# THE EFFECT OF RETAINED AUSTENITE AND CARBIDE DISTRIBUTION ON THE WEAR RESISTANCE OF BEARING STEEL

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

BY

ZEREN ÖZGENECİ

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

FEBRUARY 2017

Approval of the thesis:

## THE EFFECT OF RETAINED AUSTENITE AND CARBIDE DISTRIBUTION ON THE WEAR RESISTANCE OF BEARING STEEL

submitted by ZEREN ÖZGENECİ 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. Gülbin DURAL ÜNVER Dean, Graduate School of **Natural and Applied Sciences** 

Prof. Dr. C. Hakan GÜR Head of Department, **Metallurgical and Materials Engineering, METU** 

Prof. Dr. Bilgehan ÖGEL Supervisor, Metallurgical and Materials Engineering Dept., METU

### **Examining Committee Members:**

Prof. Dr. Rıza GÜRBÜZ Metallurgical and Materials Engineering Dept., METU	
Prof. Dr. Bilgehan ÖGEL	
Metallurgical and Materials Engineering Dept., METU	
Prof. Dr. C. Hakan GÜR	
Metallurgical and Materials Engineering Dept., METU	
Prof. Dr. A. Tamer ÖZDEMİR	
Metallurgical and Materials Engineering Dept., Gazi University	

Asst. Prof. Dr. Mert EFE Metallurgical and Materials Engineering Dept., METU

**Date:** 22.02.2017

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, Last name: Zeren ÖZGENECİ Signature:

#### ABSTRACT

# THE EFFECT OF RETAINED AUSTENITE AND CARBIDE DISTRIBUTION ON THE WEAR RESISTANCE OF BEARING STEEL

Özgeneci, Zeren

M.S., Department of Metallurgical and Materials Engineering Supervisor: Prof. Dr. Bilgehan Ögel

February 2017, 83 Pages

This study aims to investigate the effect of the amount of retained austenite and undissolved carbides on the wear resistance of the heat treated bearing steels. As inner and outer ring material 100Cr6 steel grade was used. The specimens were austenitized in the range 800°C to 1000°C, and then, quenched to room temperature in oil to produce a hard martensitic phase. A composite microstructure of undissolved carbides, martensite and retained austenite were observed in hardened steel specimens. It was seen that the amount of undissolved carbides decreases but the amount of retained austenite increases with an increase in austenitization temperature. The amount of retained austenite in all the samples were in the range of 10% to 58% in as-quenched condition. Four different tempering stages were applied to as-quenched samples: The first group and second group specimens were tempered at 180°C and at 235°C, respectively. The third group was double tempered at 190°C, and then, at 170°C. In the fourth group, a subzero treatment at -120°C was applied to the as-quenched specimen before tempering at 170°C. In all tempering operations, the tempering time was 90 minutes. The selection of different combinations of tempering yielded specimens having a very large range of retained austenite content (0%-26%) and hardness values (58 HRC-67 HRC). The highest amount of retained austenite was obtained in the samples tempered at 180°C. On the other hand the lowest amount of retained austenite was obtained in the samples sub-zero treated (-120°C) and tempered at 170°C. For this reason, the wear tests of bearings under contaminated lubrication was prepared from these steel components. It was found that maximum bearing service life time under contaminated lubrication was obtained after an austenitization treatment at 900°C and tempering at 180°C having a hardness of 63 HRC and a retained austenite content of 12%.

Keywords: retained austenite; 100Cr6; volume fraction of carbides; wear resistance; bearing ring; heat treatment.

# KALINTI ÖSTENİT VE KARBÜR DAĞILIMININ RULMAN ÇELİĞİ AŞINMA DAYANIMINA ETKİSİ

Özgeneci, Zeren

Yüksek Lisans, Metalurji ve Malzeme Mühendisliği Bölümü Tez Yöneticisi: Prof. Dr. Bilgehan Ögel

Şubat 2017, 83 Sayfa

Bu çalışma, mikroyapıdaki kalıntı östenit ve çözünmemiş karbür miktarlarının ısıl işlem sonrası rulman çeliğinin aşınma dayanımına etkisinin incelenmesini amaçlamaktadır. Rulman iç ve dış bileziğinde malzeme olarak 100Cr6 çelik sınıfı kullanılmıştır. Numuneler 800°C-1000°C aralığında ki sıcaklıklarda östenitlendikten sonra sert martensit fazı eldesi için oda sıcaklığına yağda su verilmiştir. Sertleştirilmiş çelik numunelerde, çözünmemiş karbürler, martenzit ve kalıntı östenitten oluşan kompozit bir mikroyapı gözlemlenmiştir. Östenitleme sıcaklığındaki artış ile yapıda çözünmeden kalan karbürlerin miktarının azalırken, kalıntı östenit miktarının arttığı görülmüştür. Su verme sonrası bütün numunelerin kalıntı östenit miktarları değerlendirildiğinde, %10-%58 aralığında değerler elde edilmiştir. Su verilmiş numunelere 4 farklı temper basamağı uygulanmıştır: Birinci grup numuneler 180°C'de, ikinci grup numuneler 235°C'de temperlenmiştir. Üçüncü grup numunelere, ilki 190°C'de ikincisi ise 170°C'de olmak üzere çift temperleme uygulanmıştır. Dördüncü grup su verilmiş numunelere ise 170°C'de temperleme prosesinden önce -120°C'de sıfır altı ısıl işlemi uygulanmıştır. Bütün temperleme proseslerinde, temper süresi sabit 90 dakikadır. Farklı temperleme kombinasyonlarının seçimi, numunelerin çok geniş aralıkta kalıntı östenit miktarına (%0-26%) ve sertlik değerlerine (58 HRC-67 HRC) sahip olmasına sebep olmuştur. Numunelerde en yüksek kalıntı östenit miktarı, 180°C'de temperlenen numunelerde elde edilmiştir.

Diğer yandan, en düşük kalıntı östenit miktarları ise sıfır altı ısıl işlemi (-120°C) sonrasında 170°C'de temperlenen numunelerde elde edilmiştir. Bu sebeple, rulmanların kontaminasyonlu yağlama ortamında yapılan aşınma testlerine bu gruplardan numune hazırlanmıştır. Kontaminasyonlu yağlama ortamında çalışan rulmanlarda maksimum ömür, 900°C'de östenitlendikten sonra 180°C'de temperlenerek, 63 HRC sertlik ve %12 kalıntı östenit miktarına sahip olan çelik mikroyapısında elde edilmiştir.

Anahtar Kelimeler: kalıntı östenit; 100Cr6; karbürlerin hacimsel oranı, aşınma dayanımı; rulman bileziği; ısıl işlem

To my loving husband...

#### ACKNOWLEDGEMENTS

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, support, understanding and patience. One simply could not wish for a better or friendlier supervisor.

I would also like to express my greatest appreciation to Ozan MÜŞTAK for his enthusiasm, help, support and inspiring advices, which guided me at the most critical parts of the study.

I extend my deepest thanks to my managers, Dr.-Ing Feridun ÖZHAN, Dr.-Ing Hamdullah MERDANE at ORS Bearings Inc. for their support and encouragement. I would also like to express my gratitude to my colleagues, Nazmi SAYDEMİR, Kamil KARAŞ, Mustafa HORTAÇ, Nedim Özgür ENGİN, Mustafa SARAY, Ersen ÖZÇUBUKÇU and Alperen KILIÇARSLAN on behalf of all ORS Bearings Inc. members, for their interest, help and technical assistance.

I am indebted to my parents, Uğur Cumhur TAŞKAYA, Nurten TAŞKAYA and my sister and brother Zeynep ALAY, Özcan ALAY for enlightening me with their caring, inspiration, understanding, love, guidance and wisdom through my entire life.

I also owes my deepest gratitude to my love, Mehmet Erçin ÖZGENECİ for his great love, encouragement and help. Not only my thesis but also my life would not be possible without your endless support and patience. I really appreciate his existence.

Special thanks to my friends, Burcu COŞKUN, Doğukan ŞENGÜL, Melis ÇELİKKOPARAN, Hande ADIGÜZEL and Uğur GEZER for their endless support and encourage during this research. I have been blessed with their cheerful friendship and love throughout my life.

Finally, I would like to acknowledge ORS Bearings Inc. for their financial support. Without their support, I could not have been successfully conducted all my experiments.

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## LIST OF ABBREVIATIONS

## **XRD :** X-Ray Diffractometer

**SEM :** Scanning Electron Microscope

M<sub>f</sub>: Martensite Finish Temperature

M<sub>s</sub> : Martensite Start Temperature

**RA** : Retained Austenite

**QMA :** Quantitative Metallographic Analyses

**PSD :** Position Sensitive Detector

**VFC :** Volume Fraction of Carbides

**ASTM :** American Society for Testing and Materials

**ID**: Identification

N/A : Not Applicable

#### **CHAPTER 1**

### **INTRODUCTION**

In the production of bearings, the used material is the most crucial element, which directly affects the performance and the service life. Because of that, the raw material goes through different kind of processes in order to have better material features. So hardening of steel is one of main processes, which should be conducted in rigorous way.

Hardening of steels requires heating to an austenitic phase and quenching to room temperature to produce hard martensitic phase. Due to incomplete transformation, some austenite retain at room temperature. Therefore, hardened steels actually have composite microstructures of tempered martensite and retained austenite. Owing to the instability of retained austenite, it may transform to martensite in service because of thermal cycles, plastic deformation or shock. Thus the phase transformation from austenite to martensite is connected with a volume expansion of about 5% [1].

At the end of the through hardening heat treatment, both the amount of retained austenite that remains in the microstructure and carbide distribution that depends on the hardened microstructure can affect the mechanical properties of steel such as wear resistance, fatigue strength, toughness, hardness, yield strength and also influences the dimensional stabilization of materials. Among these, wear resistance is one of the most critical property for industrial applications in various types of machine components, especially for bearings. In other words, failures originating from the wear damages are the most critical one for the bearing applications. Therefore, the presence work is aimed to study the effects of retained austenite, its dependent carbide distribution and structure on the wear resistance of the heat-treated ball bearing steels.

