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Influence of Heating Media on Mechanical and Microstructural Properties of CP Titanium Alloy

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Anahtar Kelimeler

CP Titanyum
Renk Değişimi
Mikroyapı
Yorulma
Hidrojen Miktarı

Graphical/Tabular Abstract (Grafik Özet)

In this work, CP Ti alloys were heat treated in atmosphere-controlled and vacuum media. The effects of different heating media in terms of mechanical and microstructural features were investigated. / Bu çalışmada, CP Ti alaşımları atmosfer kontrollü ve vakumlu ortamlarda ısıl işleme tabi tutulmuştur. Farklı ısıtma ortamlarının mekanik ve mikro yapısal özellikler üzerindeki etkileri incelenmiştir.

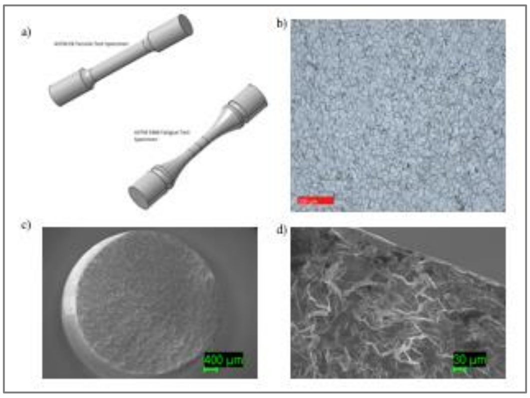

Figure A: *a) Sample types, b) Microstructure, c) Fracture macrography, d) Fracture surface /* ***Şekil A:*** *a) Numune tipleri, b) Mikroyapı, c) Kırılma makrografisi, d) Kırılma yüzeyi*

Highlights (Önemli noktalar)

- *Microstructure was observed identical in atmosphere-controlled and vauum heating media. / Atmosfer kontrollü ve vakum ısıtmalı ortamlarda mikro yapının çok yakın olduğu gözlemlenmiştir.*
- *The vacuum-heat treated samples showed 7 % higher tensile and yield strength. / Vakum ortamında ısıl işlem görmüş numuneler %7 daha yüksek çekme ve akma mukavemeti gösterdi.*
- *The vacuum-heat treated samples illustrated nearly 50 % higher fatigue life at highest stress level. / Vakum ortamında ısıl işlem görmüş numuneler, en yüksek gerilim seviyesinde yaklaşık %50 daha yüksek yorulma ömrü göstermiştir.*

Aim (Amaç): *To investigate the effects of heating media on mechanic and microstructural properties on CP Ti alloy. / CP Ti alaşımının mekanik ve mikro yapısal özellikleri üzerinde ısıtma ortamlarının etkilerini araştırmak.*

Originality (Özgünlük): *This study provides more detailed information on fatigue life of CP Ti alloys in case of discoloration. / Bu çalışma, renk bozulması durumunda CP Ti alaşımlarının yorulma ömrü hakkında daha ayrıntılı bilgi sağlamaktadır.*

Results (Bulgular): *Oxygen and hydrogen impurity content in vacuum heat-treated samples are very close to base metal. On the other hand, hydrogen content is increased in atmosphere-controlled heating media by nearly 20 ppm and oxygen 50 ppm. / Vakum altında ısıl işlem görmüş numunelerdeki oksijen ve hidrojen safsızlık içeriği, baz metale oldukça yakındır. Öte yandan, atmosfer kontrollü ısıtma ortamında hidrojen içeriği yaklaşık 20 ppm, oksijen içeriği ise 50 ppm artmaktadır.*

Conclusion (Sonuç): *Discoloration was linked to hydrogen and oxygen content increment. Reduction in mechanical properties are attributed to hydrogen content of atmosphere-controlled heat-treated samples. / Renk bozulmasının, hidrojen ve oksijen içeriğindeki artışla bağlantılı olduğu görülmüştür. Mekanik özelliklerdeki azalma ise atmosfer kontrollü ısıl işlem görmüş numunelerin hidrojen içeriğinden kaynaklanmaktadır.*

