



**AN INVESTIGATION OF THE EFFECT OF MN
AND ZR ADDITION ON THE
MICROSTRUCTURE, MECHANICAL AND
CORROSION PROPERTIES OF Ti6Al4V
TITANIUM ALLOY**

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Master Thesis

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September 2020

I certify that in my opinion the thesis submitted by Mohamed Erhaima Emahmed ERHAIMA titled “AN INVESTIGATION OF THE EFFECT OF MN AND ZR ADDITION ON THE MICROSTRUCTURE, MECHANICAL AND CORROSION PROPERTIES OF Ti6Al4V TITANIUM ALLOY” is fully adequate in scope and in quality as a thesis for the degree of Master of Science.

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“I declare that all the information within this thesis has been gathered and presented in accordance with academic regulations and ethical principles and I have according to the requirements of these regulations and principles cited all those which do not originate in this work as well.”

Mohamed Erhaima Emahmed ERHAIMA

ABSTRACT

M. Sc. Thesis

AN INVESTIGATION OF THE EFFECT OF MN AND ZR ADDITION ON THE MICROSTRUCTURE, MECHANICAL AND CORROSION PROPERTIES OF Ti6Al4V TITANIUM ALLOY

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The influence of Mn and Zr particles on both dry wear and corrosion resistance in Hank's solution of Ti-6Al-4V composite materials produced by powder metallurgy were studied systematically, and new composite materials with developed wear behaviour and corrosion resistance are promoted by micro addition amount method. Mn is effective to improve the wear behaviour of Ti-6Al-4V composite material. Moreover, Zr imparts to excellent corrosion resistance in Hank solution at 37°C. The superior properties of Ti-6Al-4V composite material with microalloying approach is highly ambitious for biomedical applications.

Key Words : Ti-6Al-4V, Wear, Corrosion.

Science Code : 91421

ÖZET

Yüksek Lisans Tezi

MN VE ZR ELEMENTİ İLAVESİNİN Ti6Al4V TİTANYUM ALAŞIMININ MİKROYAPI, MEKANİK VE KOROZYON ÖZELLİKLERİ ÜZERİNDEKİ ETKİSİNİN ARAŞTIRILMASI

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Bu çalışmada toz metalurjisi yöntemi ile üretilen Ti-6Al-4V kompozit malzemeye belirli oranlarda Mn ve Zr elementi ilavesinin kuru aşınma ve Hank's çözeltisi içerisindeki korozyon direnci üzerine etkisi incelenmiştir. Düşük oranda ilave edilen ilave elementler oluşan yeni kompozitin aşınma davranışında ve korozyon direncinde farklı sonuçlar elde edilmesini sağlamıştır. Mn, Ti-6Al-4V kompozit malzemenin aşınma davranışını iyileştirmede etkili olmuştur. Ayrıca Zr, Hank's çözeltisi içerisinde 37 °C'de mükemmel korozyon direnci sağlamıştır. Mikro alaşım yaklaşımına sahip Ti-6Al-4V kompozit malzemenin üstün özellikleri, biyomedikal uygulamalar için oldukça iddialıdır.

Anahtar kelimeler : Ti-6Al-4V, Aşınma, Korozyon.

Bilim Kodu : 91421

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SYMBOLS AND ABBREVIATIONS INDEX

SYMBOLS

Zr : Zirconium

V : Vanadium

Al : Aluminum

Ti : Titanium

Fe : Iron

PART 1

INTRODUCTION

It is possible to describe or define the biomaterials as natural or artificial materials which can be used in order to restore or support the living tissues functions which lose their functions inside human body. For this purpose, intensive studies have been conducted out for the development of new materials or the redesign of the materials [1, 2, 3, 4]. The use of metals and alloys with the greater mechanical features in biomaterials is increasing continuously because of their crystal structure. The use of these materials are wide and different where they are used as bone replacement and joint prosthesis in orthopedic applications. Also, they are used in cardiovascular surgery, jaw surgery and dental implants. At this regard, the most emphasized subjects are allergic reaction properties, surface corrosion, abrasion behavior, fatigue strength, and biocompatibility of biomaterials [5, 6, 7, 8]. In general, Co-Cr alloys, 316L stainless steels and Ti alloys are used in the biomedical applications [9]. Many reasons behind the use of titanium allows as biomaterial in orthopedic applications including their low density, good mechanical properties, easy to produce small size parts, biocompatible, corrosion resistance and most importantly the elasticity modulus is one of the metallic materials nearby to the elastic modulus of the bone [26, 27]. The microstructure of the titanium alloys divide them into four groups as α -alloys, β -alloys, α - β alloys and near α -alloys. Titanium, hexagonal tight package (HSP) structure at low temperatures and 882.5 °C has a volume centered cubic (HMK) structure beyond the temperature [10, 11, 12]. Implants used in the body environment are expected to have mechanical effects (muscle movement and body weight), chemical effects (corrosion and dissolution), temperature and osteointegration (tissue-compatible and non-toxic).

There are many reasons to use the titanium alloys in the biomaterials applications and the most important of them are the biocompatibility and corrosion resistance if compared with other metallic resources. Titanium presents high corrosion resistance against many natural environments, body fluids and sea waters [13]. These alloys present high corrosion resistance by creating a defensive passive-oxide layer (nm) over the external cover. The flow of ions and electrons in the organs or body fluid are prevented by the low electrical conductivity of this oxide layer. Consequently, the passive oxide layer (TiO_2) offers surface protection to the alloys of titanium. The stable passive oxide film ensures the excellent biocompatibility by protecting the alloys of titanium against corrosion. Moreover, the titanium is protected from both thermal and chemical impacts and acid attacks by this oxide layer. Even in case of damaging this oxide layer in anyway, it can be recreated depending on the environmental circumstances [14, 15]. Corrosion products that may occur in inserts in the environment of body enter the tissue and harm the cells. Therefore, biomaterials which are utilized in the environment of body should be tested in saliva, serum, and different synthetic buffer solutions (in vitro).

Since the modulus of elasticity of the Ti6Al4V alloy $\alpha + \beta$ is the closest alloy to the modulus of elasticity of the bone, this alloy is preferred in systems to be fixed to the bone in the body (orthopedic and dental inserts) [5]. However, alloys of titanium are deficient in the applications where mobile contact is active because of their low wear resistance and low friction [16]. It is desirable that biomaterials may exhibit biocompatibility and remain in the body of human for a long period of time without any allergic reaction though contacting the body tissue. Also with friction in the joints. As well as, it is desirable that the wear behavior and friction of the biomaterials be of superior quality, since the residues (particles) that are formed and resulting from the wear of the prosthesis because great hazards to the body. Therefore, abrasion is a crucial element that controls and decides the durable performance of metallic biomaterials [17].

The decrease in resistance of abrasion or the raise in the factor of friction bases relaxation of the biomaterials retained on the body. In addition, residues formed as a

result of wear can cause infection in the body [18, 19]. Therefore, titanium used as articulated joint material.

In this study, the Ti-6Al-4V materials produced by the PM method where the both Mn and Zr amount are determined with 0.5, 1 and 2 in wt% to be added. The electrochemical behaviour of Ti-6Al-4V, Ti-6Al-4V-xMn and Ti-6Al-4V-xZr was investigated in Hank's solution at 37 °C. The dry wear test of produced composite materials was accomplished at 25°C. To observe the microsture, scanning electron microscopy (SEM) was utilized that supported by energy dispersion spectrometer (EDS).



PART 2

BIOMATERIALS

2.1. BIOMATERIALS HISTORICAL DEVELOPMENT

Teeth, artificial eyes and nose which have been found in Egyptian mummies are indication that biomaterials have been used in ancient times. Chinese and Aztecs use the gold in dentistry from 2000 years. Serious advances and developments in the use of biomaterials in the body have been observed since the mid-19th century. For example, the first ivory prosthesis was placed in the body in 1880, and in 1938 the first metal prosthesis (vitalium) was produced. However, this prosthesis produced has been found to undergo severe corrosion in the future, and as a result, poses serious hazards to organisms. In addition, improvements in biomaterials such as the replacement of blood vessels and the development of artificial heart valves (1950), the use of hip prostheses (1960), the introduction of alumina and zirconia-based implants (1970).

In particular, thanks to developments in the past 30 years, biomaterials today are able to perform the function of a damaged or damaged organ or part, such as a hip connection implant, kidney dialysis machine, and artificial heart, to aid in the healing of damaged tissue such as bone screws and sutures, pacemakers and they are preferred for the purpose of enhancing the function of an organ such as eye lenses and for diagnosis and improvement of probes and probes. Figure 2.1 shows the biomaterials which are utilized in the human body.

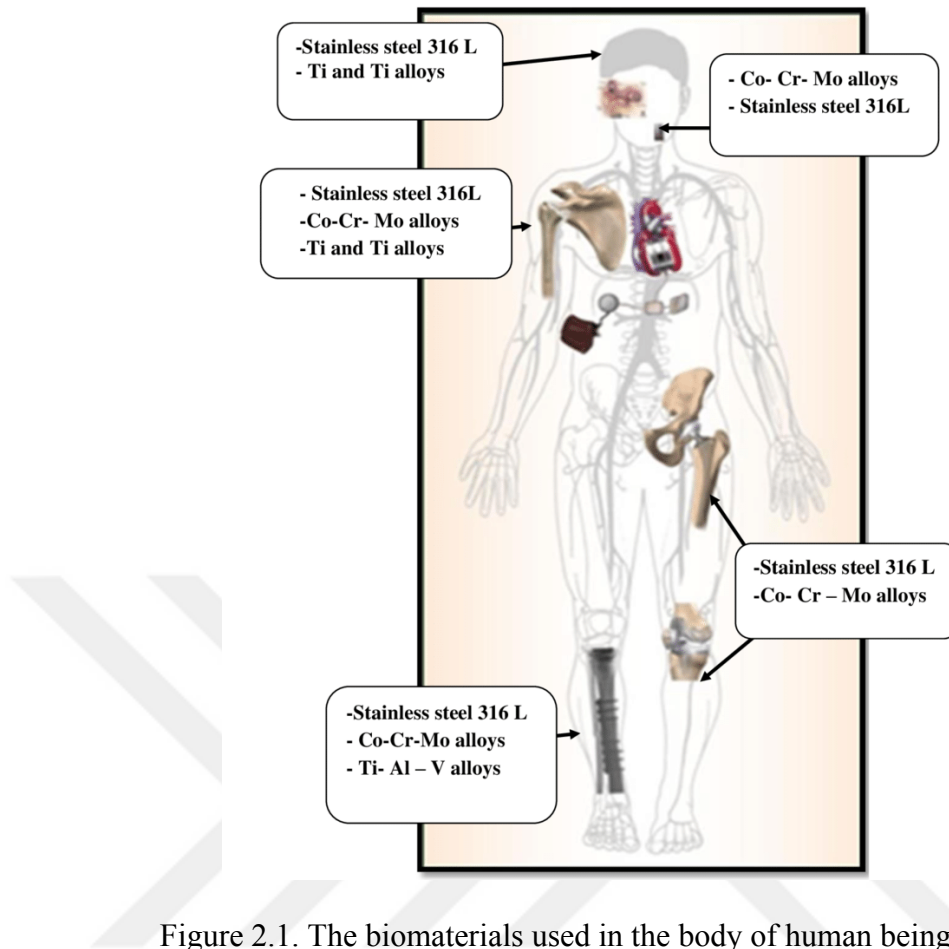


Figure 2.1. The biomaterials used in the body of human being [20].

2.2. BASIC FEATURES EXPECTED FROM BIOMATERIALS

Basic properties including biocompatibility, modulus of bone elasticity and high mechanical properties, corrosion resistance and proper design are expected from biomaterials [21, 5]. The most significant characteristic expected from biomaterials is biocompatibility. Biocompatibility means that the biomaterial used does not harm the body as a result of reactions occurring within the body. A biomaterial should not damage the surrounding tissue, interfere with its normal development and should not cause unwanted reactions (infection, clot formation, etc.) in the tissue. When a biocompatible implant material is implanted in the body, the tissue may accept, reject or retain the implant material with some complications. The main purpose, no complications or it is the acceptance of the biomaterial by the body before it occurs [22]. The new invented biomaterials may subject to exhaustive biological tests before they are released to ensure that they do not cause an undesirable effect on the body.

The most important feature of biomaterials after biocompatibility is that it has a modulus of elasticity and mechanical properties close to bone. These properties are important in orthopedic implants and dental implants because they are exposed to the most load in the body. The mechanical properties of a biomaterial that function as bone, such as modulus of elasticity, strength and toughness, should also be close to bone tissue. Otherwise, the amount of bone load increases with time and causes bone tissue to weaken over time. This is also referred to as the stress concentration. In contrast, the implant material increases the possibility of fracture over time [9, 10].

2.3. CLASSIFICATION OF METALLIC BIOMATERIALS

Biomaterials produced from metals and alloys are the group of biomaterials that best adapt to the mechanical conditions of the musculoskeletal system of the body. Metallic biomaterials, heavy, variable and sudden loads are preferred because of their mechanical properties can withstand long-term change and loss [67].

Implants manufacture use the metallic biomaterials where high tensile strength is required. Besides the biological, physical and chemical properties for these biomaterials, they have to have different desired properties. These are fatigue strength, suitable modulus of elasticity, isoelastic (such as human bone), high tensile and yield stress, sufficient elongation, high fracture toughness and wear resistance [68].

These biomaterials are classified according to the metal or alloying elements they contain. Biomaterials most commonly used as pure or alloy; Co, Ti, V, Al, Cr, W, Mo, Ni in different combinations of alloys and stainless steels. Metals are rare in their pure form because of certain properties that they cannot provide by themselves. Therefore, the use of alloys is more common and appropriate, since it can provide the expected properties with the addition of other alloying elements. Although alloys are more convenient to use, they may not meet all of the desired properties. Most commercial metallic materials are alloyed. The three basic metal groups used today in orthopedic surgery (titanium-based alloys, cobalt-chromium and stainless steels) and their derivatives are alloys. Pure titanium, stainless steels, Co-Ni-Cr-Mo-Ti, Co-Cr-WNi, Ti-Al-V alloys and Co-Cr-Mo are the most extensively used biomaterials.

