



**SYNERGISTIC EFFECTS OF MO AND TI
ELEMENT ADDITION ON MICROSTRUCTURE,
MECHANICAL AND CORROSION PROPERTIES
OF 316 L STAINLESS STEELS PRODUCED BY
PM METHOD**

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ABSTRACT

Master Thesis

SYNERGISTIC EFFECTS OF MO AND TI ELEMENT ADDITION ON MICROSTRUCTURE, MECHANICAL AND CORROSION PROPERTIES OF 316 L STAINLESS STEELS PRODUCED BY PM METHOD

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The powder metallurgy (P/M) process allows the creation of high-performance and durable components using metal powders of different sizes. The properties of these components depend on the size, composition, compression and sintering properties of the powders used. Powder metallurgy offers superior advantages such as homogeneity, economic efficiency, minimal or loss-free output during production and no need for additional processing, making it a highly recommended technique due to its excellent qualities. Stainless steels can be improved by changing their alloying elements and chemical composition. The addition of elements such as chromium, nickel, copper, titanium, molybdenum, niobium, aluminum, nitrogen, silicon and sulfur significantly improves the properties of stainless steel. This study focuses on 316L stainless steel matrix processed with specific concentrations of titanium and molybdenum elemental

powders using P/M technology, resulting in a powder mixture with the required composition. Powders were shaped into blocks using ASTM E8M powder metal drawing sample mold and cold pressed unidirectionally at 750 MPa compression pressure. After the pressing process, the samples were sintered in an atmosphere-controlled tube furnace at 1310°C for 6 hours to regain their original strength. 316L matrix alloy steel samples were examined using electron microscopy (SEM) for phase distribution and grain size analyses. In order to determine their mechanical properties, the samples were subjected to tensile testing at a tensile speed of 1 mm/min and stress-strain curves were created for each test. Additionally, the samples were subjected to microstructure examination and density measurement. This comprehensive research highlights the potential of powder metallurgy in producing high-quality stainless steel alloys with special properties through precise control of powder composition and processing parameters.

Key Word : Powder metallurgy, Stainless steel, 316L, Titanium, Molybdenum, Microstructure.

Science Code : 91437

ÖZET

Yüksek Lisans Tezi

TM YÖNTEMİ İLE ÜRETİLEN 316 L PASLANMAZ ÇELİKLERİN MİKROYAPI, MEKANİK VE KOROZYON ÖZELLİKLERİNE MO VE Tİ ELEMENT İLAVESİNİN SİNERJİK ETKİLERİ

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Toz metalurji (T/M) süreci, farklı boyutlardaki metal tozlarını kullanarak yüksek performanslı ve dayanıklı bileşenlerin oluşturulmasına olanak sağlamaktadır. Bu bileşenlerin özellikleri, kullanılan tozların boyutuna, bileşimine, sıkıştırma ve sinterleme özelliklerine bağlıdır. Toz metalurjisi, homojenlik, ekonomik verimlilik, üretim sırasında minimum veya kayıpsız çıktı ve ek işleme gerek olmaması gibi üstün avantajlar sunmakta olup, bu mükemmel nitelikleri nedeniyle şiddetle tavsiye edilen bir tekniktir. Paslanmaz çelikler, alaşım elementleri ile kimyasal bileşimleri değiştirilerek geliştirilebilmektedir. Krom, nikel, bakır, titanyum, molibden, niyobyum, alüminyum, azot, silikon ve kükürt gibi elementlerin eklenmesi, paslanmaz çeliğin özelliklerini önemli ölçüde iyileştirmektedir. Bu çalışma, P/M teknolojisi kullanılarak titanyum ve molibden element tozlarının belirli konsantrasyonları ile

işlenen 316L paslanmaz çelik matrisine odaklanmakta olup, gerekli bileşime sahip bir toz karışımı elde edilmiştir. Tozlar, ASTM E8M toz metal çekme numune kalıbı kullanılarak bloklar halinde şekillendirilmiş ve 750 MPa sıkıştırma basıncında tek yönlü olarak soğuk preslenmiştir. Presleme işleminden sonra numuneler, orijinal dayanımlarını geri kazanmak amacıyla atmosfer kontrollü tüp fırında 1310°C'de 6 saat sinterlenmiştir. 316L matris alaşım çeliği numuneleri, faz dağılımı ve tane boyutu analizleri için elektron mikroskobu (SEM) kullanılarak incelenmiştir. Mekanik özelliklerini belirlemek amacıyla, numuneler 1 mm/dak çekme hızında çekme testine tabi tutulmuş ve her deney için gerilme-şekil değiştirme eğrileri oluşturulmuştur. Ayrıca numuneler, mikro yapı incelemesi ve yoğunluk ölçümüne tabi tutulmuştur. Bu kapsamlı araştırma, toz bileşimi ve işleme parametrelerinin hassas kontrolü yoluyla özel özelliklere sahip yüksek kaliteli paslanmaz çelik alaşımlarının üretilmesinde toz metalurjisinin potansiyelini vurgulamaktadır.