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Abstract

Commercially pure titanium alloys are used as structural materials in aerospace industry. These alloys are mainly exposed to stress relief after forming operations. Heat treating of commercially pure titanium (CP Ti) alloys are generally carried out in air, atmosphere-controlled and vacuum atmospheres. This study investigates the effect of heating media on mechanical and microstructural features of CP Ti alloys. The samples were subjected to stress relief treatment by different heating media. The heating media are vacuum and atmosphere-controlled atmospheres. Discoloration was observed in atmosphere-controlled heating media. The microstructure was not influenced by heating media either near the surface or at core. Although discoloration was detected, alpha case formation was not noticed. The samples heat-treated in vacuum atmosphere showed approximately 7% higher tensile and yield strength whereas elongation percentage was not changed. Fatigue test was performed at 310, 350 and 370 MPa stress levels. The fatigue life of vacuum heat-treated samples is higher than atmosphere-controlled heat-treated samples at all stress levels. When stress level increased up to 375 MPa, the difference in fatigue life is nearly 50%. Oxygen and hydrogen contents were not changed so much in vacuum heat-treated samples whereas oxygen content was increased by 50 ppm and hydrogen content by 20 ppm in atmosphere-controlled heat-treated specimens. Increment in hydrogen content decreased mechanical properties.

Isıtma Ortamının CP Titanyum Alaşımının Mekanik ve Mikroyapı Özellikleri Üzerindeki Etkisi

Makale Bilgisi

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Öz

Titanyum alaşımları, yüksek mukavemetleri, yüksek sertlikleri ve düşük ısıl iletkenlikleri Ticari olarak saf titanyum alaşımları havacılık ve uzay endüstrisinde yapısal malzeme olarak kullanılmaktadır. Bu alaşımlar çoğunlukla şekillendirme işlemlerinden sonra gerilim giderme ısıl işlemine maruz kalırlar. Ticari olarak saf titanyum (CP Ti) alaşımlarının ısıl işlemleri genellikle hava, atmosfer kontrollü ve vakum atmosferlerinde gerçekleştirilir. Bu çalışma, ısıl işlem ısıtma ortamının CP Ti alaşımlarının mekanik ve mikro yapısal özellikleri üzerindeki etkisini araştırmaktadır. Numuneler farklı ısıtma ortamları ile gerilim giderme işlemine tabi tutulmuştur. Isıtma ortamları vakum ve atmosfer kontrollü atmosferlerdir. Atmosfer kontrollü ısıtma ortamlarında ısıl işlem gören numunelerde renk değişimi gözlenmiştir. Mikro yapı incelendiğinde ne yüzeye yakın ne de çekirdek bölgelerinde ısıtma ortamına bağlı değişim gözlenmemiştir. Atmosfer kontrollü ortamda ısıl işlem göre numunelerde renk değişimi gözlenmesine rağmen, alfa fazı oluşumu görülmemiştir. Vakum atmosferinde ısıl işlem görmüş numuneler yaklaşık %7 daha yüksek çekme ve akma dayanımı gösterirken, uzama yüzdeleri benzer değerler göstermiştir. Yorulma testi 310, 350 ve 370 MPa gerilme seviyelerinde gerçekleştirilmiştir. Vakum altında ısıl işlem görmüş numunelerin yorulma ömrü, tüm gerilim seviyelerinde atmosfer kontrollü ısıl işlem görmüş numunelerden daha yüksektir. Gerilim seviyesi 375 MPa'a kadar çıktığında, yorulma ömründeki fark yaklaşık %50 olarak gözlenmiştir. Vakumla ısıl işlem görmüş numunelerde oksijen ve hidrojen içerikleri çok fazla değişmezken, atmosfer kontrollü ısıl işlem görmüş numunelerde oksijen içeriği 50 ppm, hidrojen içeriği ise 20 ppm artmıştır. Hidrojen içeriğindeki artış mekanik özellikleri azaltmıştır.

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1. INTRODUCTION (GİRİŞ)

