

# MECHANICAL PROPERTIES OF POWDER METALLURGY (Ti-6AI-4V) ALLOY MANUFACTURED WITH HOT ISOSTATIC PRESSING

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### MECHANICAL PROPERTIES OF POWDER METALLURGY (Ti-6Al-4V) ALLOY MANUFACTURED WITH HOT ISOSTATIC PRESSING

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> > KARABÜK December 2020

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### ABSTRACT

#### Ph.D. Thesis

# MECHANICAL PROPERTIES OF POWDER METALLURGY (Ti-6Al-4V) ALLOY MANUFACTURED WITH HOT ISOSTATIC PRESSING

**Mohamed Abdulla ELFGHI** 

Karabük University Institute of Graduate Programs The Department of Mechanical Engineering

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Titanium alloys are used widely in industries due to their high performance and low density in comparison with other iron-based alloys. Applications extend to aerospace and military applications in order to utilize their high resistance for corrosion. The mechanical properties and microstructure of titanium alloys are critical to understand for performance optimization, as well as their implications on strength, plasticity and fatigue. Ti-6Al-4V is an  $\alpha + \beta$  two phase alloy and considered one of the most commonly used titanium alloys for weight reduction and high-performance criteria. To avoid manufacturing defects, such as porosity and composition segregation, hot isostatic pressing is used to consolidate alloy powder. The HIP method is also used to facilitate the manufacturing of complex structures that cannot be made with forging and casting. The current research manufactures Ti-6Al-4V alloys using the HIP method and study the impact on heat treatment under different temperatures and sintering durations on the performance and microstructure of the alloy. Results show

changes in mechanical properties and microstructure with the increase of temperature and duration.

**Keywords** : Titanium alloy: Ti-6Al-4V; Hot isostatic pressing (HIP). Science Code : 91421

### ÖZET

#### **Doktora Tezi**

# SICAK İZOSTATİK PRES İLE ÜRETİLEN TOZ METALURJİSİ (Ti-6Al-4V) ALAŞIMININ MEKANİK ÖZELLİKLERİ

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Titanyum alaşımları, diğer demir bazlı alaşımlara kıyasla yüksek performansları ve düşük yoğunlukları nedeniyle endüstrilerde yaygın olarak kullanılmaktadır. Uygulamalar, yüksek korozyon direncini kullanmak için havacılık ve askeri uygulamalara kadar uzanır. Titanyum alaşımlarının mekanik özellikleri ve mikroyapısı, performans optimizasyonunun yanı sıra mukavemet, plastisite ve yorgunluk üzerindeki etkilerini anlamak için önemlidir. Ti-6Al-4V, a +  $\beta$  iki fazlı bir alaşımdır ve ağırlık azaltma ve yüksek performans kriterleri için en yaygın kullanılan titanyum alaşımlarından biri olarak kabul edilir. Gözeneklilik ve bileşim ayrımı gibi imalat kusurlarından kaçınmak için alaşım tozunu birleştirmek için sıcak izostatik presleme kullanılır. HIP yöntemi ayrıca dövme ve döküm ile yapılamayan karmaşık yapıların üretimini kolaylaştırmak için kullanılır. Mevcut araştırma, HIP yöntemini kullanarak Ti-6Al-4V alaşımları üretmekte ve farklı sıcaklıklarda ısıl işlem üzerindeki etkisini ve alaşımın performansı ve mikro yapısı üzerindeki sinterleme sürelerini incelemektedir. Sonuçlar sıcaklık ve sürenin artmasıyla birlikte mekanik özelliklerde ve mikroyapıda değişiklikler olduğunu göstermektedir.

Anahtar Kelimeler : Titanyum alaşımı: Ti-6Al-4V; Sıcak izostatik presleme (HIP).Bilim Kodu: 91421

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### SYMBOLS AND ABBREVIATIONS

### SYMBOLS

Ann	: Annealed
$d_{av}$	: Average impact diameter in Vickers test
FeTiO <sub>3</sub>	: Iron (II) titanate (ilmenite)
GPa	: Gigapascal
$H_{v}$	: Vickers hardness
Hz	: Hertz
MgCl <sub>2</sub>	: Magnesium dichloride
MPa	: Megapascal
°C	: Degree Celsius
°F	: Degree Fahrenheit
Р	: Loaded force in Vickers hardness
Pa	: Pascal
psi	: Pound per square inch
TiCl <sub>2</sub>	: Titanium dichloride
TiCl <sub>4</sub>	: Titanium tetrachloride
TiO <sub>2</sub>	: Titanium dioxide (rutile)
wt%	: Percentage weight or weight percentage
μCΤ	: X-ray microtomography

### ABBREVIATIONS

- ASTM : American Society for Testing and Materials
- BCC : Body Centred Cubic
- CFRP : Carbon Fibre Reinforced Polymer
- EDS : Energy Dispersive Spectroscopy
- FCC : Face Centred Cubic
- FESEM : Field Emission Scanning Electron Microscope
- HCP : Hexagonal Close Packed
- HIP : Hot Isostatic Pressing (Hipping)
- ICDD : International Centre for Diffraction Data
- LCF : Low Cycle Fatigue
- PM : Powder Metallurgy
- SEBM : Selective Electron Beam Melting
- SS : Stainless Steel
- STA : Solution Treated and Aged
- VAM : Vacuum Arc Melting
- VHN : Vickers Hardness Number
- XRD : X-Ray Diffraction
- XRF : X-Ray Fluorescence

### **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1. RESEARCH BACKGROUND**

The utilization of titanium for industrial purposes became popular in the past fifty years due to competitive properties in comparison with traditionally used metals, such as iron, steel and etc. [1]. Titanium alloys are commonly used in aerospace and military applications due to their low density and high resistance for corrosion, in addition to their high performance and strength. Despite being expensive and hard to source, the alloy became the engineering choice for experts due to its high performance under high temperatures and light weight [2].

The applications of titanium alloys are correlated to their mechanical properties. Therefore, it is important to understand the factors that affect these properties in order to optimize their performance. A research found that the mechanical properties of titanium alloys, such as creep, fatigue, plasticity and strength, are highly affected by their microstructure [3]. The small size of  $\alpha$  grains are correlated to the increase in ultimate tensile strength [4].

One of the most common titanium alloys is Grade 5 (Ti-6Al-4V), which is used extensively in engineering and industrial purposes. Due to its high mechanical performance criteria and facilitation of weight reduction, industries using this alloy included marine equipment, automotive and aerospace applications. Medical and pharmaceutical applications can also be found for Ti-6Al-4V due to its exceptional biocompatibility [5]. However, the alloy has difficult machinability because of its low thermal conductivity and high strength to density ratio [6].

Ti-6Al-4V is classified as an  $\alpha + \beta$  two phase alloy with wide usage range in mechanical applications. Due to the structure complexity of the manufactured components and the defects, such as composition segregation and porosity, forging and casting is not the most preferred manufacturing method [7]. Thus, hot isostatic pressing (HIP) is used for the consolidation of alloy powder in order to facilitate manufacturing complex components and avoid those defects [8]. The HIP method has also economic advantages as it maximizes the utilization of material and reduce the costs of defected waste [9].

Through further investigation and analysis, further advantages were found for the HIP method application on Ti-6Al-4V on the microstructure level [10]. There are several studies that attempted to optimize the heat and pressure conditions of the alloy in order to reach to an optimum performance. There are studies that solely tested the heat or pressure change implications on titanium alloys, while few studies investigated the change in both parameters [11].

Xu, et al. [12] investigated the heat and pressure conditions of the HIP method, as well as the cooling rate of the Ti-6Al-4V samples on their microstructure and mechanical properties. The authors found that temperatures ranging between 900 and 940 °C and a pressure of 100 MPa yield the most optimize tensile strength; with sample holding for 3 hours in room temperature.

Cai, et al. [13] used HIP method with high temperature and pressure conditions: 1200 °C and 120 MPa, while cooling in furnace for 3 hours. The microstructure analysis showed an increase on the needle structures of Ti-6Al-4V and an increase in the nano hardness and the wear resistance of the samples.

The current study manufactures Ti-6Al-4V alloys using the hot isostatic pressing and put the samples under very high temperatures in furnace with different exposure timings. The impacts on wear resistance, microstructure and mechanical properties are investigated and compared to results of previous research for verification and possible enhancements on the properties of the alloy.

### **1.2. RESEARCH SIGNIFICANCE**

Titanium and its alloys are considered as competitive metals due to their advantageous mechanical properties, high corrosion resistance, and lightweight in comparison with other alloys like steel. The many advantages of titanium and its alloys motivated their utilization in wide range of applications, including automotive, petrochemical products, aerospace, personal products, and biomedical products [14]. Titanium provides the option to save weight due to its lower density in comparison with other metal, such as steel. Moreover, titanium preserves its strength criteria at high temperatures, which is not possible with other light weight metals like aluminum. The density of titanium is 60% higher than aluminum. Nonetheless, the dominance in the selection criteria prevails to the resistance to high temperatures with an advantage of lower density, which makes aluminum the most preferred metal in aerospace [15].

The physical and mechanical properties of the final titanium product are significantly affected by the working and treatment factors, individually or combined. The most significant factors are heat treatment, fabrication methods, mechanical working methods of ingots to turn them into mill products, the process used for melting to make the ingot, and the types and amounts of alloyants and impurities that are contained within the ingot. The environment surrounding the processing of titanium and its alloys, and its conditions, needs to be highly controlled. Although the ability to use a narrow range of titanium alloys for a wide range of applications is advantageous, further variety of titanium and its alloys can be produced by altering the conditions of mechanical or thermal processing [16].

The Ti-6Al-4V alloy exists in cast and wrought forms and is used widely in the aerospace industry. Fabricating the components made of Ti-6Al-4V is performed through hot working and machining of the wrought form in conventional applications. The strain rate of deformation and the temperature significantly impact the final microstructure of the alloy, which affect its mechanical properties subsequently. During elevated temperatures, the deformation in the alloy determines the mechanical properties, while the alloy is known for its hard-plastic deformability when compared to aluminum and steel alloys. When using conventional methods, the manufacturing

of complex parts, such as porous hip joint disk, requires high labor force and machining process [17].

In the literature, several studies have addressed processing Ti-6Al-4V with hot isostatic pressing in order to enhance the properties of the material, including the elimination of porosity to increase strength and hardness, as well as avoid negative effects imposed by macro and micro segregation and crack propagation [18]. However, there were almost no studies that studied the influence of very high temperatures on the mechanical properties and the microstructure of Ti-6Al-4V after processing by hot isostatic pressing. Thus, the current research looks to perform the necessary experiments in order to contribute to the literature with original data and expose these effects.

### **1.3. AIM OF STUDY**

The aim of the research is to investigate the effect of very high heat exposure on Ti-6Al-4V alloys processed by hot isostatic pressing through comparing samples with different exposure timing. For the achievement of the main aim, several objectives are fulfilled throughout the course of the research:

- Study and understand the properties of titanium and its alloys and the developments in titanium extraction, manufacturing, and processing.
- Investigate the physical and mechanical properties of titanium alloys and the impact of the different factors on them.
- Compare the properties of titanium alloys with pure titanium and other metals and alloys based on their yield strength, tensile strength, elasticity, and hardness.
- Study the applications of titanium alloys and the competitive properties that influence the choice of engineers and designers to utilize them into different industries.
- Understand the influence of residual and additive elements on the performance criteria and microstructure of titanium alloys.

- Investigate and analyse the metallurgical properties of titanium alloys and Ti-6Al-4V in particular, including microstructural changes.
- Review the mechanical and microstructural properties of Ti-6Al-4V from existing literature for reference and comparison.
- Understand hot isostatic pressing processing and its impact on the performance criteria of the metal.
- Fabricate Ti-6Al-4V from its powder through hot isostatic pressing and expose it to very heat in preparation for the experiment.
- Perform different experiment on the produced samples, including XRF, SEM, EDS analyses, in addition to wear, hardness tests
- Obtain the results from the experiments and compare them with the findings of the literature for further understanding of the behaviour of TI-6Al-4V after exposure to very high temperatures.

### **1.4. STRUCTURE OF STUDY**

The study is mainly split into two main segments: theoretical and literature review, and experimental application. Five chapters are reported in the research in order to cover its parts:

- Introduction: a brief review of the research subject is carried out, and the significance of the research is presented for further development of the study. Subsequently, the main aim of the study, as well as the objective of each research phase are provided.
- Literature review: a theoretical research on the history of titanium and its alloys, their physical and mechanical properties and the manufacturing processes that are used to extract and produce titanium products. The metallurgical properties of titanium alloys are researched, and the effect of composition, working and treatment are discussed in detail. The literature is reviewed for the properties of Ti-6Al-4V and its performance in comparison with other titanium alloys, as well as other metals used in different industries. The process of hot isostatic pressing is studied in order to highlight its

advantages and influences on the performance of different material, as well as the objectives of using this type of processes.

- Methodology and experiment: the chapter provides the steps that were taken to prepare the samples for the experiments, in addition to the procedures and equipment that were used in the different tests.
- Results and discussion: the results of the experiments are provided, along with a comparison and discussion in alignment with the findings of previous literature.
- Conclusions: a summary of the research is carried out, the final results of the experiment are reviewed, and recommendations for future research are given for further development.