Anahtar Sözcükler : Toz metalurjisi, Paslanmaz çelik, 316L, Titanyum, Molibden, Mikro yapı.

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ABBREVIATIONS

- BCC : Body-Centered Cubic (Gövde Merkezli Kübik)
- P/M : Powder Metallurgy (Toz Metalurjisi)
- SEM : Scanning Electron Microscopy (Taramalı Elektron Mikroskobu)
- EDX : Energy Dispersive X-ray Spectroscopy (Enerji Dağıtıcı X-ışını Spektroskopisi)
- FCC : Face-Centered Cubic (Yüz Merkezli Küp)
- MSS : Martensitic Stainless Steels (Martenzitik Paslanmaz Çelikler)
- SCC : Stress Corrosion Cracking (Gerilim Korozyonu Çatlama)

CHAPTER 1

INTRODUCTION

Materials are the building blocks that engineers use to create anything. Biomaterials are any materials that the body uses to repair or maintain tissues that function in a living environment and injured organs. A great deal of effort is invested in ensuring that components of biomedical goods adapt to biological systems without causing harm. Biocompatible, dependable, and effective biomaterials are materials used in the human body to support or carry out the functions of organs or tissues that come into contact with bodily fluids on a regular or continuous basis and that shouldn't result in cytotoxic reactions in the organism. Their significance and field of application are expanding every day [1].

Generally speaking, a biological material's performance is determined by two factors: biofunctioning and biocompatibility. While biocompatibility evaluates a substance's suitability for the body, bio functioning indicates how effectively a device can perform its intended purpose. Some important characteristics of biomaterials make them desirable. Engineers are well-acquainted with mechanical performance, which includes tensile strength, deformation, bending, fatigue, toughness, and elasticity. They are also familiar with chemical stability, such as resistance to corrosion. Additionally, biological properties and bioinertity are traits that are of interest to disciplines in the biological and medical fields [2].

One of the minerals being examined in this research is chromium, which was given the moniker Siberian red lead after it was found in a Siberian gold mine in 1766. The discovery was first attributed to the French researcher Nicholas Louis Vauquelin, who identified it in Paris in 1798 [3]. The term "chromium" is derived from the Greek word "chroma," which translates to "color." The substance is now referred to as crocoite and

belongs to the category of lead chromate [4]. This mineral may be found in several locations, including as Kazakhstan, South Africa, India, and Turkey.

Chromium is a bright, brittle, and inflexible metal. It might have a silver-gray hue and be very polished [5]. In air, it does not tarnish. The surface of the stainless steels becomes a thin, chemically stable coating of passive oxide when Cr is present. The oxide coating forms and heals itself when oxygen is present. The physical-chemical properties of this passive layer control the material's corrosion behavior, its bodily interactions, and its biocompatibility. Many in-vitro and in-vivo studies have been conducted on the corrosion of metal implants. However, the majority of in vitro studies employed physiologically realistic fluids such as Ringer's or Hanks' solutions. Reportedly, the amount of chromium in the protective coating processes that result in a coating significantly affects stainless steel's corrosion resistance [6].

The discovery that stainless steel, which is made of metalized chromium, may have been engineered to be very resistant to corrosion and discoloration, represented a dramatic shift in the production of steel. Eighty-five percent of industrial use is made up of stainless steel (chromium electroplating) and chrome coating combined. Chromium is added to steel to increase its resistance to oxidation. This resistance increases when more chromium is added. Chromium content in stainless steels must be at least 10.5% (usually 10.9 or 12.1%). This offers a notable level of overall abrasion resistance in comparison to steels that include less chromite. Stainless steel is resistant to corrosion because, as previously mentioned, its surface is coated with chromium oxide, which facilitates the material's passive self-healing properties. Stainless steel, which has a chromium concentration of more than 12% and provides better corrosion resistance, was used to manufacture the first orthopedic implants in the 1920s [7]. Owing to its superior machinability, cheap cost, enhanced ductility, and strong fatigue strength, type (AISI 316L) is frequently used in implanted devices, especially orthopedic implants [8]. Stainless steels are known for having a high level of corrosion resistance in a variety of circumstances [9]. Nonetheless, solutions containing 30% nitric acid demonstrate the exceptional corrosion resistance of chromium and chromium alloys [10].