Mostly 100Cr6 steel grade (SAE 52100) is used in bearing applications. Due to its quite well material properties such as high hardenability, high machinability, it is

known as bearing steel. The chemical composition of 100Cr6 can be examined as it is given in Table 1.

DIN	Analyse-Composition Wt%								
Symbol	С%	Si%	Mn%	P≤%	S≤%	Cr%	Mo%	Ni%	Others%
100Cr6	0.93- 1.05	0.15- 0.35	0.25- 0.45	0.025	0.015	1.35- 1.6	≤0.10	-	Al ≤0.050; Cu≤0.30; O ≤0.0015

Table 1 Standard Chemical Composition of 100Cr6 [2]

In general, bearing steel is supplied in the form of bars or tubes as a raw material. In the case of as-spheroidized seamless steel tubes usage, the first production step is turning operation. On the other hand, steel bars are used in two different ways. In the first method of steel bar usage, the drilling operation is performed to steel bars in the first stage; then turning operation is followed in order to give the rough form of bearing rings. In the second and most conventional method of steel bar usage, the form of inner and outer bearing rings is firstly given by hot forging operation. Thereafter, general process flow for bearing production is followed by spheroidization heat treatment process. Then, the rough shape of bearing rings is given by machining processes such as cold ring rolling and turning. Another important step is the through hardening heat treatment process where the hardness and final microstructure are determined in a precise way. Subsequent to the heat treatment process. Finally, ground rings are mounted together with the addition of auxiliary bearing elements such as balls, cages and cover.

In this study, 100Cr6 steel grade is used in the form of bearing ring. Since the wear mechanism characterization of 100Cr6 grade steels is directly correlated with the heat-treated microstructure, the amount of retained austenite and undissolved carbide in the bearing rings are measured in an accurate way. Therefore, all measurements of microstructure are completed by using micro and macro analysis. Microstructure characterization is handled with various surface and volume techniques such as image analyzing and processing technique, Scanning Electron Microscopy (SEM), X-Ray

Diffractometer (XRD) and Optical Microscopy. Beside these, wear tests are performed with the life test rig. Based on the results of the wear tests, the wear resistance is associated to microstructure. So, this thesis study evaluates the wear resistance of bearing rings in a comprehensive perspective.

# **CHAPTER 2**

### THEORY AND LITERATURE SURVEY

#### 2.1 Through Hardening Process

Through hardening plays a crucial role in the manufacturing of bearing rings since the hardness and final microstructure of bearing rings are determined in this process. Due to fact that the most of the important mechanical properties such as wear resistance, fatigue strength, hardenability, toughness, yield strength and dimensional stability of materials are directly associated with the phases that are distributed in the heat treated microstructure, the determination of the optimum through hardening process parameters is the milestone for bearing production.

Hardening of bearing steels requires heating to an austenitic phase and quenching to room temperature to produce a hard martensitic phase. After quenching process, due to incomplete transformation, some austenite may retain at room temperature. Therefore, composite microstructures of undissolved carbides, martensite and retained austenite can be observed in hardened steel.

Carbides give good wear resistance [3] and up to 3-4% volume percent excess carbides raise the wear performance [4]. Also, Yajima reported that carbides which are failed to dissolve during quenching affect the mechanical properties of bearing steel such as fatigue life [5].

On the other side, retained austenite is primarily martensitic microstructure based phase in bearing steel and it is essential for the service life of steel components. However, optimum characteristics of retained austenite such as volume fraction, distribution and composition have to be achieved during heat treatment process. While certain amount of retained austenite can enhance ductility and toughness, excess amount of retained austenite phase causes reduction in strength of steels [6]. In addition to these effects, the dimensional stability of bearing steel is also directly related with retained austenite. The decomposition of retained austenite into martensite that is influenced from stress or cooling below ambient temperature, induces expansion in the order of 4  $\mu$ m per 100 mm during service [7]. So, the volume expansion of approximately 4%, which is a consequence of the phase transformation from retained austenite to martensite, leads to distortion based failures [8].

Since the amount of each phase varies with changing through hardening process parameters, appropriate selection of temperature and time is significant for heat-treating procedure. The austenitizing temperature, tempering temperature, tempering time and cyrogenic/cold treatment are the main heat treatment parameters that have the most influence on the retained austenite and carbide level in the heat treatment of bearing steel [8].

#### 2.1.1 Austenitizing and Quenching

The first step of hardening of bearing steel is austenitizing process, which yields transformation from the body-centered cubic ferrite to face-centered cubic austenite, and hence the structure has homogeneous single phase with increased carbon stability. Especially the austenitizing temperature is crucial parameter for hardness, the amount of retained austenite and the volume fraction of carbides. The typical austenitizing temperature is indicated as 840°C in literature. Austenitization in excess of 1000°C results in completely dissolving the cementite in bearing steel. In particular, the calculated equilibrium compositions of austenite and cementite at 840°C are presented in Table 2.

Table 2 The calculated equilibrium compositions of austenite and cementite at 840°C[7]

	С	Si	Mn	Ni	Mo	Cr
Mean Composition (wt%)	1.04	0.25	0.35	0.125	0.05	1.45
Austenite	0.86	0.26	0.34	0.13	0.05	1.15
Cementite	6.74	-	0.66	0.02	0.17	11.00

It is generally assumed that the equilibrium point for austenitization process is achieved within 20 minutes for 52100 type steel. The measured kinetics of dissolution are given in Figure 1 [9].



Figure 1 The kinetics of cementite dissolution in 52100 type steel with respect to austenitisation temperature [9]

Quenching is a rapid cooling, which is particularly applied to the metallic alloys like steel. Cooling from austenitization temperature leads to a microstructure which is a mixture of untempered martensite, some amount of retained austenite and undissolved carbides which are failed during austenitizing [8]. In previously conducted experiment given in literature, about 6% volume of retained austenite and 3-4% of cementite particles are observed after quenching from the austenitizing process of 52100 steel [10, 11]. Austenite to martensite transformation starts at a definite temperature, known as M<sub>s</sub>, continues for a temperature range, and then ends at a definite temperature called M<sub>f</sub>. The amount of both martensite and retained austenite during cooling is depicted in Figure 2. As it is clearly seen in the Figure 2, more austenite has been transformed into martensite as the transformation temperature decreases.



Figure 2 The amount of martensite vs transformation temperature [12]

Martensite transformation, which is diffusionless/displacive transformation due to high cooling rate, leads to high hardness through material with uniform distribution [13]. An example of hardness, volume percent of retained austenite and undissolved carbides as a function of austenitizing temperature for 52100 specimens is illustrate in Figure 3. Figure 3 clearly showed that while hardness starts to reduce after austenitizing approximately at 900°C, the percentage of retained austenite increases linearly from 850°C.



Figure 3 Hardness, retained austenite and undissolved carbide as a function of austenitizing temperature for 52100 steel (Austenitizing time is 30 minutes and tempering one hour at 175°C) [4]

### 2.1.2 Tempering Process

Besides the martensite, which is available in the as-quenched state, is very hard, it is also so brittle for using most applications. In addition to that, internal stresses due to quenching have a weakening effect. Because of these, tempering process has a vital importance for the 52100 steel applications.

Tempering of steel is a heat treatment process which causes reduction in both the hardness and the amount of retained austenite while increase in ductility and toughness. The tempering process that is applied to quenched steel is performed by selecting its temperature below the lower critical temperature. It is worth emphasizing that temperature is much more effective than time in tempering process.

The transformation of martensite to tempered martensite is a diffusional process given in the below reaction (2.1) which is based on the iron-iron carbide phase diagram:

Martensite(BCT, single phase)  $\rightarrow$  tempered martensite ( $\alpha$ +Fe<sub>3</sub>C phases) (2.1)

The microstructure of tempered martensite comprises extremely small cementite particles which are uniformly distributed in continuous ferrite matrix [14].

The tempering temperature has a great impact on the amount of retained austenite as it is investigated in various studies [7, 8, 15, 4]. It has been stated that the retained austenite amount in 52100 steel does not change with tempering temperatures up to 175°C while it reduces continuously between 200 °C and 230 °C. Tempering at 260 °C and higher temperatures cause great deal of reduction in the amount of retained austenite that approaches approximately to zero [4]. Thus, the amount of retained austenite phase can be decreased or eliminated completely by selecting proper process parameters of tempering without compromising on hardness value [7]. Another study to point up the effect of tempering temperature to hardness and retained austenite was completed by Huh et.al. Figure 4 indicates that while the fraction of retained austenite goes to 0 dramatically at tempering temperature 250°C , hardness changes up to 5-6 Rockwell C scale between 150-250°C tempering temperature ranges [16].



Figure 4 Hardness and retained austenite data for 52100 steel tempered for 1 hour [16]

#### 2.2 Sub-Zero Heat Treatment

Sub-zero cooling process, namely deep cryogenic treatment, completes the transformation from retained austenite to martensite owing to fact that retained austenite is more unstable and tends to transform at low temperatures [17]. Because of austenite to martensite transformation that is achieved by sub-zero cooling process, both the hardness and the wear resistance of bearing steel increases with an increase percentage of martensite in microstructure. Resulting wear resistance difference between cryogenic treatment and conventional heat treatment is illustrated in Figure 5.



Figure 5 Wear rate of 100Cr6 bearing steel at 5 Hz frequency for the two different treatments (DCT:Deep Cryogenic Treatment, CHT: Conventional Heat Treatment) [17]

In addition to advance in the wear resistance, Collins et.al proved that cryogenic treatment process makes contribution to improving toughness by enabling finer distribution of carbides [18, 19].

This cold-treatment process is an additional step for the hardening heat treatment. As it is shown in Figure 2, it is almost impossible to have completely martensitic structure by through hardening process. In other words, some amount of retained austenite is always present in the hardened microstructure. Then, specific amount of retained austenite can be transformed into martensite by cooling the bearing steel below the martensite finish temperature  $(M_f)$ .

Figure 6 shows that  $M_f$  and  $M_s$  temperatures decreases with increased carbon content of steel. Thus, the amount of retained austenite increases together with the carbon content. So that subzero heat treatment process is one of the main solution for eliminating or reducing this amount of retained austenite [12].