### **CHAPTER 2**

#### LITERATURE REVIEW

### **2.1. PROPERTIES OF TITANIUM**

### 2.1.1. Titanium and its Production

The ore of titanium is widely dispersed in earth and it is considered the fourth most abundant metal in the crust with 0.6% level after aluminum, iron, and magnesium, respectively. Titanium in its natural form is bonded to other elements and its ores are available mostly in highly igneous rocks. A metal content analysis showed that more than 97% of igneous rocks contain titanium with different proportions [19]. Figure 2.1 shows two of the most primary sources of titanium: rutile (TiO<sub>2</sub>) and ilmenite (FeTiO<sub>3</sub>), in addition to other secondary ores, such as titanite, perovskite, brookite and anatase, shown in Figure 2.2.



Figure 2.1. Primary titanium ores: rutile [20] and ilmenite [21].



Figure 2.2. Secondary titanium ores: titanite [22], perovskite [23], brookite [24], and anatase [25] – arranged from left to right and top to bottom, respectively.

Figure 2.3 provides a summary timeline of the discovery and development of titanium. The first discovery of titanium was by William Gregor, a pastor from England, in 1791, who obtained a black sand sample with magnetic properties from Cornwall in South West of the British island. During his analysis, the reverend observed an unidentifiable residue that he recorded as a new metal. In 1793, the German chemist Martin Heinrich Klaproth identified the oxide of this new metal and named it after the Titans from the Greek mythology as Titanium. Although it was discovered in the eighteenth century and was characterized with its high strength, the full knowledge of titanium was not obtained until its pure form was produced in the early twentieth century [26].



Figure 2.3. Timeline of titanium discovery and development (Developed by researcher).

In 1910, an engineer working for Rensselaer Polytechnic Institute, named Matthew Hunter, was working with General Electric Company on developing incandescent lamps. The requirement of finding metals with high melting points drove him to suggest titanium for the filaments of the lamps. In light of this project, Hunter developed a feasible method to extract titanium from its ores, Figure 2.4. The extraction method started by blending TiO<sub>2</sub> with chlorine and coke, which is a fuel of high carbon content, then applying heat to titanium dioxide. The product of the process gives titanium tetrachloride (TiCl<sub>4</sub>). Sodium is then used to reduce this product into an alloying agent with white pigment that revolutionized the painting and steel industry [27].



Figure 2.4. Hunter's process for titanium extraction [28].

In 1938, William Kroll developed the Kroll's process (Figure 2.5), which changed the gent used for reduction to magnesium. The basis of Kroll's process remained similar to the one developed by Hunt. Other minor differences were added to the process by Kroll, including the increase in the retort size and the reaction location of vacuum distillation, which takes place with the reduction procedure [27].



Figure 2.5. Kroll's process for titanium extraction and production [29].

Despite the possibility for large production of titanium, the private labs and producers only created two tons in the next ten years. It was not until the government in the United States funded several titanium related projects in 1948, including spacecraft, missiles, and aircrafts, which had driven the attention towards its scientific, engineering, and financial advantages. In the next five years, the production of titanium increased to a million kilograms. Titanium remained exclusive for military applications, until it was introduced commercially in the 1960s. The aerospace industry was the main beneficiary from the spreading of titanium production and manufacturing, as it reached to use up around 75% of total produced titanium quantities in the United States. The biomedical industry utilized titanium since the 1960s. Since then, several medical break throughs have been observed, especially in implants and medical equipment. Several other industries have benefited also from titanium, such as cosmetics, paint, and food production [30]. The current production of titanium is led by several countries, including China, Japan, Russia, United States, Ukraine, and Kazakhstan [31], as shown in Figure 2.6.



Figure 2.6. Titanium production in leading countries between 2003 and 2010 [31].

### 2.1.2. Physical Properties of Titanium

In the periodic table, titanium is the elements number 22, and it is given the symbol "Ti". Titanium belongs to the fourth group of the table and it is one of transition metals. The atomic weight is 47.867 gram per mole. The acidic-alkaline properties of titanium have equal strengths and the metal has a solid physical state at the room temperature. The electron configuration of the metal is expressed as  $[Ar]3d^24s^2$ . Titanium has three oxidation states: +2, +3 and +4 [32].

Titanium has a white silvery color. The density of pure titanium is approximately 4.5  $g/cm^3$  at the room temperature, which is compared to half the density of iron. Thus, its light weight makes it a competitive metal in engineering and design. It has a melting temperature point of 1660 °C and a boiling point of 3287 °C. The metal is naturally ductile, with low thermal and electric conductivities. The magnetic property of titanium is described as paramagnetic, which means that it has low attraction to magnets [33]. The molecular crystalline structure of titanium depends on the alloying

elements that are added with it. Titanium dioxide  $(TiO_2)$  is one of the most common impure forms of titanium through its ore: rutile. Figure 2.7 shows a ball and stick representation of the structure of the titanium dioxide and the bonding between the titanium and oxygen atoms [34].



Figure 2.7. Structure of titanium dioxide [34].

Figure 2.8 shows the hatched and diamond structures of Ti-6Al-4V alloy, which is the most utilized titanium alloy in several industries. These structures can be manufactured in the cellular level using selective electron beam melting (SEBM). The images are taken through SEM micrograph and using  $\mu$ CT measurement. The structures have different porosity levels and pore sizes, which is a controllable and useful feature in manufacturing biomedical nanocomponents that are desired to be compatible with bone tissues. The hatched structure was found to exhibit higher strength characteristics in comparison to the diamond structure [35].



Figure 2.8. Diamond and hatched structures of a titanium alloy (Ti-6Al-4V) nanocomponent manufactured by SEBM for biomedical application [35].

#### **2.1.3. Mechanical Properties of Titanium**

The tensile strength of pure unalloyed titanium depends mainly on iron and oxygen contents and ranges between 275 MPa for iodide reduction process produced metal with high purity and 590 MPa when produced from high hardness sponge titanium [36]. For the purpose of increasing its strength, pure titanium is alloyed with other metals, such as Fe, V, Al, Sn, and Cr. Titanium alloys have higher tensile strengths ranging between 600 MPa and 1250 MPa [37]. Figure 2.9 shows a comparison of the tensile strength ranges between pure titanium and titanium alloys. Examples of these ranges are Ti-3Al-2.5V and Ti-15Mo-5Zr-3Al, respectively. Titanium and its alloys are competitive in maintaining their strength characteristics at higher temperatures

[38]. Figure 2.10 compares between titanium alloys, titanium-aluminum alloys and other alloys based on their specific strength according to temperature changes.



Figure 2.9. Comparison between yield strength of pure and alloyed titanium [30].



Figure 2.10. Change of specific strength of metal alloys and titanium alloys with temperature [36].

Arc-melted pure titanium has good ductility, which is determined by its interstitial content, that ranges between 20% and 40% in elongation and 45% to 65% in area reduction. The elongation and area reduction in iodide processed titanium can reach up to 55% and 80%, respectively. Despite the increase in strength with alloying titanium, ductility is reduced depending on its composition and content [39]. Figure 2.11 shows the tensile strength, yield strength and elongation properties of pure and alloyed titanium.



Figure 2.11. Tensile strength and elongation of pure and alloyed titanium [40].

Elasticity modulus of pure titanium is less than steel,  $15x10^6$  psi and  $29x10^6$  psi respectively; however, it can be increased through adding alloyants for up to  $18x10^6$  psi. Titanium and its alloys are competitive when compared to other metals, such as aluminum and magnesium, which have their elasticity modulus as  $10.4x10^6$  psi and  $6.4x10^6$  psi, respectively [41]. Figure 2.13 compares between elasticity modulus of titanium and other alloys.



Figure 2.12. Elasticity modulus of titanium and other alloys [30].

The hardness of titanium and alloyed steel are close to each other, while the former exceeds the hardness value of aluminum. Alloying and heat treatment can increase the hardness of pure titanium from 90 VHN for iodide pure titanium for up to 500 VHN. Figure 2.13 shows a comparison between different alloys and metals and one of most common titanium alloys, i.e. Ti-6Al-4V [42].



Figure 2.13. Vickers hardness for Ti-6Al-4V and other metals [43].

The impact strength of titanium depends on its purity, alloyant contents and treatments performed on it. High strength titanium alloys have a range of impact resistance between 1 to 2 foot-pounds, while highly pure titanium (iodide process) can reach up to 100 foot-pounds [2].

### 2.2. TITANIUM ALLOYS AND THEIR APPLICATIONS

There are several fields that take advantage of the properties of titanium alloys, such as automotive, petrochemical products, aerospace, personal products, and biomedical products. The demand on the titanium alloys is attributed to their competitive corrosion resistance, in addition to their high strength to weight ratio among other excellent properties. In aerospace, titanium is crucial due to its performance at high temperatures and its ability to maintain high specific strength. As shown in Figure 2.14 with comparison to other metallic materials, a titanium alloy (Ti-6Al-4V) has higher strength to weight ratio at operating temperatures reaching to 400-550 °C. There are several benefits from using titanium alloys in many industries [14]:

- The weight of pure titanium is 40% less of its counterpart metals, such as super alloy composites with nickel and aluminum.
- Titanium alloys is compared to martensitic stainless steel, and it is higher than austenitic and ferritic stainless steel.

- The operating temperature range of titanium alloys is high at 400-550 °C, while the strength of titanium aluminates increases at higher temperatures.
- The expansion coefficients of titanium alloys are lower than steel and is less than half of the expansion coefficient of aluminum.
- Titanium alloys are effective substitutes for aluminum at temperatures exceeding 130 °C.

The only type of material that can exceed titanium in specific strength is composites; however, in applications that require operations at high temperatures exceeding 300 °C, this advantage is diminished and titanium alloys remain the best choice for applications with higher temperatures.



Figure 2.14. Comparison between a titanium alloy with other materials based on their specific strength to temperature ratios [44].

The ability to save weight in design components is crucial for the aerospace industry. Therefore, titanium provides this option due to its lower density in comparison with other metal, such as steel. Moreover, titanium preserves its strength criteria at high temperatures, which is not possible with other light weight metals like aluminum. The density of titanium is 60% higher than aluminum. Nonetheless, the dominance in the selection criteria prevails to the resistance to high temperatures with an advantage of lower density, which makes aluminum the most preferred metal in aerospace [15].
Figure 2.15 shows the different components that are made of titanium alloys in the airplane engine, which is the most part that utilizes the properties of titanium in the design.



Figure 2.15. Airplane engine components made from titanium alloys – an example from a V2500-A5 model [45].

The biomedical field is the second most consumer for titanium alloys due to its good biocompatibility, high corrosion resistance, and excellent mechanical properties. These properties are essential for biomedical applications, as titanium components are placed near bones and tissues as substitutes to original body parts, as shown in Figure 2.16. Titanium alloys can be used as screws for bone fixation devices, artificial vascular stents, hip joints, knees and teeth [46].



Figure 2.16. Application of titanium alloys in the biomedical; (a) dental implant [47], (b) hip joint, (c) vascular stent [48].

#### 2.3. METALLURGICAL PROPERTIES OF TITANIUM ALLOYS

As shown in Table 2.1, titanium has competitive properties in comparison with the most used metals, such as iron, nickel, and aluminum. The melting temperature is 250% higher than aluminum, 15% higher than nickel and 8.6% higher than iron. The yield stress is comparable to those of iron and nickel, while its density is 43% and 49.4% lower than those metals, respectively. It has a high corrosion resistance which increases its durability in several applications.

Table 2.1. A comparison between titanium and other metallic materials based on their mechanical and metallurgical properties [49].

Property	Ti	Fe	Ni	Al
Melting temperature (°C)	1670	1538	1455	660
Allotropic transformation (°C)	882	912	-	-
Crystal structure	$\begin{array}{c} \text{HCP} \rightarrow \\ \text{BCC} \end{array}$	$\begin{array}{c} BCC \rightarrow \\ FCC \end{array}$	FCC	FCC
Room temperature E (GPa)	115	215	200	72
Yield Stress (MPa)	1000	1000	1000	500
Density (g/cm <sup>3</sup> )	4.5	7.9	8.9	2.7
Comparative Corrosion Resistance	Very high	Low	Medium	High
Comparative Reactivity with O <sub>2</sub>	Very high	Low	Low	High
Comparative Price of Metal	Very high	Low	High	Medium

Due to the costly production method used for titanium, i.e. Kroll's process, and its high reactivity with oxygen, the price of titanium and its alloys is relatively higher. The high oxygen reactivity of titanium requires using inert atmosphere or vacuum during production to eliminate the risk of oxygen contamination. The specific strength of titanium and its alloys are also higher when compared with the most used industrial metal. When exposed to atmospheric air, titanium forms a thin oxide layer immediately

at all surfaces, which provides the high resistance to corrosion and makes the metal desirable for many applications [17].

At room temperature, pure titanium exists in a hexagonal close packed (HCP)  $\alpha$ -phase and goes through allotropic phase transformation when its temperature exceeds 882 °C to a body centered cubic (BCC)  $\beta$ -phase. Figure 2.17 shows a schematic illustration, with lattice constants, of the transformation of the titanium cell from the HCP- $\alpha$  phase to the BCC- $\beta$  phase. The lattice parameters are a = 0.295 nm and c = 0.468 nm at the room temperature at the HCP  $\alpha$ -phase. The lattice parameters are a = 0.0332 nm at 900 °C at the BCC  $\beta$ -phase. Three slip systems exist for the  $\alpha$ -phase, while twelve slip systems exist for the  $\beta$ -phase, which makes contributes to the increase of ductility, softness, and plastic deformability for the  $\beta$ -phase. These properties are also results of the higher atomic density of the  $\beta$ -phase and the easiness of sliding of dislocations on the slip systems [30].



