montoncito	M_{s}	300°C	-	
51C-V4	wartensite	M_{f}	180°C	-	
510174	Doinito	0% Bainite	300°C	50 sec	
	Dannie	100% Bainite	300°C	30 min	
	Montoncito	M_{s}	250°C	-	
60SiMp5	wartensite	M_{f}	130°C	-	
0051101115	Doinito	0% Bainite	250°C	6 min	
	Daimte	100% Bainite	250°C	4 hours	

Table 4.1: Phase Transformation data of 51CrV4 and 60SiMn5

The transformation data of 60SiMn5 is summarized in Table 4.4.



- - Bainitic transformation at 200°C

..... Bainitic transformation at 180°C

Figure 4.4: Schematic cooling paths of 60SiMn5.

Heat treatment summary of 51CrV4 and 60SiMn5 steels is given at the Table 4.2.

Steel	Austenitization	Heat	Sample	Temperature	Time	
Steel	Austennization	Treatment	Name	Temperature		
			300°C/5m	300°C	5 min	
			300°C/10m	300°C	10 min	
			300°C/15m	300°C	15 min	
		Lasthampally	300°C/30m	300°C	30 min	
	880°C	Transformed	300°C/1h	300°C	1 hour	
51CrV4	880°C	Transformed	300°C/5h	300°C	5 hours	
	for 50 mm		270°C/5h	270°C	5 hours	
			240°C/1h	240°C	1 hour	
			240°C/5h	240°C	5 hours	
		Tommonod	T220°C/2h	220°C	2 hours	
		Tempered	T220°C/4h	220°C	4 hours	
			180°C/4h	180°C	4 hours	
	050°C	To oth owned live	200°C/1h	200°C	1 hour	
60SiMn5	930°C	Transformed	200°C/4h	200°C	4 hours	
	for 30 min	Transformed	250°C/1h	250°C	1 hour	
			250°C/4h	250°C	4 hours	

Table 4.2: Summary of heat treatment processes of 51CrV4 and 60SiMn5

4.1.2 Microstructural Characterization of 51CrV4 and 60SiMn5

Microstructural Characterization of 51CrV4

It is known from the TTT diagram that bainite transformation completed in 30 minutes at 300°C. As a consequence, it was expected to see fully bainitic microstructure at isothermal holding times longer than 30 minutes at 300°C.

In order to confirm the transformation times, three samples of 51CrV4 were isothermally transformed at 300°C for 30 minutes, 1 hour and 5 hours respectively. After heat treatment process, they were air cooled and their microstructure were examined. All the isothermally transformed samples 300°C/30m, 300°C/1h and 300°C/5h have fully bainitic microstructure (Figure 4.5, 4.6 and 4.7). There is no considerable difference in the microstructures.



Figure 4.5: The SEM micrograph of isothermally transformed 51CrV4 specimen at 300°C for 30 minutes. Bainite sheaves.



Figure 4.6: The SEM micrograph of isothermally transformed 51CrV4 specimen at 300°C for 1 hours. Bainite sheaves.



Figure 4.7: The SEM micrograph of isothermally transformed 51CrV4 specimen at 300°C for 5 hours. Bainite sheaves.

As mentioned previously, when the transformation time is short the bainitic transformation cannot go into completion and the austenite remained in the microstructure transforms to martensite upon cooling to room temperature. As shown above, even after 30 minutes the microstructure was nearly 100% bainitic. For this reason, after austenitization at 880°C for 30 minutes, three samples were treated isothermally at 300°C for 5 minutes, 10 minutes and 15 minutes, respectively. As the isothermally holding time decreases, the amount of bainite decreases. However, since bainite transformation starts at 50 seconds at 300°C, there is always bainite seen in the microstructures. The optical and SEM images of the samples are given in Figure 4.8, 4.9, 4.10.

In optical images, the light - banded areas are martensite while dark areas are bainite (Figures 4.8, 4.9, 4.10). Since bainite is etched faster than martensite, it appears darker in the microstructure . However; in SEM images, the martensite phase seems as dark blocky islands(flat areas)(Figure 4.10b). As seen from the micrographs, with a decrease in transformation time, the fraction of light areas increases. (Figures 4.8, 4.9, 4.10)



Figure 4.8: a)Optical and b) SEM micrographs of isothermally transformed 51CrV4 specimen at 300°C for 5 minutes.



Figure 4.9: a)Optical and b) SEM micrographs of isothermally transformed 51CrV4 specimen at 300°C for 10 minutes.



Figure 4.10: a)Optical and b) SEM micrographs of isothermally transformed 51CrV4 specimen at 300°C for 15 minutes.

Under the M_s temperature, martensite transformation begins. If the specimen is quenched to a temperature M_q which is below M_s but above M_f , some amount of martensite will form instantly and its amount is related to the quenching temperature, M_q . Upon isothermal transformation at that temperature, the austenite which is not transformed to martensite will transform to bainite. Therefore, by careful selection of the temperature, the microstructure will consist of martensite-bainite phase mixture.

A microstructure obtained by quenching to M_q +isothermal treatment can have two important results: 1. The martensite obtained upon quenching to M_q will be tempered during isothermal treatment. 2. The bainite sheaves will be much finer with respect to that of transformed above M_s . Two samples of 51CrV4 were quenched to either 270°C or 240°C and isothermally transformed. (Figure 4.11)



Figure 4.11: The SEM micrograph of isothermally transformed 51CrV4 specimen at 270°C for 5 hours. Martensite (dark blocky islands) with lower bainite sheaves.