Table 2.1. Elements used in metallic biomaterials [17].

Element	Symbol	Atom number	Atomic weight	Use of
Aluminum	Al	13	26,98	Alloying element
Kobalt	Co	27	58,93	The basic element
Krum	Cr	24	52,00	Alloying element
Iridium	<u>Ir</u>	77	192,2	Alloying element
Iron	Fe	26	55,85	The basic element
Manganese	Mn	25	55,94	Alloying element
Molybdenum	Mo	42	95,94	Alloying element
Nickel	Ni	28	58,71	Alloying element
Niobium	<u>Nb</u>	41	92,91	Alloying element
palladium	Pb	46	106,4	Alloying element
Platinum	<u>pt</u>	78	195,1	The basic element
Tantalum	Ta	73	181,0	Alloying element
Titanium	<u>Ti</u>	22	47,90	The Basic element
Tungsten	W	74	183,9	Alloying element
Vanadium	V	23	50,94	Alloying element
Zirconium	<u>Zr</u>	40	91,22	Alloying element

2.3.1. Stainless Steels

Generally, stainless steels is classified into two main groups: alloy steel and carbon steel. Carbon steel consists of manganese, iron, silicon and carbon. As well as, it includes little amount of phosphorus and sulfur. Steels with a carbon content of less than 1%, including nonmetals and other metals, are named alloy steel. If compared with carbon steels, alloy steels are considered more difficult to process and costly. Nevertheless, their thermal resistance and corrosion are higher than for carbon steels. Alloy steels, Al, Cr, Ni, Co, Mn, Mo, Si, Ti, W, V as well as elements such as Cu, S and P may contain a small amount. The addition of Al to alloy steels increases the wear resistance, while the addition of high amounts of Cr increases the corrosion and thermal resistance of the alloy. These alloy steels are named “stainless steel. 316L is an extensively used (steel grade) biomaterial. “L low means low carbon content. The

structure of 316L, which was arranged by decreasing the carbon content of 316 stainless steel (0.08% to 0.03% by weight) in the 1950s, Fe, 11th. It consists of Cr and Ni. In addition, these steels contain a small amount of N, Mn, Si, S, P and Mo [23].

The most common austenitic stainless steels used in implant making as biomaterials are 316 and 316L. The hardenability of these steels is not possible by heat treatment, but by cold treatment. The addition of Ni to the alloy stabilizes the austenite phase and increases the resistance of corrosion. A major disadvantage of stainless steels is the release of metal ions when they stop in the body for a long period time. In particular, stainless steels are used in hip prostheses, connecting wires, bone plates and screws [24].

2.3.2. Cobalt Alloys

Cobalt alloys are also referred to as Co-Cr alloys. The two significant alloys that used as biomaterials are Co-Cr-Mo alloy and Co-Cr-Ni-Mo. Co-Cr-Ni-Mo alloy is formed by hot rolling method while Co-Cr-Mo alloy is formed by molding method. Cobalt alloy biomaterials generally have high abrasion, temperature and corrosion resistance [25]. The production of dental implants is considered the first application of cobalt alloys in the medical area. Recently, CoCr-Mo alloys are used to produce the artificial joints in spite of its use for several years in the dentistry field. Co-Cr-Ni-Mo alloy is utilized in joints where the load is high (arm and leg).

Co-Cr alloys are supplied by Co (65%). The addition of Mo enhances the mechanical characteristics of the material and reduces the grains in the structure. Increasing the amount of Cr in the structure increases the alloy resistance of corrosion against solid solutions [20].

2.3.3. Titanium and its Alloys

2.3.3.1. History of Titanium

William Gregor discovered the titanium for the first time in 1791. Four years later, Martin Klaproth called this element Titanium, inspired by the story of the Titans in Greek mythology. More than a hundred years were required to obtain titanium metal from titanium oxide. The first alloys of titanium were developed in the late 1940s. The most frequently used alloys of titanium in the markets is the Ti-6Al-4V alloy. In the years that followed, a lot of work has been done to improve the density and characteristics of Ti-6Al-4V alloy and it is being continued [10]. Titanium has been used in the production of biomaterials since the late 1930s [69]. In addition, titanium is a material which has a lower density (about 56%) than steel and has a high specific strength.

Titanium alloys have been utilized as biomaterials in orthopedic applications for many years because of their easy to produce small sized parts, high mechanical properties, biocompatible, low density, corrosion resistance and most importantly, because the elasticity modulus is one of the metal materials adjoining to the elastic modulus of the bone [26, 27].

It was stated that the oxide layer designed on the surface of titanium implants having a tight package hexagonal lattice structure, melting temperature of 1668 °C and a density of 4.5 g / cm³ was similar to titanium oxide at room temperature. Furthermore, this oxide layer has been reported to alter the structure of oxides at the metal-oxide interface [28].

Recently, titanium alloys and titanium are used in many medical applications. Traditionally, the use of titanium has been concentrated in aerospace, aviation and marine applications. The durability and robust structure of titanium, high heat resistance, high corrosion resistance and low density increase its use in these areas. With the development of titanium processing methods in the last 30 years, its use in the field of biomedical products has increased. Today, titanium alloys are used in the

medical field to produce medical materials such as joint prostheses, splints, stents and binders, dental implants, crown bridges and partial dentures. Different alloying factors are used to enhance the mechanical characteristics of titanium. The most commonly used elements for this purpose are aluminum, vanadium and iron. There are three different titanium alloys in addition to pure titanium as defined by international ASTM standards. These alloys are Ti6Al4V, “extra low backlash” Ti6Al4V and TiAlNb [29]. Titanium metal is considered a very sensitive metal and forms a quick protective oxide layer on the surface. High corrosion resistance is because of this rapidly forming oxide layer. Low-density and high-strength prostheses. Titanium alloys form a significant engineering materials class in the aerospace field because of the excellent mixture of properties including low density and high strength fracture toughness [70]. Furthermore, titanium alloys are commonly used in many industries including aerospace, biomedical, automotive and petroleum industries because of its outstanding corrosion resistance, high strength, low elasticity, low density and biocompatibility modulus [30].

Chip removal is the process of removing the chip from the workpiece surface in small particles by deforming the workpiece surface using cutting tools. In this way, the part comes in the desired shape. A workpiece can be inserted into any shape with a variety of methods. However, the main issue here is whether the workpiece is brought to the desired shape, surface quality, time spent for it, cutting or shaping tools, costs and expectations are met [31].

During tool removal, all tools wear and this wear continues until the tool life end. Tool life; this is the time during which the cutting edge of the tool continues to lift the workpiece by removing the desired dimensions within the tolerance limits of the workpiece. Using the correct cutting tool during chip removal is important to ensure maximum productivity. The choice of tool material and cutting geometry is particularly important. However, if the tool was selected correctly it is not possible to achieve optimum tool life even if proper processing conditions cannot be achieved [32].

For the preservation of cutting or shaping tools and achieving ideal surface quality, the cutting fluids, which have been used for nearly 200 years in chip removal, consisted of simple oils that were first applied by brush to cool and lubricate the tool. In order to increase lubrication, animal fats were sometimes added to these fats. In the early 20th century, F.W. Taylor in his studies with tool steels, concluded that using some water increases tool life. Subsequently, in parallel with the development of metal cutting techniques, machine designs, and cutting tool materials, the coolant formulation became more complex [33].

Today, energy and time are the two most important factors in all enterprises in the industry. Producing the most production possible in less time and with less energy is one of the most important factors for the sustainability and profitability of that business. Accelerating the production within the desired tolerance limits and surface quality of the products in the machined industrial enterprises will increase the productivity and profitability of the enterprises.

In the light of this information, in this thesis, it is intended to decrease machining times by using high cutting speeds in the turning process of Ti-6Al-4V alloy that is the most common use among titanium alloys having excellent properties. However, the literature illustrations that raising the cutting speed decreases the life of the cutting tools used and also adversely affects the surface quality. In this research, it was tried to regulate the optimum critical conditions by eliminating these handicaps. For this purpose, tool life and surface quality were evaluated in cutting conditions which were formed by using different coolant / lubricant fluids at different cutting speeds. Table 2.2 shows the chemical arrangement of the Ti6Al4V alloy that used in the construction.

Table 2.2. Chemical arrangement of Ti6Al4V alloy [71].

Yield Strength. (MPa)	Tensile Strength (MPa)	Elongation Ratio (%)	Shrinkage Rate %
765	860	10	25

Table 2.3 displays the mechanical characteristics of the Ti6Al4V alloy used in the construction.

Table 2. 3. Mechanical characteristics of Ti6Al4V alloy [71].

Element	V	AL	C	H	Fe	O	N	Ti	Other
%	3,5-4,5	5,5-6,5	0,08	0,0125	0,25	0,13	0,05	Remaining	0,1-0,4

2.3.4. Illustration of Some Phase Diagrams of Ti Alloys

The phase diagrams of industrial Ti alloys includes three important areas: α phase region, β phase region and $\alpha + \beta$ phase region. Figure 2.2 shows the Ti-Al phase diagram. In the Ti-Al phase diagram, it is seen that $\alpha + \beta$ phase region is formed by the effect of Al expanding the α phase region. The β conversion temperature rises depending on the amount of Al. The melting temperature of the alloy increases when Al increases by up to 6% by weight. The stability zone and the conditions required for the formation of Ti₃Al intermetallic which is known to exist in various Ti alloys is another important phenomenon in this phase diagram [71].

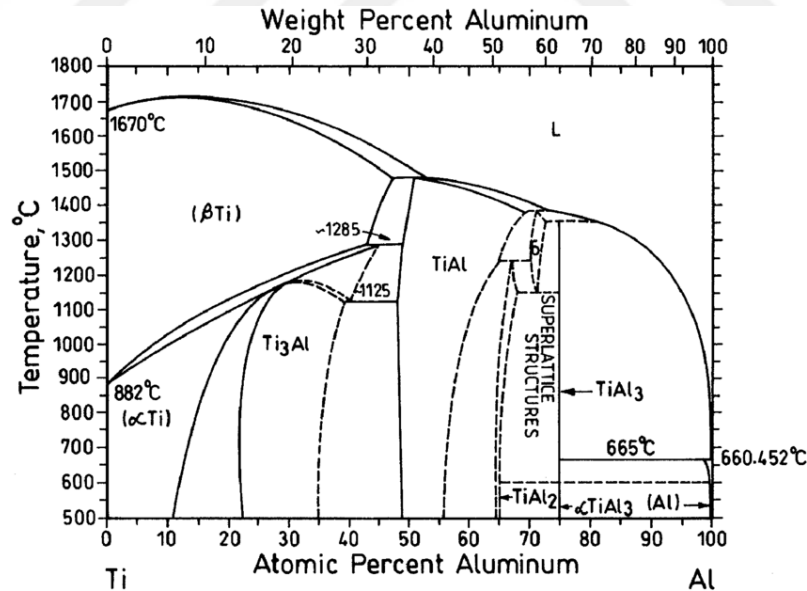


Figure 2.2. Ti-Al phase graph (ASM, 1992) [71].

Figure 2.3 shows the Ti-V phase diagram. In the Ti-V phase graph, with the effect of V, the α phase region shrinks, while the two-phase $\alpha + \beta$ and β phase regions expand. The increased amount of V up to the eutectoid composition decreases the β conversion

temperature. Due to the chemical proximity and high solubility of these two metals, intermetallic formation is not observed [10].

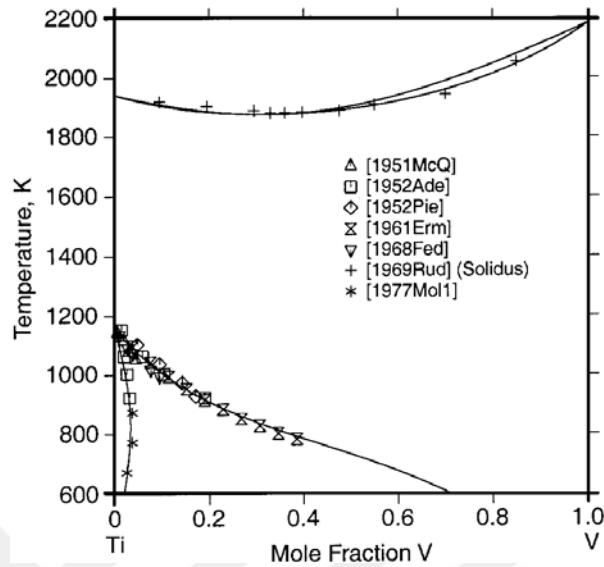


Figure 2.3. Ti-V phase graph (ASM, 1992) [71].

Figure 2.4 displays the Ti-Al-V phase graph. The Ti-Al-V system is converted to Ti-6Al-V and reduced to a binary system and the β conversion temperature decreases as the amount of V increases. This shows that despite the α -forming effect of Al, the β -forming effect of element V is dominant. While the β conversion temperature of pure Ti is 882 °C (Figure 2.3), in Ti6Al4V composition, this temperature rises to 985-995 °C. With the isomorphous effect of V, an expanding $\alpha + \beta$ and β phase region emerges and the α region narrows [10].

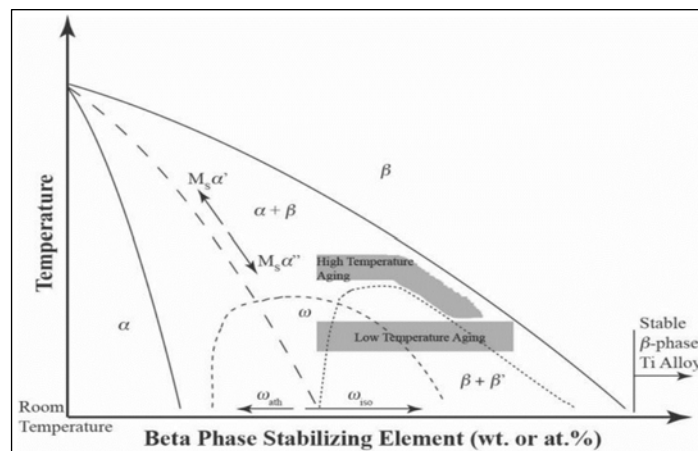


Figure 2.4. The sub-eutectoid region of the Ti-Al-V ternary system transformed into a binary system between Ti6Al and V [10].