Figure 2.17. Titanium allotropic phase transformation from  $\alpha$ -phase (a) to  $\beta$ -phase (b) [30].

The alloyants that are used with pure titanium contribute into changing its allotropic transformation temperature, which is 882 °C, while the magnitude of the change is dependent on the amount of interstitial and substitutional alloyants. Figure 2.18 shows the impact of several alloyants on the change in titanium phase diagram and the transition from the  $\alpha$ -phase to the  $\beta$ -phase.



Figure 2.18. The change in titanium phase diagram with different alloyants [30].

Based on its high solubility in both the  $\alpha$ -phase and the  $\beta$ -phase, Aluminum is the most frequent substitutional alloyant used with titanium. The ability of other alloyants like Ga, Ge and B to raise the  $\beta$  transition temperature makes them part of the group of alternatives used, besides aluminum; however, their rarity in nature and lower solubility in titanium are less competitive in comparison to aluminum. Oxygen, carbon, and nitrogen are interstitial elements that are used to stabilize the  $\alpha$ -phase. Despite the high stabilizing impact of oxygen, the increase in its content reduces the ductility and increases embrittlement significantly [50]. Hydrogen is the only interstitial element that is able to reduce the transition temperature from the  $\alpha$ -phase to the  $\beta$ -phase, which makes it has a  $\beta$ -phase stabilization impact. Other  $\beta$ -phase stabilization substitutional elements are used, such as Si, Cr, Mo, Nb, and V. It is possible to stabilize the  $\beta$ -phase at the room temperature with the usage of the adequate amounts of the latter elements [51]. Other alloyants, such as Sn, Hf and Zr, have no stabilization impact. While Sn has no effect on the transition temperature from the  $\alpha$ phase to the  $\beta$ -phase, it is used as an  $\alpha$ -phase stabilization element in several applications with the presence of Al in the titanium alloy structure due to its substitution in the Ti3Al phase with hexagonal order [52].

 $\alpha$  and near- $\alpha$  alloys, as well as alloys with alloyants used for  $\beta$ -phase stabilization, are stable at the room temperature. The presence of  $\alpha$ -phase is dominant in the microstructure of ( $\alpha + \beta$ ) alloys, with a lesser amount of the  $\beta$ -phase. A titanium alloys if considered as a  $\beta$ -Ti alloy, if its quenching for  $\beta$ -phase field retains 100%  $\beta$ -phase [53]. Titanium alloys are classified into  $\alpha$  and near- $\alpha$  alloys,  $\alpha+\beta$  alloys, and  $\beta$  and near- $\beta$  alloys. The different properties of these alloys according to their classification and their type and amount of alloyants is shown in Figure 2.19.

α alloys	Unalloyed titanium Ti-5Al-2.5Sn	-Higher density -Increasing heat treatment	1
Near-α	Ti-8Al-1Mo-1V Ti-6Al-2Sn-4Zr-2Mo	response -Higher short time strength -Increasing strain rate sensitivity	
$\alpha + \beta$ alloys	Ti-6Al-4V Ti-6Al-2Sn-6V	-Improved fabricability	
Near-B	Ti-6Al-2Sn-4Zr-6Mo Ti-3Al-10V-2Fe		-Higher creep strength
$\beta$ alloys	Ti-13V-11Cr-3Al Ti-8Mo-8V-2Fe-3Al	<b>↓</b>	weldability

Figure 2.19. Properties and classification of titanium alloys [54].

#### 2.3.1. α and near α Alloys

Titanium alloys made with  $\alpha$ -phase stabilization elements, as well as commercially pure titanium are classified under this type. A titanium alloy is classified as a near- $\alpha$ alloys, if it has 100% HCP at low temperatures. Interstitial elements, like nitrogen, carbon and oxygen, and substitutional alloyants, like aluminum and tin (Sn), are soluble in titanium HCP  $\alpha$ -phase [17]. Several  $\alpha$ -phase titanium alloys are used in applications, especially in gas turbines where high creep resistance is essential, such as Ti-6Al-2Sn-4Zr-2Mo, Ti-8Al-1Mo-1V, Ti-5Al-2.5Sn, and Ti-3Al-2.5V. Heat treatment does not affect these alloys, which allows them to operate at high temperatures better than  $\alpha + \beta$  alloys, like Ti-6Al-4V.  $\alpha$ -phase titanium alloys require strict treatment with solution annealing close to the 100%  $\beta$ -phase field in order to be used. In comparison with other titanium alloys, the  $\alpha$ -phase titanium alloys have acceptable weldability and competitive corrosion resistance [55]. Creep resistance is improved for some  $\alpha$ -phase titanium alloys with Si and  $\beta$ -phase stabilization elements, like in the case of Ti-0.3Mo-0.8Ni. Dissolving aluminum in the  $\alpha$ -phase titanium alloys enhances their mechanical properties significantly through forming hexagonal Ti3Al phase as an effective strengthening mechanism [56].

#### 2.3.2. $\alpha + \beta$ Alloys

Titanium alloys that are classified under  $\alpha + \beta$  alloys contain  $\alpha$ -phase and  $\beta$ -phase stabilization elements, where aluminum is added as an  $\alpha$ -phase stabilization element with one or more  $\beta$ -phase stabilization elements, such as Mo and V. One of the most performing alloys in this category is the Ti-6Al-4V, which is formed primarily from aluminum as an  $\alpha$ -phase stabilization element and vanadium as an  $\beta$ -phase stabilization element and vanadium as an  $\beta$ -phase stabilization element. The combination between the  $\alpha$ -phase and the  $\beta$ -phase stabilization allows for a combination of high strength and enhanced creep resistance provided by both phases, respectively [14].

The capability of heat treatment for the  $\alpha + \beta$  alloys increases proportionally with the content of  $\beta$ -phase stabilization elements in it, while heat treatment is mostly performed for these alloys through solution treatment below the temperature of  $\beta$  transition. The strength, hardenability, and plastic deformability of  $\alpha + \beta$  alloys highly depend on the type and amount of alloyants used in them. Figure 2.20 shows the properties of the  $\alpha + \beta$  alloys, which are the center of many research and applications due to their combined nature and advantages. Several alloys fall under this classification, including Ti-6Al-4V, Ti-8Mn, Ti-3Al-2.5V, Ti-6Al-7Nb, Ti-7Al-4Mo, Ti-6Al-6V-2Sn, and Ti-6Al-2Sn-4Zr-6Mo [54].



Figure 2.20. Characterization of classification of titanium alloys [14].

## 2.3.3. β Alloys

When a titanium alloy has the ability to retain all its  $\beta$ -phase field at quenching without turning to martensite, it is considered as a  $\beta$ -alloy. Following heat treatment,  $\beta$  alloys with sufficient  $\beta$  stabilization elements have the ability to have high strength reaching to 1400 MPa. The mechanical properties of the  $\beta$  alloys are wide in range due to their readiness for heat treatment, while solution treatment is performed at temperatures exceeding the  $\beta$  transition point [14]. When compared to other titanium alloys,  $\beta$  alloys have much higher strength, good formability caused by the BCC  $\beta$ -phase and causing easier cold rolling. Nevertheless, it is not possible to weld the  $\beta$  alloys, in addition to their limited creep resistance, when compared to the  $\alpha$  alloys. Using heating at elevated temperatures or cold working can transform  $\beta$  alloys towards the  $\alpha$ -phase due to being metastable at the room temperature. Several alloys fall under this classification, including Ti-10V-2Fe-3Al, Ti-3Al-8V-6Cr-4Mo-4Zr, Ti-8Mo-8V-2Fe-3Al, Ti-15V-3Cr-3Al-3Sn, Ti-15Mo-2.7Nb-3Al-0.2Si [54].

# 2.4. MANUFACTURING AND PROCESSING OF TITANIUM ALLOYS

There are several steps that take place in order to transform row titanium ore to pure metal or alloy. Figure 2.21 shows the steps of processing and manufacturing, while Figure 2.22 shows the processing into final products. The steps taken to transform row titanium into functional metal or alloy are as follows [16]:

- Production of a porous type of titanium (sponge) through reducing titanium ore.
- Ingot is formed by melting sponge with a master alloy.
- General mill product types are produced from ingots through primary fabrication.
- Finished metal shapes are produced from mill products in the secondary fabrication.



Figure 2.21. Schematic of titanium production (primary fabrication) [16].



Figure 2.22. Schematic of titanium alloys processing (secondary fabrication) [16].

The composition of the ingot is highly dependent on the specifications of the titanium sponge used in its production. Therefore, extremely strict control must be carried out on the complex titanium oxynitride particles, titanium nitrides, and brittle, hard and refractory titanium in order to keep them at the zero level. The oxynitride particles can be crack origination sites in final metal produced if they are retained in following melting processes. Iron, Silicon, nitrogen, carbon, and oxygen are the most common residual elements that are found in titanium sponge that need to be kept at the lowest level. Figure 2.23 illustrates graphically the effect of some residual elements on lowering the ductility and increasing the strength of the final titanium product [16].



Figure 2.23. Impact of residual elements on unalloyed titanium ductility and strength [16].

As shown from Figures 2.22 and 2.23, rutile titanium ore is chlorinated (TiCh), followed by adding sodium or magnesium metals to reduce the resulting TiCU, in order to manufacture titanium sponge. NaCl is removed from the sodium-reduced sponge through leaching it with acid, while MgCF is removed from magnesium-reduced sponge through leaching it either with vacuum distillation or sweeping with inert gas. High ingot quality is assured in current titanium production methods through the use of advanced melting techniques that works on removing volatile substances from titanium sponge [57].

The main technique used for producing titanium alloy ingots is vacuum arc melting (VAM), which performs refinement of ingot diameter sizes by melting and re-melting that range originally between 70 mm and 1.5 m. VAM uses either argon's low partial pressure or direct current arc in vacuum with a consumable electrode for the melting process, while copper crucibles that are water-cooled are used to solidify the molten metal. A few factors affect the rate of melting and solidification, including the heat transfer properties in the interface between the ingot and the crucible, the

thermophysical properties of ingot, and the melting power derived from pressure, current or voltage [58]. The VAM technique eliminates several manufacturing issues that affect the quality of titanium ingots, including macro-segregation, high vapor pressures from unwanted trace elements, and dissolved gases such as nitrogen and hydrogen. The cooling rate during solidification dominantly determines the grain structure, where homogenous as-cast microstructure and fine grain structure are the results of higher cooling rates. VAM melting produces titanium ingots with three zones defining the as-cast macrostructure of the metal:

- During rapid cooling, the liquid metal forms fine solid equiaxed grains at the surface region, which is the closest to the bottom and wall of the water-cooled copper crucible.
- Large columnar grains, that form the inter-middle region with the size of 1000s of microns, which run in parallel to the temperature gradient and grow into the bulk from the outer rim.
- Equiaxed grains are formed at the axis of the ingot during solidification, which results into flecks: a type of macro-segregation observed especially in titanium alloys with β stabilization elements, such as copper and chromium, which has unity coefficients that are significantly higher than partitioning coefficients. During solidification, β stabilization elements partition to a liquid phase, which lowers melting point and cause segregation at the axis of ingot. At heat treatment below nominal β transition temperature of the titanium alloy or during its working, β flecks made of single β-phase material are formed having lower β transition temperature. It is difficult to eliminate chemical inhomogeneities occurring in thermomechanical processing due to its large scale. Nonetheless, it is possible to control macro-segregation resulting from forming β flecks through increasing the rate of solidification, while taking into consideration that an unbalanced increase can cause compositional variation at the solid state of the ingot caused by variation in chemical composition leading to micro-segregation in the molten metal [59].

Primary fabrication commences after the formation of ingot, where inhomogeneous and course is preheated and forged at 100 °C to 150 °C above  $\beta$  transition temperature

in a single  $\beta$ -phase field in order to break it. Table 2.2 shows the near- $\alpha$  and  $\alpha + \beta$  titanium alloys with their optimum hot working temperatures. The most common hot working machines are hydraulic presses, which are selected based on their moderate deformation rates. In conventional titanium alloys, there are two types of breakdown forging: upsetting and cogging, which are distinguished based the working of the forging dies. In cogging, the flat dies press on the sides of the round ingot along its length. In upsetting, the pressing is performed along the axis of the ingot. The heat radiation from ingot to its surroundings and the contact between the ingot and the cooler forging dies form the two sources of heat loss during the breaking down of the ingot. During forging, the heat at the center of the ingot is subject to increase caused by the effect releasing deformation energy, while temperatures at the surfaces are subject to higher heat losses. The temperature of the ingot is maintained above the  $\beta$  transition temperature in order to be able to perform adequate hot work for the heat treatment [59].