Figure 6 Carbon content vs M<sub>s</sub> and M<sub>f</sub> temperatures of steel [12]

According to thesis study of Mustak, O., the martensite finish temperature is believed to be less than -90°C for bearing steel and the related experimental result was plotted in Figure 7 [13].



Figure 7 Martensite phase transformation (%) vs temperature plot of sub-zero test and kinetic model fittings [13]

Due to the retained austenite stabilization caused by tempering process, its transformation to martensite seems to be very difficult. So, sub-zero cooling seems to be not very effective on decreasing the amount of retained austenite in quenched and tempered steel [7]. In that case, sub-zero cooling process should be applied in the temperature range of -70°C to -120°C for 1 hour per 25mm of cross section of material after the quenching process [20]. Therefore, it can be stated that the last step of heat treatment must be tempering process to prevent the newly formed untempered martensite (fresh martensite) remaining in heat-treated structure.

#### 2.3 Carbide Staining Process

As it is previously stated, the heat-treated microstructure directly affects the wear resistance of bearing steel. Besides the unknown phases in the heat treated microstructure, the amount of carbide dissolution in the microstructure should also be taken in consideration for the accurate measurement of retained austenite levels, especially with XRD. The optimum process parameters should be determined based on retained austenite and the amount of carbide dissolution in heat-treated structures to obtain better wear resistance.

There are various methods for the retained austenite measurements based on metallographic, magnetic and X-Ray Diffraction techniques. The accuracy of quantitative metallographic analysis strongly depends on the form and the amount of retained austenite in the heat-treated structures. On the other hand, the accuracy of magnetic method is directly related with the presence of nonmagnetic carbides [6]. Owing to its less than 1% uncertainty, X-Ray Diffraction technique gives more accurate and reliable results compared with other techniques [21].

In calculation of the amount of retained austenite by X-ray diffraction technique, the measurement of the volume fraction of carbides accurately is of prime importance for adequate peak evaluation. In the case of high carbon contents and low-alloy chromium steels like 100Cr6, carbides precipitate in the form of ((Fe,Cr)<sub>3</sub>C) and ((Cr, Fe)<sub>7</sub>C<sub>3</sub>) [3].

The volume fraction of carbides in the heat-treated structures is determined by quantitative metallography. After etching the heat-treated structures with selective etchant, different colors make us to distinguish the phases with the aid of optical contrast. The quantitative measurements can be carried out by using optical microscope and the image analysis software. Therefore, the amount of retained austenite calculation by deducting the carbide percentage at first gives more accurate results.

Given these points, the volume fraction of carbides is calculated with quantitative metallography technique, whereas the amount of retained austenite is determined by X-Ray techniques in this thesis project.

#### 2.4 Volume Fraction of Carbide Calculation

Manual stereological methods and conventional image analyzer are both tough and tedious processes, especially for fine scale or complicated microstructures in metals. In that respect, quantitative metallography has a key position in material science and engineering. In order to overcome these complicated tasks, computer assisted quantitative analysis has been developed at the beginning of 2000's and late 1990's.

Although the usage of computer-assisted quantitative analysis provides great convenience, the microstructure should be etched properly to obtain high contrast level. For example outlined, colored or brightened structures are appropriate for computer-assisted quantitative analysis due to their high contrast.

G. F. Vander Voort darkened the matrix of specimens by using certain selective etchants for the staining process of specific carbides. Then they assessed the volume fraction, number per unit area and size of the carbides with the help of image analysis program [22].

Chaix and his colleagues performed a study [23], which investigated austenite and carbides coarsening in a high speed steel. Digital images, which were taken by Jeol JSM 6400 microscope, were evaluated on the image analysis system.

Nowadays, there are widely used softwares both for image improvement and for quantitative analysis. So that volume fraction of phases, recrystallization dynamics and grain growth in steel microstructures can be examined by these image analysis software (Image Tool Software) in an accurate way.

#### 2.5 Retained Austenite Measurement by XRD

Although, common techniques such as quantitative metallographic analysis (QMA), magnetic methods, and dilatometer techniques have been utilized in years, they have important handicaps [6]. Jacques and his colleagues reported the results of blind round

robin test which includes various techniques such as light microscopy, X-ray diffraction, electron backscattered diffraction, magnetic saturation, thermal diffusivity and laser ultrasonic for the detection of the retained austenite content in steels. The results show that, while the slopes of mentioned techniques are differ from each other, there is a strong correlation between magnetic saturation and XRD [24]. So the limitations related to mentioned techniques leave XRD as the most confidential method for the retained austenite measurement [25].

As Mason and Prevey [8] argue that quantitative microscopic examination can be used for the detection of retained austenite amount when its amount is as high as 15% or above. However, the related austenite content could be very low in bearing steel. In this circumstance, X-ray diffraction (XRD) is again more reliable technique for RA analysis.

In the study of Magner et al [26], early (1930-1950) examinations, later (1950-1970) studies and finally recent (1970-2000) investigations with regard to X-ray diffraction methods were explained. Briefly, X-ray diffraction methods have been utilized for the determination of retained austenite amount in steel more than 50 years [27, 28, 29]. Besides, it is also highlighted that the RA content below about 0.5% can be detected by automated procedure of XRD in these days.

According to ASTM standard (E975) [30], although cobalt, copper or molybdenum Xradiations can be utilized for ferrous materials, chromium radiation is generally preferred to gain the best resolution of X-ray diffraction peaks in steel. In steel specimens, X-rays can penetrate to a maximum depth of approximately 10 micron below the surface. Therefore, any local abrasion has a great effect on the resolution of diffracted beam, and hence the amount of retained austenite. So that, it is worth to emphasize that sample preparation for the X-ray diffractometer has vital importance. Since heat effects from cutting process or deformation from grinding process may cause the transformation of RA, attentive sample preparation such as electropolishing or chemically polishing are prerequisite for accurate retained austenite measurement [31].
# 2.6 The Effects of Retained Austenite on Mechanical Properties of Bearing Steel

The amount and stability of retained austenite is important as it affects the rolling friction, contact plastic deformation and elastic limit for bearing steel. At the beginning of 60's, it has been examined that the structure having zero retained austenite content has the least rolling friction [32].

As early as 1970, Yajime et al have studied the effects of retained austenite on the rolling fatigue life of bearing steel. They highlighted that the retained austenite makes a noticeable but auxiliary addition to the fatigue life of bearings by decreasing the irregularity of the fatigue life [5]. Subsequently, Bogachev has examined the amount and the stability of retained austenite on the wear resistance of 9Kh18 steel under dry sliding friction. In his study, it was stated that raising the quenching temperature yields as an increase in the amount and stability of retained austenite. Also the wear resistance under the thermal wear condition increases relatively [33].

During 80's and 90's, fatigue behavior of the bearing steel SAE 52100 has been examined in more detail. One of the main output of these studies has been accomplished by C.A. Stickels. According to his research, increasing the austenitization temperature from 843°C to 900°C, raises the retained austenite content and improves fatigue life [34].

At the end of 90's, You Wang emphasized the influence of austenitizing temperature and the microstructure on wear behavior of 52100 steel. He pointed out that raising the austenitizing temperature to 1050°C yields as dissolution of carbides. So that wear resistance of the materials may increase due to the increased carbon content and hardness of matrix. He also proved that the wear resistance of various heat treated microstructures increases in the following order: martensite + carbide + RA, spheroidized structure, martensite, bainite, lamellar pearlite [35]. Moreover, Furumura et al. studied the relationship between the amount of retained austenite, hardness and minimum expected life ( $L_{10}$ ) for bearings. They concluded that increment in neither retained austenite nor hardness was effective on fatigue life lonely. These properties should be taken into consideration together [36]. The relationship between mentioned properties and the minimum expected life is given in the Figure 8 and Figure 9.



Figure 8 Relationship between fatigue life and retained austenite [36]



Figure 9 Relationship between fatigue life and hardness (RA: 27-32%) [36]

Retained austenite may prevent crack extension in the specimen because of its high toughness. In that sense, Dong et al. studied the strengthening effects of retained austenite together with residual stress alteration and the processes that cause crack in the specimen. They stated that the contact fatigue resistance increases with the increasing amount of retained austenite in the microstructure. This result can be beneficial to improve the performance of loaded friction pairs such as gears [37]. In another work on gears, Fernandes and McDuling showed that the amount of retained austenite between 10-20% helps to restrain contact fatigue by plastic deformation of retained austenite in the microstructure to form larger contact areas [38].

#### 2.6.1 The Wear Behavior of 52100 Steel

The wear mechanism in bearings is generally fatigue occurring in the layers of bearing steel surface and near surface. While two metal surfaces are sliding each other as in bearings, frictional heating can change both mechanical and metallurgical properties of sliding surfaces.

Though they have important consequences, the effects of microstructure, hardness, austenitizing temperature, isothermal treatment and thermal conductivity on the wear behavior of steels are least understood.

Wang et al have studied the wear resistance of different microstructures such as martensite + carbide + retained austenite, spheroidized structure, martensite, bainite, lamellar pearlite. They found that martensite+carbide+retained austenite microstructure has the least wear resistance while lamellar pearlite microstructure has the best performance in pin-on-ring testing at room temperature without lubrication [35].

It is important to emphasize that quenching from the austenitization temperature leads to a microstructure containing martensite, retained austenite and undissolved spheroidized carbides. In order to answer the question of "How much retained austenite is sufficient for good wear resistance?" an optimum balance should be provided between tempered martensite, retained austenite and residual carbide in the heat-treated microstructure. A better approach to make this optimization requires an optimized heat treatment process that will improve the wear resistance of bearing steel [39].

#### 2.6.1.1 Wear Fatigue Tests under Contaminated Lubrication

Bearing producers have been focused on many researches about their products in order to make them work in tough conditions such as contaminated lubrication, high loading and temperature [40]. Contaminated lubrication leads significant failures that reduce the operating time of bearings and these type of failures has a substantial part in the list of bearing failures as it is illustrated in Figure 10. The reason of this decrease in the fatigue life of bearings under contaminated lubrication is the contact stress concentration on the edges of the indentation. Solid particles which are called debris mostly cause the contamination of the lubricant and this leads to fatigue cracks generation in a short time interval [41]. In order to cope with debris denting, one of the main solution is using through hardened steel with a high level of stable retained austenite [42, 43].