2.3.5. Developments Resulting from Titanium Alloys

The Ti6Al4V alloy in $\alpha + \beta$ phase Ti alloys was first developed as structural material and then started to be used as biomaterial. The β phase stabilizer causes the formation of vanadium toxic wastes. Therefore, instead of V in Ti6Al4V alloy, Fe, a β phase balancing element, was added to the composition and Ti5Al2,5Fe alloy with similar characteristics has been used in implant applications. Mark and Niiomi studied Ti5Al2,5Fe $\alpha + \beta$ and Ti6Al7Nb type alloys by the use of niobium and iron in place of vanadium because of the negative impacts of vanadium. In their studies, despite the fact that these alloys exhibit tribologically and mechanically similar properties to the Ti6Al4V alloy (due to the presence of aluminum in its content), they have indicated that the problem for biomedical applications still persists [34].

New $\alpha + \beta$ alloys such as Ti5Al2,5Fe and Ti6Al7Nb have been constructed. Similarly, various biomaterial titanium alloys such as Ti6Al6Nb1Ta and Ti6Al2Nb1Ta have been developed. Subsequently, vanadium or aluminum-free Ti15Zr was introduced in alloys such as Ti15Sn. Since the elasticity modulus of biomaterials is greater than natural bone, it causes stress accumulation in the bones. Modulus of elasticity of Ti6Al4V alloy, cobalt alloys and stainless steels.

However, the elasticity modulus of natural bone is less than that of Ti6Al4V. Modulus of elasticity for $\alpha + \beta$ alloys is higher than β alloys. Therefore, β phase Ti alloys such as Ti13Nb13Zr and Ti15Mo have been developed [35]. Júnior et al. In their study, Ti-15Mo alloy produced by the results of cytotoxicity and cell viability tests stated that the alloy gives good results [36].

2.3.6. Titanium Alloys and its Properties

Generally, titanium alloys are commonly existed in food maritime and industries, chemical units and medicine and aerospace industry. Titanium-based products are high-cost materials, such as the need for complex processes for extraction and separation, the absence of a large number of mines, and consequently the insufficient investment of the facilities required for extraction and separation, and the consequent

excess production costs. However, titanium, which has high production costs, maintains its charm thanks to its superior properties. The superior properties of titanium can be listed as follows [72]:

- Excellent low temperature resistance
- High precise strength
- Low precise gravity
- High resistance of temperature
- Low current conductivity
- High resistance against thermal shrinkage
- Excellent corrosion resistance
- Very low magnetism
- Over conductivity (seen in Ti-Nb based alloys),
- Hydrogen absorption,
- High chemical reactivity

In titanium alloys researches, researchers focus on β alloys. Features including better adaptation to bone tissue give better results compared to alloy types α and $\alpha + \beta$, increased corrosion resistance and lower modulus of elasticity [9, 37]. Binary Ti-Mo alloy systems are studied by several researchers and in their studies, micro-structure of the two Ti-Mo alloys, they stated that relies on the amount of Mo in the alloy [38, 4, 39]. However, in a study of Ho, Ti15Mo alloy made a comparison between pure-Ti, Ti7,5Mo, Ti15Mo and Ti6Al4V according to the corrosion resistance and mechanical characteristics. The elasticity modulus of Ti7.5Mo is lower than of this alloy, although it is smaller than pure Ti and Ti6Al4V. However, Ti15Mo alloy among these alloys, has the best corrosion resistance stated [40].

Yalçın and Varol examined the mechanical properties of Ti6Al4V and Ti5Al2,5Fe alloys used as aerospace and biomaterials by powder metallurgy method. As a result of the studies, Ti5Al2,5Fe alloy produced under the same conditions, Ti6Al4V alloy has a structure that is more durable and more ductile properties indicated [73].

Investigated the characterization of Ti6Al7Nb alloy used in dental applications. Weight loss was measured during the wear test of Ti6Al7Nb alloy produced by pure titanium and casting method. The weight loss of Ti6Al7Nb alloy was found to be low. When the results were examined, it was found that the mechanical strength and wear resistance of the Ti6Al7Nb alloy produced by casting method were suitable for dental prosthesis production [41].

Choubey et al. in their study, some of the important biomaterials commercial titanium, Ti6Al4V, Ti5Al2,5Fe, Ti13Nb13Zr and Co28Cr6Mo examined the tribological behavior. In their study, they carried out tests in Hanks balanced salt solution against bearing steel over 10 N load and stated that the lowest friction factor was obtained with Ti5Al2,5Fe and steel pair and the coefficient of friction was high for other titanium alloys. In addition, they stated that abrasive wear mechanism occurs in all alloys [42].

Iwabuchi et al. studied the wear behavior of Co29Cr6Mo, Ti6Al4V and AISI304 stainless steel alloys in the Hanks solution. According to the data obtained, Ti6Al4V alloy shows good friction resistance compared to standard AISI 304 stainless steel alloy, but it is weak due to wear [43].

2.3.7. Commercial Purity Titanium

Commercial purity titanium, which contains 99.5% to 99.635% titanium, is in the strict order hexagonal structure, ie, in the α phase and transforms into the volume-centered β phase at 885 °C. However, there is also a small temperature range in which both phases coexist during this transformation [44].

In general, commercial purity titanium is desired in the applications which do not need high strength but the resistance of corrosion is at the forefront [45].

2.3.8. Titanium Alloys According to Phase Structures

There are three main groups to grouping α , β alloys and $\alpha + \beta$ alloys. The whole structure of α alloys constitutes the α phase whereas the structure of β alloys is

generally β phase. This structure is reached by cooling from the dissolution temperature. $\alpha + \beta$ alloys include both β and α phase at room heat. Alloys comprising more than α phase in their structure are named near-alpha alloys, and the names super-alpha or lean-beta can be used for these alloys [46].

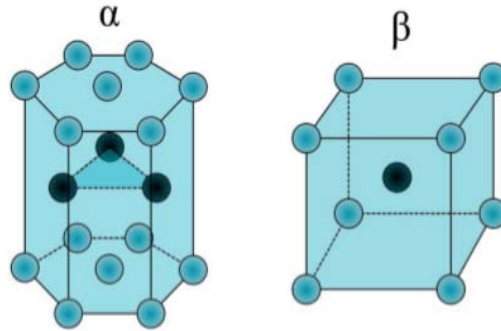


Figure 2.5. Micro internal structure of titanium alloys. The α phase is hexagonal tight package and the β phase is volume centered cubic [47].

2.3.8.1. α Titanium Alloys

The well-known α alloys all include 5% Al and 2.5% Sn. Both of these present solid melt strength to α . These alloys maintain their strength at high temperatures. Besides, it has good corrosion and oxidation resistance and weldability. It also has good formability properties. Alpha alloys are hardened at high temperatures in the β -zone and then cooled. Fast cooling provides a very fine needle-shaped α grain structure. Cooling in the furnace is more like a plate forms the internal structure [74]. Alloys of Ti-6Al-2Sn-4Zr-2Mo and Ti-8Al-1Mo-1V are in the α titanium alloys group.

2.3.8.2. β Titanium Alloys

The addition of a large amount of vanadium and molybdenum produces a completely β -structure at room temperature. However, at these amounts, no one of the so-called β alloys are really alloyed. These alloys are rich by β stabilizers. Rapid cooling therefore forms an unstable phase, all of which are β . Strength is the result of solid melt strengthening. Its alloys can be aged to provide high strength. High hardness, cold formability and high corrosion resistance are general characteristics [48]. Applications

include high strength fasteners, beams and connections in space applications [74]. Ti-15V-3Cr-3Al-3Sn and Ti-10V-2Fe-3Al alloys are in the group of β titanium alloys.

2.3.8.3. $\alpha + \beta$ Titanium Alloys

With the appropriate balance of α and β stabilizers, a mix of α and β is generated at room temperature. It forms a mixture of good hardness, identical characteristic and high ductility. More alloyed α - β alloys are heat treated to get high strength. The alloy can be dissolved at a temperature close to the conversion temperature to β . The alloy is then rapidly cooled to form an unstable excess saturated solid melt β' or titanium martensite α' . The alloy is then aged or tempered at temperatures close to 500 °C. These low density materials maintain their corrosion resistance and strength even with the high temperatures. Landing gear and parts, jet engines, Aircraft skeleton and rockets for these are distinctive applications in temperature treated alpha-beta alloys [74].

The most properly used Ti-6Al-4V alloy fits to this group. The three-dimensional phase diagram of titanium alloys is shown in Figure 2.6. This diagram displays whether the structure containing Al and V will be α and β .

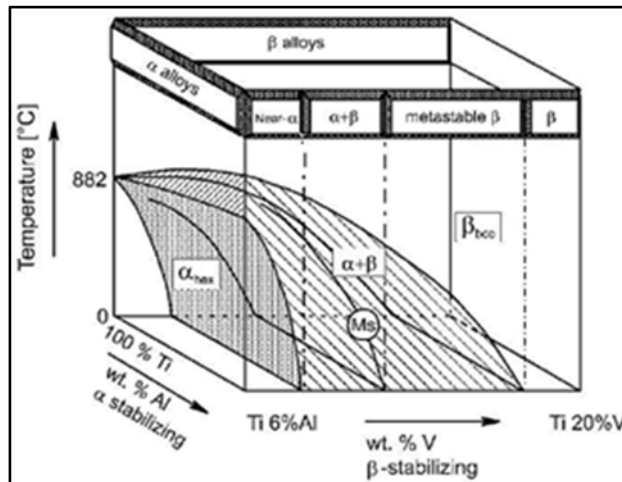


Figure 2.6. Three-dimensional phase graph of titanium alloys [10].

2.3.9. Ti-6Al-4V Alloy

Ti – 6Al – 4V alloy represents more than 50% for total production of titanium [10]. It is the most properly used titanium alloy and belongs to the $\alpha + \beta$ alloy group. Ti-6Al-4V alloy contains α phase with 6% aluminum stabilizer and β phase with 4% vanadium stabilizer [75]. Since this alloy contains more α stabilizers (Al), α is located in the near region in the phase diagram. Aluminum stabilizer with α stabilizer provides solid solution strengthening. In order to neglect the configuration of the α_2 phase, the aluminum content is generally kept around 6%. This α_2 phase causes a decrease in ductility, corrosion resistance and toughness of the Ti-6Al-4V alloy. Oxygen, which is always present in titanium, acts as the α -phase solid solution strengthener [49].

In this alloy, the β stabilizer vanadium (V) is insoluble in the α phase and is excreted from this phase. Vanadium therefore accumulates in small regions of the β phase. β phase, α matrix. It is regularly distributed. Since the α and β stages are different in content, these two stages are very stable against grain growth. That is, more diffusion is required for grain growth. As a result, alloys including Ti-6Al-4V have a reasonably thin microstructure and generally provide their strength from grain boundary strengthening [50].

It is difficult to process the titanium because of its modulus of elasticity, high chemical reactivity, thermal resistance and low thermal conductivity. The high temperature formed in the cutting area be transmitted slowly in the material is caused by the low thermal conductivity.

The Young's elasticity modulus (E) can be defined as the relationship between strain and stress, is about half that of most steels in titanium alloys. Although titanium alloys are chemically steady at the low temperatures, they react with cutting tool materials at temperatures above 500 ° C. The high temperature resistance means that the material is resistant to fractures at the high temperatures [47].

2.4. CORROSION AND WEAR BEHAVIOR OF TITANIUM AND ALLOYS

2.4.1. Corrosion Behavior of Titanium

Titanium is used in many fields due to its mechanical properties. In addition, titanium metal exhibits superior corrosion resistance. Titanium metal is naturally a very reactive metal and material when left in a damp environment or in a reducing environment, a continuous and stable thin layer of protecting film is configured over the surface. This the film layer increases the corrosion titanium resistance. For instance, pure titanium can resist corrosion in seawater, body fluids and natural environmental environments. High resistance. A piece of titanium held in sea water for 18 years only color change was observed on the surface [4].

2.4.2. Effect of Corrosion on Titanium and Alloys of Media

a) Effect of Gases: Titanium has an excellent resistance to the corrosive effect of oxygen and air up to 370 °C. Between 370 °C and 450 °C, a colored oxide layer forms slowly thickening on the surface of the titanium. If the temperature of titanium alloys are above 650 °C, leaving long-term oxidation resistance they become hard because of the increased diffusion of oxygen in the metal [4].

Oxygen reacts more quickly with titanium than nitrogen. Nevertheless, at high temperatures of 800 °C, metal brittleness occurs in the material due to the excessive spread of nitrogen to the metal. Under normal atmospheric conditions, dry or liquid ammonia solutions have not an abrasive impact on titanium [76]. The oxide layer formed on titanium shows a very high resistance to hydrogen gas. The penetration of hydrogen gas into the metal occurs only when the oxide layer is mechanically or chemically damaged or removed. Also, moisture in the medium allows the oxide layer formation on the surface of hydrogen.

It prevents penetration to metal even at high temperatures and pressures. However, precipitation of the titanium hydride phase in the material can be seen by the penetration of pure and dry hydrogen into the titanium at elevated pressures and

temperatures, which leads to brittleness. Hydrogen in high temperature titanium and alkaline media in industrial applications brake formation [76].

Titanium is also resistant to sulfur gases, stress corrosion cracking in the sulfur environment and the formation of sulfur at operating temperatures in industrial applications. Table 4.2 shows that sulfur dioxide and hydrogen sulfite do not affect titanium in dry and humid conditions.

Table 2.4. Corrosion resistance of pure titanium to sulfur containing gases [78].