This study aims to investigate the effects of Mo and Ti addition on the mechanical properties and microstructure of 316L stainless steels. It is thus thought that these steels produced using the new P/M can have improved mechanical properties. It is commonly recognized that if P/M is used to make these steels, the number of samples required will determine how much the production costs will decrease. The object that is formed will be guaranteed to have a high-quality surface, and some metals that are difficult to produce and process using traditional methods can be made.

As a result, several combinations of stainless steel 316L with the addition of Mo and Ti can be formed. During the production process, sintering time is going to be used to shape them. The optimal sintering temperature for 316L structure steels produced by powder metallurgy (P/M) will be identified by subjecting the fabricated components to a series of tests, including as cutting, mounting, grinding, polishing, imaging, SEM analysis of the microstructure, SEM analysis of the surface fraction, and energy EDS analysis. The examinations will also assess the mechanical properties of the components.

CHAPTER 2

STAINLESS STEEL

2.1. DESCRIPTION OF STAINLESS STEELS

Stainless steel, a type of iron alloy, contains a minimum of 11.2% chromium, which provides heat resistance and prevents corrosion [11]. Various steel materials include elements such as carbon (ranging from 0.03% to more than 1.1%), nitrogen, aluminum, silicon, sulfur, titanium, nickel, copper, selenium, niobium, and molybdenum [12]. Some types of stainless steel, such as 340 stainless, are commonly identified by three-digit AISI designations [12]. The resistance of stainless steel to aging is due to its chromium component, which forms a passive layer that protects the underlying material from corrosion and has the ability to self-heal in the presence of oxygen [6].

To increase corrosion resistance even further, try the following techniques:

- Increase the amount of chromium to 11.2 percent and nickel to at least 8%.
- Deal with molybdenum.
- The addition of nitrogen improves the strength and anti-corrosion qualities of the structure.

Because various settings have different requirements for molybdenum and chromium, there are several varieties of stainless steel. Stainless steel is an ideal material for tools that require both strength and resilience to corrosion due to its well-known brilliance, low maintenance needs, and resistance to corrosion and stains. Additionally, stainless steel can be rolled into various shapes, which can be applied to different items, including surgical instruments.

Considerable technical progress in the 1950s and 60s allowed for the production of a large range of steel materials. Among the various types of steel, the most widely used is austenitic stainless steel [13]. Austenitic stainless steels are composed of austenitic minerals and have a face-centered cubic crystal structure. Steel is melted with sufficient amounts of nitrogen, manganese, and nickel to maintain an austenitic microstructure across the entire temperature range, from the softening point to the cold zone [14].

Thermal treatment does not strengthen austenitic stainless steels since their microstructures remain the same at room temperature. This characteristic makes them unique among stainless steels, as their mechanical properties are consistent regardless of thermal processes.

2.2. STAINLESS STEEL CLASSIFICATION

While all stainless steels contain the same amount of chromium (Cr), other alloying elements are commonly added to improve their properties. Stainless steels are classified differently from other metals due to their crystalline structure, or microstructure, which describes the arrangement of the atoms that make up the steel's grains. Based on their chemical composition, stainless steels fall into two categories: those that are primarily chromium and those that are chromium-nickel.

The microstructure is the primary determinant of the precipitation hardening behavior of various basic families, such as duplex austenitic-ferritic, ferritic, martensitic, and austenitic-ferritic types. The mechanical properties of stainless steel include elongation, yield strength, elastic modulus, and ultimate tensile strength, among others. As seen in Table 2.1, the alloy's microstructure and chemistry dictate its properties. A peculiar property of stainless-steel alloys, the "Young's modulus" is very sensitive to their microstructure and chemistry [15].

Table 2.1. Properties of stainless steel [16].

| AISI stainless steel | Microstructure properties | Mechanical properties | | | Physical characteristic |
|----------------------------------|---------------------------|-----------------------|----------------|----------------------|---|
| | | Elastic strength | Yield strength | Protraction – 50 mm% | |
| Austenitic | Austenite | 480-870 | 200-580 | 32-65 | Non-heat able & non-magnets |
| Ferritic | Ferrite | 410-640 | 280-575 | 12-30 | Magnets, non-heat able, & chloride-resistant. |
| Martensitic | Martensite | 485-999 | 277-870 | 13-28 | heat-treated, heat-enabled hardness |
| Duplex | Austenite & ferrite | 688-910 | 409-912 | 15-45 | High strength and heat-intolerant |
| Precipitation hard enable | Austenite & martensitic | 890-995 | 271-992 | 10-32 | high strength heat-treated hardening |

2.2.1. Austenitic Stainless Steels

Austenitic stainless steels are the largest and most varied class of stainless steels, making up around 70% of all industrial output of stainless steel [17]. The microstructure of these steels is composed of the face-centered cubic (FCC) crystal structure of the stable austenite phase. Austenitic stainless steels are preferred over other types because they are non-magnetic, more corrosion-resistant, and easier to weld. They can be significantly strengthened through a cold metal-forming process [18].