Figure 10 The percentage share of bearing failure causes [44] 20

By decreasing both the amount and the size of inclusions of lubricant, the quality of bearing steel has been enhanced significantly in recent years. However, it is still not possible to arrange a completely clean lubrication system for bearing operations. Since failures such as surface damage generated by debris are getting more prominent for the evaluation of wear test results of bearings, there is a need for contamination-tolerant bearings.

Due to the low yield stress of retained austenite, plastic deformation starts under the high stress concentration. Based on this fact, Murakami and Furumura et al suggest that, there is a strong interaction between the retained austenite contents and the life under contaminated lubrication [41, 45]. According to their study, the extended fatigue life can be achieved with an increase in both the amount of retained austenite and hardness under circulated and contaminated oil with debris size between 74-147  $\mu$ m [41].

Böhmer and Eberhard claims that overrolling and plastic deformation of raised edges around the indentations of debris particles are the main factor of having a failure because of the debris contaminated lubrication. Furthermore, they found that the composition of the heat-treated material microstructure, such as the amount and stability of the retained austenite, has a great impact on the deformation behavior of bearings under contaminated lubrication. In their study, they also emphasized that it cannot be achieved all benefits when the retained austenite amount is below 5% for bearings made from 100CrMn6. However, an exact value for the amount of retained austenite could not be given for standard bearing steel SAE 52100 due to that retained austenite is highly unstable in this composition [44].

There should be a balance between some metallurgical properties for bearings to make them operate in a specific harsh environment. To obtain an optimized balance that maximizes the service life of bearings, heat treatment process has to be optimized. Moreover, when the debris particle is pushed between rolling elements, plastic deformation occurs into the raceway surface. To analyze the relation of these two issues, Carlson et al made indentations into sample surfaces, which are heat treated in different ways and changes in the dent morphologies with retained austenite are further discussed in their study. So their motivation was to increase the retained austenite content by making no concessions to the surface hardness, wear resistance and near surface residual stress for maximizing the damage tolerance of bearings. In their study, VimCru20 powdered metal particles in the size range of 90-105  $\mu$ m were added in a filtered grease to obtain contaminated lubrication for wear tests. At the end of these tests, higher dent lip height was observed in hardened and tempered bearing steel which has low retained austenite amount, while lower dent height was seen in the same steel with high amount of retained austenite [39].

The use of solid particles, debris, is highly recommended for endurance tests under contaminated lubrication. In order to generate an artificial contaminated condition for the assessment of rolling contact behavior of raceways through a quick and inexpensive means, various debris types are used. Generally, gas-atomized high-speed steel powders are selected for the fatigue life tests that are conducted under solid contaminated lubrication conditions. Ooki and his colleagues and also Ohki et al selected these gas atomized particles which have the grain size from 100  $\mu$ m to 180  $\mu$ m and HV800 hardness values. On the other hand, Carlson et al used VimCru20 particles to obtain contaminated lubrication condition for the life test of bearings [46, 47, 39]. For mixing these steel particles, previously conducted experiments in the literature recommend the usage of turbine oil as a lubricant [47, 46].

#### 2.6.1.2 Worn Surface Analysis

While two metal surfaces slide against each other, both the mechanical and the metallurgical features can alter the wear behavior. The reason of these alterations is the increase in temperature, which is converted through friction. As indicated by You Wang et al [35] that the microstructures that have higher stability cause higher wear resistances in dry sliding conditions in unlubricated tests. They studied the wear resistance of different microstructures and showed that both the types and distributions of carbides in the microstructure have great importance.

Bohmer and Eberhard [44] showed that the amount and the stability of retained austenite affect the related plastic deformation behavior. Consequently, the focus of the authors is the overrolling of raised edges around the indentations, the re-flattening and the material response to those two issues. They also proved that when the retained austenite amount is too low, the lifetime ( $L_{10}$ ) reduces and its scatter increases. Moreover, they argue that [44] the present carbide network in microstructure does not have an effect on the crack progression and pitting origination according to their SEM investigations.

Tanaka and Tsushima [48] conducted endurance tests under contaminated lubrication conditions on the type 6206 which is conventionally heat treated SUJ2 steel. Upon completion of the tests, many dents, which are formed due to flaking, were detected on the bearing raceways. They also claimed that the smaller the bearing load, the larger the reduction from the basic rating bearing life under contaminated lubrication.

Carlson et al [39] compared the differences between a controlled indenting methodology and a debris denting methodology by testing under the same speed and load. According to their expectations, failure mechanism should be same and start from the trailing edge of a raceway dent. They measured the actual dent lip height by using light interferometry, which is a non-contact surface measuring equipment. In the end, they concluded that the dent lip heights of hardened and tempered 52100 bearing steel with low retained austenite are higher than the dent lip heights of the same steel with high retained austenite.

### **CHAPTER 3**

### **EXPERIMENTAL SETUP AND PROCEDURE**

Experimental part of this study consist of heat treatment processes, microstructural analysis by optical microscope and SEM, image analyzing and processing techniques by ImageJ program, retained austenite measurement by XRD, hardness measurements, wear test applications of bearing under contaminated lubrication.

### 3.1 Material

In this thesis study, 100Cr6 (SAE 52100) steel grade was used. Table 3 shows the chemical composition of the used 100Cr6 raw material.

	C	Cr	Si	Mn	Al	Cu	Mo	Ni	0	Р
Wt (%)	0.99	1.41	0.26	0.37	0.016	0.13	0.03	0.1	4x10 <sup>-6</sup>	0.02

Table 3 Chemical composition of used 100Cr6 raw material

#### 3.1.1 Sample Preparation

Preliminary heat treatment and retained austenite (RA) measurement tests were done on flat specimens. After completing the parametric trend analysis, actual heattreatment trials were conducted on the bearing ring samples.

#### 3.1.1.1 Flat Samples

In order to determine the RA content by X-Ray analysis, parametric trend analysis was completed by using flat samples. Flat samples were cut from pieces that are forged and then spheroidized in an atmosphere controlled furnace at 790°C for 16 hours. As seen in Figure 11, the flat samples are cut in the direction of rolling from disc shaped parts having dimension of 30mm x 18 mm x 7 mm (LxWxT).



Figure 11 Flat sample preparations from forged and spheroidized discs (A-the form of forged and spheroidized piece B-cutting direction C-sectioned face of the flat piece)

### **3.1.1.2 Bearing Ring Samples**

After the inner and outer rings were obtained by hot forging operation of 100Cr6 raw steel bar, spheroidization heat treatment process was applied. Then the bearing steel with type number 632605 was machined to produce 500 pieces of inner and outer rings. These rings are heat treated with the same parameters of flat specimens. The inner and outer ring are illustrated in Figure 12.



Figure 12 The illustration of inner and outer ring 26

#### 3.2 Heat Treatment Processes

### **3.2.1 Heat Treatment Parameters**

Austenitizing and tempering temperatures have the most influence on the retained austenite and hardness of the final product. For this reason, flat specimens are used for different combinations of austenitization and tempering treatments. The samples were divided into five different groups and each group was austenitized for 35 minutes at 800°C, 850°C, 900°C, 950°C and 1000°C. All of the groups were quenched in the oil bath of vacuum furnace at 50°C. To inspect the effect of tempering process on the amount of retained austenite, these five group specimens were tempered at either 180°C or 235°C for 90 minutes. In addition to these tempering processes, some of the samples, which were austenitized at 850°C for 35 minutes and oil quenched at 50°C, were divided into two groups. First group was sub-zero treated at -120°C for 120 minutes and then tempered at 170°C for 90 minutes. No sub-zero treatment was applied to the second group.

By going through all results that were collected up to this point, the new experiment procedure was decided in order to find out a relation between the microstructure and the wear behavior. This new experiment procedure proposed to have 20 different bearing ring groups by performing different heat treatment operations. So, identifications and applied heat treatment operations of 20 groups are shown in Table 4. Each group consists of 20 inner and 20 outer rings which yielded 20 bearing after assembly.

After measuring amount of retained austenite of each group, grinding and assembling operations were followed to make bearings ready for the wear tests under contaminated lubrication.

Austenitizing Temperature (°C)	Process Flow of Heat Treatment	Sample ID
	Temper at 180°C for 90 minutes	800A-180T
	Temper at 235°C for 90 minutes	800A-235T
800	Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes	800A-120SZ-170T
	Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes	800A-190T-170T
	Temper at 180°C for 90 minutes	850A-180T
	Temper at 235°C for 90 minutes	850A-235T
850	Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes	850A-SZ120-170T
	Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes	850A-190T-170T
	Temper at 180°C for 90 minutes	900A-180T
	Temper at 235°C for 90 minutes	900A-235T
900	Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes	900A-SZ120-170T
	Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes	900A-190T-170T
	Temper at 180°C for 90 minutes	950A-180T
	Temper at 235°C for 90 minutes	950A-235T
950	Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes	950A-SZ120-170T
	Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes	950A-190T-170T
	Temper at 180°C for 90 minutes	1000A-180T
	Temper at 235°C for 90 minutes	1000A-235T
1000	Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes	1000A-SZ120-170T
	Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes	1000A-190T-170T

# Table 4 Process flows and sample identifications of ring samples

### **3.2.2** Heat Treatment Equipment

To perform these processes, a vacuum furnace, a chamber type tempering furnace and a sub-zero furnace, which are all located in ORS, were used respectively.

The through hardening heat treatment process were conducted in BBC Vacuum Furnace (Figure 13). Some of the properties of the used vacuum furnaces are summarized in Table 5.



Figure 13 Vacuum Furnace for through hardening process

	150 kg at 1250°C	
Maximum charge weight	250 kg at 1100°C	
Charge size	450 mm x 350 mm x 600 mm	
Charge size	(width x height x length)	
Maximum furnace temperature	1300°C	
Maximum heating temperature	1250°C	
Maximum hardening gas	900 mB	
pressure		
Quench medium	Oil	
Oil bath volume	$\sim 2 \text{ m}^3$	
Oil batch temperature	50 °C	
Oil cooling rate	100 °C/s	

# Table 5 Properties of vacuum furnace

Tempering process was completed by using AICHELIN chamber type tempering furnace. The photo of the furnace and its properties are given in Figure 14 and Table 6 respectively.