Gas	Temperature (o C)	Corrosion Rate (mm / year)
SO ₂ (Dry)	21-100	0.00
SO ₂ (Saturated)	21-100	<0.003
H ₂ S (Saturated)	21-100	<0.12

b) Effect of water: Titanium has excellent resistance to high purity water and body fluids. 300 o C and at temperatures of up to titanium it does not affect the water and water vapor. Pollutants such as iron and manganese oxide, sulphides, sulphates and chloride in natural water vapor and chlorination to prevent water contamination do not affect the corrosive properties of titanium against water [76].

c) Effect of Halogen Compounds: Titanium alloys show excellent corrosion resistance to iodine, bromine, liquid chlorine and other chlorine compounds. Due to these properties, it replaces stainless steel and other metals which have been used against various regional corrosion and stress corrosion in many chloride and bromide-containing environmental conditions. The oxidizing effect of moist chlorine causes corrosion at low speeds in titanium [76].

Titanium; Excellent resistance to chlorite, hypochlorite, chlorate, perchlorate and chlor-di-oxide solutions. In the pulp and paper industry, no traces of corrosion were found in titanium in environments used to hold such chemicals [76].

Titanium is used in saline solutions at high temperatures and it has been observed that saline solutions with a pH value of 3-11 are subjected to very low corrosion. In environments where metallic chlorides such as FeCl_3 , NiCl_2 , or CuCl_2 are used, the resistance of titanium to corrosion continues even at lower pH. However, low pH solutions including boiling 70% CaCl_2 and boiling 30% AlCl_3 may also undergo titanium corrosion. Acids such as HCl and oxalic acid in hot environments cause great damage to titanium in terms of corrosion [4].

d) Effect of Salt Solutions: Titanium alloys have great resistance to all salt solutions practically in an extensive range of pH and temperature. High against sulphates, sulphides, borates, phosphates, cyanides, carbonates and bicarbonates corrosion resistance [76].

e) Effect of Acids: Titanium has great corrosion resistance to oxidizing acids including chromic and nitric in different concentrations over a wide temperature range. Titanium has low corrosion ratios in nitric acid environment. The corrosion resistance of titanium at the boiling point of the solution and at higher temperatures is related to the purity of nitric acid. The more metal ions the solution contains, the greater corrosion titanium resistance [76]. As well as, titanium alloys show great corrosion resistance in organic acid environments including amines, aldehydes, fluorocarbons, ethers, esters, ketones and hydrocarbons. The titanium corrosion resistance in the organic acid environment varies according to the acid concentration, temperature and presence of corrosion inhibitor in the environment [76].

Alkali containing hydroxide based solution such as titanium, NaOH , KOH . It is also highly resistant in environments. However, in high temperature alkali environments, hydrogen embrittlement occurs in titanium alloys due to the excessive diffusion of hydrogen into metal [76].

2.4.3. Gap Corrosion in Titanium

Titanium is exposed to gap corrosion in oxygen-containing saline solutions. This results in the fact that the oxygen in the saline solution is consumed faster than in the

solution and the corrosion prospective of the metal in the range region is greater electronegative than solution. The metal in the gap turns as an anode and melts at the end of galvanic cell formation. This leads to the formation of extremely positive ions at the anode that is offset by the chloride ions arrival in the gap region. The titanium chloride reaching the gap zone is unsteady and ready to react to form small amounts of HCl. These initial reactions are very slow, but within a limited volume of the crack, the pH is reduced to as low as 1. This accelerates until the newly formed corrosion potential becomes severe. Titanium has more corrosion resistance this is also seen in iodide, bromide and sulphate solutions. Increase the ambient temperature, increase the focus of chloride ions, and decrease the dissolved oxygen concentration and increasing pH values increase the sensitivity of the range corrosion. There is no gap corrosion in titanium as long as the temperature does not exceed 120 °C in natural pH solutions. At low pH values, range corrosion may occur below 120 °C [4].

2.4.4. Hollow Corrosion in Titanium

Hollow corrosion is a regional corrosion type associated with gap corrosion. These two types of corrosion are found in major passive metals including titanium, stainless steel and aluminum. The pits begin where the layer of oxide on the surface of titanium is discontinuous. Active ions, such as chlorine, condense in this region and remain there until they are replaced by oxygen in the passive film layer. Subsequently, an insoluble corrosion product, TiO₂, forms small cracks and fills into the pit. Therefore, diffusion in the rising well is prevented and the formation of acid solutions is allowed. Titanium should not be left in the iron particles both during processing and assembly.

In the construction of titanium pipes, the pit marks in the form of scratches can be seen especially in the places where iron is present, which is caused by iron particles which penetrate into the passive TiO₂ layer in some way. The difference in corrosion prospective between pure titanium and low carbon steel is nearly 0.5 V in boiling saturated saline solutions. This difference is sufficient to form an electrochemical cell where the iron is consumed in the anode. A pit is formed in the titanium and starts to grow before the iron is consumed completely. After a pit is formed, the acid medium also develops in the pit. This acid medium prevents the TiO₂ layer from regenerating

in the region and corrosion continues until the material is pierced. As a result, both during processing of titanium and during the assembly phase, loading the part as an anode or eliminating iron particles on the passive oxide layer will prevent pit formation [4].

2.5. WEAR BEHAVIOR OF TITANIUM ALLOYS

Although titanium alloys have great physical and mechanical characteristics including high corrosion resistance, low weight and high strength, they are insufficient in applications involving friction and wear. Since the strong adherence of titanium to the counter material during sliding, the friction factor during sliding is not constant and reaches very high values [16, 51].

The reasons why titanium exhibits poor tribological properties; it is due to the electron configuration of titanium, its crystal structure, and the lack of lubricants [16, 52]. The electron formation of titanium has low d-band character. Therefore, the surface of titanium is extremely reactive. Although metals with SPH structure have good friction, this is not the case for titanium. This is due to the ideal tight package ratio (c/a) of 1.633 in metals, while this value is 1.588 in titanium. This encourages the sliding of the prismatic and pyramidal shift planes as well as the base planes. Increased sliding systems increase ductility and plastic deformation on the surface to which abrasion is exposed. Lubricants are another important reason why titanium has poor tribological properties. Lubricants that are successful in most metals are inadequate when used in titanium alloys. It also reduces the impact of lubricants on the low thermal conductivity of titanium. One purpose of using lubricants is to prevent heat from friction between surfaces with high thermal conductivity. However, the low thermal titanium conductivity prevents lubricant from performing this function [16, 51].

2.6. THEORETICAL FOUNDATIONS

2.6.1. Powder Metallurgy

Powder Metallurgy (TM) is a different manufacturing method that allows the manufacture of parts close from the last shape and the production of high-tech materials-alloys [45]. TM is a manufacturing process that aims to manufacture and characterize various metal and ceramic powders and to combine these powders by applying different thermal and mechanical deformation principles and converting them into useful engineering parts [53, 54]. This conversion mainly involves the steps of producing powder, mixing the desired powders in a homogeneous manner, compressing the powder mixture in the mold and performing atmospherically controlled and required temperature-time sintering to bond the powder particles with each other. In a general statement covering this transformation, TM; It is described as the technique of producing parts by weighting homogeneous mixing of powder with different size, type, shape and compressibility feature, homogenous mixing, compressing the mixture in the appropriate medium and gaining density by converting to the desired shape or form and then sintering to increase the strength and density [55].

2.6.2. Production of Titanium Powders

Powder production, which is the first step of TM process, is produced by two methods, mechanical and chemical. These methods are also divided into many parts. The most common mechanical approaches are electrolysis, milling, gas atomization and water and mechanical alloying. Among chemical methods, reduction (hydrogen reduction, hydro-chemical reduction, carbon reduction and metal reduction, etc.) is the most commonly used method [77].

In particular, Ti and titanium alloy powders are produced by chemical reduction, gas atomization and mechanical alloying methods [45]. The techniques for producing titanium powder are summarized as follows after literature review.

2.6.2.1. Atomisation

Today, 80% of the metal powders produced are produced by atomization [78]. The basic principle is that the molten metal is poured into a very fine strip and is cooled by breaking into very small particles with a water or gas jet. It is divided into three parts: atomization, melting, shredding molten metal drops into small dimensions and solidification / cooling. Air, nitrogen and argon are commonly used gases, and water is the preferred liquid. Water Atomization method; It is the most widely used method in terms of production amount due to low installation and operating costs in the industrial sense. The molten metal poured vertically from the ladle outlet is disintegrated by high pressure and specially designed water jets. With a water pressure of 6–21 MPa, a speed of 70–250 m / sec is provided per second and under these conditions 10–100 kg / min. for metal flow, there is a liquid flow consumption of 0.1–0.4 m³ / min. The particle size of the powders produced by water atomization is in the range of 30–100 μm . The gas atomization (GA) method (Figure 2.7) Is similar to water atomization in principle, but instead of water, air, nitrogen, argon and helium are used as the fluid to break down the molten liquid.

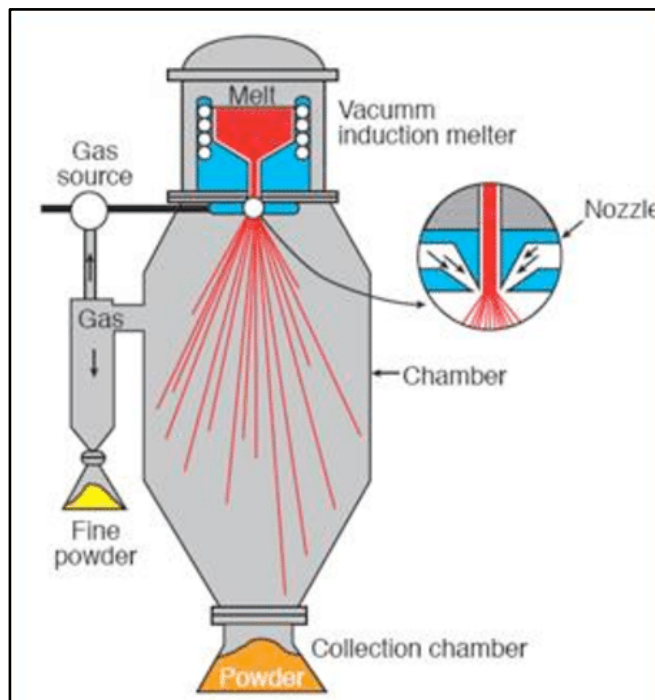


Figure 2.7. Titanium gas atomization unit [53].

In gas atomization (GA) method; similar to water atomization in principle, but instead of water as a fluid for shredding molten liquid, air, nitrogen, argon and helium gas is used. For Ti powders, a special Titanium Gas Atomization (TGA) method has been developed [45] and the solid Ti material in the melting pot is heated and melted by loading the vacuum induction current. After full melting is achieved, it is kept in the crucible to a certain time to guarantee the uniformity of the melt and long-term crucible is the most important advantage of this method. In this method, a secondary induction coil is designed in the center of the crucible for the first flow of the melt through the nozzle, allowing the melt to settle in the zone of the nozzle. The liquid metal is poured from the nozzle through the free flow into the atomization chamber. In this chamber, the high-pressure gas is formed by a high-pressure gas torch to melt the molten Ti into small pieces. In the cooling tower, the melt Ti, which becomes particulate, solidifies. In the TGA process, dust particles have a spherical structure and good free flow [45]. The TGA method is widely used to produce commercially pure Ti and α / β and β alloy powders. The size of powders produced by this method is between 20–300 μm . As shown in Figure 2.8, the grain shape is regular and close to the sphere.

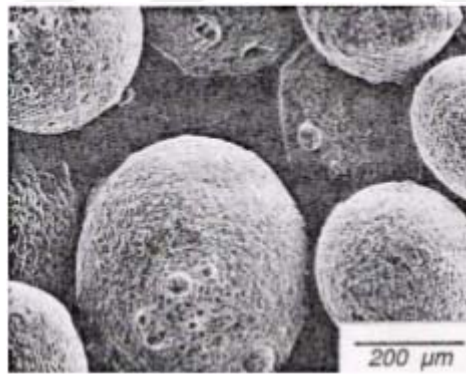


Figure 2.8. Particle shape and size of Ti powders produced by gas atomization [45].

2.6.2.2. Powder Production by Chemical Process

Powder production by chemical method, solution, chemical precipitation, chemical vapor deposition, reduction, separation, reduction and electrolysis methods. Generally, it is produced in tube furnaces by a reduction method at a reaction temperature of 1100–1200 $^{\circ}\text{C}$. In most cases the material to be reduced is solid. Very fine and pure powders can be obtained by hydrogen reduction.

Important parameters affecting the powder characteristics for the production of powder by the reduction method are described below [45]:

- Larger dust size, lower unit surface area and residual oxygen as the reduction time increases,
- Low reduction temperature leads to low dust size and high surface area, increasing residual oxygen content,
- A high hydrogen flow rate results in a high reduction rate and some oxidation during cooling with low residual oxygen.

2.6.2.3. Hydride / Hydride Method

It is a technique of producing Ti powder by reacting Ti with a reversible reaction with hydrogen. Ti has a very high chemical stability for hydrogen, and Ti can be easily hydrogenated by heating in the hydrogen atmosphere. Hydrogen can be easily removed by Ti powders heated in a dynamic vacuum. The minimum hydrogenation temperature of commercial pure Ti is made at a positive hydrogen pressure of 400°C and 0.007 MPa. As shown in Figure 2.10., b, the Ti powders produced by this method have a angular structure with an average order of 80–10 10m. Hydride / hydride (hydride / dehydride) produced by the pure Ti powders, HIP-CIP methods are used to produce alloy of Ti-6Al-6V-2Sn and Ti-6Al-4V [45].

2.6.2.4. Plasma-Rotary Electrode Method (PDEY)

PDEY method is defined as the centrifugal atomization method developed by Starmet. In this method, an arc is formed between an electrode (anode) made of Ti to be obtained powder and an insoluble tungsten electrode (cathode). It is used to melt the helium plasma Ti electrode. The molten Ti electrode is rapidly spun under a helium atmosphere and under the effect of rotation the molten Ti electrode is scattered around in powder form.

Melting and atomization are maintained in a stainless steel tank of approximately 2440 mm diameter. The electrodes are 60–65 mm in diameter and 15000 rpm. quickly

rotated. As shown in Figure 2.9, a, PDEY Ti powders are generally spherical and show good flow characteristics [45].