Many of the materials required for aerospace applications are high-performance materials that can sustain very high thermal cycling, corrosive environments, high mechanical stresses, and weight restrictions, while maintaining strength over long operational periods [1,2]. In this challenging context, titanium alloys have emerged as important materials due to their ideal balance of strength-to-weight ratio and corrosion resistance [3,4]. CP Ti maintains its mechanical strength from cryogenic temperatures to 300°C, allowing it to be used in a wide range of aerospace applications [5]. Although they are not as temperature resistant as some titanium alloys, enough performance can be provided by CP Ti grades on many structural and non-structural aerospace components used within this temperature range [6]. The heat treatment process is essential for improving the properties of CP Ti for aerospace applications. However, it has fewer options than the respective treatment for titanium alloys due to the very low proportion of alloying additions [7,8]. Stress relieving, recrystallizing, and controlling the grain structure, rather than phase transformations, are the primary heat treatment processes applicable to CP Ti [9]. Stress-relief annealing is the most frequently performed heat treatment on CP Ti aerospace parts. This operation is carried out at temperatures ranging between 480 and 595°C (900 and 1100°F) for 15 min−4 h, depending on the part thickness [10]. Processing allowed it to reduce manufacturing-induced residual stresses, and the mechanical properties and microstructure were relatively unaffected [11]. For dimensionally stable aerospace systems, stress relief annealing is an essential processing technique to prevent distortion in service [12]. The process is especially significant in the case of thin-walled components, such as ducting, brackets, and sheet-metal assemblies, which, due to the presence of residual stresses, may fail at earlier stages of life or undergo dimensional variation under operational loads [13]. The heat treatment of CP Ti requires conscientious atmosphere control. The high reactivity of CP Ti with oxygen, nitrogen, and hydrogen makes the heat treatment of this alloy sensitive to atmosphere conditions [14]. Hydrogen especially affects the mechanical performance of titanium alloys known as hydrogen embrittlement. Hydrogen can enter titanium through heat treatment, welding operations and moisture environments. Hydrogen atoms diffuse through the lattice and form titanium hydrides resulting in changing mechanical behavior [15]. Vacuum or controlled atmosphere (argon) heat treatments are necessary to avoid contamination that would compromise the mechanical properties and corrosion resistance. Even trace pollution would be highly detrimental to fatigue properties; hence, atmospheric control is of great importance for aerospace applications [16]. The alpha case, which occurs during the pick-up of oxygen and nitrogen during heat treatment, must be prevented or controlled [17]. This hardened surface layer has lower ductility and fatigue resistance, which may eliminate the reliability of the parts in aerospace service [18]. Mechanical or chemical removal is usually necessary before the component can be used where an alpha case is formed. Not as sensitive to cooling rates as most titanium alloys because they contain no phase transformation [19]. However, cooling speed may affect the final structure size and the level of stress relaxation [20]. Furnace cooling is used for maximum stress relief and grain growth, whereas air cooling further accelerates processing with somewhat higher residual stress levels [21]. For applications in aerospace components that experience thermal cycling, controlled cooling rates are necessary to maintain microstructural stability and uniform mechanical properties throughout the part's life [22]. Mpumlwan et.al [23] studied high temperature heat treatment cycles of CP Ti and found that the microstructure was negatively affected whereas tensile strength and hardness were developed. Park et.al applied cyclic heat treatment to electron beam manufactured CP Ti alloys up to 1000 °C. After heat treatment, they observed that mechanical properties were diminished whereas grain size was refined. Lee et.al [24] worked on rapid electric current heat treatment on CP Ti alloy. According to their findings, electrically heat-treated samples demonstrated more uniform grain size and mechanical properties were improved when compared with conventional heat treatment. In a study [25], CP Ti alloys were exposed to annealing at 700 °C and 900 °C. At higher annealing temperature oxidation was detected and mechanical properties were reduced. Park [26] repeated heat treatment on CP Ti alloys up to 7 cycles. After the cycles, the grain size was diminished, tensile strength was developed, and elongation was held. Mguni studied the effects of oxygen and hydrogen on mechanical properties of CP Ti and Ti6Al4V alloys. They observed that hardness and compressive strengths were improved by increasing quench temperature [27]. An et.al [28] worked on fatigue behavior of CP Ti under cryogenic low cycle fatigue where they observed that cryogenic conditions increase fatigue life. However, according to our knowledge, there is not enough information on how mechanical properties of CP Ti alloys are affected in case of discoloration. This study makes an important contribution to literature by investigating the effects of discoloration, analyzing microstructures and mechanical values and setting an example for industrial applications.

2. MATERIALS AND METHODS (MATERYAL VE METOD)

The chemical composition of the CP Ti alloys used in this study was determined by X-Ray Fluorescence (XRF) and the analysis results are given in Table 1. The analysis results were found to be in compliance with the AMS 4901 standard [29] used in the aerospace industry. Two different sample types were prepared. One sample type is fatigue test specimen according to ASTM E466 M16, the other sample type was prepared for tensile testing according to ASTM E8. The drawings of the samples are shown in Figure 1. In this study, Cp Ti alloys were heat treated in atmosphere controlled and vacuum furnaces. Inert gas, argon mixture was present in an atmosphere-controlled furnace. Stress relief treatment was applied at 540 °C and the soaking time is 150 minutes for both heating media. Furnace cooling was employed below 200 °C for 8 hours. Load thermocouple was used to control the temperature of the parts.