	β-transus	Ingot	Secondary	
Alloy	temperature	breakdown	hot working	
	(°C)	(°C)	(°C)	
Ti-5Al-2.5Sn	1040	1120-1175	900-1010	
Ti-8Al-lMo-lV	1040	1120-1175	930-1010	
Ti-6A1-4V	995	1095-1150	860-980	
Ti-6Al-2Sn-4Zr-2Mo	990	1095-1150	920-975	
Ti-6Al-2Sn-2Zr-2Mo-2Cr	980	1070-1130	870-955	
Ti-6Al-6V-2Sn	945	1035-1095	845-915	
Ti-6Al-2Sn-4Zr-6Mo	940	1030-1090	850-910	
Ti-4.5Al-5Mo-l.5Cr	925	1015-1070	850-910	
Ti-17 (Ti-5Al-2Sn-2Zr-4Cr-4Mo)	885	990-1050	800-865	

Table 2.2. Recommended hot-working temperatures and  $\beta$  transition temperatures for  $\alpha + \beta$  and near- $\alpha$  titanium alloys [59].

The amount of primary or globular  $\alpha$  and transformed  $\beta$  throughout the ingot section microstructure can experience variations caused by the variation in temperature during

breakdown processing. Therefore, heat treatment is applied with a temperature varying between 50 °C and 75 °C above  $\beta$  transition temperature after breakdown forging in order to minimize this microstructural variation. It is essential to keep the heating time to a minimum during this process to avoid excess growth of grain, while its main objective is to simulate recrystallization to be completed with a grain size should have an order of 500 urn. Widmanstatten transformation occurs during the cooling that follows heat treatment as  $\alpha$ -phase is formed within the boundaries and platelet of  $\beta$ grain. A high cooling rate is recommended to minimize the thickness of the harmful grain boundary  $\alpha$ -phase that is damaging to consequent sub-transition temperature hot workability. The high cooling rate also affects the prior  $\beta$  grains through producing thinner platelet structure [59].

The physical and mechanical properties of the final titanium product are significantly affected by the abovementioned factors, individually or combined. The most significant factors are heat treatment, fabrication methods, mechanical working methods of ingots to turn them into mill products, the process used for melting to make the ingot, and the types and amounts of alloyants and impurities that are contained within the ingot. The environment surrounding the processing of titanium and its alloys, and its conditions, needs to be highly controlled. Although the ability to use a narrow range of titanium alloys for a wide range of applications is advantageous, further variety of titanium and its alloys can be produced by altering the conditions of mechanical or thermal processing [16].

#### 2.5. Ti-6Al-4V ALLOY

The Ti-6Al-4V alloy is formed with alloyant content of  $\alpha$  stabilization element (Al; 6% wt.) and  $\beta$  stabilization element (V; 4% wt.), which makes it an  $\alpha + \beta$  alloy. At room temperature, the HCP  $\alpha$ -phase (90% wt.) is stable, while the BCC  $\beta$ -phase (10% wt.) is metastable when slowly cooled down from the  $\beta$ -phase region high temperature. Variations in the microstructure and the amount of  $\alpha$ -phase and  $\beta$ -phase occur based on the heat treatment type and rate of cooling, which can also include Widmanstatten structures, platelets, martensitic structures, grain boundary allotriomorph  $\alpha$ , and globular or primary  $\alpha$  [4].

The change in alloying elements within Ti-6Al-4V affects its  $\beta$  transition temperature, while 995 ± 20 °C is considered as the required temperature to transform the  $\alpha$ -phase and the  $\beta$ -phase into a 100%  $\beta$  system [60]. The  $\beta$ -phase transforms into globular  $\alpha$ -phase when the alloy is cooled at a very slow rate from  $\beta$  transition temperature. If the cooling rate is increased, Figure 2.24, a casting Widmanstatten microstructure occurs due to the emerging of acicular  $\alpha$  plates into the boundaries of  $\beta$  grain [60].



Figure 2.24. Casting Widmanstatten structure with  $\alpha$ -phase in light color and  $\beta$ -phase is dark color [61].

The cooling system used for the titanium alloy determines the length and width of the  $\alpha$  plates. Therefore, the increase in the cooling rate enhances the nucleation rate into primary  $\beta$  grains from the  $\alpha$  plates [60]. Additionally, the transformation to  $\alpha'$ -phase or HCP martensite from the  $\beta$ -phase through the martensite-start phase is observed in quenching. Orthorhombic martensite or  $\alpha'$ -phase, and hexagonal martensite or  $\alpha'$ -phase, are the two martensite forms that exist in titanium, which their amount and ratio depend mainly on the amount of  $\beta$  stabilization element in the titanium alloy [62]. A  $\geq 10\%$ wt. vanadium, or with the addition of hydrogen, condition with both martensite types leads to the transformation of 100%  $\beta$ -phase field to a martensite of the type  $\alpha''$ -phase following carrying out quenching [63]. Figure 2.25 shows quenching at different temperatures of Ti-6Al-4V with the phases that form the alloy in various conditions.



Figure 2.25. Phase constituents of Ti-6Al-4V alloy upon quenching [63].

Lutjering [4] examined Ti-6Al-4V alloy for the relationship between its microstructure and mechanical properties, where the author found that the mechanical properties of the alloy are highly influenced by the colony size of  $\alpha$  plates. Mechanical properties of Ti-6Al-4V, including ductility, crack propagation resistance, yield strength, and low cycle fatigue strength (LCF), are significantly improved with the decrease of  $\alpha$  colony size, which also reduce macro crack propagation resistance and fracture toughness. The results of the study highlight the criticality of understanding the microstructure of Ti-6Al-4V in order to determine its mechanical properties for different applications. The main determinants of the size of  $\alpha$  colony are the fabrication and heat treatment processes, which affect the cooling rate from the  $\beta$  field, and subsequently determines the microstructure of the alloy [4].

The Ti-6Al-4V alloy exists in cast and wrought forms and is used widely in the aerospace industry. Fabricating the components made of Ti-6Al-4V is performed through hot working and machining of the wrought form in conventional applications. The strain rate of deformation and the temperature significantly impact the final microstructure of the alloy, which affect its mechanical properties subsequently. During elevated temperatures, the deformation in the alloy determines the mechanical

properties, while the alloy is known for its hard-plastic deformability when compared to aluminum and steel alloys. When using conventional methods, the manufacturing of complex parts, such as porous hip joint disk, requires high labor force and machining process [17].

# 2.6. THERMOMECHANICAL TREATMENT, MICROSTRUCTURE AND MECHANICAL PROPERTIES

The heat treatment and processing history of the titanium alloys highly influence their microstructure and mechanical properties [64]. As shown in Figure 2.26, a duplex microstructure is developed with primary  $\alpha$  and transformed  $\beta$  when an  $\alpha + \beta$  alloy, like Ti-6Al-4V, is hot worked and heat treated below the  $\beta$  transition temperature from the  $\alpha + \beta$  field. The morphology of the primary  $\alpha$  plates, which is developed by nucleation and grows throughout the  $\alpha + \beta$  working operations, becomes globular and equiaxed due to recrystallization during heavy working and elongated during light working. Moreover, the globular plates of the primary  $\alpha$  is improved with a very low cooling rate from elevated temperatures, in addition to recrystallization and deformation processes, as shown in Figure 2.27 [65].



Figure 2.26. Bimodal microstructure of Ti-6Al-4V with transformed  $\beta$  surrounding primary  $\alpha$  shown through optical micrograph [64].



Figure 2.27. More than 90% primary α forming on the top surface of Ti-6Al-4V after a heating-cooling cycle [64].

Regions with  $\beta$ -phase change to transformed  $\beta$  due to temperature of hot working, annealing temperature, or solution treatment temperature. Based on the composition and cooling rate, the morphology of the transformed  $\beta$  regions may vary with different combination of these two factors. Slow cooling rates transform  $\beta$ -phase into  $\alpha$  plates with Widmanstatten structure through growth and nucleation, while fast cooling rates transform it into martensitic  $\alpha$ , as shown in Figure 2.28. Based on the alloy composition, metastable  $\beta$ -phase can maintain its structure when cooled to room temperature. The microstructure can all be formed of transformed  $\beta$ , if the temperatures of solution treatment or finishing exceeds the  $\beta$  transition temperature, as shown in Figure 2.29.



Figure 2.28. Two different magnification SEM images (left 480X and right 1440X) of Ti-6Al-4V showing ultrafine martensitic  $\alpha$  and retained  $\beta$  surrounding globular  $\alpha$  after holding the alloy at 950 °C isothermally for 15 minutes and then quenching.



Figure 2.29. Examples of top surface samples of titanium alloys after holding them isothermally at 795°C after equilibration at 950°C.

In the following stages of aging, the decomposition of  $\alpha$  martensite leads to the precipitation of fine  $\beta$ , which leads to increasing the strength of the alloy [64]. Due to titanium's low thermal conductivity, it is difficult for martensite to form, especially in thick sections. The quick formation of Widmanstatten structures through nucleation and growth in alloys with lean  $\beta$  stabilization elements does not allow for the formation of martensite in them.

Based on the cooling rates of the alloy, the morphology of Widmanstatten  $\alpha$  can vary widely. Colonies of aligned  $\alpha$  platelets mixed with prior  $\beta$  grain boundaries are more likely to form with slow cooling, while basketweave structure types are more likely to form with  $\beta$  stabilization elements and faster cooling. There is a debate in the literature regarding the distinction between Widmanstatten and basketweave morphologies. While some sources consider the latter a finer type of Widmanstatten [64], other sources distinguish between them through their nucleation and growth patterns. It is observed that in basketweave  $\alpha$  plates grow within  $\beta$  grains, besides from  $\beta$  grain boundaries [66].

The term "basketweave" provides a reference to the morphology of the structure with stranded waves interlocking with each other, which makes their unique feature as the orientation between the  $\alpha$  plate colonies [67]. Based on that description, the characterization of basketweaves can be determined through the structure as a whole, rather than with a single colony. The contradiction between the sources may have emerged through the interpretation approach adopted by different authors.

In the following stages of aging, the decomposition of metastable  $\beta$  leads to the precipitation of fine  $\alpha$ , which subsequently leads to increasing the strength of the alloy. The improvement in the strength is referred in other sources to the formation of lamellar structures from the precipitation of finer secondary  $\alpha$ , which forms bilamellar microstructure. The higher level of  $\alpha$ -phase precipitation, leading to the increased strength, is correlated to weaker  $\beta$ -phase stabilization [68].

#### 2.6.1. Mechanical Properties of a Alloys

These alloys are characterized with good weldability, creep resistance, toughness and strength. Alloys with BCC are usually related to the lack of ductile-to-brittle fracture behavior. Ti-5Al-2.5Sn is one of the titanium alloys that fall under this category and its favorable in cryogenic applications [69]. Table 2.3 shows examples of common  $\alpha$  and near-  $\alpha$  alloys along with their composition and strength features.

Alloy	Nominal Composition (%wt.)				Maximum Impurities (%wt.)				wt.)	Strength (MPa)		
Alloy	Al	Sn	Zr	Mo	Others	N	С	Н	Fe	0	YS	TS
Ti-0.3Mo- 0.8Ni	-	-	-	0.3	0.8Ni	0.03	0.10	0.015	0.30	0.25	380	480
Ti-5Al- 2.5Sn	5	2.5	-	-	-	0.05	0.08	0.02	0.50	0.20	760	790
Ti-5Al- 2.5Sn-ELI	5	2.5	-	-	-	0.07	0.08	0.0125	0.25	0.12	620	690
Ti-8Al- 1Mo-1V	8	-	-	1	1V	0.05	0.08	0.015	0.30	0.12	830	900
Ti-6Al-2Sn- 4Zr-2Mo	6	2	4	2	-	0.05	0.05	0.0125	0.25	0.15	830	900
Ti-6Al- 2Nb-1Ta- 0.8Mo	2.25	11	5	1	0.2Si	0.02	0.03	0.0125	0.12	0.10	690	790
Ti-2.25Al- 11Sn-5Zr- 1Mo	5	5	2	2	0.25Si	0.04	0.04	0.008	0.12	0.17	900	1000

Table 2.3. Examples of common  $\alpha$  and near-  $\alpha$  alloys with their chemical and mechanical characteristics [69].

## **2.6.2.** Mechanical Properties of α + β Alloys

The precipitation of  $\beta$  elements and the adjustment of the microstructure of  $\alpha + \beta$  alloys is determined by their heat treatment process, which also determines its mechanical properties [69]. This feature allows for various mechanical properties options for the  $\alpha + \beta$  alloys, which make them competitive in terms of their strength at moderate elevated temperatures, strength at high room temperatures and good fabricability [16]. Table 2.4 shows examples of common  $\alpha + \beta$  alloys along with their composition and strength features.

Alloy	No	Nominal Composition (%wt.) Maximum Impurities (%wt.)				wt.)	Stree (M	ngth Pa)				
Anoy	Al	Sn	Zr	Мо	Others	Ν	С	Н	Fe	0	YS	TS
Ti-6Al- 4V <sup>*</sup>	6	-	-	-	4V	0.05	0.10	0.0125	0.30	0.20	830	900
Ti-6Al- 4V-ELI <sup>*</sup>	6	-	-	-	4V	0.05	0.08	0.0125	0.25	0.13	760	830
Ti-6Al- 6V-2Sn <sup>*</sup>	6	2	-	-	0.75Cu 6V	0.04	0.05	0.015	1.0	0.20	970	1030
Ti-8Mn <sup>*</sup>	-	-	-	-	8Mn	0.05	0.08	0.015	0.50	0.20	760	860
Ti-7Al- 4Mo <sup>*</sup>	7	-	-	4	-	0.05	0.10	0.013	0.30	0.20	970	1030
Ti-6Al- 2Sn-4Zr- 6Mo <sup>**</sup>	6	2	4	6	-	0.04	0.04	0.0125	0.15	0.15	1100	1170
Ti-5Al- 2Sn-2Zr- 4Mo-4Cr <sup>**</sup>	5	2	2	4	4Cr	0.04	0.05	0.0125	0.30	0.13	1055	1125
Ti-6Al- 2Sn-2Zr- 2Mo-2Cr <sup>*</sup>	5.7	2	2	2	2Cr 0.25Si	0.03	0.05	0.0125	0.25	0.14	970	1030

Table 2.4. Examples of common  $\alpha + \beta$  alloys with their chemical and mechanical characteristics [69].