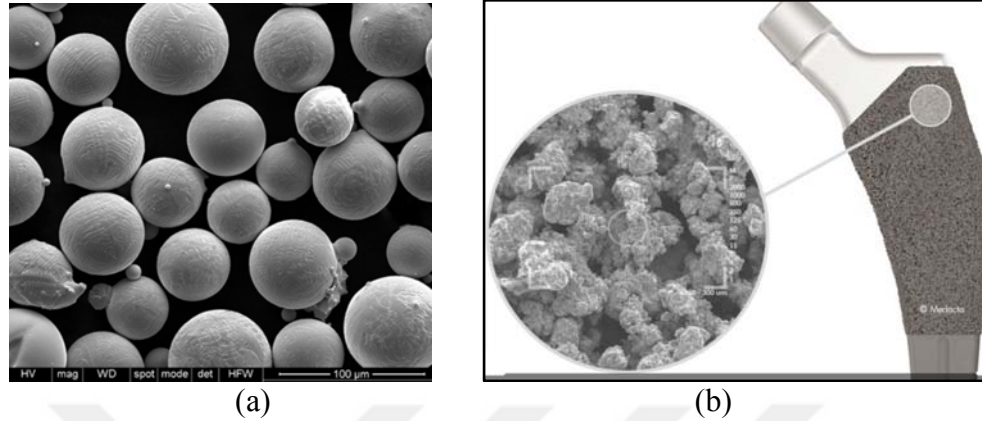


Figure 2.9. a) Form of Ti-6Al-4V powders created by PDEY method b) Porous coating with Ti-6Al-4V [45].

The powder size of particle relies on the turning speed and time and electrode diameter. The Ti-6Al-4V powders generated by this method are in the range of 100–300 μm. Compared to gas atomization, coarse-grained powders are produced. Ti powders produced by this method are generally used for porous coating of hip prostheses (Figure 2.9. b).

2.6.3. Physical and Chemical Properties of Powders

The basic properties of parts manufactured by TM method rely on the chemical and physical characteristics of the powders forming the part and the applied compression pressure and sintering processes. Powder grains are composed of a single grain. The grains may be of regular structure or amorphous structure. The beads may be single-crystalline or multi-crystalline. Even dust grains can form secondary grains. This structure is called agglomeration [45]. Agglomeration occurs unintentionally in uncontrolled powder production processes where multiple particles co-exist in a single solid state.

An important parameter used to determine the free flow of powders in the mold is the powder flow or flow rate (gr / s). In principle, it is calculated by determining how long

(sec) the powder passes at a constant weight (50 g) through a conical container. The ISO 4490 standard is used to determine the powder flow rate. The compressibility of powders depends on the physical properties of the powders. The movement of the powder during compression depends on its ability to be shaped (ie the stability of the shape and edges of the part obtained by compression) and the compression index (ie the density of the sample obtained by applying a pressure). The compressibility of spherical powders is known to be lower than that of powders having angular structure [64]. In Figure 2.10, the compression-crude strength relationship of the angular powders produced by hydrogen reduction method and spherical powders obtained by atomization method are given.

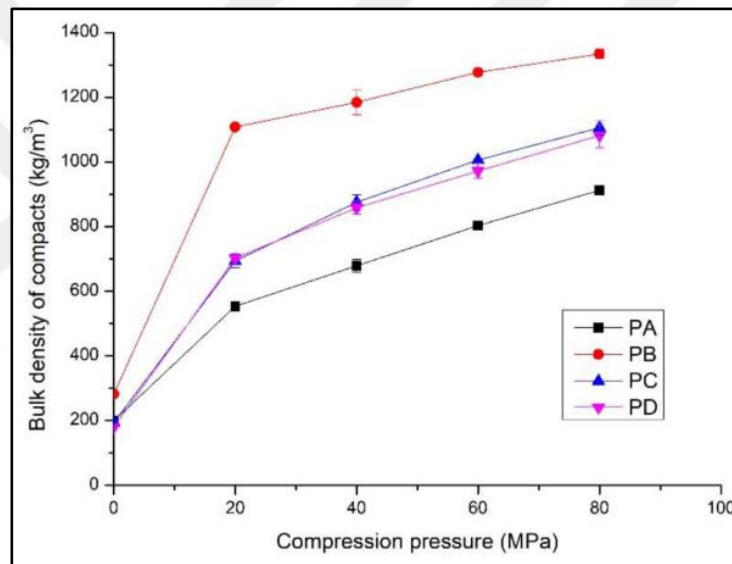


Figure 2.10. Bulk density of the compacts at different compression pressures; hydrothermal synthesis of the powders [56].

Another element for the production of metal-based powders and strength alloys is their chemical properties and purity. Purity can be determined by chemical analysis and the mechanical and physical properties of sintered bodies affect first degree [56]. The purity of metal-based powders largely relies on the purity of the alloying factors. For instance, the purity of tungsten, cobalt and iron powders obtained by the reduction of their oxides with hydrogen depends on the purity of the oxide practically used. It is also important that the factors including as carbon and oxygen are presented in the material. Metallic powders prepared by reduction of oxides generally contain uniform

oxide residues. In powders obtained by electrolysis or atomization, oxygen is generally present as oxide residue, carbon graphite, carbide or solid solution.

2.6.4. Powder Mixing

An important step of TM technology after powder production is the homogeneous mixing. It is mixed with metallic powders, lubricants and optional alloying elements to obtain a homogeneous mixture. The mixture is formed by determining the weight of the mixture elements in% based on the weight of the part to be manufactured. Generally 0.5-1.5% lubricant is added to the mixture. Zinc stearate, stearic acid, metallic stearates and paraffin are the most commonly used lubricants. In particular, binders such as paraffin, polyvinyl alcohol (PVA) are frequently used in the packaging of spherical powders. The main purpose of the lubricant is to decrease the friction between the grains of powder and the surfaces of tool and mold walls, and to allow the powders to slip easily during compression [57]. Mixing is carried out by prismatic, V-shaped and double-sided conical containers as shown in Figure 2.11.

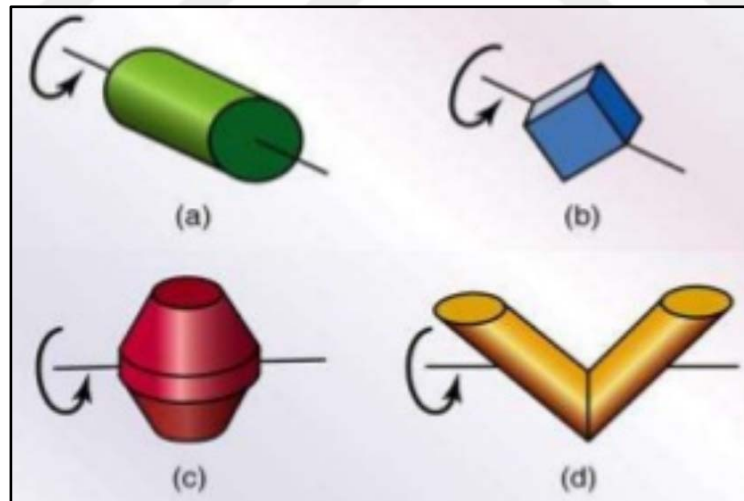


Figure 2.9. Some types of mixers used in powder mixing (a. cylindrical shape, b. Cubic, c. Double-sided conical, d. V-shaped) [53].

In mixing process, it is recommended to have a minimum filler ratio of 30-40% [53]. The mixing cycle is carried out at 20-30 rpm (Figure 2.11.-a) for 20-30 minutes. The optimum mixing process affects the raw density and the density after further sintering. If proper mixing is not performed, mechanical interlocking occurs between the

particles, resulting in a larger porous structure after sintering that leads to decline the mechanical characteristics. High-speed mixing eliminates homogeneity and can produce plastic deformation of dust particles. The higher the amount of the lubricant added, the lower crude density values (Figure 1.10.) And raw briquettes were found to undergo more deformation during stripping [53].

2.6.5. Compression

The most important and third process step in powder metallurgy is compression. Metal powders are made into briquettes by pressure effect in specially prepared mold. The main purpose of compaction (compaction) is to give the structure sufficient density to carry its own weight to convert the powder particles into the desired shape. Compression is performed in three different methods: axial, isostatic and volume deformation. Axial compression; it can be single and double acting as well as cold, hot and warm compression. Isostatic compression can be applied in two different ways: cold and hot. Volumetric deformation processes are applied as powder forging, powder extrusion and powder rolling [45]. The following parameters are taken into consideration when deciding the forming and compacting method:

- Type of powder to be used (spherical, sponge, flake) and powder manufacturing method.
- Use of binder and lubricant.
- Use of rigid mold.
- Desired properties of the material to be produced.

Generally, in industrial applications, a double-acting compression process, which is systematically clarified in Figure 2.12, is used.

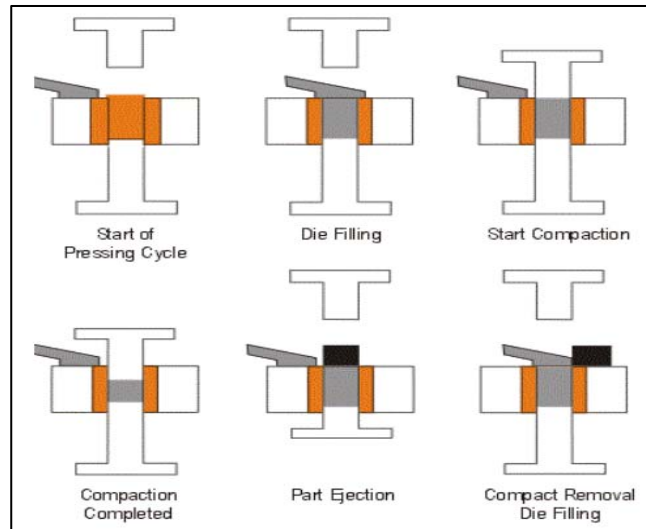


Figure 2.10. Compression steps of powdered metal parts [53].

In double-acting clamping, both punches are movable. Figure 2.13., A is the first stage, and the mold of the part to be manufactured is already designed and the mold is mounted to the press. In the second step of Figure 2.13 b, the powder particles are filled in the free flow from the mold filling shoe to the mold. In the third stage, the powder filled into the mold cavity, the vibration mechanism of the system by means of the vibration mechanism prior to compression, is achieved by placing the filled powder in the mold properly. In the meantime, increasing dust on the mold surface by the punch is pushed into the other powder chamber and the female is placed in the mold cavity to compress the upper punch (Figure 2.13.). In the fourth step, the compression is performed. At this stage, powder particles become briquettes by gaining density and strength at a certain value under the effect of compression. In the fifth step, the raw briquette with strength and density is removed from the mold cavity by the lower punch with a certain stripping force. In the sixth and final step, the raw briquette removed from the mold cavity is pushed by the powder filling lug and powder filling is performed to press the second part. As can be seen in Figure 2.13, the compression process can be handled in three metallurgical terms [53, 77]:

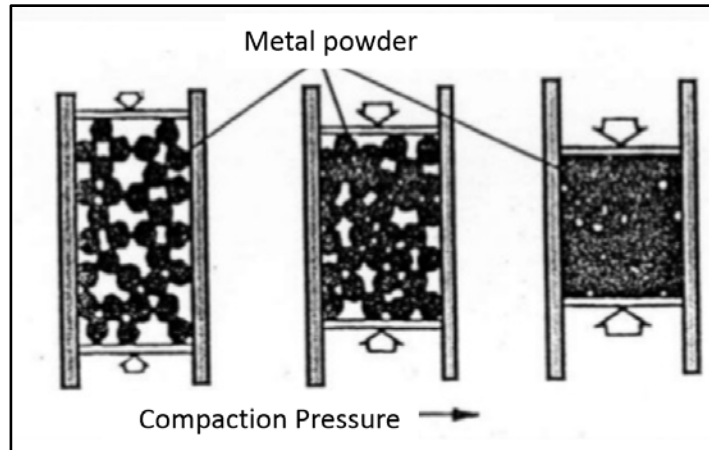


Figure 2.11. Metallurgical compression process [53].

- In the first stage, after the application of the compression pressure, the powder particles are displaced and re-determined. At this stage, there is no plastic forming, but some powder grains may break. Powder distribution of size, Particle size, shape and intergranular friction play an important role.
- In the second stage, elasticity and plastic deformation factors are dominant. Bonds may form due to cold forming between particles. In addition, mechanical interlocking of the beads and grain-to-grain interactions occur at this phase.
- In the final phase of compaction, compression pressure increases and fracture of the powder grains, filling the voids and cold welding between the powder grains occur and raw density is obtained.

The main problem in powder compaction is the friction between the powders and the mold walls. The frictional force causes the applied force to decrease in the lower region. The relation between strength and pressure-density in the powder compresses depends on the rate of strengthening of the molding factual, the surface coefficient friction and tendency of chemical reaction with the powder particles. In addition, compressibility is associated with powder shape and size, lubricant condition and compression ratio.

2.6.6. Sintering

Another important step in TM technology is sintering heat treatment. The sintering process can be defined as the transport of thermally activated material which causes the porous structure of the shaped powders to shrink to the specific surface area, to raise the particle contact zone and consequently to reduce the pore volume. In single component systems, $2/3$ or $4/5$ of the absolute melting temperature of the metal is generally taken as the sintering temperature. In multi-component systems including Ti-6Al-7Nb and Ti-6Al-4V. It is recommended that the sintering temperature, the ratio of these alloys to the melting temperature (T / T_E) of above 0.75 [58]. Some porous bronze and bronze-like alloys are between 600°C-800°C, iron group metals are between 1000°C-1300°C, hard alloys are between 1400°C-1600°C, refractory metals (molybdenum, tungsten, tantalum) are in the temperature range of 2000°C-2900 °C and Ti6Al4V is 1100 °C. It is recommended to sinter with vacuum or argon shielding gas for two hours between 1350 °C (Schatt and Wieters, 1997; [45]). The connection between the pressed powder particles is weak bonds such as mechanical locking, adhesion, and the like, which is weak relative to the bond strength in the crystal lattice system. Each particle is independent of the other, even if the compacted powder particles are in contact or permeation. By sintering, it is provided to decrease the specific surface area as a result of the growth of grain contact points, decrease or serialization of pore volume, decrease the atomic cavities that may occur in the structure, dislocation. This creates a physical bond between atoms and ions. This bond is similar to the bond in the crystal lattice system.