Figure 4.12: The SEM micrographs of isothermally transformed 51CrV4 specimen at 240°C for 1 hour. Martensite (dark blocky islands) with lower bainite sheaves.



Figure 4.13: The SEM micrograph of isothermally transformed 51CrV4 specimen at 240°C for 5 hours. Martensite (dark blocky islands) with lower bainite sheaves.



Figure 4.14: Area fractions of 240°C/1h, 240°C/5h and 270°C/5h specimens calculated by ImageJ. Dark area is bainite, white area is martensite.

In Figure 4.14, the ratio of area fraction of bainite and martensite for specimens 240°C/1h, 240°C/5h and 270°C/5h can be seen. Dark areas on the Figure 4.14 illustrates bainite while white areas are martensite. It can be seen that approximately 57% of austenite transforms to bainite at 240°C at 1 hour. Moreover, bainite area fraction increases as time and temperature increase, as expected.

To see the difference between martensite formed during isothermal holding and tempered martensite, three samples of 51CrV4 were austenitized at 880°C for 30 minutes and then directly quenched in oil. After quenching the specimens were tempered at 220°C for either 2 hours or 4 hours respectively. The microstructures of T220°C/2h and T220°C /4h was investigated under SEM which is given in Figure 4.15. Both samples have tempered martensitic microstructure.



Figure 4.15: SEM micrograph of 51CrV4, oil quenched tempered at 220°C for a)2 hours and b) 4 hours. Tempered martensite.

Microstructural Characterization of 60SiMn5

As mentioned in previous section, bainite transformation of 60SiMn5 should be completed in 4 hours at 250°C. According to this, it can be said that there can be expected fully bainitic microstructure at this temperature after 4 hours. A sample is isothermally transformed for 4 hours at 250°C and as expected, it has fully bainitic microstructure which can be seen in Figure 4.16 and 4.17



Figure 4.16: The optical micrograph of isothermally transformed 60SiMn5 specimen at 250°C for 4 hours. Lower bainite sheaves.



Figure 4.17: The SEM micrograph of isothermally transformed 60SiMn5 specimen at 250°C for 4 hours. Carbide-free lower bainite sheaves.

In order to obtain martensitic-bainitic microstructures different routes were applied. After the samples are austenitized at 950°C for 30 minutes, they were isothermally transformed for 1 hous at 200°C and for 4 hours either 200°C or 180°C, respectively. After heat treatment processes, all samples were air cooled and their microstructure were examined. It was expected 200°C/1h to get mainly martensitic microstructure with some bainite in. The SEM images of the samples were illustrated in Figure 4.18. On the other hand, the 200°C/4h revealed mostly bainitic microstructure (Figure 4.19,4.20). The martensite phase could not be resolved. However, in specimen 180°C/4h martensite islands can be seen easily (Figure 4.21).



Figure 4.18: SEM micrograph of isothermally transformed 60SiMn5 specimen at 200°C for 1 hour.



Figure 4.19: The SEM micrograph of isothermally transformed 60SiMn5 specimen at 200°C for 4 hours.



Figure 4.20: The optical micrograph of isothermally transformed 60SiMn5 specimen at 200°C for 4 hours. Bainite with austenite/martensite matrix.



Figure 4.21: The SEM micrograph isothermally transformed 60SiMn5 specimen at 180°C for 4 hours.

A short period of transformation just above M_s can also yield a microstructure consisting of bainite-martensite mixture. For this purpose, a sample of 60SiMn5 isothermally transformed for 1 hour at 250°C. Within 1 hour, the bainitic transformation cannot be completed and the microstructure expected as martensite/austenite. It can be seen from the Figure 4.22, microstructure has mainly bainite with some martensite/austenite island.



Figure 4.22: SEM micrograph of isothermally transformed 60SiMn5 specimen at 250°C for 1 hours. The flat regions are most probably martensite islands.

4.1.3 Mechanical Test Results of Heat Treated 51CrV4 and 60SiMn5

Overall results of mechanical tests are given in Table 4.3.

Stool	Heat	Sample	Dhaca	Hardness	CV	UTS	YS	%
Sleel	Treatment	Name	r nase	(HV30)	(J)	(MPa)	(MPa)	Elong.
		300°C/5m	B+M	532	-	-	-	-
	tion	300°C/10m	B+M	531	25	1810	1236	11.6
	rmat	300°C/15m	B+M	529	-	-	-	-
	nsfor	300°C/30m	100% B	527	-	-	-	-
_	Iraı	300°C/1h	100% B	530	19	1720	1396	13.6
CrV4	nal	300°C/5h	100% B	524	18	1695	1431	13.7
51C	herr	270°C/5h	B+M	559	14	1910	1553	12.4
	Isot	240°C/1h	B+M	584	-	-	-	-
		240°C/5h	B+M	574	12	2065	1575	11.4
	ing	T220°C/2h	ТМ	624	15	1570	-	1.0
	ıper	T220°C/4h	ТМ	615	-	-	-	-
	Tem	T450°C/2h	ТМ	-	23	1545	1405	13.9
		250°C/1h	B+M	664	10	2060	1556	10.9
NO	al tion	250°C/4h	100% B	618	8	1995	1740	9.8
Mn	erma	250°C/12h	100% B	-	6	2015	1756	10.4
60Si	othe	200°C/1h	B+M	683	-	-	-	-
	Is Trai	200°C/4h	B+M	637	10	2285	1358	7.15
		180°C/4h	B+M	720	9	2225	-	4.3

Table 4.3: Mechanical test results of 51CrV4 and 60SiMn5 samples.