\*. Annealed condition. Strength can be increased with solution treatment and aging. \*\*. Solution treatment and aging. Annealing is not possible.

### 2.6.3. Mechanical Properties of β Alloys

Despite of their low basic strength,  $\beta$  alloys are characterized with their good toughness, good ductility, and excellent formability. Over-aging treatment and stabilization are essential for the use of  $\beta$  alloys at elevated temperatures, as  $\alpha$ 

precipitates with solution treatment at these temperatures. Nonetheless, when compared to aged  $\alpha + \beta$  alloys with similar yield strength, aged  $\beta$  alloys are more competitive in terms of their toughness [69]. Table 2.5 shows examples of common  $\beta$  alloys along with their composition and strength features.

Allow	Nominal Composition (% wt.)				Maximum Impurities (%wt.)				Strength (MPa)			
Anoy	Al	Sn	Zr	Мо	Others	Ν	С	Н	Fe	0	YS	TS
Ti-10V- 2Fe-3Al <sup>**</sup>	3	-	-	-	10V	0.05	0.05	0.015	2.5	0.16	1100	1170
Ti-13V- 11Cr- 3Al**	3	-	-	-	11Cr 13V	0.05	0.05	0.025	0.35	0.17	1100	1170
Ti-8Mo- 8V-2Fe- 3Al**	3	-	-	8	8V	0.05	0.05	0.015	2.5	0.17	1100	1170
Ti-3Al- 8V-6Cr- 4Mo-4Zr**	3	-	4	4	6Cr 8V	0.03	0.05	0.020	0.25	0.12	830	900
Ti- 11.5Mo- 6Zr- 4.5Sn <sup>*</sup>	-	4.5	6	11.5	-	0.05	0.10	0.020	0.35	0.18	620	690

Table 2.5. Examples of common  $\beta$  alloys with their chemical and mechanical characteristics [69].

\*. Annealed condition. Strength can be increased with solution treatment and aging. \*\*. Solution treatment and aging. Annealing is not possible.

## 2.7. TITANIUM POWDER METALLURGY

### 2.7.1. Processing, Pressing and Applications

Titanium metal powder is produced through several methods, which are classified into groups [70]:

• Methods that produce titanium powder through extraction from the reduction of primary metals such as titanium dioxide (TiO<sub>2</sub>) or titanium tetrachloride (TiCl<sub>4</sub>)

• Methods producing powder from titanium scrap metals, titanium mill products, titanium alloy ingots, or titanium sponge.

Despite the various numbers of techniques that are used in these two groups of methods, the commercial restrictions allowed for few of them to be used in an industrial scale [70]. Table 2.6 shows a summary of the processes used for the production of different forms of titanium powder in an industrial level.

Process	Titanium Product	Deploying Organizations		
Electrolytic reduction of		TIMET/ Quinetiq/		
partially sintered TiO <sub>2</sub>	Powder block	Cambridge university/		
electrode in molten CaCl <sub>2</sub>		FFC		
Liquid Na reduction of TiCl <sub>4</sub>	Describen	International Ti Powder/		
vapor	Powder	Armstrong		
Anode reduction of TiO <sub>2</sub> ,				
transport through mixed	Solid slab, flake, or	MED Corporation		
halide electrolyte and	powder	MER Corporation		
deposition on cathode				
Fluidized bed reduction of Ti	Granula nouidar	SDI International		
halide	Granule, powder	SKI International		
Hydrogen reduction of TiCl <sub>4</sub>	Douvdor	Idaho Titanium		
plasma	rowder	Technologies		
Electrolytic reduction of	Tanned or solidified			
TiCl <sub>4</sub> vapor dissolved in	Ti liquid	Ginatta		
molten electrolyte	IIIquiu			
Calciothermic reduction of	Powder	Kyoto University/		
TiO <sub>2</sub>	Towaci	Suzuki/ Ono		
$I_2$ reduction of TiO <sub>2</sub> in	Particles	MIR Chemical		
shaking reactor	1 ditienes	White Chemical		
H <sub>2</sub> reduction of TiCl <sub>4</sub>	Sponge	CSIR		
Electrolytic reduction of Ti	Ti liquid	Quebec Fe & Ti		
slag	IIIquiu			
Electrolytic cell between	Compact of Ti	University of Tokyo/		
TiO <sub>2</sub> and liquid Ca alloy	powder with high	MSE/ FMR		
reduces TiO <sub>2</sub>	porosity			
Reduction of TiO <sub>2</sub> reduction	Compact of Ti	Preform Reduction		
by Ca	powder			
Gaseous reduction of TiCl <sub>4</sub>	Powder	Vartech		
vapor	100000	Varteen		
Mechanochemical reduction	Powder	Idoho Research		
of liquid TiCl <sub>4</sub>	IOWUCI	Foundation		

Table 2.6. Commercially used processes used for the production of titanium products and powders [70].

Titanium powder is directly produced through metallurgic extraction processes from purified  $TiO_2$ ,  $TiCl_4$ , or 90%  $TiO_2$  content in upgraded titanium slag, which is produced from titanium ores (ilmenite and rutile) using carbothermal reduction. Two classifications of reduction processes are identified: thermochemical and electrochemical [70]. Table 2.7 shows a summary of the reduction processes of  $TiO_2$  and their parameters under the two categories.

Catagory	Identifian	Raw	Reducing	Product	Temperature
Category	Identifier	Material	Agent	Morphology	(°C)
	MHR	TiO <sub>2</sub>	CaH <sub>2</sub>	Irregular sponge	1100-1200
	Calciothermic reduction	TiO <sub>2</sub>	Ca	Irregular sponge	900-1200
Thermoch	Preform reduction process	TiO <sub>2</sub>	Ca	Irregular sponge	>900
Thermoen	EMR	TiO <sub>2</sub>	Ca	Irregular sponge	>900
	Combustion synthesis	TiO <sub>2</sub>	$TiO_2 \qquad Mg + Ca \qquad Irregular and porous$		850-1000
	DRTS/HAMR	Upgraded Ti slag	Mg + Ca	Dense and globular powder	< 800
	OS	TiO <sub>2</sub>	Electron/ Ca	Irregular sponge	>900
	FFC	TiO <sub>2</sub>	Electron	Irregular and porous	800-1000
Flootro	MER	TiO <sub>x</sub> C <sub>y</sub>	Electron	Irregular and porous	Approx. 900
Electro	Chinuka	TiO <sub>x</sub> C <sub>y</sub>	Electron	Irregular and porous	700-900
	USTB	TiO <sub>x</sub> C <sub>y</sub> N <sub>z</sub>	Electron	Irregular and porous	Approx. 800
	QIT	Ti slag	Electron	Solid ingot	>1400
	SOM	TiO <sub>2</sub>	Electron		1100-1300

Table 2.7. Difference in characteristics between the reduction processes of TiO<sub>2</sub> [70].

Titanium powder is solidified to fit different applications through several pressing, molding, and melting methods. There are six main applications that are used for that purpose: hot isostatic pressing, electron beam melting, metal injection molding, cold spray, direct energy deposition, and selective laser melting. The size of the titanium particles plays a major role in the selection of the sintering method, as well as oxygen content, chemical composition, flowability, and particle size distribution [71]. As shown in Figure 2.30, hot isostatic pressing is the most compatible method with the wide range of titanium powder particle sizes.



Figure 2.30. Titanium powder particle size requirements for different sintering methods [71].

The use of different compaction methods affects the mechanical properties of titanium alloy [72], as shown in Figure 2.31, where it illustrates the stress/strain graph for titanium powder compacted by different methods.



Figure 2.31. Stress/strain graphs for titanium alloys compressed with different methods [72].

#### 2.7.2. Hot Isostatic Pressing (HIP)

During metal casting, several defects can arise during solidification, including shrinkage and pores, which emerge from the bond between different material and creep vacancies. Therefore, pressing methods, like hot isostatic pressing (HIP), are used in order to achieve densification of the material that reduces metal surface areas and the surface energy of the pores, subsequently. The concept of hipping (referring to HIP) is based on utilizing isostatic pressure that is produced by the molecules of the trapped gases towards the surface, which makes each one of them act as an individual hot forge. The uniform pressure allows for the gas molecules to collide with the surface material and reach all of its parts, including angles, which makes hipping a reliable and consistent processing method, and independent of the shape. The uniformity of hipping is a result of similar velocity and direction of all moving gas atoms [73].

In old types of pressing, the pressure from the ram is exerted on a single axis, while a die contains the pressed material. The friction between the material in the die and the ram contributes to the distribution of various pressure force in different material parts in a nonuniform way causing variations in densification. In isostatic pressing, a larger size of the desired material is placed in an envelope made of glass or metal. The envelope material shall be selected from a material type that plastically deform under the applied pressure and temperature of hipping. During the densification process, shrinkage in the compacted powder occur, as well as the material of the envelope in all directions with the same extent. The densified powder takes the shape of the envelope ultimately, while both reduce their shape photographically [74], as shown in Figure 2.32.



Figure 2.32. Photographic reduction of HIP envelope and contained material [74].

Hipping achieves total void removal from the processed powder, which cannot be achieved with other pressing methods, while its isostatic nature facilitates the formation and maintenance of complex shapes. Table 2.8 shows the standard temperatures and pressures that are used for different materials during hot isostatic pressing, which can be compared with the melting point and yield stress of each one of them. The most used pressure used in hipping is 100 MPa, which is similar to pressures exerted by the ocean water on the bottom of the deepest point in ocean floor. In the hipping cycle, the heated gas in the enclosure with the constant volume and the mechanical compressor work in conjunction to achieve the developed gas pressure. Although the relationship between the temperature and pressure, as per ideal-gas laws, breaks down with high pressure, the fixed volume lead to the increase of the pressure with the rise of the temperature [75].

Material	Melting point (°C)	Yield Stress at 20 °C (MPa)	Temperatur e of Hipping (°C)	Pressure of Hipping (MPa)
Al and Al alloys	660 (Al)	100 to 627	500	100
Al/ Al <sub>2</sub> O <sub>3</sub>	-	-	300	350
Cu and Cu alloys	1083 (Cu)	60 to 960	800 to 950	100
Be and Be alloys	1289 (Be)	240	900	103
Nimonic and superalloys	1453 (Ni)	200 to 1600	1100 to 1280	100 to 140
Hydroxyapatite	-	-	1100	200
Mg/ Zn ferrite	-	-	1200	100
TiAl	-	-	900 to 1150	35 to 200
T <sub>3</sub> Al	-	-	925	200
Ceramic superconductors	-	-	900	100
Steels	1536 (Fe)	500 to 1980	950 to 1160	100
Ti and Ti alloys	1670 (Ti)	180 to 1320	920	100
$Al_2O_3$	2050	5000	1500	100
Al <sub>2</sub> O <sub>3</sub> / glass	-	-	1400	100
Al <sub>2</sub> O <sub>3</sub> / TiC	-	-	1935	150
Al <sub>2</sub> O <sub>3</sub> / ZrO <sub>2</sub>	-	-	1500	200
SiC	2837	10000	1850	200
B4C	-	6000	2000	200
WC/Co	2867		1350	100

Table 2.8. Standard temperatures and pressures that are used for different materials during hot isostatic pressing [76].

The temperature of hot isostatic pressing is often greater than 0.7 of the melting point temperature of the material. For instance, hipping of turbine blades made of cast superalloys is carried out temperatures between 1100 °C and 1280 °C. The melting-point-constituent of some material is considered relatively low, which helps removing pores while performing the process between the melting point and the temperature specified in the matrix. Hipping at high temperatures raises diffusivities and lower yield strength of material, which allows a timely closure of pores [77].

The development of hot isostatic pressing process incepted to remove pores in hard material and to bond nuclear reactor components by diffusion [78]. Nonetheless,

densifying high-performance casting and consolidating metal powders are the main focus of commercial activities of hipping these days. Several industries use hot isostatic pressing in their manufacturing and processing, including mining, metal working, telecommunications, microelectronics, defense, medical, automotive, power generation, marine, and aerospace [79]. Table 2.9 shows the range of applications of hot isostatic pressing the objectives of each application and the materials that are associated with each one.