Table 1. Chemical composition of CP Ti alloy (CP Ti alaşımının kimyasal kompozisyonu)

Element	**Ti**	**Cr**	**Co**
Results (wt. %)	99.85	0.08	0.02

After heat treatment, the samples were subjected to tensile and fatigue testing. Tensile Testing was carried out with Instron 5985 equipped with automatic extensometer. Strain-controlled method given in ASTM E8, which are 0.005 mm/mm/min-1 strain rate until yield strength, and 0.05 mm/mm/min-1 strain rate until fracture was utilized. Fatigue Testing was performed according to EN 6072 standard with Instron 8801 Equipment and the parameters are 310,350,375 MPa as maximum stress values, R=0.1, 20 Hz frequency and Kt=1.0. To examine the microhardness properties, the materials were prepared metallographically. Sample cutting was performed with ATM Brilliant 250 device and the cut samples were bakelite with Struers CitoPress 30 device. The samples were also sanded and polished using Struers Laboforce-100 device. Optical microscopy examinations were performed with Zeiss Smartzoom 5 microscope and scanning electron microscope (SEM) analysis were conducted by Zeiss EVO 10. Impurities content in terms of H and O was measured by LECO ONH836 equipment.

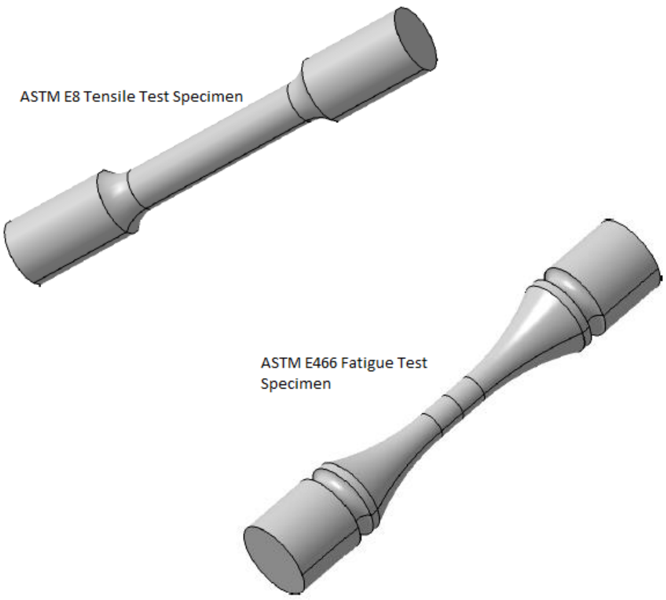

Figure 1. Drawings of the CP-Ti test samples (CP Ti test numunelerinin çizimleri)

2. RESULTS and DISCUSSION (BULGULAR ve TARTIŞMA)

Figure 2a) illustrates the samples before heat treatment. One set of samples were heat treated in an atmosphere-controlled furnace where the surface of the samples has discoloration after heat treatment as shown in Figure 2b) that is a sign of oxidation. It is known that oxidation may reduce mechanical properties and may form alpha case leading to brittle nature on the surface [30]. On the other hand, the samples heated in vacuum atmosphere are metallic gray implying that the surfaces of these samples were not oxidized as shown in Figure 2c.

Figure 2. a) Samples before heat treatment, b) Atmosphere-controlled heat-treated samples b) Vacuum heat-treated samples (a) Isıl işlem öncesi numuneler, b) Atmosfer kontrollü ortamda ısıl işlem görmüş numuneler, b) Vakum ortamında ısıl işlem görmüş numuneler)

As seen in Figure 3, the microstructural analysis was performed both near the surface and at the core. Both regions of the samples show identical microstructure that consists of equiaxed alpha grains. Alpha case, generally formed on the surface as oxide scale, was not observed on the surface of both samples so it can be deduced there was not enough oxygen environment for alpha case formation [31].