4.1.3.1 Hardness Tests

Hardness of 51CrV4

Hardness test results of heat treated 51CrV4 are given in Table 4.4.

As seen, the hardness values of all bainitic samples are at around 530HV.

The specimens isothermally transformed at 300°C for 5 minutes and 5 hours yielded hardness values of 530HV and 524HV respectively. It seems the martensite present in the microstructure did not affect the hardness considerably in bainitic specimens. However, the samples isothermally transformed at either 270°C and 240°C yielded higher hardness values than that of 300°C.

		Isothermal Tra	Hardness	
Sample Name	Quenching	Temperature	Time	(HV30)
300°C/5m	Salt Bath	300°C	5 minutes	532 ± 7
300°C/10m	Salt Bath	300°C	10 minutes	531±6
300°C/15m	Salt Bath	300°C	15 minutes	529±7
300°C/30m	Salt Bath	300°C	30 minutes	527±7
300°C/1h	Salt Bath	300°C	1 hour	530±5
300°C/5h	Salt Bath	300°C	5 hours	524±3
270°C/5h	Salt Bath	270°C	5 hours	559±7
240°C/1h	Salt Bath	240°C	1 hour	584±14
240°C/5h	Salt Bath	240°C	5 hours	574±7
		Tempering		
		Temperature	Time	
OQ	Oil	-	-	690±3
T220°C/2h	Oil	220°C	2 hours	624±6
T220°C/4h	Oil	220°C	4 hours	615 ± 4

Table 4.4: Hardness values of heat treated 51CrV4
The hardness of isothermally transformed samples are plotted in Figure 4.23. It is seen all the hardnesses are in a 10HV band.

The specimens directly quenched to oil yields a hardness of 690HV. This hardness drops to 624HV and 615HV upon tempering at 220°C for 2 hours and 4 hours respectively.

Hardness of 60SiMn5

Hardness test results of heat treated 60SiMn5 are given in Table 4.5. Results show that hardness of the isothermally transformed 60SiMn5 are between 620 -720 HV. An isothermal holding temperature just above M_s will cause formation of bainite. The longer holding times will obviously increase the amount of bainite formed. The fully bainitic sample 250°C/4h has the lowest hardness. When isothermal holding times are compared, it can be clearly seen that increasing holding time decreases hardness. This can be explained by the amount of bainite, such that bainitic transformation cannot be completed at short time. The remaining austenite will transform to martensite upon cooling and probably will cause a hardness increase.

Another point is that when 200° C/4h and 180° C/4h samples are compared which both isothermally treated under the M_s temperature, the 180° C/4h sample has dramatically higher hardness value than 200° C/4h which indicates that the martensite content is considerably high.

Sample Name	Quenching	Isothermal Transformation		Hardness
		Temperature	Time	(HV30)
250°C/1h	Salt Bath	250°C	1 hours	664±4
250°C/4h	Salt Bath	250°C	4 hours	618±5
200°C/1h	Salt Bath	200°C	1 hour	683±7
200°C/4h	Salt Bath	200°C	4 hours	637±7
180°C/4h	Salt Bath	180°C	4 hours	720±15

Table 4.5: Hardness values of heat treated 60SiMn5



Figure 4.23: Hardness of 51CrV4 and 60SiMn5 samples.

Hardness results of both 51CrV4 and 60SiMn5 samples are illustrated in Figure 4.23.

4.1.3.2 Tensile Tests

Tensile Test Results of 51CrV4

Tensile test results of 51CrV4 heat treated samples are given in Figure 4.24.

Sample that tempered at 220°C for 2 hours does not yield and shows brittle fracture with lowest tensile strength, 1569 MPa. On the other hand 450°C/2h tempered sample yields at 1405 MPa and shows maximum elongation with a low ultimate tensile strength(UTS) of 1545 MPa. Fully bainitic samples 300°C/5h and 300°C/1h show similiar UTS and % elongation values as expected. 300°C/10m sample that have bainite and martensite in structure has higher UTS than fully bainitic samples. Moreover, their yield strengths are 1431 MPa and 1396 MPa, respectively.



Figure 4.24: Tensile test results of 51CrV4 samples.

270°C/5h sample that treated under M_s temperature and have 78% bainite - 22% martensite phase mixture, has higher UTS and yield strength than fully bainitic samples. Furthermore, 240°C/5h sample have 65% bainite - 35% martensite phase mixture shows best tensile properties.

Tensile Test Results of 60SiMn5

60SiMn5 samples have higher UTS than 51CrV4 samples. On the other hand, their elongations are lower than 51CrV4. Fully bainitic samples 250° C/4h and 250° C/12h shows similar % elongation and ultimate tensile strength. Bainitic - martensitic sample 250° C/1h that transformed at M_s temperature for 1 hour also have close results with fully bainitic ones. However, samples that transformed under M_s temperatures gives higher UTS with lower % elongation. 200° C/4h sample have the higher UTS with 2286 MPa with lowest yield strength. Moreover, 180°C/4h sample have 2226 MPa UTS with the lowest % elongation result which is 4.30 and show no yield.



Figure 4.25: Tensile test results of 60SiMn5 samples.