Figure 14 Chamber type tempering furnace

Furnace dimension	2430 mm x 2705 mm x 3563 mm
Charge size	1100 mm x 600 mm x 600 mm
Maximum furnace temperature	750°C
Maximum operating temperature	300-700°C
Maximum charge capacity	650 kg
Protective gas	Nitrogen (N <sub>2</sub> )

Table 6 Properties of tempering furnace

Sub-zero heat treatment process was completed by using AICHELIN SLX-680 Liquid  $N_2$  furnace. This furnace is shown in Figure 15 and its properties are listed in Table 7.



Figure 15 Sub-zero heat treatment furnace

Furnace dimension	2000 mm x 3630 mm x 1900 mm
Charge size	850 mm x 850 mm x 700 mm
Minimum furnace temperature	-180°C
Operating temperature	-120°C
Maximum charge capacity	1000 kg
Cooling system	Liquid Nitrogen (N <sub>2</sub> )

Table 7 Properties of sub-zero heat treatment furnace

Bearing ring and flat samples with different heat treatment parameters were prepared by using these furnaces. Quenching from 840°C and then tempering at a lower temperature 160-180°C is a common heat treatment technique in bearing industry. In order to understand the influence of microstructure on wear behavior of 100Cr6 better, austenitizing temperatures of this thesis study were selected in the range 800°C -1000°C. The heat treatment processes for flat and bearing ring samples are explained in the following subsections.

#### 3.3 Microstructural Examination

Microstructural analysis of the samples was performed using optical microscopy and SEM in this thesis work. Both optical microscopy and SEM studies were carried out on as polished and on etched surfaces.

After the heat treatment, the samples were ground, polished and edged using the standard metallographic procedures [49]. For general microstructural observation, 5%Nital is used.

In order to find the volume fraction of carbides, the stained specimens were examined under optical microscope. OLYMPUS BX51M microscope equipped with DP25 camera, which is located in ORS laboratories was used for studying the heat-treated microstructures.

The JEOL JSM6400 at acceleration voltage of 20kV, was used in SEM studies. The heat-treated flat samples were examined with SEM before and after the tempering

processes. Before these analyses, all microstructures were etched by using Nital etchant.

### **3.4 Hardness Measurement**

Hardness measurements were completed for 20 different heat-treated bearing rings samples by using INDENTEC type 4151BK Rockwell hardness testing machine (Figure 16).



Figure 16 Rockwell hardness testing machine

### 3.5 Image Processing and Analyzing

Accurate determination of chromium carbide percentages is very important in RA measurements. The amount of undissolved alloy carbides decrease with an increase in austenitization temperature. For this reason, the percentage of carbides must be measured independently for each specimen and included to the RA measurements. The etchants in Table 8 was used for carbide staining. Various types of etchant were used such as such as Alkaline Sodium Picrate, Klemm's Tint Etching and Nital (5%) in this thesis study. The amount of undissolved carbide values that were calculated after etching with nital (%5) were more comparable to the ones given in the literature, so it was decided to use nital (%5) for microstructural observations.

Table 8 Etchant used in this study

Alkaline Sodium Picrate	100 ml water,2 g picric acid, 25 g NaOH; use 90-100°C
Klemm's Tint Etching	50 ml of saturated aqueous sodium thiosulfate solution and 1 g of sodium disulfide
Nital (5%)	100 ml ethanol, 5 ml nitric acid

After the carbides are stained, the volume percentages of carbides are obtained with the assistance of freeware image processing program, ImageJ.

In order to calculate the volume fraction of carbides accurately, a minimum three images were taken randomly from each specimens by using optical microscope. Then 500x500 pixels (71x71  $\mu$ m) were cut from the each well-contrasted image at a magnification of 100x and this process was repeated for the randomly taken images of each specimen. Finally, the volume fraction of chromium carbides was computed by taking the average of three calculated values for each different heat-treated structure.

### 3.6 XRD Analysis

The XRD system, which is used in this thesis study, located in the R&D Department of ORS Bearings Inc. After calculating the percentages of undissolved carbides by ImageJ analysis, the RA content of heat treated samples were analyzed by Seifert XRD 3003 PTS.

The related XRD system is shown in Figure 17. As it is indicated in Figure 17, X-Ray generator tube, X-Ray detector and goniometer are the three main parts of this system. X-Rays are produced by X-Ray generator tube (Cr) and they are canalized to the measurement point on specimens through a collimator. This XRD system has 7-axis goniometer. Since 2 $\Theta$  angle should be preserved constantly between detector axis and X-Ray tube axis at the point of measurement, the specimen acts in compliance with detector by the aid of goniometer. In addition to these, Position Sensitive Detector was used in XRD system and diffracted X-Rays are collected from the point of measurement. All measurement parameters for XRD analysis are listed in Table 9.



Figure 17 X-Ray Diffractometer (XRD), Seifert 3003 PTS. (1) Goniometer, (2) X-Ray Generator Tube and (3) Position Sensitive Detector 35

XRD Measurement Parameters			
Collimator	3 mm		
Number of Scans:	4		
Scan Ranges:	102.00-168.00		
Step Width:	0.02		
Time:	300s		
Scan Axis:	2:1 Absolute		
Scan Mode:	Stepscan		

Table 9 XRD analysis parameters

# 3.7 Wear Test Under Contaminated Lubrication Conditions

An experiment is designed to find a relation between the amount of retained austenite and undissolved carbides on wear properties of the bearing steels. As shown in Figure 18, the wear test rig that was used in this thesis study applies the radial load evenly to two test bearings.



Figure 18 ORS wear test rig for ball bearing

The wear test was performed by applying a predetermined radial load on the test bearings, which are lubricated with the help of oil bath system. Test conditions are given in Table 10.

Radial load Fr (kN)	4.41 (450 kg)
Radial internal space (MM)	19
Maximum contact pressure (GPa)	2.5
Rotation speed (min-1)	2000 rpm (inner ring rotation)
Lubrication oil	Turbine oil (VG56), oil bath, approx.30 ml
Amount of contamination	1.0 g/L
	Gas-atomised particles of high speed tool
Type of contamination	steel Grain size:108 to 180 µm
	Hardness approx. HV800

Table 10 Test conditions of wear testing under contaminated lubrication

As it is seen in Table 10, the inner ring of the bearings rotate at 2000 rpm by refilling 30 ml of contaminated oil before each wear test application. In this thesis study, always the same test rig was used for the wear tests of all specimen groups in order to avoid the possible variation of test parameters between different test rigs. In this test rig, both vibration and temperature were kept under control during the wear tests.

In general, heat-treated bearing rings meet the standardized values at operating temperatures up to 120°C. At higher operating temperatures, some changes such as a decrease in hardness and dimensional stability may occur. So operating temperature should be monitored during the wear tests. For this reason, the thermocouples were set to 100°C to be on the safe side. When the thermocouples detected a temperature over 100°C, the test was terminated. In addition to temperature controls, the vibration velocity sensors, which have effective frequency bandwidth between 10 Hz and 1 kHz, were used during the tests. When the vibration velocity reached at 6.5 mm/s, the related test again was terminated.

To obtain a contaminated lubrication in oil bath, the gas atomized particles of highspeed tool steel, SPM30, were mixed with the oil. Thus, the tests of the bearings were carried out in a contaminated oil with SPM30 particles. After the predetermined amount of metal particles were put into the oil bath, life tests were started. The related metal particles were agitated in the oil bath due to the rotation of bearings. Chemical composition and particle size distribution of SPM 30 particles are given in Table 11 and Table 12 respectively. The related gas atomized particles were purchased as a certified materials from SANYO Special Steel Co. LTD

u	С	Si	Mn	Р	S	Cr	Mo	V
nical ositic s%)	1.288	0.28	0.31	0.012	0.008	4.05	5.1	3.02
Chen ompo (mas	W	Co	Fe					
ĞĞ	6.02	8.08	Rest					

Table 11 Chemical composition of SPM30

Table 12 Particle size distribution of SPM30

Sieve Size(µm)	Cumulative	Cumulative
	Passing(%)	Retained(%)
500.00	100.00	0.00
350.00	100.00	0.62
250.00	99.38	5.40
180.00	93.98	10.79
125.00	83.19	24.43
106.00	58.76	48.99
88.00	9.77	8.99
62.00	0.78	0.78
44.00	0.00	0.00
31.00	0.00	0.00
22.00	0.00	0.00
16.00	0.00	0.00
11.00	0.00	0.00
7.80	0.00	0.00
5.50	0.00	0.00
3.90	0.00	0.00
2.80	0.00	0.00
1.90	0.00	0.00
1.40	0.00	0.00
0.90	0.00	0.00

### **CHAPTER 4**

### **EXPERIMENTAL RESULTS AND DISCUSSION**

The effect of undissolved carbides and heat treatment parameters on retained austenite were studied in this thesis. For this purpose, several specimens are heat treated at different austenitization and tempering temperatures. Sub-zero treatment – tempering combinations were also applied. After evaluation of microstructures, the selected heat treatment parameters were applied to the bearing rings. Wear tests were performed on those several heat-treated samples. At the end of the study, the relation between heat treatment parameters and wear properties was discussed.

#### 4.1 Preliminary Test Results

The preliminary tests were carried out to decide through hardening process parameters and to interpret the effect of these parameters on volume fraction of undissolved carbide and retained austenite. These tests were conducted on flat samples, which were described briefly in section 3.1.1.1. The details and results of preliminary test are given in the following sections.

#### 4.1.1 Through Hardening Processes

In this part of the study, the main purpose was to examine the effect of austenitizing temperature on carbides and the amount of retained austenite. So, flat samples were divided in 5 different groups. Each group was austenitized at a different temperature, namely 800°C, 850°C, 900°C, 950°C and 1000°C for 35 minutes. Then the specimens are oil quenched at 50°C.