Tensile testing results are presented in Table 2. The results are given as average where 5 samples of each set were tested. The average yield strength of atmosphere-controlled heat-treated samples is 520 MPa whereas vacuum heat-treated samples have an average of 558 MPa yield strength. The same trend is observed in tensile strength of the samples where vacuum heat-treated samples show an average of 40 MPa higher than the atmosphere-controlled heat-treated samples. The reason may originate from hydrogen and oxygen content of the heat-treated samples in different heating media. Although there is difference in strength values, elongation of the samples has not affected. Shi et. al [32] suggested that oxygen does not have a significant effect on grain refinement so microstructure similarity could be explained. Also, according to this study, increment in oxygen concentration may increase yield strength without altering elongation below 0.8 wt. %. On the other hand, the harmful results of hydrogen on CP titanium happen at lower hydrogen concentrations and are much more effective [ref 3]. Therefore, lower tensile properties of atmosphere-controlled heat-treated specimens could be explained by increment in hydrogen content.

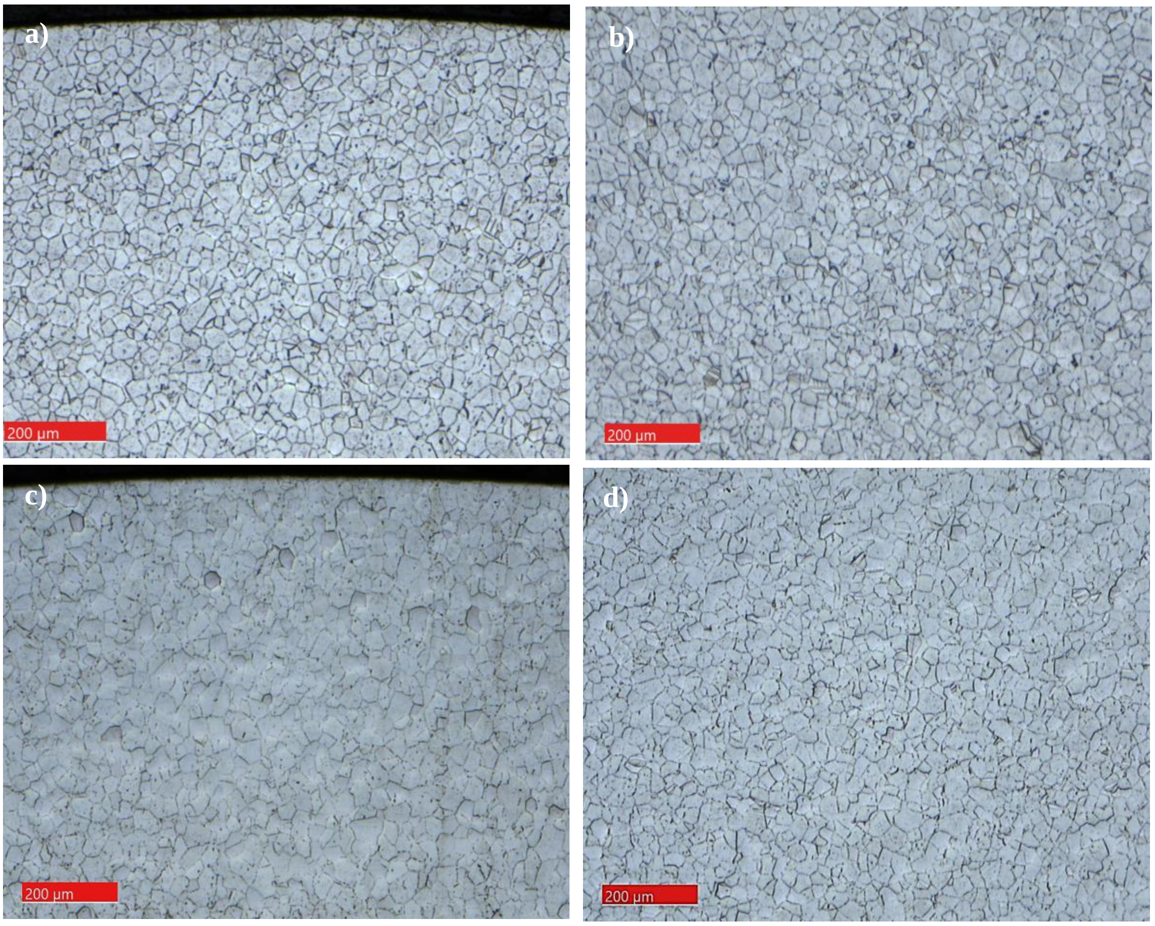

Figure 3. a) Vacuum heat-treated sample near surface area, b) Vacuum heat-treated sample core area, c) Atmosphere-controlled heat-treated sample near surface area, d) Atmosphere-controlled heat-treated sample core area (a) Vakum ortamında ısıl işlem görmüş numunenin