4.1.3.3 Charpy Impact Tests

Charpy Impact Test Results of 51CrV4

The charpy impact toughness results of 51CrV4 are given in Figure 4.26. 300°C/10min sample yields 25 J which is very close to tempered martensite sample T450°C/2h (23 J). Fully bainitic samples 300°C/5h and 300°C/1h show similiar toughness values as expected.

Samples that isothermally transformed below M_s temperature has the lowest impact energy. The reason of this is most probably the presence of martensitic microstructure besides bainite. Also, 220°C tempered sample have similar toughness properties with 270°C/5h and 240°C/5h.

As all the heat treated samples were compared with the untreated sample, it can be clearly seen that heat treatment increases the toughness of the material dramatically.



Figure 4.26: Charpy impact test results of 51CrV4 samples.

Fracture surfaces of tested samples were examined under SEM.



Figure 4.27: The SEM micrographs of 300°C/10min sample's fractured surfaces.



Figure 4.28: The SEM micrographs of T450°C/2h sample's fractured surfaces.

Fracture surface of sample 300°C/10min shows ductile fracture. When it is compared with T450°C/2h which have similar results, it can be seen that both have dimples and some cleavages. Also, the SEM images of 300°C/5h fracture surface have same characteristics in common with 300°C/10min and T450°C/2h. Since their absorbed energy values are close, it is a predictable result.



Figure 4.29: The SEM micrographs of 300°C/5h sample's fractured surfaces.



Figure 4.30: The SEM micrographs of 240°C/5h sample's fractured surfaces.

As fracture surface of 240°C/5h sample was analyzed, it can be seen that there is more clevage area than others. This can be the result of the mostly martesitic microstructure that gave brittleness to the sample.

Charpy Impact Test Results of 60SiMn5

Toughness of 60SiMn5 specimens were measured by V- notched charpy impact test. The results are given in Figure 4.31.



Figure 4.31: Charpy impact test results of 60SiMn5 samples.

All samples show similar toughness values. There is no dramatical differences between the fully bainitic samples (250°C/12h, 250°C/4h) and bainitic/martensitic samples (180°C/4h, 200°C/4h, 250°C/1h).

As all the heat treated samples were compared with the untreated sample, there is considerable difference observed according to their toughness.

Fracture surfaces of tested samples were examined under SEM.



Figure 4.32: The SEM micrographs of 200°C/4h sample's fractured surfaces.



Figure 4.33: The SEM micrographs of 250°C/1h sample's fractured surfaces.



Figure 4.34: The SEM micrographs of 250°C/12h sample's fractured surfaces.

All fracture surfaces shows brittle fracture. Since their absorbed energy values are low, the result is predictable.

CHAPTER 5

DISCUSSION

In this study, 51CrV4 and 60SiMn5 spring steels were used in order to obtain 100% bainitic and bainitic-martensitic mixture structures. The mechanical properties of these steel were tried to be improved by different heat treatment applications. The two main parameters during these processes are isothermal transformation time and temperature which have direct effect on microstructure, i.e mechanical properties.

5.1 Microstructural Characterization

It is known that bainitic transformation of 51CrV4 steel is completed in 30 minutes at 300°C. Fully bainitic microstructure is expected for the isothermal transformation times longer than 30 minutes at 300°C. As the micrographs of 300°C/30m, 300°C/1h and 300°C/5h are investigated, it can be said that there is no considerable difference in the microstructures and all of them have fully bainitic structure. These bainites are considered as lower bainite since the transformation is carried out at 300°C, just above the Ms temperature. Moreover, lower bainite is noted for having carbide precipitation not only at the sheave boundaries but also within the sheaves of bainitic ferrite. In Figure 4.7, carbide precipitates in bainitic ferrite can be seen. On the other hand, 60SiMn5 steel completes bainitic transformation at 250°C in 4 hours. 250°C/4h sample has fully bainitic microstructure as can be seen in Figure 4.17. The bainite formed at 250°C is expected to be lower bainite. However, there is no sign of carbides in SEM micrograph of sample as seen in Figure 4.17. This can be explained by the presence of silicon in the steel which suppresses the cementite precipitation in ferrite [24].

The transformation times shorter than 100% bainite formation i.e. 30mins) lead to formation of martensite and bainite. When three samples of 51CrV4 (300°C/5m, 300°C/10m and 300°C/15m) are examined, it is seen that all of them have bainite and martensite in their structure, simultaneously (Figure 4.8, 4.9,4.10). Bainite is seen as dark areas while martensite is light banded which happens due to the slower etching rate of martensite than bainite. According to Goulas et al. study of isothermal transformation of 51CrV4 at 300°C for 1 hour, the microstructure of steel has homogeneous, fully bainitic structure with carbide precipitation which is consistent with the results of this study [9]. On 60SiMn5 steel, 250°C/1h sample shows bainiticmartensitic microstructure since bainitic transformation cannot be completed in 1 hour. In Figure 4.22, bainitic sheaves can be seen clearly. The remaining microstructure expected as martensite/austenite (M/A) islands. M/A is the phase where austenite retains at room temperature due to carbon enrichment. This retained austenite partially turns into martensite creating M/A. As it is possible that the retained austenite can transform to martensite, these islands are named as M/A islands (Martensite/Austenite) [42].