2.6.6.1. Sintering Mechanisms

Metallurgically, sintering takes place in six steps. These can be listed as the formation of initial powder grain bonds, formation of bridge bonds called intergranular neck, spherical pores, closure of pore channels, shrinkage of pores, and volume increase and density increase. However, for material transfer mechanism, surface diffusion, grain boundary diffusion, evaporation-condensation and volume diffusion are required [96]. Sintering process, sintering temperature and time directly, particle size and type, the amount of subsequent pore compression, pre-neutralization affects indirectly [45].

Sintering mechanisms generally accepted for solid state sintering are examined in three basic groups [77]. This situation is given in Figure 2.15.

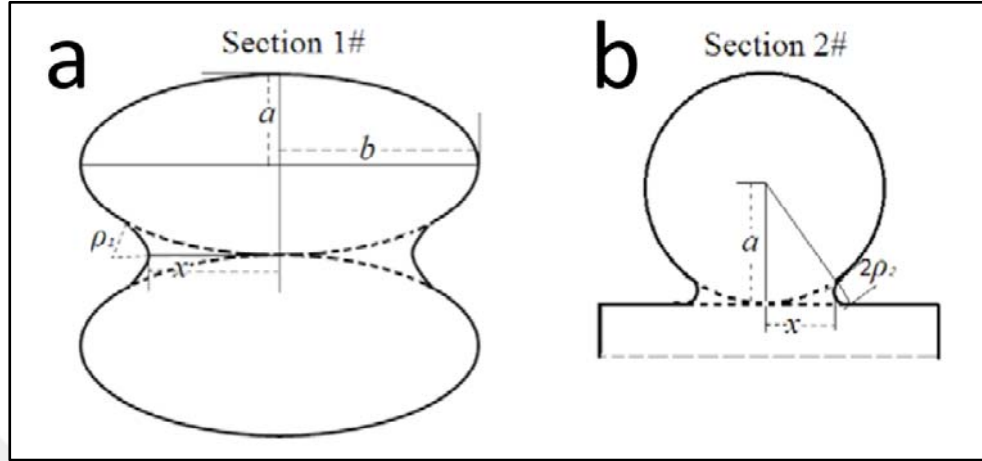


Figure 2.12. The basic model of sintering fibers neck; (a) Top vision for sintering joint; (b) Section for sintering joint [58].

- Stage:** Particle contact becomes sinter bridges called neck. At this stage, the powder particles are still independent of each other and formed by the effect of compression. However, the grain boundary begins to form on the contact surfaces between the two grains. The centers of the particles approach each other in a very small amount and form the onset of volumetric contraction (surface diffusion). Surface diffusion for contact between two is characterized by the following empirical formula [58].

$$K = \left(\frac{1}{\rho} - \frac{1}{r} \right) + 2/R \quad (2.1)$$

Here, ρ represents the radius of the sinter neck curve, r represents the radius of the sinter neck, and R is the radius of the sinter dust grains.

- Stage:** After the formation of the strength neck region between two powdered grains, if the r / R ratio exceeds a certain value, the individual grain shape disappears. A regular void network is formed and the so-called grain growth microstructure. At this stage, the majority of the binders and lubricants introduced into the powder mixture evaporate and are the stage in which

volumetric contraction is most observed (Evaporation and condensation). This stage is considered sinter neck development and there is an empirical formula that gives this expression [58]:

$$r^n F_{(s)} \chi t \quad (2.2)$$

Here, r , sinter neck radius, t , sintering time, $F(S)$, temperature dependent function and particle size geometry, n , the value of the material transfer mechanism, and according to some scientific studies surface diffusion value is between 6.5-7.5, grain boundary diffusion 6, volume diffusion value is considered to be 4-5 [58].

Stage: Closed pore rate decreases rapidly. Isolated pores turn into spherical form. The desired theoretical density in the sintered structure cannot be achieved when the gas and gas pressure generated during the removal of the lubricant and binding elements and the pressure resulting from the surface strain take the balance state within the pores. With vacuum sintering, if there is no gas in the pores or if the gas can be diffused easily from the main structure, the density increase can continue and the theoretical density can be reached to 9099% (Mass and grain boundary diffusion). Figure 2.15 shows the intergranular bond formation (sinter neck), condensed and non-condensed structures.

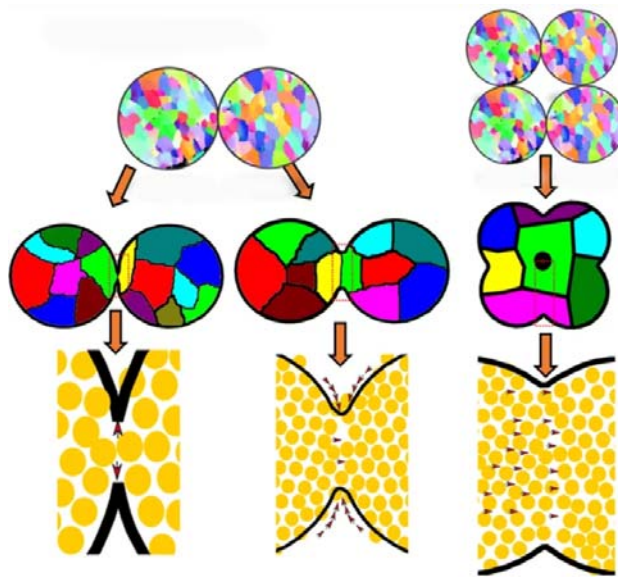


Figure 2.13. Development of intergranular bonds during sintering [79].

2.6.7. Advantages and Limits of TM Technology

In order to understand the advantages and limitations of parts manufacturing by TM method, six important parameters should be reviewed. These can be characterized as shape dimensions, shape complexity, tolerances, material systems, mechanical and physical properties, quality and cost [45]. When these parameters are evaluated, TM technology has many advantages compared to manufacturing methods such as casting and forging, but has some limitations [60]. These are described as economic and feature advantages as follows [61]:

2.6.7.1. Mechanical Properties and Economic Advantages:

- Elimination of melting losses by using 99% of all raw materials at the beginning, which is more economical and comparable in quality compared to casting and forging methods.
- To produce self-lubricated bearings by providing the desired porous structure, to produce implant materials suitable for porous bone structure.
- Reducing material consumption by minimizing secondary operation such as chip removal.
- Production of complex and small shaped parts with great efficiency compared to other approaches.
- It is the only economic method for the production of refractory metals (tungsten, tantalum, molybdenum, etc.) and hard metals with high melting temperature, which are almost impossible to manufacture by other methods.
- Microcrystalline and amorphous structures with high ductility, strength and thermal struggle can only be produced through this approach (Aircraft parts with significant structural weight).
- Good surface quality and narrow dimensional tolerance.
- Controlled pore structure, infiltration of pores according to the desired mechanical and physical properties.
- The ability to produce composite materials the above-mentioned advantages have made the powder metallurgy technique preferable to other conventional methods, especially in the realization of high quality and fast part manufacturing and meeting

the requirements. However, since each manufacturing method has its own specific application.

2.6.7.2. TM Technology has Some Advantages as Well as Its Limitations

- If mass production is not performed, pore filling equipment, which is defined as initial investment cost and secondary investment cost, such as compression and sintering units, is high in design and manufacturing costs of molds [62].
- The cost of metal powders is more expensive than ingot material
- Poor mechanical properties and variable density values compared to other methods, if no secondary treatment such as pore filling and infiltration is applied to parts requiring strength.
- Low corrosion resistance and low plasticity properties due to pore.
- Failure to manufacture large volumes of parts, design limitations or the use of new cost-effective TM techniques (SLS).

2.6.8. TM Technology Development and Application Areas

The output and development of TM technology is given in Table 2.5.

Table 2.5. Developments by years in the field of TM [45].

Year	TM Developments	Location
3000 BC	Construction of hand tools made of sponge iron	Egypt, Africa,
1200 Ms	Cemented platinum grains	South America
1822	Production of platinum powders from solid ingots	France
1826	Compression, sintering of platinum powders commercially	Russia
1830	Sintering of various metal powders	European
1870	Initial self-lubricating bearings are disclosed in	U.S.A.
1878-1900	Making of lamp filaments	U.S.A.
1915-1930	Manufacture of cemented carbides	Germany
1900	Manufacture of composites and porous materials	U.S.A.
1940	Development of iron powder technology	European
1970	Manufacture of TM tool steels, super plastic alloys with hot isostatic compression	U.S.A.
1980	Development of metal injection technology	U.S.A.
1990	Intermetallics, metal matrix composites, nano powders and hot compression technologies	U.S.A.

Egyptians, BC in 3000, they produced various hand tools made of sponge iron. The New Delhi Colon weighs 6,5 tons and is the M.S. In 300, the sponge was shaped from iron. As the first commercial development, in the early 1800's in the United States, carbon (C), zirconium (Zr), vanadium (V), tantalum (Ta) and tungsten (W) was introduced into the industry by manufacturing material such as filament and in 1826 The first industrial application of platinum money TM, which was put into circulation in Russia, was [63, 45]. All these developments show that the refractory materials such as Ti, Ta, W, V, Nb, Mo can be produced with TM technology. Another important development was the production of important composites such as electrodes, contact materials and carbide cutting tools with TM technology.

Krupp, on the other hand, tried hard metal production in 1914 and launched it in 1927. In the method of TM, great improvements have been achieved in World War I. Porous tools, magnets and impregnated iron powder parts were produced during these years. In the 1940s, copper based self-lubricating bearings were produced as TM products. From the 1950s onwards, gears, cams, connecting rods, valves and various automotive parts made of iron and steel powders have increased the use of the TM method, particularly in the automotive sector. In the 1950s and 1960s, the disadvantages of TM products compared to full dense parts were determined and intensive studies were carried out to obtain full dense TM products. Some of them are HIP-CIP, MIM, PIM and powder forging. In 1990–1995, superalloys, tool steels, carbide grade cutting tools, porous products, abrasive materials, electronic applications, alloys with fast solidification rate, most automotive parts and light metals can be manufactured with TM technology. Production has increased to one million tons [63].

An important feature of the TM technique is that it can be applied to most engineering materials that cannot be alloyed or allowed in other conventional methods. For example, it is possible to manufacture parts with aluminum, copper, titanium, niobium, iron, tantalum, zirconium, tungsten, refractory materials, superalloys, tool steels and powder mixtures that they can make with each other.

Thanks to these developments, 60% of TM technology is used in automotive parts (manifold, gear, water pump, etc.), aerospace and defense industry, biomaterials and

carbide cutting tool industry, electrical-electronic and magnetic applications, mold and bearing manufacturing. It is widely used in the manufacture of abrasive abrasives and impellers compared to other methods [55]. Carbides, nitrides, borides and aluminum oxides in the production of creep resistance parts are also produced by this method. Important industrial applications of TM in general.

Titanium has a density of 4.51 g / cm³ and is a 57% light metal compared to steel (Table 2.6.). Ti's heat transfer property, magnetism and electrical conductivity are relatively low material compared to steel [65].

Ti and alloys it is used effectively in many industrial applications where special properties are advantageous. The main reason for the rapid spread of Ti in the industry is its corrosion resistance and high strength structure despite its 57% lower density than metal. Because of their low density, it is a preferred material in weight-critical aircraft constructions and implant applications [66]. In addition, there is no limit in their ability to produce ingot, rod, sheet, shell, sponge and powder in various product forms.

Table 2.6. Mechanical properties of titanium as an element [46].

Properties of Ti	Value
Elasticity module (GPa)	120
Tensile strength (MPa)	240
Hardness (HRB)	70-74
Poisson's ratio	361
Density (g / cm ³)	4.51
Melting point (°C)	1668
Thermal conductivity ($Wm^{-1}K^{-1}$)	11.4 (at room temperature)
Electrical conductivity	According to 3% Copper (100%)
Calculated atomic diameter (meters)	$176 \cdot 10^{-12}$
Crystal Lattice Structure (α - β)	Tightly Packed if <882.5> Hexagonal-HMK if 882.5

The melting temperature is higher than that of steel and has a thermal resistance up to a maximum temperature of 538–427 °C in structural applications and TiAl alloys are used in industrial applications up to 760 °C. Pure Ti does not contain toxic and some

Ti alloys including Ti-5Al-2.5 Fe, Ti-6Al-7Nb and Ti-6Al-4V are biocompatible with human tissue and bone.



PART 3

EXPERIMENTAL STUDY

The composite samples with Ti6Al4V matrix obtained in the study were produced at the rates determined by the powder metallurgy method. The effects of Mn and Zr elements added in different proportions on mechanical and corrosion properties and changes in microstructure were investigated.

3.1. MATERIALS

In this study, Ti6Al4V powders and Manganese alloys, were used in the starting materials. The materials were produced by the powder metallurgy method. Ti6Al4V powders; titanium powder (99.7%, d <149 μm , Nanografi), aluminum (91%, d <50 μm , Nanografi), vanadium (99.5%, d <44 μm , Nanografi), manganese (99.5%, d <44 μm , Nanografi) and zirconia (99.5%, d <44 μm , Nanografi) for Ti6Al4V alloy powders were used. The Powder chemical compositions are given in Table 3.1.

Table 3.1. The powder chemical compositions.

Alloys	C	Fe	N	Al	V	Mn	Zr	Ti
Ti6Al4V	0,08	0,25	0,05	6	4	-	-	Balance
Ti6Al4V-0,5Mn	0,08	0,25	0,05	6	4	0,5	-	Balance
Ti6Al4V-1Mn	0,08	0,25	0,05	6	4	1	-	Balance
Ti6Al4V-2Mn	0,08	0,25	0,05	6	4	2	-	Balance
Ti6Al4V-0,5Zr	0,08	0,25	0,05	6	4	-	0,5	Balance
Ti6Al4V-1Zr	0,08	0,25	0,05	6	4	-	1	Balance
Ti6Al4V-2Zr	0,08	0,25	0,05	6	4	-	2	Balance

3.2. PRODUCTION OF COMPOSITE

3.2.1. Mixing

The chemical powders of Table 1 were calculated and mixed with the Mixing Machine given in Figure 3.1. Each powder mixture was mixed in the mixing device for 30 minutes. Before mixing, the balls and containers to be used were cleaned with alcohol.