yüzey alanına yakın kısmı, b) Vakum ortamında ısıl işlem görmüş numunenin çekirdek alanı, c) Atmosfer kontrollü ortamda ısıl işlem görmüş numunenin yüzey alanına yakın kısmı, d) Atmosfer kontrollü ortamda ısıl işlem görmüş numunenin çekirdek alanı)

Table 2. Tensile properties of the samples after heat treatment (Isıl işlem sonrası numunelerin çekme sonuçları)

Property (Avg.)	**Atmosphere-controlled heat-treated samples**	**Vacuum Heat-Treated Samples**
Yield Strength (MPa)	520	558
Tensile Strength (MPa)	629	669
Elongation (%)	30.92	30.98

Fatigue tests were employed at 310,350 and 375 MPa stress levels. The stress levels were below yield strength due to investigating high cycle fatigue (HCF) properties. The results are given in Figure 4. The fatigue life of vacuum heat-treated samples is higher than the samples heat treated in atmosphere-controlled heating media at all stress levels. When looked at detailed at the graph, the fatigue life difference is getting smaller at 310 MPa stress level but the difference is getting higher at higher stress levels. When stress level is enhanced to 375 MPa, fatigue life difference is nearly 50 %. The trend is similar to the study given in the literature [33]. The fatigue life of vacuum heat-treated samples matches well with values of previous study done by Hosseini et.al [34]. To understand the failure mechanism of the samples, scanning electron microscope (SEM) analysis was conducted.

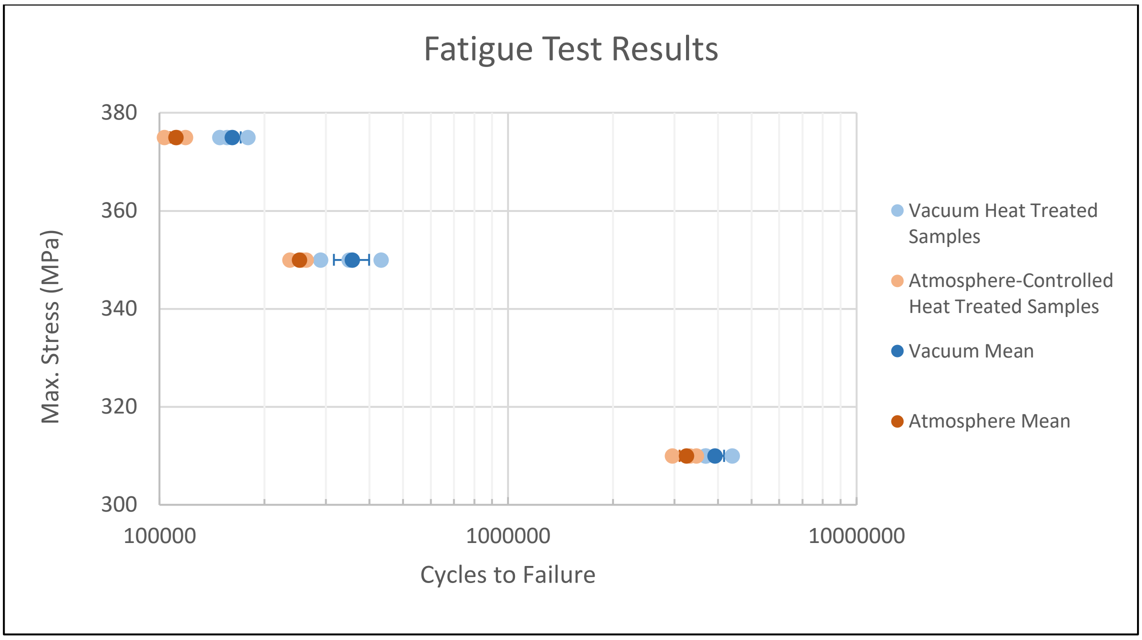

Figure 4. Fatigue test results (Yorulma testi sonuçları)