HIP Application	Main Objective	Processed Materials
Reactive hipping	Synthetization of compounds using an exothermic reaction	Intermetallic compounds, like Nb <sub>3</sub> Al and TiB <sub>2</sub>
Interface bonding	Join similar and dissimilar materials through diffusion by joining and uniaxial techniques. Also,	Ni alloys with steel Bronze with steel Ceramic-metal bonding
	Densify and bond coatings	Ta, Ti, Al and W in Al housing for sputter targets
Consolidation of encapsulated powders	Achievement of full theoretical density, elimination of segregation, and avoidance of excessive grain growth	Fe-TiC Ceramic superconductors Magnetic material Ceramic-ceramic composites Metal matrix composites Ceramics High-speed tool steel
Densification of pre-sintered powders	Achievement of full theoretical density, elimination of segregation, and avoidance of excessive grain growth, which are not possible through conventional techniques	Be alloys Si <sub>3</sub> N <sub>4</sub> , Al <sub>2</sub> O <sub>3</sub> , ZrO <sub>2</sub> Advanced ceramics WC-based hard metals
Densification of castings	Removal of micro and macro porosities	Cu alloys Ti alloys Steels Al alloys Ni- and Co-based superalloys
Specialized applications	Removal of gaseous impurities and pores from optical material	Lanthanates and aluminates Sn-In-O ZnS

Table 2.9. Applications of hot isostatic pressing (HIP), their objectives and processed materials [77].

Hot isostatic pressing is able to remove micro and macro porosities. In casting, the molten metal cools and solidifies, which causes gas evolution and shrinkages that develop into micro porosities. As shown in Figure 2.33, toughness of casted metals is negatively influenced in a significant manner, which also applies to other dynamic and static mechanical properties, including fatigue, creep, and strength. In traditional practices, hot working, such as forging, has been used in order to remove pores from casted metals. However, the use of hot working compromises the shape and cost of the metal piece [80].



Figure 2.33. Impact of micro porosity on the toughness of cast steel [80].

Hot isostatic pressing and similar techniques are preferred for the removal of porosity as they do not have adverse effects on the micro-structure of the metal, such as precipitate arrangement, phases present and grain structure. Therefore, the use of hipping is considered significant as it reduces scrap rates in manufacturing and increase the reliability of service. Moreover, The mechanical properties of metals are enhanced through the use of hipping [81], as shown in Figures 2.34, 2.35 and 2.36 that compare hipped and forged components based on their strength and elongation, fatigue life and creep life, respectively.



Figure 2.34. Comparison of casted and hipped Ni-Al bronze based on ultimate tensile strength and elongation (porosity range 10% to 20%) [81]



Figure 2.35. Comparison of casted and hipped Ti-6Al-4V based on fatigue life [82].



Figure 2.36. Comparison of casted and hipped Ti-6Al-4V based on creep life [18].

In addition to mechanical properties, the property scatter is also enhanced through hot isostatic pressing [83]. The average values of several specimens can be scattered through a range of values can be enhanced through hipping, in addition to the decrease in distribution width, as shown in Figure 2.37. Such a property allows for the prediction of important mechanical properties, especially creep and fatigue life, which enables better scheduling for maintenance. Furthermore, this property is important for designers to use higher design values confidently, as the scatter provide the mean values for the material [84].



Figure 2.37. Comparison of transverse rupture strength before and after hipping of metal sample [84]

### **CHAPTER 3**

#### METHODOLOGY AND EXPERIMENT

## **3.1. MATERIAL**

The Ti-6Al-4V alloy used in the experiment is prepared through the cost-effective blended elemental PM technique [85]. The hydrogenated titanium powder (3.5% H wt; Ti particle size 100 µm) is mixed with 60Al-40V master alloy powder with particle size ranging between 40 to 63 µm in order to obtain the required alloy composition [86], as shown in Table 3.1.

Table 3.1. Chemical composition of Ti-6Al-4V alloy by % wt.

Ti	Al	V	Fe	0	Ν	Н
Balance	6.21	4.12	0.0	0.18	0.04	0.004

#### **3.2. SAMPLE PREPARATION**

The powder was blended for 1200 seconds (20 mins) and die pressed under 300 MPa at a temperature of 500 °C for 4 hours to form green compacts of 60 x 60 x 3 mm, as shown in Figure 3.1. The approximate density of the sintered alloy is determined through hydrostatic weighing to be 4.42 g/cm<sup>3</sup>: corresponding to 97.73% of the theoretical density of the cast alloy. The compacted specimens are shown in Figure 3.2. For each produced sample, 50 gram of alloy powder was used, while the final average weight of each of nine produced sample is 47.844 gram. Samples are produced in the laboratory of Gazi University.



Figure 3.1. Hot isostatic pressing of Ti-6Al-4V powder.



Figure 3.2. Specimens produced after hipping.

The sintering of the specimens is performed in a vacuum furnace at a vacuum pressure of 10<sup>-3</sup> Pa. The nine specimens were divided into three groups, as shown in Table 3.2. Three temperatures were applied: 1200 °C, 1300 °C and 1400 °C, at 2, 4 and 8 hours for each group. The described properties of powder metallurgy compacts are not comparable to the obtained wrought metal. It is expected the sintered metal would have lower ductility, for both alloyed and unalloyed titanium, than the wrought metal [87].

Group	Specimen	Furnace Temperature (°C)	Sintering Duration (Hours)
	A1		2
А	A2	1200	4
	A3		8
	B1		2
В	B2	1300	4
	B3		8
	C1		2
С	C2	1400	4
	C3		8

Table 3.2. Specimens and sintering conditions.

Five tests are performed on each sample: wear resistance test, microhardness test, XRF analysis, XRD analysis, and SEM analysis. The results are presented in the next section of this article and discussed along with relevant literature.

# **3.3. PHASE ANALYSIS & MICROSTRUCTURAL CHARACTERIZATION**

For phase identification, Rigaku ULTRA IV Diffractometer is used the XRD analysis. Samples are tested in the laboratory of Karabuk University. The settings of the diffractometer were set, as follows:

- 20 mA current
- Cu-K $\alpha$ -X-ray radiation below 40 kV acceleration voltage
- 3 degrees per minutes as the measurement scan speed
- Scan range is 20° to 90°

For identification of the material's Xray diffraction pattern, the ICDD database was used.



Figure 3.3. Rigaku ULTRA IV diffractometer.

Three microstructural characterization tested were performed: Field Emission Scanning Electron Microscopy (FESEM) with Carl Zeiss ULTRA PLUS FESEM, and chemical composition assessment with Energy Dispersive Spectroscopy (EDS) with Carl Zeiss EVO 18. Samples are tested in the laboratory of Karabuk University.

## **3.4. HARDNESS TEST**

Q10 A+ QNESS microhardness testing machine was used to perform Vickers microhardness test with a 1-kilogram load and 15 seconds holding time. Samples are tested in the laboratory of Karabuk University. Three readings have been taken from the surface of the sample and the points A, B, and C, as shown in Figure 3.4, where the distance Between (A, B) and (B, C) was up to 4 mm. The microhydraulic hardness test was performed based in the following equation:

$$H_{\nu} = \frac{1.8544 \, P}{(d_{a\nu})^2} \tag{3.1}$$

Where,

 $H_v$ : Vickers hardness (kg/ mm<sup>2</sup>) P : Loaded force (kg)  $d_{av}$ : Average impact diameter (mm)

The cleaning and polishing process was done then washed with water, alcohol and then dried using warm air to avoid surface oxidation of the sample. The average measurement at three different points was calculated to produce the hardness value at one indentation per point.



Figure 3.4. Position of microhardness measurements.

## 3.5. WEAR TEST

A pin-on disk type wear apparatus was used to perform dry sliding wear test (Figure 3.5). The wear tests were performed according to ASTM-G99-05 standards. An AISI 4423 steel disc of 64 HRc was used as the counter surface for wear test of the AMCs produced in the diameter of 10 mm and in the height of 6 mm. Before the wear tests, the surfaces of the samples were ground using 1200 grade SiC sandpaper in order to provide full contact with the steel disc surface. The wear test was performed with a 200 m sliding distance for all samples, at a sliding speed of 56 mm\s and under 20 N load. The wear samples were weighed in a digital balance with a sensitivity of  $\pm$  0.0001 g before and after each test and the weight losses were determined. Samples are tested in the laboratory of Karabuk University.


Figure 3.5. Pin-on disk type wear apparatus.

# **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### 4.1. WEAR TEST

The specimens were subjected to wear resistance test, where machine parameters were set as shown in Table 4.1. Raw data is also available in Appendix A.

Parameter Load	Lood	Strolzo	Test	Sliding	Sliding	Engguenav
	Load	SUOKE	cycles	Distance	Speed	riequency
Unit	Ν	mm	No.	М	mm/s	Hz
Value	20	7	14285.71	200	56	4

Table 4.1. Wear resistance test parameters.

As shown in Figure 4.1, the friction coefficient for specimens in group A differed with the sintering duration. While maximum friction coefficient increased from 3.054 in A1 to 3.882 in A2, the maximum friction coefficient decreased to 1.929 in A3, which corresponds to the longest sintering duration. In A1, fluctuations with an increase in friction appear between zero to 40 meters. Smooth sliding gaps started appearing afterwards with the maximum friction towards the end of the distance. Specimen A2 showed high fluctuation rate with a steady overall increase in friction, while A3 showed a steady friction coefficient with a wave of higher friction between 5 to 35 meters.



Figure 4.1. Wear resistance friction coefficient change for Group A.

Results for group B specimens are presented for comparison in Figure 4.2. A reduction in overall friction coefficient is found in B2. Specimen B1 showed an increase of friction between zero to 30 meters and continued a maximum coefficient of 3.07. Specimens B2 and B3 showed more steady fluctuations at periodical intervals, with maximum coefficients of 2.109 and 3.382, respectively.



Figure 4.2. Wear resistance friction coefficient change for Group B.

# 4.2. MICROHARDNESS

Vickers microhardness test is conducted through applying a load of 1 kilogram for 15 seconds by a ball indicator. Values are measured through a calibrated optical microscope for the stress in MPa, as shown in Table 4.2. The average hardness values are presented in Figure 4.3. Hardness value for group A increased with the increase of sintering time between 2 hours and 4 hours, while it dropped at 8 hours. For group B, the hardness value increased at both 2 and 4 hours, while it decreased for both sintering times for group C. Generally, microhardness values increased with the increase of heat treatment temperatures, while increased sintering times decreased hardness values. Wang and Langdon [88] and Abdalla, et al. [89] showed the increase in microhardness values between heat treated and untreated Ti-6Al-4V samples. However, Ahmadi, et al. [90] showed decrease in the hardness values with the increase of heat treatment temperatures without specifying changes in sintering durations.

Temperature	Time	Value (1)	Value (2)	Value (3)	Average
	2 h	331	333	319	327
1200 °C	4 h	389	398	405	397
	8 h	375	332	364	357
1300 °C	2 h	403	387	379	389
	4 h	433	454	481	456
	8 h	488	491	499	485
	2 h	599	533	599	577
1400 °C	4 h	427	481	477	461
	8 h	494	424	461	459

Table 4.2. Hardness test values.



Figure 4.3. Average hardness values for groups A, B and C.

#### 4.3. XRF ANALYSIS

X-ray fluorescence (XRF) is a microscopy method that is used to determine the concentration of metals in an alloy in a specified area in the testing. The method is based on the interaction between the x-ray arrays with the matter through dislodging electrons from atoms and measuring the energy resulting to correlate it with a specific element [91]. Table 4.3 shows the results of the XRF analysis in the tested alloy. As seen from the results of the XRF analysis. The analysis depth ranged between 0.0030 to 0.8550 mm. Titanium percentage reduced from 58.85% to 47.79%, while the percentages of Al and V were reduced from 5.12% to 4.16% and from 8.15% to 6.62%, respectively. The 90:6:4 ratio is confirmed for the Ti-6Al-4V in different studies. Hamad and Salloom [92] tested four samples of Ti-6Al-4V and confirmed the ratio with error margins ranging between 0.13% and 10%. Lee, et al. [93] identified 5.5% Aluminum, 3.87% Vanadium and 0.22% iron by weight through an XRF analysis.

Component	Result	Unit	Det. Limit	EL line	Intensity	w/o normal	Analyzing depth (mm)
В	0.7751	Mass%	0.11345	B-KA	0.1816	0.9545	
С	3.1998	Mass%	0.03225	C-KA	4.4768	3.9402	
Ν	13.1932	Mass%	0.15751	N-KA	3.9806	16.2459	
0	30.8619	Mass%	1.09191	O-KA	0.4586	38.0030	
Na	0.0814	Mass%	0.01053	Na-KA	0.1706	0.1002	0.0030
Mg	0.0781	Mass%	0.00738	Mg-KA	0.5700	0.0962	0.0046
Al	0.4155	Mass%	0.00380	Al-KA	9.1576	0.5116	0.0069
Si	0.1610	Mass%	0.00156	Si-KA	3.7107	0.1982	0.0099
Р	1.8753	Mass%	0.00281	P-KA	121.6887	2.3092	0.0137
S	0.1542	Mass%	0.00082	S-KA	8.6498	0.1899	0.0174
Cl	0.0401	Mass%	0.00255	Cl-KA	0.6165	0.0493	0.0240
K	0.0041	Mass%	0.00221	K-KA	0.1213	0.0051	0.0506
Ca	0.0198	Mass%	0.00266	Ca-KA	0.6777	0.0244	0.0658
Ti	47.7941	Mass%	0.02561	Ti-KA	432.8491	58.8530	0.1127
V	0.6622	Mass%	0.05760	V-KA	0.6306	0.8154	0.0402
Fe	0.6684	Mass%	0.00446	Fe-KA	13.8373	0.8230	0.0619
Ni	0.0041	Mass%	0.00247	Ni-KA	0.1708	0.0050	0.0919
Cu	0.0047	Mass%	0.00249	Cu-KA	0.2469	0.0057	0.1119
Mo	0.0071	Mass%	0.00126	Mo-KA	2.8908	0.0088	0.8550

Table 4.3. XRF Analysis of the alloyed Ti-6Al-4V.