#### 4.1.1.1 Microstructural Development in Heat Treated Samples

In Figure 19 - Figure 23, the microstructures of bearing steel are seen after austenitized at different temperatures. After austenitized at 850°C and quenched in oil, the microstructure consists of alloy carbides distributed in a martensitic matrix (Figure 20). The undissolved alloy carbides are seen as white, small spherical particles (Figure 20). As the austenitization temperature is increased to 900°C, the microstructural development is very similar to that of the sample treated at 850°C. However, a further increase in austenitization temperature to 950°C caused dissolution of most of the alloy carbides in the austenitic matrix. This is more evident in the specimen austenitized at 1000°C such that there is no undissolved carbide left in the matrix. The microstructure consists of only martensite needles (and most probably bulky RA). It is generally very difficult to differentiate retained austenite under SEM after etching with Nital.

In order to quantify the amount of retained austenite and undissolved carbides, the XRD and color etching method was used respectively which will be discussed in the following sections.



Figure 19 The SEM image of the specimen after austenitized at 800°C and quenched in oil (Untempered)



Figure 20 The SEM image of the specimen after austenitized at 850°C and quenched in oil (Untempered)



Figure 21 The SEM image of the specimen after austenitized at 900°C and quenched in oil (Untempered)



Figure 22 The SEM image of the specimen after austenitized at 950°C and quenched in oil (Untempered)



Figure 23 The SEM image of the specimen after austenitized at 1000°C and quenched in oil (Untempered)

### 4.1.1.2 Quantitative Analysis of Alloy Carbides

After austenitizing at five different temperatures and oil quenched, the carbide staining process is used to calculate the volume fraction of undissolved carbides. In literature, alkaline sodium picrate and nital etchants were utilized for carbide staining [49, 22]. After etching with either Nital or Alkaline etchant solutions, the microstructures were analyzed under optical microscope. The outputs of optical microscope were used as an input to ImageJ analysis as described in section 3.5. In nital etch, the white contrast of the carbides are introduced as an input to the image analyzer. On the other hand, for alkaline etch, the dark areas are assumed as undissolved carbides. All of the optical microscope and ImageJ images are given in Table 13 and Table 14 together with the calculated volume fraction of undissolved carbides.

Austenitizing Temperatures	Microstructure – Optical Microscope	Microstructure – ImageJ (500px -500px)	Volume Fraction of Undissolved Carbides
800 °C			24.5% (±1.5%)
850 °C			10.8% (±1.0%)
900 °C			9.9% (±1.1%)
950 °C			7.1% (±0.3%)
1000 °C			6.6% (±0.3%)

Table 13 Microstructure analysis and calculated undissolved carbides using alkaline sodium picrate etchant

Austenitizing Temperatures	Microstructure – Optical Microscope	Microstructure – ImageJ (500px -500px)	Volume Fraction of Undissolved Carbides
800 °C			8.6% (±0.2%)
850 °C			7.8% (±0.4%)
900 °C			6.4% (±0.3%)
950 °C			4.2% (±0.04%)
1000 °C			1.1% (±0.06%)

Table 14 Microstructure analysis and calculated undissolved carbides using nital (%5) etchant

As can be seen in Table 13 and Table 14, the amount of undissolved carbides decreases with an increase in austenitization temperature as expected. Additionally, Figure 24 compares the volume fraction of carbides calculation results for alkaline sodium picrate and nital etchants.



Figure 24 Volume fraction of undissolved carbides calculation by using different etchants

As seen in Figure 24, there is a remarkable difference between the volume fraction of undissolved carbides in alkaline sodium picrate and nital etchants. It is thought that alkaline sodium picrate stains a larger area than the real area fraction of the carbides. The comparison of the retained austenite measurements of this study with that of literature also supported this observation and will be discussed in the next section.

#### 4.1.1.3 Retained Austenite Measurements with XRD Analysis

The amount of retained austenite in through-hardened samples were calculated by using the amount of undissolved carbide values as an input. Due to fact that X-rays do not generally penetrate more than 10  $\mu$ m into steel, the amount of retained austenite and the resolution of X-ray diffraction are highly sensitive to any local deformation on the sample surface [31]. For this reason, flat samples are electropolished approximately 70  $\mu$ m prior to diffraction and the XRD measurements were handled with this careful sample preparation in order to obtain more reliable results. In calculation of retained austenite content of bearing steels by XRD, the percentage of undissolved carbides is an important input. The retained austenite contents of the specimens are calculated by using the both data, i.e. from alkaline sodium picrate etch and from Nital etch. These results are tabulated in Table 15 and Table 16.

Austenitizing Temperatures	Volume Fraction Of Carbides	Retained Austenite% (As quenched samples) XRD Measurement from electropolished surface
800°C	24.5% (±1.5%)	9.2% (±0.7%)
850°C	10.8% (±1.0%)	14.2% (±0.9%)
900°C	9.9% (±1.1%)	18.0% (±0.6%)
950°C	7.1% (±0.3%)	28.2% (±1.5%)
1000°C	6.6% (±0.3%)	48.8% (±3.5%)

Table 15 Retained austenite measurements of alkaline sodium picrate etched samples

Austenitizing Temperatures	Volume Fraction Of Carbides	Retained Austenite% (As quenched samples)
		XRD Measurement from electropolished surface
800°C	8.6% (±0.2%)	10.0% (±0.8%)
850°C	7.8% (±0.4%)	14.4% (±0.8%)
900°C	6.4% (±0.3%)	18.4% (±0.7%)
950°C	4.2% (±0.04%)	29.0% (±1.7%)
1000°C	1.1% (±0.06%)	58.8% (±5.8%)

Table 16 Retained austenite measurements of nital etched samples

When Table 15 and Table 16 are compared, the undissolved carbide values that were calculated after etching with nital (%5) are more comparable to the ones given in the literature [4, 7]. The microstructural observations also dictated a similar result: For example, the SEM analysis of the samples indicate that there is no undissolved carbide left after austenitizing at 1000°C (Figure 23). This result is reflected best in image analysis of Nital etched samples which yields less than 1% carbide. For this reason, the volume fraction of carbides and the amount of retained austenite that are shown in Table 16 were used in the following sections. In addition to that, only nital etchant was used for the following microstructure analysis in this thesis.



Figure 25 The amount of retained austenite vs. Austenitizing temperatures

When Figure 25 is considered, it is seen that the amount of retained austenite increases with an increase in austenitization temperature. The amount of retained austenite increases from approximately 10% to 58%, as the austenitization temperature increases from 800°C to 1000°C. This is an expected result, since higher temperatures enhances carbide dissolution in the matrix and causes a decrease in both  $M_s$  and  $M_f$  temperatures. As a result higher amount of austenite retains at room temperature.

### 4.1.2 Tempering Process and Its Effect on Retained Austenite

The effect of tempering process on retained austenite content was examined in this section. Samples from 5 different austenitizing temperature groups, as described in section 4.1.1, were tempered in two groups;

- 1<sup>st</sup> Group: Tempered at 180°C for 90 minutes
- 2<sup>nd</sup> Group: Tempered at 235°C for 90 minutes

It is known that tempering process has no effect on the amount of undissolved carbides. This was also verified during experimental studies of this thesis. So, Figure 26 clearly indicates that tempering process does not play critical role in the volume fraction of undissolved carbides. It was experienced that a vital factor in the volume fraction of carbides is the austenitizing temperature, not tempering process. Because of this reason, the volume fraction of undissolved carbides values in Table 14 are used for the further retained austenite measurements with XRD analysis in 180°C and 235°C tempered samples.



Figure 26 Volume fraction of undissolved carbides for tempered and untempered samples

Because of the fact that tempering process did not show a major effect on undissolved carbides, SEM analysis was conducted just for illustration. So only samples that were austenitized at 1000°C and tempered were selected for this illustration. Figure 27 and Figure 28 show the images taken under SEM. As seen in Figure 27 and Figure 28, the effect of tempering on microstructure is not very clear. This would be an expected result since the tempering temperatures are very low for a change in acicular morphology of martensite.


Figure 27 The SEM images of tempered samples at 180°C after austenitized at 1000°C and quenched in oil



Figure 28 The SEM images of tempered samples at 235°C after austenitized at 1000°C and quenched in oil

In order to measure the amount of retained austenite, the XRD analysis was completed with the identical procedure described in 4.1.1.3. The retained austenite contents of tempered specimens are compared with that of untempered specimens in Table 17. The graphical representation of the results are plotted in Figure 29.

	Retained Austenite Analysis by XRD				
Austenitizing Temperatures	Untempered	Tempered at 180°C for 90 minutes	Tempered at 235°C for 90 minutes		
800 °C	10.0% (±0.8%)	10.0% (±0.8%)	0.0% (±0.0%)		
850 °C	14.4% (±0.8%)	11.6% (±0.8%)	3.0% (±0.1%)		
900 °C	18.4% (±0.7%)	19.1% (±1.2%)	5.8% (±0.2%)		
950 °C	29.0% (±1.7%)	25.2% (±1.2%)	12.3% (±0.2%)		
1000 °C	58.8% (±5.8%)	56.1% (±1.3%)	36.0% (±1.3%)		

Table 17 The amount of retained austenite for tempered and untempered samples



Figure 29 Graph for the amount of retained austenite for tempered and untempered samples

As seen in Figure 29, the retained austenite content of untempered samples were approximately same with the samples tempered at 180°C for 90 minutes. On the other hand, when the samples were tempered at 235°C for 90 minutes, the amount of retained austenite reduces progressively. This indicates that if tempering operation is carried at a sufficiently high temperature, the retained austenite re-conditions and transforms to fresh martensite upon quenching. Otherwise, a low tempering temperature seems to be not enough for retained austenite conditioning. These results show that the retained austenite content of the samples can be controlled by adjusting the tempering temperature. In this study, the re-conditioning could be achieved when a tempering temperature above 200 °C is selected.

Also the hardness measurements of these sample groups are given in Table 18 and plotted in Figure 30. When Figure 30 is considered, the lowest hardness values were observed at 800°C. On the other hand, while the peak hardness values have obtained at 900°C, the hardness values have followed a decreasing trend after 900°C for all specimen groups. Due to previously mentioned effect of tempering process on retained austenite, the reduction of hardness values of the tempered samples is an expected observation.