It is a known fact that martensite transformation begins at M_s temperature. The martensite amount depends on the quenched temperature which has to be below M_s . The remaining austenite that cannot be transformed into martensite alters into bainite by isothermal transformation. This phenomenon is seen in 270°C/5h, 240°C/1h and 240°C/5h samples of 51CrV4 steels. Figures 4.11, 4.13 and 4.12 that show the microstructure of the samples, indicate that bainitic structure is observed with blocky martensite. The amount of martensite increases as noticed from the micrographs. Also, area fraction analyses of the 270°C/5h, 240°C/5h and 240°C/1h prove that amount of martensite increases as transformation temperature and time decreases (4.14). Moreover, it is clearly seen that 270°C/5h, 240°C/5h and 240°C/1h structures become finer than 300°C samples. It is more probably due to the decrement in mobility of growth mechanism.

In a similar way, 200°C/4h, 200°C/1h and 180°C/4h samples of 60SiMn5 is expected to have bainitic-martensitic microstructure. SEM micrographs of 200°C/1h and 180°C/4h support this claim (Figure 4.21, 4.18). 180°C/4h which is isothermally transformed much under the M_s , indicated that martensite island formation with some

lower bainite surrounding. Likewise, main structure of 200°C/1h structure is identified as martensite with some bainite formation in it. However, when micrograph of 200°C/4h is examined, it is observed that sample has mostly bainitic structure (Figure 4.19). The martensite phase, which should be seen together with bainite, cannot be revealed. However, when the optical micrograph of 200°C/4h (Figure ??) is investigated, bainite sheaves with a shiny white background is seen which is revealed as the austenitic/martensitic matrix.

Heat treatment process is applied to the two samples of 51CrV4 to get tempered martensite. As gathered from literature, tempering process relieves the internal stress of martensite and toughness increase. This is complimented by very fine carbide precipitation within martensite. The SEM micrographs of T220°C/2h and T220°C/4h samples proove that both samples have tempered martensitic microstructure. (Figure 4.15).

5.2 Mechanical Properties

Hardness

As mentioned previous, fully bainitic structure is formed after isothermally treated 30 minutes at 300°C for 51CrV4. According to the results that are shown in Table 4.3 it is not a surprise that fully bainitic structured samples 300°C/30m, 300°C/1h and 300°C/5h have same results which is 530 HV. On the other hand, reducing the isothermal transformation time is the reason of the uncompleted bainitic transformation. The untransformed austenite turns into martensite during cooling. It is a common fact that martensite increases hardness of steel due to its fine microstructure and internal stress. So, it is expected that 300°C/5m, 300°C/10m and 300°C/15m samples which have martensite in them, have higher hardness values. However, there is no considerable difference between the hardnesses of the samples which can be an indication that either there is no martensite or the amount of martensite is very low.

In a similar way, decreasing the isothermal transformation temperature yield a result of hardness increment. At low temperatures, thickness of bainite reduces as mentioned previously. Fine grained steels have higher amount of grain boundaries than coarser ones which prevents dislocation to move easily and so increases strength. Regarding this information, 270°C/5h, 240°C/1h and 240°C/5h samples that isothermally transformed under M_s have higher hardness level than samples that treated 300°C as seen in Table 4.4. All samples have fine bainite with martensite in microstructure. 270°C/5h has a hardness of 559HV while 240°C/5h has 574HV. This increment can explained by the finer microstructure of 240°C/5h since it transforms at lower temperatures. When 240°C/1h and 240°C/5h samples are compared, it is noted that 240°C/1h has 584HV which is probably due to higher amount of martensite amount in microstructure. According to area fraction analysis, 240°C/1h has 42.9% martensite whereas 240°C/5h has 34.8% and support this claim.Besides isothermal transformed samples, the sample directly quenched to oil yields a hardness of 690HV. This hardness drops to 624HV and 615HV upon tempering at 220°C for 2 hours and 4 hours respectively. Reduction in hardness is expected due to the longer tempering times.

Similar results are observed in samples of 60SiMn5 steel. 100% bainitic sample, 250° C/4h has the lowest hardness value of 618HV. As the temperature difference between the M_s and bainitic tranfomation temperature increase the hardness of specimens increase: The 200°C/4h specimen has a hardness value of 637 HV, while 180°C/4h has dramatically higher hardness value, i.e. 720HV. This results indicates that 180°C/4h has high martensite content. As 250°C/1h and 250°C/4h is compared, 250°C/1h has higher value due to the more martensitic content. Same results are valid for 200°C/1h and 200°C/4h samples which have hardness of 683HV and 637HV respectively.

Tensile Strength

When the tensile properties of 51CrV4 samples are compared, it is seen that 300°C/1h and 300°C/5h samples have similar results as expected. Since both of the sample have fully bainitic microstructure, their %elongation is highest among other samples and yield around 13,0%. Moreover, they have UTS values in the range 1800 MPa - 1700 MPa. As the transformation time decreases, 100% bainitic structure could not be be obtained. The 300°C/10m sample has 1809 MPa UTS which is higher than fully bainitic samples (from the samples transformed for 15min, 30min, 1hr and 5hrs).

On the other hand, it has % elongation of 11.64% which is lower than fully bainitic samples most probably due to the presence of martensite. According to the results of Lambers et al., 100% bainitic samples has highest % elongation since elongation is dependent on the microstructure and shows increament with the increase of bainitic fraction [37].