Figure 3.1. Fritsch branded mixing device.

3.2.2. Cold Pressing Process

The powders were cold pressed to $\text{Ø}12 \times 7$ mm for experimental studies. Figure 2 shows the mold used in cold pressing. Pressing process was performed by applying pressure of 620MPa with Hidroliks brand press which is given in Figure 3 in Karabük University Faculty of Margem laboratory.



Figure 3.2. Metal mold.



Figure 3.3. Hydraulic press machine.

3.2.3. Sintering Process

The sintering process for Ti6Al4V alloys was carried out at a heating rate of 5 °C/min, at 1400 °C and under vacuum of 10^{-6} mbar for two hours with Protherm Brand which is given in Figure 3.4.



Figure 3.4. Image of vacuum heat treatment furnace with sintering operations.

3.2.4. Density Measurements

Density measurements were made according to the Archimedes principle on the PRECISA XB200 h brand density measuring device with a precision of 1/10000 g which can measure a maximum weight of 220 g. The density of three samples from each group was calculated and the average of these values was calculated.

3.2.5. Hardness Measurements

Hardness measurements were made in Shimadzu brand micro hardness tester (under 0.5 N load), which is shown in Figure 3.5 in Karabük University Faculty of Technology Manufacturing Engineering Laboratory. In the hardness measurements, the average of these values was calculated by making measurements from 10 different regions of three samples from each group which is given Figure 3.5.



Figure 3.5. Image of microhardness tester for hardness measurements.

3.2.5. Sample Preparation for Metallographic Examinations

In order to carry out metallographic examinations, samples were cold embedded. Fig. 3.6. Cutting operations were performed on the Struers cutting device and Metallography operations were performed on the sanding and polishing process. After this procedure, all samples were prepared by standard metallographic procedures and distributed for 60-90 sec with 1 ml HF, 1.5 ml HCl, 2.5 ml HNO₃ and 95 ml H₂O solution (Keller's).



Figure 3.6. Cutting and Grinding - Polishing Machine.

3.2.6. Wear Test

Wear tests carried out at 25°C (± 2). 8 mm bulk materials were exposed to wear along 40 m under load 60 N (6kg) with 0,5mm/s wear speed at 25 °C according to ASTM-G133 which was conducted in a tribometer. The counterbody component was ceramic ball (Al_2O_3). Before the wear test, the sample surfaces were first sanded with 1200 grit and dried with alcohol before the abrasion test. After the wear test, microstructure images of the samples were taken by digital microscope (Nikon SHUTTLEPIX Digital Microscope) and SEM (Carl Zeiss Ultra Plus Gemini Fesem). Wear behavior in terms of metal loss was examined.



Figure 3.7. Wear test machine (Tribometer).

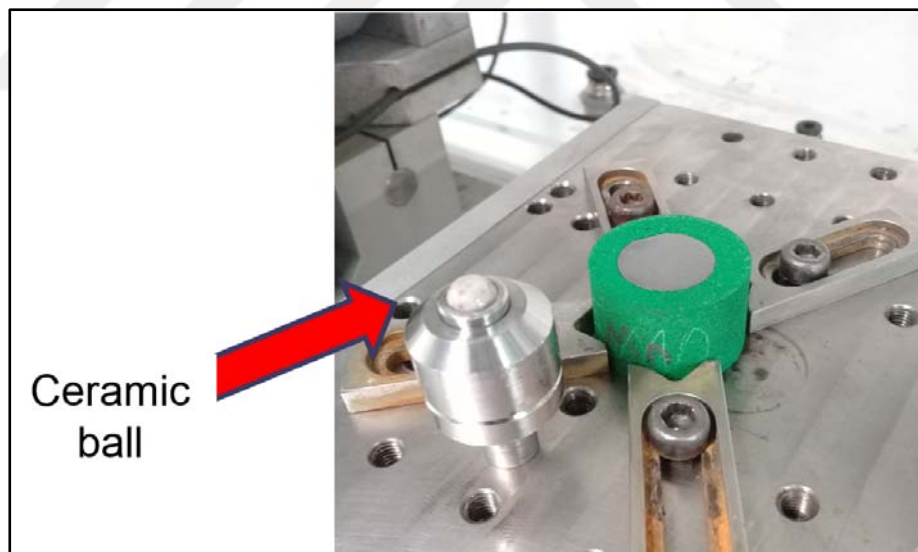


Figure 3.8. Wear test sample.

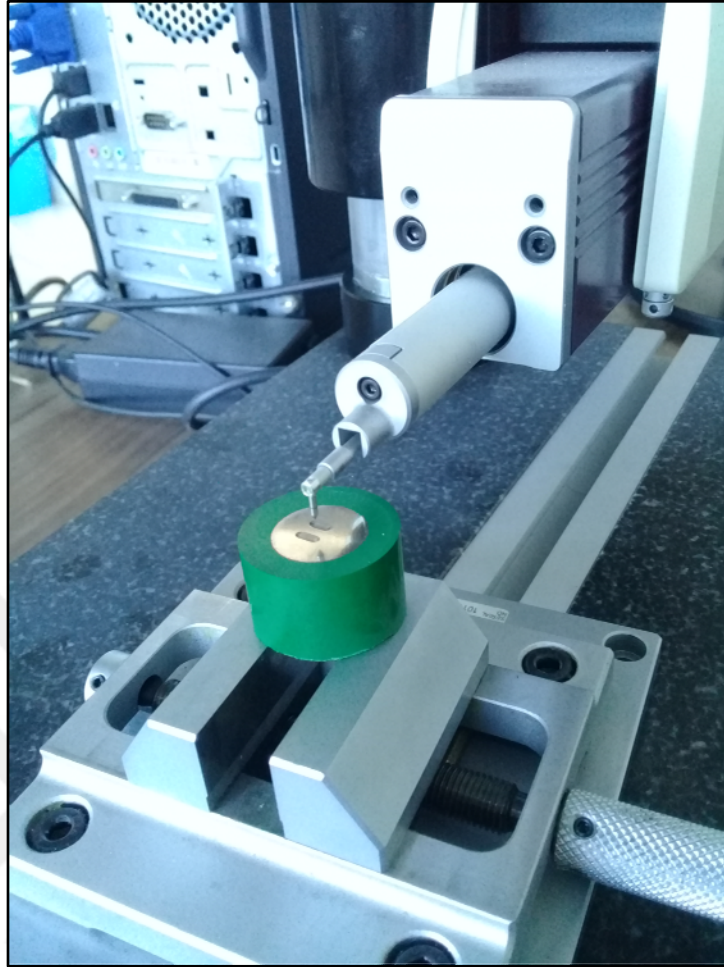


Figure 3.9. Surface roughness measurement machine.

3.2.7. Corrosion Test

Electrochemical experiments were performed for the Ti-6Al-4V-xMn and Ti-6Al-4V-xZr composite materials at temperature of $36,5^{\circ} (\pm 1)$. Potentiodynamic tests were performed with parstat 4000 potentiostat machine which based on the three electrode setup, where specimens, Ag / AgCl (3,5 mol KCl) and a pair of graphite rods are the working electrode, reference electrode and counter electrode, respectively. The sample preparation was accomplished by following procedure. Firstly, the samples was grinding by 2500 grit SiC and polished with diamond suspension of $3\mu\text{m}$. Secondly, the surface cleaning was finished by ultrasonic cleaning in acetone and deonize water. The test duration is determined as 900s at scan rate of $2\text{ mV} / \text{s}$ in scanning interval of- 0.75 V vs. Ag/AgCl to $+0.50\text{ V}$ vs. Ag/AgCl. Hank's solution [82] is applied as corrosive environment.

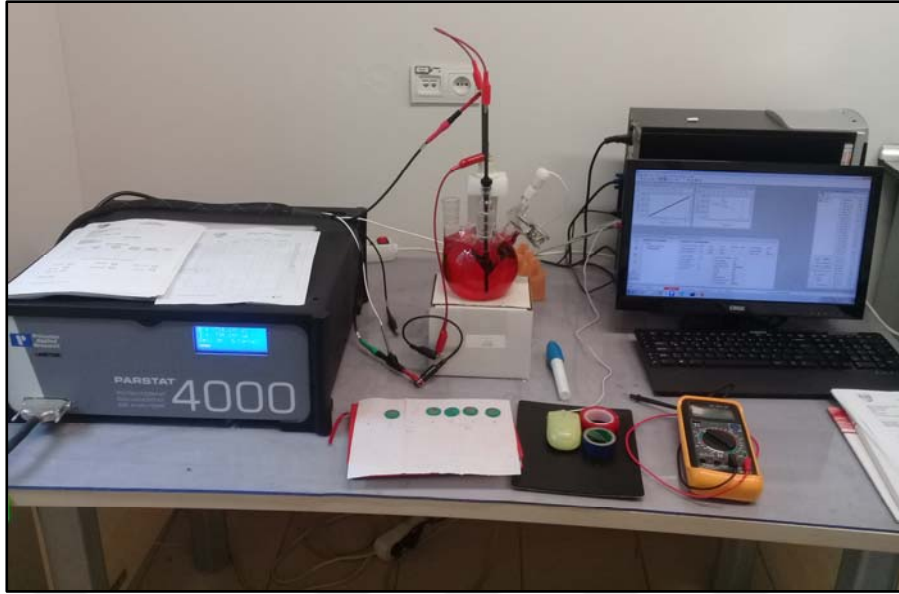


Figure 3.10. Parstat 4000 Potentiostat Galvanostat corrosion machine.

3.2.8. Scanning Electron Microscope (SEM + EDS) Analysis

The microstructure and EDS of the samples were examined by Zeiss brand Ultra / Plus (FEG) model scanning electron microscope (SEM) in MARGEM Laboratory of Karabük University Iron and Steel Institute. In addition, abrasion and corrosion tests were carried out by SEM.

PART 4

RESULTS AND DISCUSSION

4.1. MICROSTRUCTURE

The phases were determined with α and β phases for both investigated of Mn and Zr added composite materials which is given in Fig. 4.1. [83] As to all investigated composite materials, the dark region is obtained as β phase, however, the light appears as α phase. It is shown that base labelled composite material contains the fine acicular α -phases and broader β -phase are around by lath- α phases. As to adding of Mn to Ti-6Al-4V, the amount of 0,5 wt% Mn enhanced the formation of broader α -phase that encircled the equiaxed β -phase. As for the Mn, addition is 1 wt%, the microstructure is reverted to the initial micrograph of Ti-6Al-4V which has the fine α -phase placed on the boundaries of β -phase. Regarding the 2 wt% Mn addition, the β -phase formed clearly which is surrounded by lath- α phase. On the other hand, the β -phase became more dominant phase than α -phase on the microstructure of 0,5Zr labelled composite material where the formed acicular α -phase has outstretched arms along β -phase.

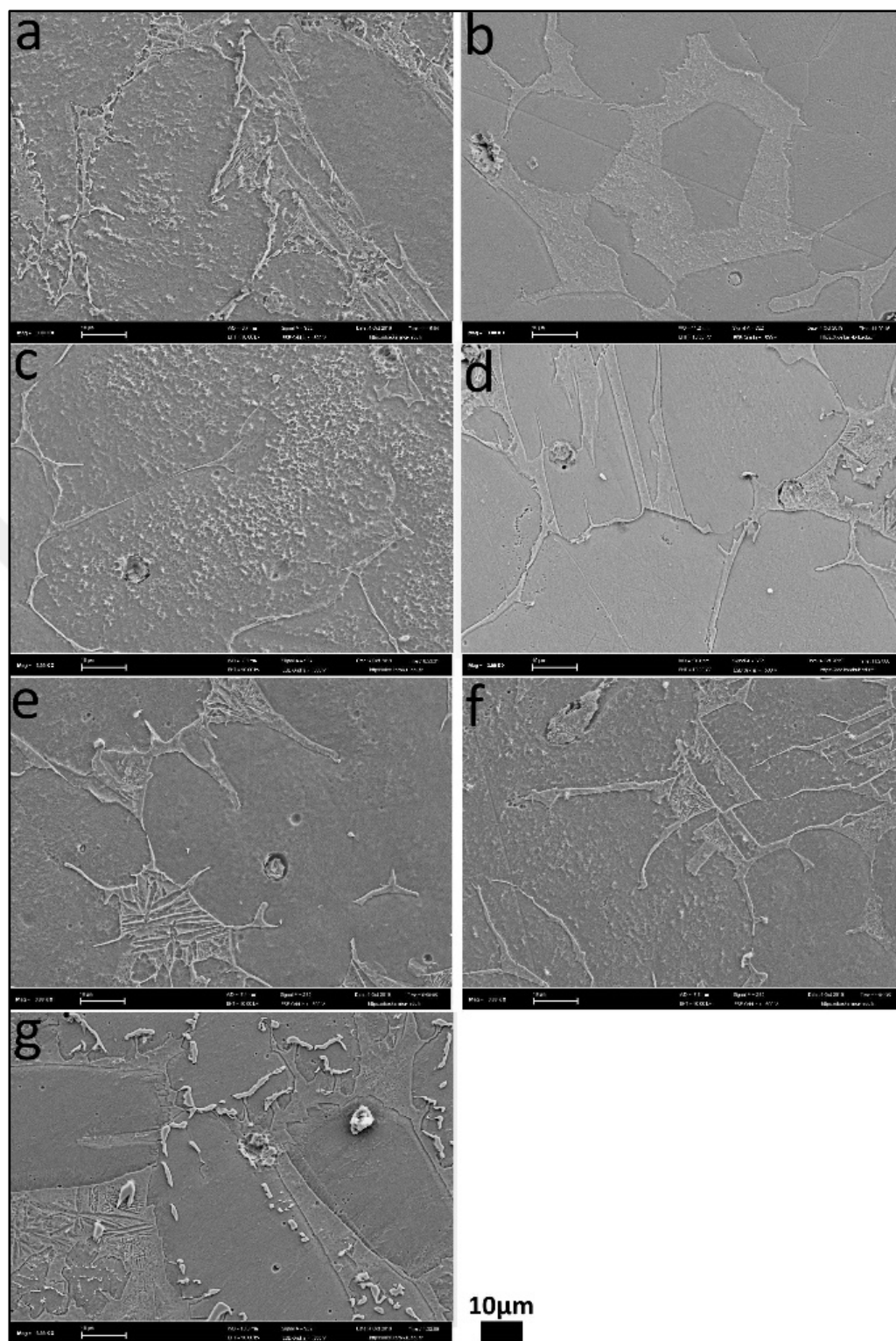


Figure 4.1. SEM micrographs of (a) base, (b) 0,5Mn, (c) 1Mn, (d) 2Mn, (e) 0,5Zr, (f) 1Zr and (g) 2Zr composite materials.