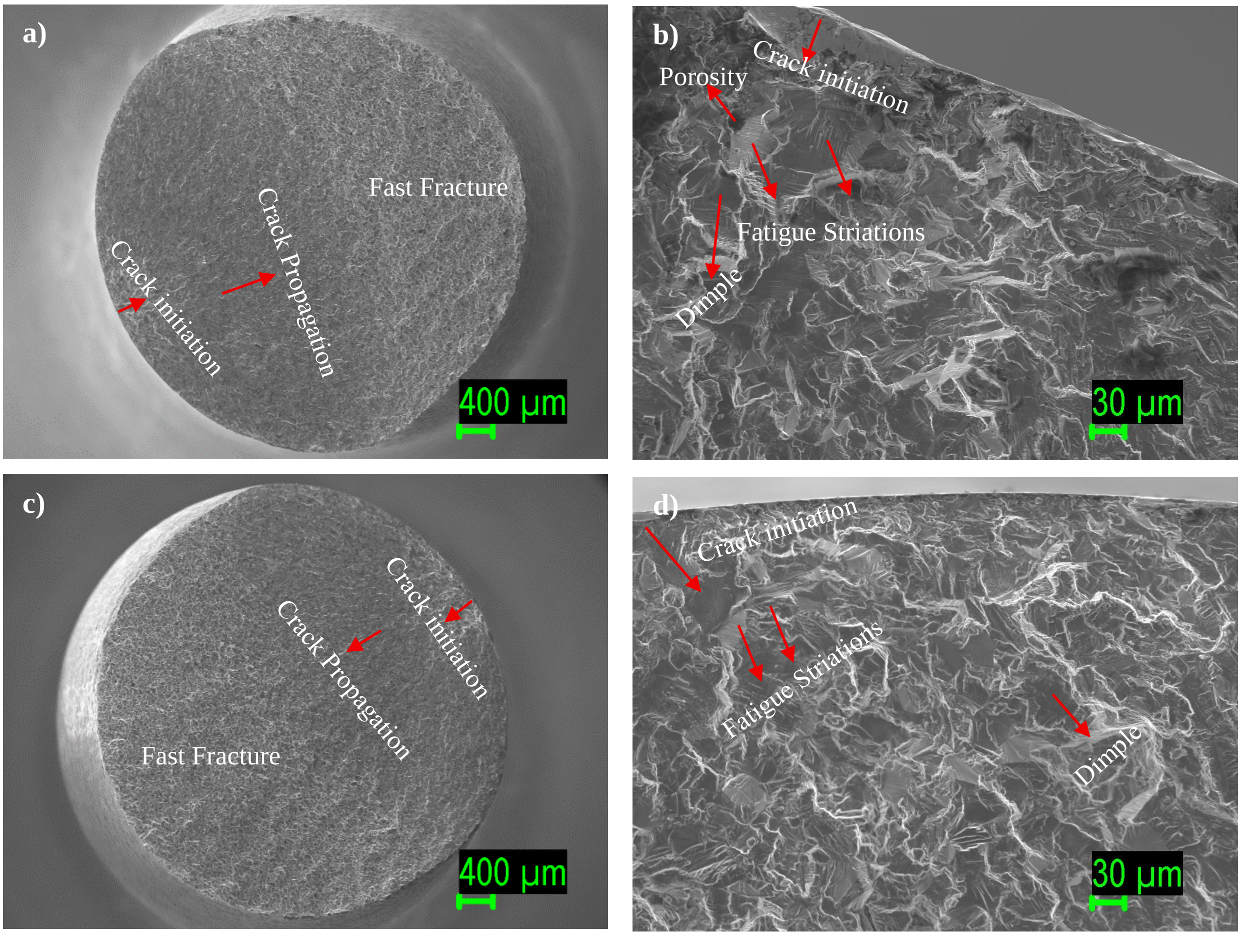

Figure 5. a) Atmosphere-controlled heat-treated sample fatigue macrography, b) Atmosphere-controlled heat-treated sample detailed crack initiation area, c) Vacuum heat-treated sample fatigue macrography, d) Vacuum heat-treated sample detailed crack initiation area (a) Atmosfer kontrollü ortamda ısıl işlem görmüş numunenin yorulma makrografisi, b) Atmosfer kontrollü ortamda ısıl işlem görmüş numunenin detaylı çatlak başlangıç alanı, c) Vakum ortamında ısıl işlem görmüş numune yorulma makrografisi, d) Vakum ısıl işlem görmüş numunenin detaylı çatlak başlangıç alanı)