Samples that transformed under M_s temperature has higher UTS results as the amount of martensite increases. 270°C/5h sample an UTS of 1912 MPa with a %elongation of 12.38%. Besides, 240°C/5h sample has an UTS of 2065 MPa which is the highest UTS value among 51CrV4 samples. It also has an %elongation of 11.37% which is barely lower than fully bainitic ones. So, it can be said the a mixture of martensite and fine bainitic structures improve the tensile properties. The stress on the martensite that formed during quenching releases during isothermal holding. This cause decrement in brittelness of the samples and improves ductility. Moreover, the martensite and fine bainite in structure act as obstacles to the dislocation motion since the amount of grain boundary increases with decreasing grain size. On the other side, quenched and tempered samples shows the lowest UTS values. Sample that tempered at 220°C for 2 hours has 1569 MPa UTS while 450°C tempered for 2 hours sample has 1545 MPa which is the lowest tensile strength among samples. Furthermore, T220°C/2h sample is very brittle and shows no yield while T450°C/2h has an elongation of 13.87%. High carbon in 51CrV4 cause the increament of the amount of the embedded carbon in martensite which increases the distortion in the matrix. This distortion prevents the movement of dislocations and improves strength. However, the more the dissolved carbon in martensite, the more the notch - sensitive the steel become. After a critical point, notch sensitivity gets dominant over strength mechanism and consequence as failure without yielding [37].

Alp and Wazzan studied the tensile properties of as rechieved, dual phase (ferriticmartensitic) and tempered martensitic SAE 6150 steel which is another grade of 51CrV4. The samples show UTS of 758 MPa, 590 MPa and 819 MPa respectively. When it is compared with the UTS values of Alp and Wazzan study, it can be said that UTS results of isothermally heat treated 51CrV4 samples has quite better. It is obvious that bainitic - martensitic structure improves UTS of steel dramatically. Since 60SiMn5 has 1.2% Si in it, which suppresses cementite and cause rejection of carbon from bainitic ferrite into the untransformed austenite and saturates the austenite with respect to carbon. This austenite retains between the bainite sheaves as retained austenite at room temperature. This is probably why 60SiMn5 steel has higher UTS values than 51CrV4. However, it shows worse plasticity than 51CrV4 samples. Fully bainitic samples of 60SiMn5, 250°C/4h and 250°C/12h, have an UTS values of 1995 MPa and 2015 MPa with respectively. As the transformation time and temperature decreases, bainitic transform cannot be completed and martensite occurs in structure which explains the increase in strength and reduction in plasticity. However, 250°C/1h sample which has bainitic - martensitic structure, has higher UTS and %elongation than fully bainitic samples with a value of 2059 MPa and 10.93% respectively. As the amount of martensite increases, the strength of samples increase. 200°C/4h sample has 2286 MPa UTS value while 180°C/4h has 2226 MPa. On the other hand, they have %elongation of 7.15% and 4.3% respectively.

Charpy Impact Toughness

Similar to hardness, impact toughness results of 100% bainitic 51CrV4 samples are close to each other. (Figure 4.26) The toughness of 250°C/5h is 19 J while 250°C/1h is 18 J. Both results shows an increment when it is compared with a base steel of 51CrV4 with a toughness of 8 J. It can be said that higher amount of bainite in the microstructure increases toughness. On the other side, one of the higher toughness is observed at 300°C/10m sample with 25 J. The reason for this can be explained by the high amount of bainite and autotempered martensite. The toughness of 300°C/10m is very close to toughness of tempered martensite sample which is T450°C/2h with 23 J. Higher toughness is expected to observed since tempering is relaxed the sample's stress which increases the absorbed energy. Besides, T220°C/2h has lower values than 450°C tempered sample which is probably due to higher tempering temperature. What is interesting that, samples transformed under M_s has the lowest toughness values. 240°C/5h and 270°C/5h samples have a result of 12 J and 14 J respectively. Even though these results are higher than base metal, still it is the lowest among the heat treated samples. The explanation of this result can be built on the higher amount of martensite. Although the martensite is expected to autotempered during 5 hour treatment, it is obvious that the temperature is not enough for improving toughness.

Moreover, as the fracture surfaces are investigated, dimples are seen in Figures 4.27, 4.28 and 4.29 which are indicated the ductile fracture. For 240°C/5h, there is more cleavage areas than others which fits with the lower toughness (Figure 4.30).

As it comes to 60SiMn5 samples, there is no noticeable difference between the charpy impact test results. The results are approximately 9 J seen in Figure 4.31. It is clarified by the reaction of retained austenite under stress. As it gathered from literature, silicon suppresses cementite formation which causes carbon enrichment in austenite and lower the M_s . As a consequence, unstable austenite can be retained at lower temperatures. Under a mechanical stress, it transforms into brittle martensite phase which lowers the toughness of the steel [22,23]. On the other hand, the presence of austenite should improve the toughness values as in the case of TRIP steels [43]. However, this was not the case for the 60SiMn5 steel. This can be due to the high carbon content of the 60SiMn5 steel (Figure 4.32,4.33 and 4.34).

CHAPTER 6

CONCLUSION

In this study, 51CrV4 steel and 60SiMn5 steel were used in order to obtain bainitic and bainitic-martensitic structure. Heat treatment process were done by altering times and temperatures. Quenched and tempered samples was done for comparison of mechanical properties. Microstructural and mechanical properties were investigated. The following conclusions are drawn from this study:

- 1. The average hardness of 100% bainitic structure of 51CrV4 is 527HV. As the amount of the martensite in the structure increases in the bainitic martensitic structure, the hardness values increases to a range 530HV-584HV. An intermediate quenching to a temperature between M_s and M_f is found to be more efficient than short isothermal transformation times in controlling the ratio of bainite/martensite.
- 2. In 60SiMn5 steel, 100% bainitic sample yields a hardness of 618HV. By intermediate quenching or by applying short transformation times the hardness of bainitic-martensitic structures varies in the range 637HV-720HV, which is higher than that of 51CrV4.
- 3. Both in 51CrV4 and 60SiMn5 steels, the samples with bainite-martensite mixed structures show higher UTS values than 100% bainitic ones. The reason of this is the presence of hard martensite phase in the structure.
- 4. In 51CrV4 samples, the ductility (%elongation) of fully bainitic samples are comparable to martensite-bainite mixed structures. In the case of 60SiMn5 samples, the ductility of both 100% bainitic and bainite-martensite mixed structures are at around 10% which is somewhat lower than that of 51CrV4 samples.