Given the widespread use of magnetic resonance imaging techniques, the non-magnetic nature of austenitic stainless steels makes them ideal for orthopedic implant materials, where ferromagnetism is a desirable state [19]. One of the most important properties of a material for orthopedic applications is excellent corrosion resistance in body fluids, which austenitic stainless steels provide. These qualities make them a suitable choice for orthopedic implants.

However, austenitic stainless steels are more susceptible to pitting corrosion and hostile environments. They also frequently show reduced resistance to stress corrosion cracking (SCC) and crevice corrosion [20]. Despite these limitations, the overall benefits make austenitic stainless steels a preferred material in many industrial and medical applications.

2.2.2. Steel with Ferritic Properties

Ferritic stainless steels feature a ferrite microstructure, which is a body-centered cubic (BCC) crystal structure at all temperatures, much like carbon steels do. Ferritic grades can have Cr concentrations ranging from 10.5% to 30.0%, while carbon content is kept at a minimum of 0.08%. Even while in many ferritic stages of an alloy chromium is the main metallic component, some of them also contain (Mo 4%), very little Ni, or none at all. Certain grades, like austenitic stainless steels, cannot be strengthened or toughened by heat treatment because they are not heat treated. Due to their magnetic properties similar to carbon steel, ferritic grades are preferred when longevity is not the primary requirement. But ferritic stainless steels only resist moderate amounts of corrosion, particularly when combined with stress and certain corrosive environments, which causes a phenomenon called SCC. Hot water tanks, exhaust systems, and automobile trim are common applications for ferritic stainless steels [21].

2.2.3. Stainless Steels in Duplex Form

In duplex stainless steels, austenite and ferrite are both present in the microstructure (FCC, BCC). The goal is to have the equal amount of ferritic and austenitic elements in the alloy, however in commercial alloys, the ratio is often 40/60. Consequently, duplex ferritic-austenitic steels include a number of the best qualities from both types of steel. For example, they may experience SCC, but it's not nearly as bad as ferritic steels. Even when compared to austenitic steels, their resistance to SCC is lower. The magnetic properties and almost twice as big elastic limit of duplex stainless steels set them apart from austenitic stainless steels. Magnetic and offering about double the yield strength of austenitic stainless steels, duplex stainless steels outperform their counterparts in terms of corrosion resistance. In addition, austenitic stainless-steel types 304 and 316 benefit greatly from their complex microstructure, which offers superior protection against corrosion and resistance to chloride stress corrosion. These alloys may include molybdenum at a level of 4.7% and cadmium at a level of 0.03%. Many different industries use duplex stainless steels, including those dealing with heat exchangers, chemicals, water filtration stations, maritime settings [22].

2.2.4. Martensitic Stainless Steels

Martensitic stainless steels (MSS) are metal compounds primarily composed of 11.5–18% Cr, 0.1–4.2% Ni, and 0.11–1.2% C. These steels may also include elements such as V, Mo, Nb, Al, and Cu. At high temperatures, MSS exhibit an austenitic structure, which transforms into martensite when cooled to a sufficiently low temperature. Based on their composition and thermal history, MSS can contain δ -ferrite, undissolved carbides, and martensite. Heat treatment significantly impacts the properties of MSS. To ensure the transition to a homogeneous martensitic structure rather than ferrite during the cooling phase, MSS must undergo careful heat treatment to avoid carbide precipitation and δ -ferrite formation [23].

MSS are chromium and carbon alloys with a body-centered tetragonal structure [24]. The carbon content in the alloy determines the achievable strength through heat treatment [25]. However, the corrosion resistance of MSS is generally poor. These steels can be significantly hardened by cold working [26]. When tempered and hardened, these alloys exhibit good ductility and hardness properties. Applications of MSS include surgical instruments, propellers, creep-resistant steels, stainless tool steels, cutting blades, and other items requiring high tensile strength, fatigue strength, and other mechanical properties [21].