	Hardness (HRC)				
Austenitizing Temperatures	Untempered	Tempered at 180°C for 90 minutes	Tempered at 235°C for 90 minutes		
800 °C	58.0 (±2 HRC)	53.8 (±1 HRC)	46.0 (±8 HRC)		
850 °C	63.0 (±3 HRC)	62.2 (±0.2 HRC)	60.0 (±1 HRC)		
900 °C	66.8 (±2 HRC)	64.1 (±0.1 HRC)	61.4 (±1 HRC)		
950 °C	65.5 (±0.5 HRC)	62.2 (±0.2 HRC)	60.8 (±0.5 HRC)		
1000 °C	61.8 (±2 HRC)	59.0 (±0.6 HRC)	58.6 (±1 HRC)		

Table 18 Hardness measurements of tempered flat samples



Figure 30 The hardness measurements change with the austenitizing temperatures for flat samples

### 4.1.3 Sub-zero Heat Treatment Process and Its Effect on Retained Austenite

To observe the effect of sub-zero heat treatment process on the amount of retained austenite, flat samples were subjected to sub-zero heat treatment process. Since, the optimum austenitization temperature is 840°C for the bearing steel, these sub-zero heat treatment trials were conducted on previously austenitized specimens at 850°C.

After a sub-zero heat treatment at -120°C for 120 minutes the specimens are tempered at 170°C for 90 minutes to minimize the brittleness of martensite.

The amount of retained austenite of sub-zero heat treated samples were calculated by using the respective volume fraction of carbides given in Table 16. Table 19 summarizes the amount of retained austenite in sub-zero heat-treated sample together with tempered at 180°C sample.

Retained Austenite (%)				
Austenitized at 850°C	Austenitized at 850°C	Austenitized at 850°C, then		
(In as-quenched	and tempered at 180°C	sub-zero heat-treated (-120°C) and tempered at 170°C		
condition)	and tempered at 100 C			
14.4% (±0.8%)	11.6% (±0.8%)	2.8% (±0.2%)		

Table 19 The comparison of the amount of retained austenite between sub-zero treated, single tempered (180°C) and as-quenched samples

As it is seen in Table 19, the amount of retained austenite is slightly above the detectability limit 2% after a sub-zero treatment. Therefore, it can be stated that a sub-zero heat treatment step substantially decreases the amount of retained austenite by transforming it into martensite. Therefore, sub-zero heat treatment seems to be more effective than a single step tempering process (at 180°C) in terms of retained austenite reduction.

# **4.1.4 Double Tempering Heat Treatment Process and Its Effect on Retained Austenite**

Besides sub-zero heat treatment process, the effect of double tempering treatment on the amount of retained austenite was also examined. For this purpose, the flat samples that were austenitized at 850°C for 35 minutes and oil quenched were subjected to double tempering at 190°C for 90 minutes and then 170°C for 90 minutes. Between tempering cycles, the specimen is cooled to room temperature before tempering at 170°C.

Similar to the retained austenite measurement of sub-zero heat treated samples, the retained austenite amount of double tempering heat treated samples was calculated by using the respective volume fraction of carbides value given in Table 16. Table 20 compares the amount of retained austenite in double tempered sample (190°C and 170°C) with that of single tempered specimen (180°C) and as-quenched specimen.

Table 20 The comparison of the amount of retained austenite between double tempered, single tempered at 180°C and as-quenched samples

Retained Austenite (%)				
Austenitized at 850°C	Austanitizad at 850°C and	Austenitized at 850°C and then double tempered at 190°C and at 170°C		
(in as-quenched condition)	tempered at 180°C			
14.4% (±0.8%)	11.6% (±0.8%)	8.3% (±0.4%)		

As seen in Table 20, the amount of retained ustenite decreases from 14% to 8% upon a double tempering operation. Although a double tempering treatment causes a decrease in the amount of retained austenite, it's effect is not as significant as sub-zero treated samples. Nevertheless, the reason behind the double tempering is to reduce the amount of retained austenite and to make the fresh martensite tougher. Because of this reason, double tempering may be carried out just for being on the safe side for bearing steel applications.

### 4.2 Bearing Ring Tests

Accounting for all results that were obtained in section 4.1, 20 different set of bearing rings were produced with different heat treatment parameters. So, this section focuses on the relationship between microstructure, hardness and wear behavior of these set of bearings.

### 4.2.1 Ring Samples Preparation

In order to evaluate the relation between different heat treated microstructures and the wear resistance, all tempering processes that were experimented in the previous sections were applied after five different austenitizing temperatures; 800°C, 850°C, 900°C, 950°C and 1000°C. These set of bearing groups and their heat treatment processes are summarized in Table 21.

Sample ID	Process Flow of Heat Treatment		
800A-180T	Austenitized at 800°C, Temper at 180°C for 90 minutes		
800A-235T	Austenitized at 800°C, Temper at 235°C for 90 minutes		
800A-120SZ-170T	Austenitized at 800°C, Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes		
800A-190T-170T	Austenitized at 800°C, Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes		
850A-180T	Austenitized at 850°C, Temper at 180°C for 90 minutes		
850A-235T	Austenitized at 850°C, Temper at 235°C for 90 minutes		
850A-SZ120-170T	Austenitized at 850°C, Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes		
850A-190T-170T	Austenitized at 850°C, Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes		
900A-180T	Austenitized at 900°C, Temper at 180°C for 90 minutes		
900A-235T	Austenitized at 900°C, Temper at 235°C for 90 minutes		
900A-SZ120-170T	Austenitized at 900°C, Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes		
900A-190T-170T	Austenitized at 900°C, Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes		
950A-180T	Austenitized at 950°C, Temper at 180°C for 90 minutes		
950A-235T	Austenitized at 950°C, Temper at 235°C for 90 minutes		
950A-SZ120-170T	Austenitized at 950°C, Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes		
950A-190T-170T	Austenitized at 950°C, Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes		
1000A-180T	Austenitized at 1000°C, Temper at 180°C for 90 minutes		
1000A-235T	Austenitized at 1000°C, Temper at 235°C for 90 minutes		
1000A-SZ120-170T	Austenitized at 1000°C, Sub-zero treatment at -120°C for 120 minutes and then Temper at 170°C for 90 minutes		
1000A-190T-170T	Austenitized at 1000°C, Temper at 190°C for 90 minutes and then Temper at 170°C for 90 minutes		

Table 21 Set of bearing groups and their heat treatment processes

### 4.2.2 Microstructural Development in Ring Samples

Before going forward with the retained austenite measurements, the volume fraction of carbides calculation of the newly produced bearing rings were verified with the ones in section 4.1. Since all tempering experiments in section 4.1 (tempering at 180°C, at 235°C, sub-zero heat treatment and double tempering) were applied for samples that were austenitized at 850°C, ring groups that were austenitized at 850°C were selected for the comparison and verification of undissolved carbides values. So, ring sample and their respective flat samples are listed in Table 22.

Table 22 clearly indicates that undissolved carbide values obtained from ring samples are approximately same with that of flat samples given in Table 16. The respective volume fraction of carbides values given in Table 16 were used in retained austenite calculation of all ring sample groups.

Sample ID	Image of Optical Microscope	Image of ImageJ Analysis	VFC (Ring Samples)	VFC (Flat Samples)
850A-180T			7.2% (±1.7%)	
850A-235T			6.8% (±0.23%)	7.8% (±0.4%)
850A-SZ120- 170T			7.1% (±0.21%)	
850A-190T- 170T			7.1% (±0.53%)	

Table 22 The verification of undissolved carbide calculation

# 4.2.3 Retained Austenite Measurement with XRD

The retained austenite amount of 20 different sample groups were calculated by considering undissolved carbide values as it is explained in the previous section 4.2.2. These measurements can be seen in Table 23.

		Austenitizing Temperatures				
		800A	850A	900A	950A	1000A
	1907	6.2%	9.2%	11.6%	16.4%	26.0%
70	1001	(±0.2%)	(±0.0%)	(±0.2%)	(±0.8%)	(±3.4%)
ering Process	235T	1.9%	2.3%	3.1%	4.4%	16.4%
		(±0.0%)	(±0.0%)	(±0.1%)	(±0.0%)	(±0.8%)
	S7120 170T	0.6%	1.5%	2.0%	4.2%	6.5%
Femp	52120-1701	(±0.0%)	(±0.1%)	(±0.0%)	(±0.1%)	(±0.5%)
Γ.	100T 1 <b>7</b> 0T	3.9%	7.5%	8.4%	12.5%	18.9%
	1901-1701	(±0.1%)	(±0.1%)	(±0.0%)	(±0.4%)	(±0.9%)

Table 23 The retained austenite amount of sample groups for wear tests

For a better visualization, the amount of retained austenite for all the sample groups are plotted in Figure 31. As seen in Figure 31, a tempering at 180°C (180T) has the maximum, whereas a sub-zero treatment (SZ120-170T) has the minimum amount of retained austenite. Under those circumstances, it was decided that the wear tests to be performed with 180T and SZ120-170T samples of each five different austenitizing temperatures in the following sections of this study.



Figure 31 Retained austenite values of sample groups for wear tests

### 4.2.4 Hardness Measurements

The hardness measurements of all sample groups are given in Table 24 and displayed in Figure 32.

	Hardness (HRC)				
Austenitizing Temperatures	180T	235T	SZ120-170T	190T-170T	
800 °C	62.1	57.7	60.4	58.3	
	(±0.2 HRC)	(±0.5 HRC)	(±0.8 HRC)	(±0.5 HRC)	
850 °C	63.2	59.9	63.5	61.6	
	(±0.3 HRC)	(±0.5 HRC)	(±0.2 HRC)	(±0.6 HRC)	
900 °C	62.9	60.9	66.1	62.6	
	(±0.2 HRC)	(±0.1 HRC)	(±0.1 HRC)	(±0.2 HRC)	
950 °C	62.1	60.3	66.5	61.7	
	(±0.1 HRC)	(±0.1 HRC)	(±0.1 HRC)	(±0.1 HRC)	
1000 °C	60.6	59.8	67.0	59.6	
	(±0.3 HRC)	(±0.3 HRC)	(±0.1 HRC)	(±0.2 HRC)	

Table 24 Hardness measurements of ring samples



Figure 32 The hardness measurements change with the austenitizing temperatures

The trend in hardness variation of bearing samples is very similar to that of flat samples. It is worth to note that austenitizing at 800°C is not sufficient to obtain a complete transformation from body-centered cubic ferrite to homogeneous face centered cubic austenite by increasing the stability of carbon yielding the lowest hardness value was obtained at 800°C. Morever, it can be deduced that peak hardness is achieved at austenitizing temperatures between 850°-900°C after tempering processes. The lower hardness values of the tempered bearings after an austenitization treatment at 950°C and 1000°C can be related to the dissolution of residual carbides and causing a reduction in both M<sub>s</sub> and M<sub>f</sub> temperatures. On the other hand, hardness continues to increase above 950°C and 1000°C for sub-zero treated samples which is most probably due to the transformation of the retained austenite to martensite.