4.2. CORROSION

The potentiodynamic polarization curves enable to investigate the effect of Mn and Zr addition to the corrosion resistance of Ti-6Al-4V composite materials in the simulated body fluid environment. The potentiodynamic polarization curves for investigated composite materials that base, Mn and Zr added composite materials were illustrated in Fig. 4.2 and the test results were given in Table 4.1. As seen in Fig. 4.2, the noticeable transition region not obtained from active and passive that of curves. On the other hand, when we look at the Table 4.1, the corrosion current density (CCD) of 2Zr labelled composite materials has the lowest value of 0,004 $\mu\text{A} / \text{cm}^2$ that followed by 2Mn and base labelled composite materials that values of 0,350 and 0,526 $\mu\text{A} / \text{cm}^2$, respectively. However, the highest ones as CCD was presented by 0,5Zr and 1Zr composite materials with the values of 1,173 and 1,480 $\mu\text{A} / \text{cm}^2$, respectively. As to the corrosion rate values that are proportional to the CCD, as seen in Table 4.1. The different behaviour of corrosion resistance of investigated composite materials could have resulted from the changing of microstructure that showed distinctive phases formed based on the amount of %wt Mn and Zr. The role of the occurred phases here could be explained by the role of corrosion barrier where the homogeneously distributed phases impart to more resistance regions to corrosive attacks due to anodic and cathodic behaviour of materials was changed by these regions [85]. The addition of 0,5 %wt Mn to base changed the α phase as broader and therefore worse corrosion resistance was obtained than the base. The finer α phase gives rise to better corrosion resistance that is approved by 1Mn composite material containing both microstructure and CCD values closely with base composite material.

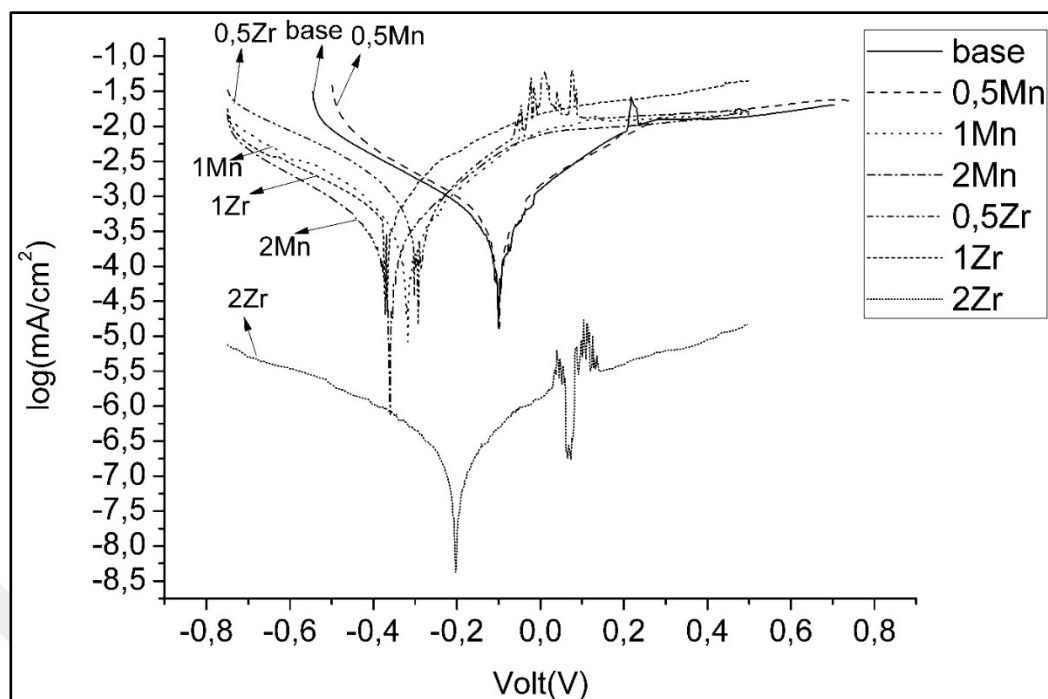


Figure 4.2. The potentiodynamic polarization curves of tested composite materials in Hank's solution.

Table 4.1. The potentiodynamic corrosion test results.

Materials	S.A (cm ²)	d (g / cm ³)	E.W	CC (μA)	CCD (μA / cm ²)	CR (mm / year)	E _{corr} (V)
Base	0.283	4.22	23.10	0.149	0.526	0.009	-0.10
0,5Mn	0.283	4.30	23.25	0.204	0.721	0.013	-0.12
1Mn	0.283	4.17	23.35	0.152	0.537	0.010	-0.32
2Mn	0.283	4.27	23.60	0.099	0.350	0.006	-0.36
0,5Zr	0.283	4.31	23.25	0.332	1.173	0.021	-0.29
1Zr	0.283	4.28	23.35	0.419	1.480	0.026	-0.37
2Zr	0.283	4.30	23.60	0.001	0.004	8x10 ⁻⁶	-0.20

4.3. HARDNESS

The hardness test results of the investigated composite materials are presented in Table 4.2. As seen from Table 4.2, the highest and the lowest values were obtained from 0,5Zr (359HV) and 2Zr (419HV) labelled composite materials, respectively. When we investigate the effect of the amount Mn to hardness, there is an increase with the addition of 0,5 wt. % Mn (383HV) which is similar with almost 1 wt. % Mn (380HV)

added one due to the enlarged α -phase and β -phase. Further, the hardness is increasing with the addition of the 2 wt. % Mn (417HV) to base composite material (376HV) as the lath α -phase. It is known that the grain boundaries impart to more hindering to dislocation movement which is attributing to the rising of hardness. However, the smooth character of the α and β phase gives rise to lower hardness to Ti composite materials.

Table 4.2. The micro vickers hardness test results of investigated composite materials.

Materials	Average Hardness Values (HV)
Base	376
0,5Mn	383
1Mn	380
2Mn	417
0,5Zr	359
1Zr	374
2Zr	419

4.4. WEAR

The wear volume of the investigated composite materials was illustrated in Fig. 4.4. Moreover, the SEM images are providing the plastic deformation and oxidation type wear mechanisms were occurred during the wear test for all specimens. The morphology of wear tracks belong to all specimens are obviously similar that indicates the wear mechanism mostly were imparted by adhesive wear process. To understand the relationship wear volume loss of investigated composite materials between microstructure, the EDS study were utilized. The results obtained by EDX analysis shown that the oxide formation with the addition of Mn and Zr particles changed the wear volume proportionally with the MnO or ZrO₂. As to Mn addition between 0,5wt% and 2%wt, the MnO formation is firstly is increased but after that, it is decreasing and lastly again increasing that were observed for 0.5%wt Mn, 1%wt Mn and 2%wt Mn addition, respectively. In addition, the ZrO₂ formation showed similar behaviour with the addition of %wt Zr addition, where the amount of ZrO₂ is following as 2Zr>1Zr>0,5Zr. It is could be said that the stable oxide particle resists to the high contact pressure during wear test.

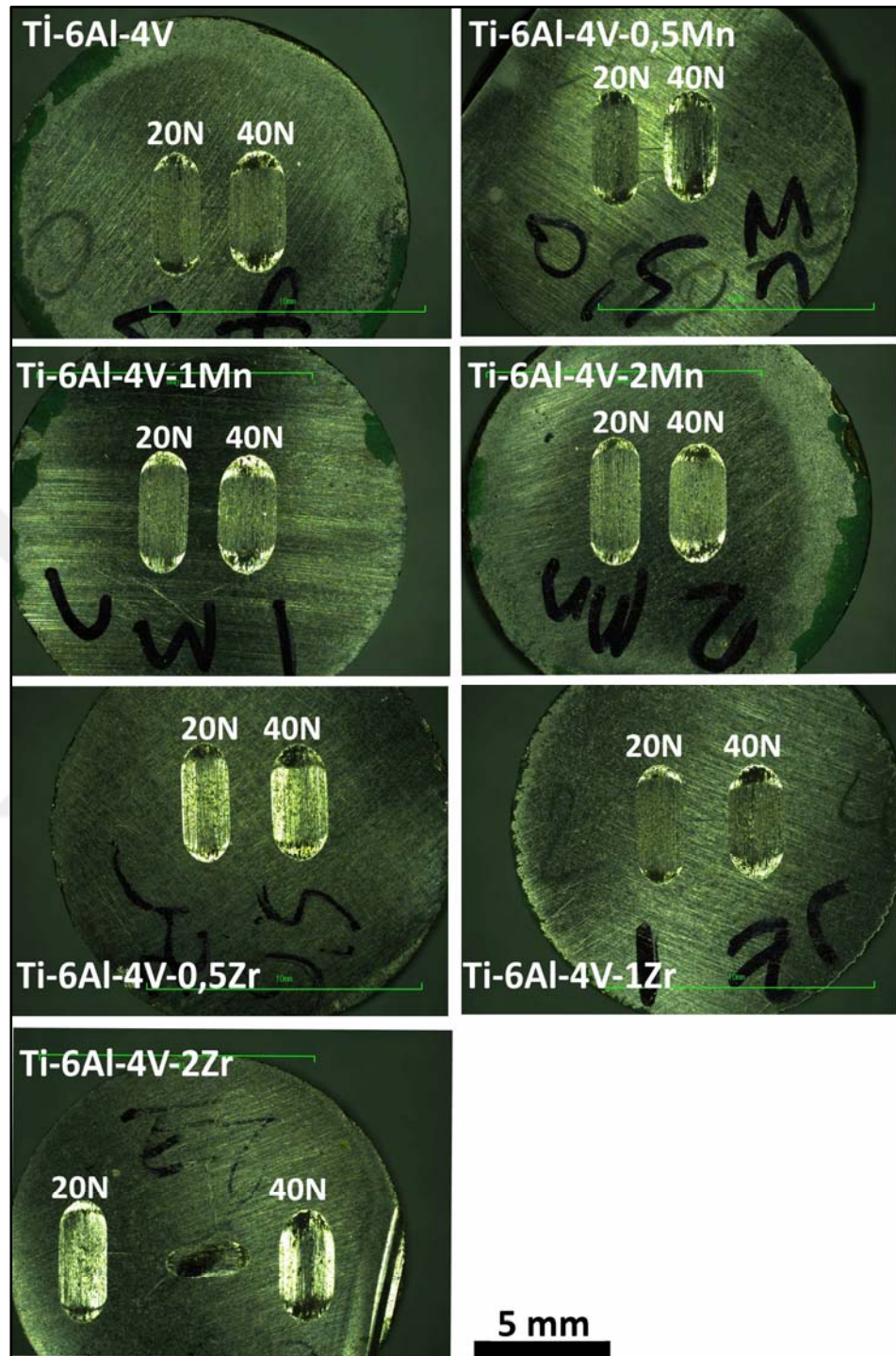


Figure 4.3. The macro worn surface.

The worn scars of the base, 2Mn and 2Zr labelled composite materials is given in Figure 4.4 shows that oxidation type wear formed on the surface of base labelled composite material were, more region exposed the oxidation, however, a small section

of surface harmed by adhesive type wear. On the other hand, parallel lines are placed which resulted from the adhesive wear on the nearly all section of surface 2Mn labelled composite material. Nevertheless, mainly material-removing occurred at the surface of 2Zr labelled composite material which emerged from the abrasive wear as the characteristic of ZrO_2 formation.

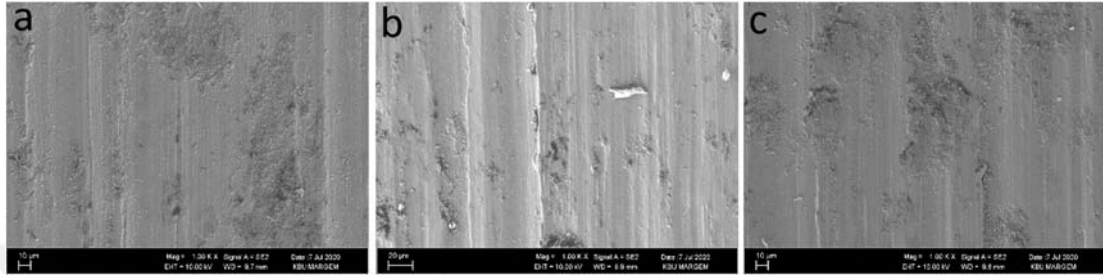


Figure 4.4. The worn surface of (a) base, (b) 2Mn and (c) 2Zr labelled composite materials.

PART 5

CONCLUSIONS

In a way that depends on differences in amounts of Mn and Zr particles on the Ti-6Al-4V composite material, the stable MnO and ZrO₂ formation gives rise to better hardness and consequently which the more wear resistance was obtained. The microstructure of lath α -phase was formed by 2Mn wt% addition to Ti-6Al-4V impart to the best corrosion resistance where the homogeneous formation on grain boundaries of β -phase mainly affects the corrosion attacks rate negatively. Similarly, the Laves phase was occurred by addition of Zr particles as wt%2 amount on the grain boundaries of β -phases hindered the corrosion attacks as it makes a role as a barrier to them.

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RESUME

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