As shown in Figure 5, failure mechanisms are the same in both vacuum and atmosphere-controlled heat-treated samples. Crack initiation, crack propagation and fast fracture areas were observed in macro view of the samples. When comparing detailed views of atmosphere-controlled heat treated and vacuum heat-treated samples, crack initiation, regions, fatigue striations can be observed. The main difference of the samples is that porosity is observed in atmosphere-controlled heat-treated specimen that may originate from impurities such as H and O of the sample [35]. The impurities may reduce fatigue life of atmosphere-controlled heat-treated specimens when compared to vacuum heat-treated samples.

Table 3. Analysis results of impurity elements (Safsızlık elementlerinin analiz sonuçları)

Impurity	Base Metal	Vacuum heat-Treated Samples	Atmosphere-controlled heat-treated samples
O (ppm)	440±5	461±5	488±5
H (ppm)	77±1	80±1	98±1

Hydrogen and oxygen impurities are the main reasons for discoloration after heat treatment. However, oxygen and hydrogen are permitted in material specifications to a certain limit. Oxygen is permitted up to 0.40 % and hydrogen is allowed up to 0.015 %. In addition, oxygen content is controlled due to enhancement of mechanical properties. On the other hand, hydrogen is associated with diminishing strength and fatigue life [36,37]. To comprehend the situation, base metal and heat-treated samples in different heating media are subjected to impurities testing by LECO ONH836 equipment. Tests were performed with five samples for each condition, and the average value was taken. The standard deviation is ±1 ppm for hydrogen content and ±5 ppm for oxygen content so they are negligible. The results are given in Table 3. As it is seen in Table 3, oxygen and hydrogen impurity content in vacuum heat-treated samples are very close to base metal that implied vacuum heating media prevented formation of oxygen and hydrogen considerably. On the other hand, hydrogen content increased in atmosphere-controlled heating media by nearly 20 ppm and oxygen 50 ppm. So, discoloration is linked to hydrogen and oxygen content increment. Reduction in mechanical properties are attributed to hydrogen content of atmosphere-controlled heat-treated samples.

3. CONCLUSIONS (SONUÇLAR)

CP Ti alloys were treated in atmosphere controlled and vacuum atmospheres. Discoloration has been observed in atmosphere-controlled heat-treated samples, on the other hand vacuum heat-treated samples showed no discoloration. Microstructure and mechanical properties in terms of tensile and fatigue have been examined. Atmosphere-controlled and vacuum heat-treated samples illustrated identical microstructures.

Tensile properties were compared after microstructure testing. The difference in tensile and yield strength values was about 7% on the other hand, elongation percentages were nearly similar. Vacuum heat-treated samples showed higher strength values than atmosphere-controlled specimens. The differences in strength values were linked to impurity content of the samples. Although oxygen content has some increment effects on strength, increment in hydrogen content decreased mechanical properties.

Fatigue testing was performed at stress levels of 310, 350, and 370 MPa that are below yield stress. fatigue tests were conducted. At all stress levels, the fatigue life was reduced. Fatigue life was reduced by almost 50% when stress levels are raised to 375 MPa. To understand the differences, failure mechanisms were examined by SEM. Fatigue striations and dimples were observed in both samples. The main difference was in atmosphere-controlled heat-treated specimens where porosity was also observed that might originate from increment in impurity contents. This was also proved by impurity analysis. Atmosphere-controlled heat-treated samples had nearly higher 20 ppm in hydrogen and 50 ppm in oxygen when compared to base metal. Mechanical properties decreased as the hydrogen content increased.

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DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission.

Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

AUTHORS' CONTRIBUTIONS (YAZARLARIN KATKILARI)

Erdem MERMER**:** He conducted the experiments, analyzed the results and performed the writing process.

Deneyleri yapmış, sonuçlarını analiz etmiş ve makalenin yazım işlemini gerçekleştirmiştir.

Nurdan EVCİMEN**:** She analyzed the results and performed the writing process.

Deney sonuçlarını analiz etmiş ve makalenin yazım işlemini gerçekleştirmiştir.

Hanifi ÇİNİCİ**:** He reviewed the article.

Makaleyi gözden geçirmiştir.

CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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