## 4.4. XRD ANALYSIS

Identification of crystalline material in the samples that were subject to heat treatment is carried out through an x-ray powder diffraction (XRD) analysis. Figure 4.4 shows the peaks of eight samples, which are identical in phase pattern and position, while differing in intensity. The higher temperature and duration of the heat treatment the higher is the intensity.



Figure 4.4. Diffractogram of XRD analysis.

The shift in the peaks with the increase of the heat treatment conditions is caused by the creation of equilibrium phases of the lamellar structure  $\alpha+\beta$ . The decrease in the width accompanied with the increase in intensity of the peaks is caused by the non-

equilibrium  $\alpha$  phase of the non-treated samples and the transformation to the equilibrium phase with heat treatment [94].

# 4.5. SCANNING ELECTRON MICROSCOPY (SEM)

As shown in figures 4.5 to 4.12, the specimens A1 to C2 analyzed through scanning electron microscopy. The behavior of Aluminum and vanadium vary under heat treatment. While Al tends to enter the  $\alpha$  phase, the V tends to enter the  $\beta$  phase [95]. In the images taken for the eight specimens at the same magnification, the dark regions show the  $\alpha$  phase, while the light regions present the  $\beta$  phase [96]. The presence of delamination wear, abrasion and debris in the SEM pictures of Ti-6Al-4V is similar to the test results of other titanium alloy testing, such as Ti-5Al-3V-2.5Fe [97].



Figure 4.5. FESEM images of heat-treated specimens at x500 for A1 (Layer thickness:  $A1=8.8 \ \mu m$ ).



Figure 4.6. FESEM images of heat-treated specimens at x500 for A2 (Layer thickness: A2=8.9  $\mu$ m)



Figure 4.7. FESEM images of heat-treated specimens at x500 for A3 (Layer thickness: A3=11.5  $\mu$ m)



Figure 4.8. FESEM images of heat-treated specimens at x500 for B1 (Layer thickness:  $B1=11.0 \ \mu m$ ).



Figure 4.9. FESEM images of heat-treated specimens at x500 for B2 (Layer thickness: B2= 10.4  $\mu m$ ).



Figure 4.10. FESEM images of heat-treated specimens at x500 for B3 (Layer thickness:  $B3 = 10.4 \mu m$ ).



Figure 4.11. FESEM images of heat-treated specimens at x500 for C1 (Layer thickness:  $C1=10.5 \mu m$ ).



Figure 4.12. FESEM images of heat-treated specimens at x500 for C2 (Layer thickness:  $C2 = 9.7 \ \mu m$ ).

# **CHAPTER 5**

#### CONCLUSIONS

Titanium and its alloys are considered as competitive metals due to their advantageous mechanical properties, high corrosion resistance, and lightweight in comparison with other alloys like steel. The many advantages of titanium and its alloys motivated their utilization in wide range of applications, including automotive, petrochemical products, aerospace, personal products, and biomedical products. Titanium provides the option to save weight due to its lower density in comparison with other metal, such as steel. Moreover, titanium preserves its strength criteria at high temperatures, which is not possible with other light weight metals like aluminum. The density of titanium is 60% higher than aluminum. Nonetheless, the dominance in the selection criteria prevails to the resistance to high temperatures with an advantage of lower density, which makes aluminum the most preferred metal in aerospace.

The physical and mechanical properties of the final titanium product are significantly affected by the working and treatment factors, individually or combined. The most significant factors are heat treatment, fabrication methods, mechanical working methods of ingots to turn them into mill products, the process used for melting to make the ingot, and the types and amounts of alloyants and impurities that are contained within the ingot. The environment surrounding the processing of titanium and its alloys, and its conditions, needs to be highly controlled. Although the ability to use a narrow range of titanium alloys for a wide range of applications is advantageous, further variety of titanium and its alloys can be produced by altering the conditions of mechanical or thermal processing.

The applications of titanium alloys are correlated to their mechanical properties. Therefore, it is important to understand the factors that affect these properties in order to optimize their performance. A research found that the mechanical properties of titanium alloys, such as creep, fatigue, plasticity, and strength, are highly affected by their microstructure. The small size of  $\alpha$  grains are correlated to the increase in ultimate tensile strength.

One of the most common titanium alloys is Grade 5 (Ti-6Al-4V), which is used extensively in engineering and industrial purposes. Due to its high mechanical performance criteria and facilitation of weight reduction, industries using this alloy included marine equipment, automotive and aerospace applications. Medical and pharmaceutical applications can also be found for Ti-6Al-4V due to its exceptional biocompatibility. However, the alloy has difficult machinability because of its low thermal conductivity and high strength to density ratio.

Ti-6Al-4V is classified as an  $\alpha + \beta$  two phase alloy with wide usage range in mechanical applications. Due to the structure complexity of the manufactured components and the defects, such as composition segregation and porosity, forging and casting is not the most preferred manufacturing method. Thus, hot isostatic pressing (HIP) is used for the consolidation of alloy powder in order to facilitate manufacturing complex components and avoid those defects. The HIP method has also economic advantages as it maximizes the utilization of material and reduce the costs of defected waste.

Through further investigation and analysis, further advantages were found for the HIP method application on Ti-6Al-4V on the microstructure level. There are several studies that attempted to optimize the heat and pressure conditions of the alloy in order to reach to an optimum performance. There are studies that solely tested the heat or pressure change implications on titanium alloys, while few studies investigated the change in both parameters.

The aim of the research is to investigate the effect of very high heat exposure on Ti-6Al-4V alloys processed by hot isostatic pressing through comparing samples with different exposure timing. The current research manufactured Ti-6Al-4V alloys using the HIP method and studied the impact on heat treatment under different temperatures and sintering durations on the performance and microstructure of the alloy. Three temperatures were tested (1200 °C, 1300 °C and 1400 °C) with the durations of 2, 4 and 8 hours. Results show that sintering temperatures and durations had significant effects on wear resistance and hardness properties. The higher temperatures and longer durations reduced wear resistance and hardness. The XRD analysis showed a shift in the peaks with the increase and temperature and sintering durations, while equilibrium in lamellar structure  $\alpha+\beta$  was achieved. The peaks were increased in intensity and reduced in width. Moreover, the SEM analysis showed that the  $\alpha$  and  $\beta$  phases were balanced with the heat treatment.

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APPENDIX A.

WEAR TEST RAW DATA

Sliding Distance			Sam	ples		
(m)	A1	A2	A3	B1	B2	B3
0	0,8826656	0,0156224	-0,4530496	0,0078112	-0,039056	0,2109024
1	-0,9295328	1,796576	-0,2499584	0,4139936	-1,2341696	-1,9996672
2	0,7733088	-2,0387232	0,8357984	-0,9139104	1,2029248	2,0074784
3	-0,7264416	-1,9449888	-1,3122816	0,3202592	-1,3200928	0,898288
4	1,484128	0,6795744	-1,5075616	-1,0701344	0,7654976	-0,6717632
5	-1,1170016	0,7576864	1,5934848	1,093568	-0,3358816	1,7653312
6	1,2263584	1,75752	-1,5778624	-0,2109024	1,2419808	-2,2574368
7	0,312448	-2,34336	1,0076448	-0,039056	1,5934848	2,3980384
8	0,5545952	0	-1,75752	1,0232672	-1,445072	1,5388064
9	1,327904	1,3747712	-1,6637856	-1,3200928	1,6715968	-2,0387232
10	-1,1091904	-2,5073952	1,4919392	1,0701344	-0,7733088	1,56224
11	1,17168	2,4683392	-0,7733088	-1,093568	1,327904	-2,3667936
12	-0,58584	-2,5542624	0,3983712	0,4139936	1,5388064	2,030912
13	1,679408	-0,1640352	1,6169184	-0,9139104	-0,0312448	-0,3046368
14	-1,9762336	-1,5700512	-1,7106528	-1,0857568	1,56224	2,265248
15	-1,0779456	2,4527168	1,5856736	0,9451552	-1,7887648	-2,5230176
16	1,0701344	1,4138272	-0,7498752	-1,5153728	1,7028416	0,312448
17	-2,1402688	-0,4061824	0,3436928	2,069968	0,7108192	-1,6247296
18	1,9606112	1,6247296	-1,6169184	-1,3357152	0,0859232	2,53864
19	-1,8434432	-2,3902272	1,1873024	1,3982048	-1,2654144	2,3589824
20	-1,0388896	2,8198432	1,5309952	0,7967424	-1,36696	-2,5933184
21	0,0468672	0,9295328	1,5934848	-0,2499584	-1,406016	0,5545952
22	0,6405184	-1,5700512	-0,468672	1,327904	-1,4763168	-1,4685056
23	2,069968	1,0779456	-0,5702176	-1,4919392	1,6247296	2,1246464
24	-1,2263584	-2,6011296	-0,2968256	1,132624	-1,8043872	2,030912
25	-0,2577696	2,812032	-1,8121984	0,5155392	-1,36696	-2,2027584
26	-0,2655808	-2,3355488	1,4919392	0,19528	0,8514208	1,4919392
27	0,9842112	-1,1170016	-1,5544288	1,3513376	-0,8357984	-1,7809536
28	1,5466176	-1,4294496	-0,156224	-1,5075616	1,640352	2,3511712
29	-1,3435264	-2,2574368	-1,5934848	1,0779456	-1,484128	1,5700512
30	0,39056	1,3591488	-0,742064	-2,4058496	1,7653312	-1,17168
31	-1,679408	-0,0624896	1,3200928	1,7340864	-0,3827488	0,1718464
32	2,4058496	0,8514208	-1,3747712	-1,9996672	0,2030912	1,6325408
33	1,3825824	-0,4218048	0,7498752	-1,1638688	-1,5388064	1,6247296
34	-1,2732256	2,5073952	-1,5700512	1,484128	-1,406016	1,640352

Table A.1. Raw data obtained from the wear test and used in the analysis.

35	0,8357984	1,8434432	-1,5700512	-2,34336	-0,8357984	-0,8904768
36	-2,2183808	-2,3121152	1,3982048	2,1012128	-1,5856736	0,2187136
37	1,679408	1,2263584	-1,17168	-1,3747712	0,703008	-2,9292
38	0,859232	-2,616752	1,1248128	-0,9920224	-0,0156224	2,4527168
39	0,3202592	-2,0934016	1,3903936	0,0078112	0,312448	1,3122816
40	-1,0310784	1,1248128	1,1482464	-1,7497088	-0,6717632	-0,2265248
41	-1,4216384	-0,6327072	1,3982048	1,288848	-1,679408	-0,156224
42	1,7106528	2,53864	-0,8748544	-1,2341696	1,7262752	-2,2964928
43	-0,2812032	-2,7104864	0,9607776	1,210736	-1,4216384	2,2183808
44	0,7576864	2,8979552	-1,36696	-1,8043872	1,5934848	1,406016
45	-1,8668768	0,4764832	1,5309952	2,421472	-1,4763168	0,2968256
46	-1,3122816	0,3593152	-0,58584	1,1404352	-1,445072	-0,703008
47	1,3200928	-0,9529664	0,1718464	-1,4997504	1,6169184	2,2886816
48	-1,2263584	-2,0777792	-1,4216384	2,0934016	-1,2966592	1,8121984
49	0,8826656	2,3902272	-1,718464	-2,304304	0,5780288	-1,718464
50	-2,030912	-2,3511712	1,3903936	2,2886816	-0,9295328	0,6951968
51	-1,2654144	-0,2577696	-0,2187136	1,5075616	0,9998336	-1,874688
52	1,327904	-2,8667104	0,703008	-0,9451552	1,2341696	2,4449056
53	0,0312448	-1,4606944	1,4528832	2,3199264	0,039056	1,5934848
54	2,2574368	1,8824992	-1,5466176	-2,14808	1,2029248	-0,6170848
55	-1,9996672	-0,7967424	1,36696	1,56224	-1,3825824	1,5700512
56	1,7887648	0,1093568	1,0467008	1,6559744	-1,5388064	-2,6870528
57	0,1249792	-2,890144	-0,1874688	1,0623232	-0,1249792	2,4917728
58	0,3671264	2,8198432	-1,4138272	-2,3511712	-0,0156224	1,2263584
59	-1,6559744	0,7889312	-1,1482464	-1,8043872	-0,3749376	-0,0859232
60	-1,3435264	0,1406016	1,3591488	1,2341696	-1,5466176	-0,1093568
61	1,4528832	2,6323744	-1,4216384	-0,9451552	1,6325408	-2,3746048
62	-0,1327904	-2,5855072	1,445072	2,3667936	-1,640352	2,069968
63	1,8434432	-0,6405184	-1,3903936	-2,6792416	0,0468672	0,4764832
64	-2,4292832	0,9998336	-0,2265248	-1,7887648	-0,7108192	0,4608608
65	-1,2810368	0,39056	-0,351504	0,9920224	-1,5856736	-0,8436096
66	1,7262752	-1,640352	0,1093568	-2,069968	1,5934848	-2,0777792
67	-0,1249792	-2,0152896	1,3513376	2,5933184	-1,36696	1,7731424
68	1,8434432	1,8903104	-1,7731424	-2,3667936	0,6014624	0
69	-2,1168352	-0,5155392	-0,1484128	-1,4138272	-1,9528	0,4530496
70	-1,7809536	2,0387232	-1,406016	1,2341696	-1,5700512	1,5153728
71	0,5311616	1,6637856	0,7342528	0,78112	1,3122816	-2,0074784
72	0,4218048	-1,601296	1,3591488	0,351504	0,0468672	1,5153728
73	1,6481632	0,8357984	-1,2732256	-2,7104864	1,7262752	-0,7498752