- 5. The bainitic-martensitic 51CrV4 samples yield around 25 J and have the same toughness value with that of martensite tempered at 450°C. 100% fully bainitic sample exhibit a toughness of approximately 19 J. The samples that transformed under M_s have lowest impact toughness values compared to other heat treated samples due to the presence of higher amount martensite which increases the notch-sensitivity of the samples.
- 6. Whole 60SiMn5 samples yield low toughness values in the range 6 J-10 J. The samples do not reflect the effect of heat treatment on toughness.

REFERENCES

- H. K. D. H. Bhadeshia and J. Christian, "Bainite in steels," *Metallurgical trans*actions A, vol. 21, no. 3, pp. 767–797, 1990.
- [2] H. Beladi, I. B. Timokhina, P. D. Hodgson, and Y. Adachi, "Characterization of nano-structured bainitic steel," vol. 5, pp. 1–8, 2012.
- [3] I. Timokhina, H. Beladi, X.-Y. Xiong, Y. Adachi, and P. D. Hodgson, "Nanoscale microstructural characterization of a nanobainitic steel," *Acta Materialia*, vol. 59, no. 14, pp. 5511–5522, 2011.
- [4] F. Caballero and H. Bhadeshia, "Very strong bainite," *Current Opinion in Solid State and Materials Science*, vol. 8, no. 3-4, pp. 251–257, 2004.
- [5] M. Soliman and H. Palkowski, "Development of the low temperature bainite," *Archives of Civil and Mechanical Engineering*, vol. 16, no. 3, pp. 403–412, 2016.
- [6] W. D. Callister, D. G. Rethwisch, et al., Materials science and engineering: an introduction, vol. 7. John wiley & sons New York, 2007.
- [7] X. Long, J. Kang, B. Lv, and F. Zhang, "Carbide-free bainite in medium carbon steel," *Materials & Design*, vol. 64, pp. 237–245, 2014.
- [8] C. Garcia-Mateo, G. Paul, M. Somani, D. Porter, L. Bracke, A. Latz, C. Garcia De Andres, and F. Caballero, "Transferring nanoscale bainite concept to lower c contents: A perspective," *Metals*, vol. 7, no. 5, p. 159, 2017.
- [9] C. Goulas, A. Kumar, M.-G. Mecozzi, F. M. Castro-Cerda, M. Herbig, R. H. Petrov, and J. Sietsma, "Atomic-scale investigations of isothermally formed bainite microstructures in 51crv4 spring steel," *Materials Characterization*, vol. 152, pp. 67–75, 2019.
- [10] K. Abbaszadeh, H. Saghafian, and S. Kheirandish, "Effect of bainite morphology on mechanical properties of the mixed bainite-martensite microstructure in

d6ac steel," *Journal of Materials Science & Technology*, vol. 28, no. 4, pp. 336–342, 2012.

- [11] H. Bhadeshia, "Case study: design of bainitic steels," 2000.
- [12] T. Furuhara, H. Kawata, S. Morito, and T. Maki, "Crystallography of upper bainite in fe–ni–c alloys," *Materials Science and Engineering: A*, vol. 431, no. 1-2, pp. 228–236, 2006.
- [13] L. Leach, *Modeling Bainite Formation in Steels*. PhD thesis, KTH Royal Institute of Technology, 2018.
- [14] S. Samanta, P. Biswas, S. Giri, S. B. Singh, and S. Kundu, "Formation of bainite below the ms temperature: Kinetics and crystallography," *Acta Materialia*, vol. 105, pp. 390–403, 2016.
- [15] M. J. Peet, Transformation and tempering of low-temperature bainite. PhD thesis, University of Cambridge Cambridge, UK, 2010.
- [16] L. Fielding, "The bainite controversy," *Materials Science and Technology*, vol. 29, no. 4, pp. 383–399, 2013.
- [17] H. Bhadeshia, "Nanostructured bainite," *Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences*, vol. 466, no. 2113, pp. 3– 18, 2009.
- [18] Z. Öztürk, An Investigation of Bainitic Transformation in Low Carbon Alloy and High Carbon Railway Steels. PhD thesis, Middle East Technical University, 2018.
- [19] J. L. Dossett and H. E. Boyer, *Practical heat treating*. ASM International, 2006.
- [20] A. Handbook, "Heat treating, vol. 4," ASM International, Materials Park, OH, vol. 860, 1991.
- [21] A. Navarro-López, J. Hidalgo, J. Sietsma, and M. Santofimia, "Characterization of bainitic/martensitic structures formed in isothermal treatments below the ms temperature," *Materials Characterization*, vol. 128, pp. 248–256, 2017.