Figure 33 The retained austenite measurements change with the hardness values

Figure 33 plots the hardness vs. retained austenite contents of all samples. The parameters that gave the lowest retained austenite content and the highest hardness belongs to subzero treated samples. In addition to this, both the lowest retained austenite content and the lowest hardness values were obtained after austenitizing at 800°C, due to previously mentioned partial transformation.

# 4.2.5 Comparison of Hardness and Retained Austenite Content of Flat and Bearing Ring Samples

Figure 35 and Figure 34 compares the hardness and retained austenite contents of the flat and bearing ring samples. As discussed in section 4.1, the preliminary experiments are carried out in order to organize the heat treatment cycle of bearings which will be used in wear tests. As seen in Figure 34 and Figure 35, the hardness values of flat

samples and bearing samples are comparable if the austenitization is carried out in the range 850°C - 1000°C. However, for the samples austenitized at 800°C, the flat samples yield somewhat lower hardness values with respect to bearing samples. The size of the flat specimens is approximately 3 times thicker with respect to bearing rings. It is believed that the sample size affects the amount of retained austenite content and hence the hardness values at lower austenitization temperature. This effect can be both due to the heating rate at a given austenitization temperature or due to the cooling rate from that temperature.



Figure 34 Comparison of hardness of flat and bearing ring samples



Figure 35 Comparison of retained austenite content of flat and bearing ring samples

## 4.2.6 Wear Tests Under Contaminated Lubrication

After finishing retained austenite measurements, grinding process is applied to obtain the final form of inner and outer rings with precise tolerances. These inner and outer ground rings from 20 different heat treated groups given in Table 21 are assembled together with an additional auxiliary bearing elements namely balls, cages and cover. This assembling process is depicted in Figure 36. In this way, bearings were prepared for wear tests under contaminated lubrication.



Figure 36 Assembling of bearing rings with auxiliary elements

As indicated in section 4.2.3, 180T and SZ120-170T samples of each austenitizing temperatures were used for wear tests. The details of the wear test conditions and its equipment were described in section 3.7.

The failure criteria of wear test was determined as either heating of the bearing over temperature of 100°C or the vibration velocity over 6.5 mm/s. The bearing life time has been recorded until one of these failure criteria occurs. So this recorded life time (in hours) was accepted as an indication of the wear performance of bearings.

Wear test results under contaminated lubrication were presented in Table 25 together with their retained austenite content and hardness values. All of the 950A-SZ120-170T and 1000A-SZ120-170T bearing rings were broken during mounting process, so that their bearings could not be assembled. So, the testing of these groups are cancelled.

	Wear Test	Retained	Hardness
SAMPLE ID	Result (h)	Austenite (%)	(HRC)
800A-180T	102.41	6.2% (±0.2%)	62.1 (±0.2)
800A-SZ120-170T	67.29	0.6% (±0.0%)	60.4 (±0.8)
850A-180T	270.00	9.2% (±0.0%)	63.2 (±0.3)
850A-SZ120-170T	94.28	1.5% (±0.1%)	63.5 (±0.2)
900A-180T	328.79	11.6% (±0.2%)	62.9 (±0.2)
900A-SZ120-170T	103.13	2.0% (±0.0%)	66.1 (±0.1)
950A-180T	312.94	16.4% (±0.8%)	62.1 (±0.1)
950A-SZ120-170T	N/A	4.2% (±0.1%)	66.5 (±0.1)
1000A-180T	234.82	26.0% (±3.4%)	60.6 (±0.3)
1000A-SZ120-170T	N/A	6.5% (±0.5%)	67.0 (±0.1)

Table 25 Wear test under contaminated lubrication results for bearing groups

As it was presented in Figure 25, at austenitizing temperatures higher than 900°C, the amount of retained austenite increases exponentially due to the higher carbide dissolution in the matrix, thereby decreasing M<sub>f</sub> and M<sub>s</sub> temperatures. The transformation of this higher amount of retained austenite to martensite by sub-zero cooling process causes higher hardness values with an increase in the percentage of martensite in microstructure. Although the principle of tempering application after sub-zero cooling process is to reduce hardness and to increase toughness, it is thought that a tempering temperature of 170°C was not sufficient to decrease the brittleness of the martensite in sub-zero treated specimen. At high austenitization temperatures the austenite will be highly saturated in terms of carbon and Cr due to the excessive dissolution of alloy carbides. Hence, the martensite formed from this highly supersaturated austenite will also contain too much carbon and become very brittle. It must be noted that a sub-zero treatment minimizes the amount of retained austenite which otherwise would contribute to toughness. That's why Table 25 points that 950A-SZ120-170T and 1000A-SZ120-170T are the hardest specimens among all the bearing groups.

For better illustration, wear test results and the amount of retained austenite were plotted in Figure 37. Moreover, the relation between the hardness values and wear test results were also depicted in Figure 38.



Figure 37 Wear test result vs amount of retained austenite



Figure 38 Wear test results vs hardness values

It is a well-known fact that wear resistance of steels are affected from both the hardness and the amount of carbides in the matrix. Generally, the higher the hardness and amount of alloy carbides, the higher the wear resistance. However, the selection of austenitization temperature and tempering route can make the microstructure more complicated. For example, an austenitization treatment at 1000°C and tempering after a sub-zero treatment yields highest hardness values but at the same time lowest percentage of alloy carbides in the matrix.

When both Figure 37 and Figure 38 are analyzed, tempering at 180°C is more successful than sub-zero cooling process in terms of wear life time. In addition, it is seen that there is strong correlation between the amount of retained austenite, hardness and wear test results. Figure 37 shows that when the amount of retained austenite increases, the wear life time of bearings also increases up to 900°C austenitizing temperature. On the other hand, hardness starts to decrease at austenitizing

temperatures higher than 900°C for the 180T bearings. Because of these results, it is thought that the amount of retained austenite should be increased up to some level without compromising hardness in order to maximize the wear life time of bearings. The effect of alloy carbides on wear resistance is reflected in specimens austenitized at high temperatures. After an austenitization treatment at 950°C and 1000°C, the amount of undissolved carbides decreases to 4% and 1% respectively. Therefore, the poor performance of the bearing austenitized at 1000°C and tempered at 180°C can be attributed to the lower content of carbides in the matrix.



Figure 39 3D scatter plot for retained austenite, wear life time and hardness

Figure 39 plots the results of wear life time, the amount of retained austenite and hardness measurements together. Based on the results of Figure 39, it is possible to claim that the maximum wear life time is achieved when the bearings have 10-15% amount of retained austenite and 62-63 HRC hardness. Moreover, increasing the hardness values to 66 HRC by sub-zero cooling process is not beneficial to wear life time.

Consequently, the optimum heat treatment parameters appear to be austenitizing at 900°C and then tempering at 180°C (900A-180T) for maximum bearings service life time under contaminated lubrication.

### **CHAPTER 5**

### CONCLUSION

In this study, the effect of undissolved carbides and retained austenite on the wear life of bearings produced from 100Cr6 steel under contaminated oil conditions were investigated. The following conclusions are withdrawn from this study:

1) The volume fraction of carbides were calculated using an image analyzer after carbide staining process by alkaline sodium picrate and nital etchants. It is found that nital etching yields more reliable results in terms of volume fraction of carbide evaluations.

2) The specimens are austenitized at either 800°C, 850°C, 900°C, 950°C or 1000°C and then quenched to room temperature in oil to produce a hard martensitic phase. It is seen that the amount of undissolved carbides decreases but the amount of retained austenite increases with increasing austenitization temperature from 800°C to 1000°C. The amount of retained austenite in all the samples are in the range of 10% to 58% and the corresponding hardness values are in the range of 58 HRC - 67 HRC. The peak hardness is obtained for the specimen austenitized at 900°C.

3) Tempering at 180°C or a double tempering at 190°C, and then, at 170°C do not affect the amount of retained austenite considerably. On the other hand, after tempering at 235°C, conditioning of the retained austenite takes place and a considerable amount of it transforms to martensite. The retained austenite content of the double tempered samples are in the range 4% - 19%. In the sub-zero cooled + tempered samples, most of the retained austenite transforms to martensite, such that the amount of retained austenite decreased to a range of 0%-6%. The highest hardness is attained in the specimen austenitized at 1000°C and tempered at 170°C. However, at this condition, the rings are so brittle that they are broken at the assemble stage. 4) The wear test is applied to the bearings which are either tempered at  $180^{\circ}$ C or sub-zero cooled + tempered at  $170^{\circ}$ C. It seems that a tempering at  $180^{\circ}$ C is more successful than the sub-zero cooled + tempered specimens in terms of wear life time. The maximum wear life time is achieved when the bearings have a retained austenite content in the range of 10%-15% and 62-63 HRC hardness.

5) Increasing the hardness values to 66 HRC makes the inner and outer rings very brittle which leads to failure of them during assembly.

### **Future Works**

As a future work, the worn surfaces of bearings can be examined under SEM. A relation between plastic deformation behavior such as pitting, dent origination, crack progression etc. and the amount of retained austenite may be established. This could help to investigate the failure mechanism of bearings with different heat treated microstructures. In addition to this, wear tests were only performed for 180T and SZ120-170T samples of each five different austenitizing temperatures in this thesis. Besides this, the wear test of the rest bearing groups can be completed. So that the results can be validated by increasing the number of tests.

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