74	-1,4997504	0,234336	-0,0078112	0,2968256	-1,484128	-1,874688
75	-0,663952	3,3744384	-1,4685056	-0,8826656	0,468672	0,273392
76	0	-2,9292	1,1560576	1,679408	-0,0078112	-2,1012128
77	0,7967424	-0,6873856	1,4528832	1,8824992	0,0156224	1,5544288
78	1,2966592	0,234336	-1,445072	-1,835632	1,7653312	0,3593152
79	-1,4138272	-2,4527168	1,3903936	0,4842944	-1,6637856	1,7418976
80	0,156224	3,2650816	-1,679408	-1,5778624	1,6481632	-0,9764
81	-0,4608608	0,8357984	1,3513376	2,2574368	-0,0859232	-2,2886816
82	1,6872192	0,1327904	-1,7418976	1,6169184	0,2421472	-0,0546784
83	1,3982048	-0,58584	-0,234336	-1,523184	1,7106528	1,132624
84	-1,7106528	2,53864	-0,9451552	1,2654144	-1,6169184	1,8512544
85	1,3435264	1,5700512	0,5233504	-2,5073952	1,56224	-2,1012128
86	-1,7262752	-1,2966592	1,56224	2,0934016	-1,6091072	1,718464
87	1,56224	3,2494592	-1,5544288	1,288848	1,7731424	-0,6014624
88	1,7106528	-1,8434432	0,2187136	-0,3436928	-1,5075616	1,5544288
89	-1,288848	-2,109024	-1,4216384	-0,0703008	-1,249792	1,8824992
90	0,5780288	1,3044704	1,2185472	1,36696	0,5155392	-2,0777792
91	-1,6325408	-0,7576864	1,3591488	2,109024	-1,249792	1,835632
92	1,445072	0,117168	-0,1406016	-2,030912	1,4997504	-2,2105696
93	1,6872192	-2,7885984	0,6717632	0,3671264	-1,6637856	2,1949472
94	-0,2655808	3,3900608	-1,6325408	-1,1170016	-0,5311616	1,9528
95	-0,0624896	0,7498752	1,4997504	2,5230176	-0,2812032	-1,9449888
96	-1,6091072	0,6092736	-0,1327904	1,7809536	-1,7106528	1,9059328
97	1,288848	2,5464512	-0,0156224	-1,5153728	1,7653312	-2,2964928
98	0,7342528	-2,6479968	1,327904	0,5233504	-1,5856736	2,3746048
99	2,109024	1,3982048	-1,3903936	-2,2886816	0,703008	-1,7497088
100	-1,6637856	-1,3982048	1,484128	2,4292832	-1,8200096	1,9293664
101	-1,2341696	1,8043872	-0,4452384	1,3982048	1,288848	-0,312448
102	1,36696	-2,851088	0,9451552	-0,1484128	1,6247296	-1,9840448
103	-1,2576032	-1,9215552	-1,6559744	-0,351504	-1,4685056	1,9606112
104	1,3200928	1,132624	-1,3591488	-2,577696	1,0467008	-2,1246464
105	-2,8745216	-0,2577696	1,5153728	2,14808	-0,9295328	2,1324576
106	-1,5153728	3,1713472	-1,0779456	0,2421472	1,5934848	-0,5624064
107	1,4997504	-3,1322912	1,0076448	0,117168	1,6169184	2,2886816
108	-0,6170848	3,0619904	-1,601296	-0,6951968	-1,3435264	1,4685056
109	1,6325408	0,6014624	-0,3827488	-2,6792416	1,2732256	-2,2183808
110	-1,75752	0,2265248	-0,2499584	2,34336	-1,1248128	0,9217216
111	1,36696	3,0541792	0,3202592	-0,0312448	1,6950304	-1,9762336
112	0,4999168	-2,4683392	1,4372608	0,5233504	0,9998336	-0,0312448

113	0,5155392	1,4763168	-1,6637856	-2,1402688	0,9529664	-1,9762336
114	1,5778624	0,2499584	-1,1404352	-1,445072	-0,9529664	-0,6327072
115	-1,9215552	0,4218048	-0,4218048	1,3357152	-1,5700512	0,039056
116	1,3825824	-1,5466176	0,117168	-1,0076448	1,6559744	-1,7809536
117	-1,8121984	-2,3980384	1,5544288	1,4528832	-0,5936512	1,7653312
118	0,5155392	1,2185472	-1,2966592	-2,460528	0,937344	-2,0465344
119	1,3357152	-3,0385568	0,507728	-1,523184	-1,679408	0,1796576
120	-1,2263584	1,5778624	-1,4216384	0,9842112	-1,601296	-0,5780288
121	1,5934848	1,7497088	0,9920224	-0,1327904	1,36696	-1,8981216
122	-2,3746048	-1,523184	1,445072	2,109024	-0,9998336	1,3903936
123	2,5073952	0,8826656	-0,2421472	-2,8198432	1,5153728	-2,6011296
124	1,3982048	-0,078112	0,624896	-0,9295328	-1,640352	-1,6247296
125	-1,3825824	3,2885152	-1,5466176	0,0312448	-1,3044704	0,58584
126	1,4372608	-3,1947808	1,445072	1,8512544	0,039056	-2,0152896
127	-2,1558912	0,3046368	0,5545952	1,718464	1,1248128	1,4528832
128	2,1168352	0,58584	0,2187136	-0,3280704	1,7340864	-1,054512
129	-1,9528	-3,3900608	-0,703008	-1,718464	-1,6637856	2,7495424
130	0,5624064	2,3121152	-1,6950304	-0,0312448	0,4218048	-2,499584
131	0,5155392	-2,34336	1,5466176	0,7264416	-0,1406016	1,9449888
132	-2,0777792	1,3044704	-0,39056	1,3357152	1,132624	1,1013792
133	1,2966592	-0,2812032	0,859232	-1,6091072	1,2341696	0,2421472
134	-1,56224	1,1248128	-1,3044704	1,0076448	-1,7262752	2,7651648
135	1,1170016	1,7340864	-1,3435264	-1,6559744	0,9685888	-2,3511712
136	-1,7887648	-1,9996672	1,3357152	2,5073952	-1,6637856	2,1246464
137	-1,4919392	2,8745216	-0,3358816	1,1404352	1,1951136	-2,5620736
138	1,327904	-1,0310784	0,5936512	-1,3200928	1,796576	1,1170016
139	-1,640352	-2,0934016	-1,6559744	0,0624896	-1,3357152	1,8824992
140	2,2886816	1,6637856	1,0467008	-2,499584	0,7967424	-2,1715136
141	1,5544288	-1,3357152	1,4528832	1,9606112	-1,7887648	1,4138272
142	-0,5545952	0,5702176	-1,3435264	1,1170016	1,5700512	-2,7807872
143	1,9293664	-3,1322912	-0,3358816	-0,9607776	-2,0621568	3,0932352
144	-1,7340864	2,968256	-1,835632	0,117168	-0,0312448	-2,6714304
145	0,3593152	0,2812032	1,1013792	-2,1402688	-1,3982048	-2,7261088
146	-0,5545952	-0,6405184	0,546784	1,9371776	0,9529664	1,7809536
147	1,4606944	3,5619072	0,1718464	1,796576	1,8668768	-2,4683392
148	1,7028416	-3,0307456	-0,7186304	-0,7186304	-1,7028416	2,2027584
149	-2,187136	2,4370944	-1,640352	-0,859232	0,3046368	-1,8590656
150	1,5544288	-2,0934016	1,6559744	-1,5778624	-1,7653312	0,3983712
151	-2,8042208	0,1249792	-0,2499584	1,1638688	1,1482464	0,0624896

152	1,56224	-0,6873856	0,5545952	-1,5934848	1,7262752	-2,6011296
153	-1,5075616	-2,3121152	-1,3982048	2,3746048	-1,6637856	2,1793248
154	1,5778624	1,523184	-1,4997504	-1,0857568	1,1951136	-2,421472
155	-2,2105696	-2,2418144	1,5856736	-1,718464	-1,6247296	-1,7887648
156	-1,835632	3,4056832	-0,2577696	1,0310784	1,6325408	-2,9292
157	1,5856736	-3,6009632	0,6561408	-1,3435264	0,1015456	2,4761504
158	-0,8904768	2,9292	-1,6091072	0,3749376	0,1249792	0,8045536
159	2,34336	1,1091904	-1,2029248	-2,3667936	-1,0857568	-1,8200096
160	-2,2574368	-0,4530496	0,4530496	2,3667936	-1,7731424	0,6951968
161	1,6481632	3,475984	0,2109024	1,1013792	1,640352	-2,6011296
162	-0,2968256	-3,12448	1,6091072	0,0234336	-0,3280704	1,9996672
163	-0,8670432	2,2496256	-1,6091072	2,3667936	0,3280704	-2,2964928
164	1,796576	0,6561408	-0,4139936	-2,1402688	-1,913744	-2,382416
165	-2,030912	0,039056	-0,1718464	1,4606944	-1,8121984	1,7262752
166	1,601296	3,0698016	0,7967424	1,9371776	1,7653312	-2,851088
167	1,015456	-2,890144	1,6169184	-1,0701344	-0,8279872	1,8043872
168	0,234336	1,5388064	-1,6325408	-1,1482464	0,7967424	-2,14808
169	-2,9213888	-2,5308288	1,5544288	-1,718464	-1,6715968	2,14808
170	-1,6950304	3,5384736	-1,445072	1,2732256	-1,4216384	-2,577696
171	1,1091904	-3,6400192	0,2577696	-1,4997504	0,1015456	2,7807872
172	-2,4839616	-1,9996672	1,445072	2,5152064	0,9060992	1,0779456
173	2,109024	1,2576032	-1,5309952	-2,3511712	0,6873856	-1,8668768
174	-2,187136	-3,4213056	1,1482464	-1,5700512	-1,718464	-0,820176
175	-1,601296	3,7649984	-0,0703008	1,2732256	-0,0156224	0,9529664
176	1,3122816	-3,4525504	0,1640352	-0,3671264	-0,0703008	0,1796576
177	-2,030912	-1,8824992	-1,6325408	2,7182976	0,312448	2,9838784
178	2,4839616	1,015456	-1,0857568	-2,0152896	1,4528832	-2,8042208
179	0,2265248	-3,6165856	-0,156224	-1,4138272	-1,9996672	0,6014624
180	0,2968256	2,0855904	-1,6637856	1,5934848	-0,0468672	-2,6792416
181	0,6951968	-3,397872	1,6247296	-2,3902272	-0,2265248	1,9606112
182	-2,34336	0,234336	1,445072	2,0074784	0,7576864	1,17168
183	1,6247296	-0,156224	-0,117168	-1,7340864	1,6247296	1,6091072
184	-1,9528	2,4839616	-0,2030912	1,288848	-1,5309952	-2,5620736
185	0,8357984	3,4213056	0,8123648	-1,7497088	0,2109024	-2,2886816
186	-2,3355488	-2,4839616	1,5153728	-0,0156224	-0,3983712	1,718464
187	2,73392	0,0468672	-1,4528832	1,5466176	1,1013792	-0,703008
188	1,4216384	-2,6089408	1,5153728	-1,6715968	1,7028416	2,2183808
189	-1,5856736	3,7181312	-0,9842112	1,9528	-0,8357984	-2,14808
190	1,6950304	1,2341696	-1,5153728	-2,6245632	-0,039056	-1,640352

191	-2,6401856	-3,2104032	1,601296	2,4917728	-1,327904	1,5856736
192	-1,3044704	1,1638688	-1,1013792	1,5075616	1,4294496	-1,6715968
193	1,3357152	-3,4291168	-0,0546784	-1,5700512	-1,7106528	2,499584
194	-1,6169184	3,6946976	-1,3591488	1,1638688	0,0234336	-2,5933184
195	2,53864	3,046368	1,679408	-2,9057664	-0,5389728	-1,5309952
196	-2,4292832	-1,991856	0,8279872	2,2964928	-1,7262752	1,2341696
197	1,601296	0,0546784	0,0078112	-2,4683392	1,601296	0,3280704
198	0,156224	-3,2650816	1,6325408	-1,7418976	-1,5466176	2,0621568
199	0,7733088	3,7649984	-1,8043872	-1,991856	1,1013792	-2,2105696
200	-1,4216384	0,6014624	1,6325408	-1,9606112	-0,8045536	-0,351504

## RESUME

Mohamed ELFGHI was born in Terhona, Libya in 1975 and he obtained his Bachelor of Science in mechanical engineering (Power) from College of Engineering Technology – Janzour-Libya in 1999. He also completed his master's in mechanical engineering with a focus on advanced manufacturing systems from Teesside University in the United Kingdom in 2008. He is currently pursuing the completion of his PhD degree at the Institute of Science and Technology, Department of mechanical engineering at Karabük University, Turkey.

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