- [22] Y. Tsai, H. Chang, B. Huang, C. Huang, and J. Yang, "Microstructural characterization of charpy-impact-tested nanostructured bainite," *Materials Characterization*, vol. 107, pp. 63–69, 2015.
- [23] X. Long, F. Zhang, J. Kang, B. Lv, and X. Shi, "Low-temperature bainite in low-carbon steel," *Materials Science and Engineering: A*, vol. 594, pp. 344– 351, 2014.
- [24] L. Qian, Q. Zhou, F. Zhang, J. Meng, M. Zhang, and Y. Tian, "Microstructure and mechanical properties of a low carbon carbide-free bainitic steel co-alloyed with al and si," *Materials & Design*, vol. 39, pp. 264–268, 2012.
- [25] F. C. Campbell, *Elements of metallurgy and engineering alloys*. ASM International, 2008.
- [26] J. Guimaraes and P. Rios, "Unified description of martensite microstructure and kinetics," *Journal of materials science*, vol. 44, no. 4, pp. 998–1005, 2009.
- [27] R. Grange, C. Hribal, and L. Porter, "Hardness of tempered martensite in carbon and low-alloy steels," *Metallurgical Transactions A*, vol. 8, no. 11, pp. 1775– 1785, 1977.
- [28] Z.-j. Luo, J.-c. Shen, H. Su, Y.-h. Ding, C.-f. Yang, and X. Zhu, "Effect of substructure on toughness of lath martensite/bainite mixed structure in low-carbon steels," *Journal of Iron and Steel Research International*, vol. 17, no. 11, pp. 40– 48, 2010.
- [29] Y. Nie, W. Fu, W. Hui, H. Dong, and Y. Weng, "Very high cycle fatigue behaviour of 2000-mpa ultra-high-strength spring steel with bainite-martensite duplex microstructure," *Fatigue & Fracture of Engineering Materials & Structures*, vol. 32, no. 3, pp. 189–196, 2009.
- [30] G. F. Vander Voort, Atlas of time-temperature diagrams for irons and steels. ASM international, 1991.
- [31] W. You, W.-h. Xu, Y.-x. Liu, B.-z. Bai, and H.-s. Fang, "Effect of chromium on cct diagrams of novel air-cooled bainite steels analyzed by neural network," *Journal of Iron and Steel Research International*, vol. 14, no. 4, pp. 39–42, 2007.

- [32] M. Zhou, G. Xu, J. Tian, H. Hu, and Q. Yuan, "Bainitic transformation and properties of low carbon carbide-free bainitic steels with cr addition," *Metals*, vol. 7, no. 7, p. 263, 2017.
- [33] J. Tian, G. Xu, M. Zhou, H. Hu, and X. Wan, "The effects of cr and al addition on transformation and properties in low-carbon bainitic steels," *Metals*, vol. 7, no. 2, p. 40, 2017.
- [34] L. Bracke and W. Xu, "Effect of the cr content and coiling temperature on the properties of hot rolled high strength lower bainitic steel," *ISIJ International*, vol. 55, no. 10, pp. 2206–2211, 2015.
- [35] C. Garcia-Mateo, L. Morales-Rivas, F. Caballero, D. Milbourn, and T. Sourmail, "Vanadium effect on a medium carbon forging steel," *Metals*, vol. 6, no. 6, p. 130, 2016.
- [36] T. Sourmail, C. Garcia-Mateo, F. Caballero, S. Cazottes, T. Epicier, F. Danoix, and D. Milbourn, "The influence of vanadium on ferrite and bainite formation in a medium carbon steel," *Metallurgical and Materials Transactions A*, vol. 48, no. 9, pp. 3985–3996, 2017.
- [37] H.-G. Lambers, S. Tschumak, H. Maier, and D. Canadinc, "On the bainitic and martensitic phase transformation behavior and the mechanical properties of low alloy 51crv4 steel," *The International Journal of Structural Changes in Solids*, vol. 3, no. 1, pp. 15–27, 2011.
- [38] T. Alp and A. Wazzan, "The influence of microstructure on the tensile and fatigue behavior of sae 6150 steel," *Journal of materials engineering and performance*, vol. 11, no. 4, pp. 351–359, 2002.
- [39] T. Sourmail and V. Smanio, "Low temperature kinetics of bainite formation in high carbon steels," *Acta Materialia*, vol. 61, no. 7, pp. 2639–2648, 2013.
- [40] D. B. Santos, R. Barbosa, P. P. de Oliveira, and E. V. Pereloma, "Mechanical behavior and microstructure of high carbon si-mn-cr steel with trip effect," *ISIJ international*, vol. 49, no. 10, pp. 1592–1600, 2009.

- [41] G. Gao, H. Zhang, X. Gui, P. Luo, Z. Tan, and B. Bai, "Enhanced ductility and toughness in an ultrahigh-strength mn–si–cr–c steel: the great potential of ultrafine filmy retained austenite," *Acta Materialia*, vol. 76, pp. 425–433, 2014.
- [42] J. M. Reichert, T. Garcin, M. Militzer, and W. J. Poole, "Formation of martensite/austenite (m/a) in x80 linepipe steel," in 2012 9th International Pipeline Conference, pp. 483–489, American Society of Mechanical Engineers Digital Collection, 2013.
- [43] K.-i. Sugimoto, M. Tsunezawa, T. Hojo, and S. Ikeda, "Ductility of 0.1-0.6 c-1.5 si-1.5 mn ultra high-strength trip-aided sheet steels with bainitic ferrite matrix," *ISIJ international*, vol. 44, no. 9, pp. 1608–1614, 2004.