2.2.5. Stainless Steels Through Precipitation Hardening

Precipitation-hardening stainless steels (PH) are alloys including nickel and chrome steels that have the potential to achieve exceptionally high tensile strengths. The primary benefits offered by this category of stainless-steel varieties are their moderate resistance to corrosion and ease of production [21]. In addition to the low mass percentage of C and the extensively alloyed structure, significant strength is gained by the use of precipitation hardening of the crystalline phase [27]. Precipitation hardening stainless steels have comparable corrosion resistance to austenitic steels, despite the possibility of precipitation hardening them to even higher strengths than other martensitic varieties [28]. Austenitic or martensitic microstructures are the first state of hardening stainless steels. Austenitic grades are heat-treated to become martensitic

grades prior to precipitation hardening. When martensitic is heated to a degree where hard intermetallic compounds form precipitates out of the array of crystals, precipitation hardening occurs. Aviation and other high-tech industries frequently utilize precipitation-hardening stainless steels [21]. The most common grade in this category is 17-4 PH, often known as grade 630. Its composition is approximately 18% Cr, 5% Ni, 4.3% Cu, and 0.33% Nb [28].

2.3. MECHANISMS TO INCREASE STEEL STRENGTH

The behavior of metallurgical structures in materials greatly influences their mechanical behavior. One may argue that as a material's metallurgical structure changes as a result of thermal, mechanical, and chemical operations applied to it, the material's mechanical properties likewise rely on these conditions. One of a material's most important properties is strength. Certain attributes vary based on strength. The best way to characterize the resistance, according to materials science, is the substance's resistance to plastic deformation. Linear defects propagate and cause plastic deformation in metals. Therefore, the quantity of dislocations in the internal structures of metals and their interactions with other faults are explained by mechanical properties such as hardness, ductility, and strength. The following is a list of procedures that increase strength.

- Aging Deformation.
- Aging due to precipitation hardening.
- Grain Size Reduction for Hardening.
- Martensitic transformation-induced hardening.
- Dispersion Hardening.
- Cold Process.
- Hardening of the alloy.

A detailed knowledge of the interplay between mechanical characteristics and microstructure is crucial for optimizing the enhanced steels and enhancing some of the favorable attributes seen in micro alloyed steels. The tensile strength of micro alloy steels is improved by the hardening procedures used in them, including strain

hardening, precipitation hardening, solid melt hardening, and grain size hardening. In contrast, the grain reduction technique strengthens and increases toughness throughout the hardening processes [9].

2.4. PROS AND CONSIDERATIONS OF STAINLESS STEEL

Because of its many advantageous qualities, stainless steel is frequently employed in the manufacture of parts and components across various manufacturing industries. Above all, its exceptional level of corrosion resistance is attributed to the chromium element. Steel that contains at least 10.5% chromium resists corrosion significantly better than steel that does not. Customers also benefit from its remarkable durability and toughness in both hot and cold conditions, improved processability and manufacturing simplicity, minimal maintenance requirements, extended lifespan, appealing appearance, environmental sustainability, and recyclable nature [29]. Stainless steel does not require treatment, coating, or painting once it is used.

- Austenitic stainless steels have several advantages, including:
Excellent Machinability: Due to their FCC structure, austenitic stainless steels have a restricted ratio of yield strength to tensile strength and excellent machinability.
- Enhanced Strength: Strength can be increased through gradual strain aging and cold working. Austenitic stainless steels are also capable of withstanding corrosion well.

Non-Magnetic: Predominantly, austenitic stainless steels are non-magnetic.

Regarding the negative aspects:

While 316L stainless steel is resistant to many types of corrosion when used as an implant, it is still susceptible to stress corrosion cracking, splitting, and pitting [30]. Research indicates that 316L can corrode in the body's environment, releasing chromium, nickel, and iron [31]. Furthermore, it contains 11.0–16.0% nickel, which can be hazardous to humans if released. Excessive tissue concentrations of nickel ions have been shown to induce skin contact allergies and, in some cases, cancer [32].

2.5. STAINLESS STEEL USAGE AREAS

Alloy steel, a very valuable combination of metals, is used in the creation of several indispensable goods that we use in our everyday lives. These items include communication devices, steam boilers, vehicles, airplanes, dairy equipment, leather, paper, soap, small household appliances, nuclear engineering, and exhaust production devices.

Stainless steel is particularly significant in the production of medical equipment and food preservation tools. In the medical field, stainless steel is used to manufacture needles, screws, prostheses, kneecaps, and scalpels. Additionally, stainless steel plates are preferred in oven molds, coated pots, and storage containers for food and beverages because they preserve the food's flavor and color.

2.6. ALLOYING ELEMENTS

Most metals aren't used in their pure form; instead, alloying elements are added to alter their properties for specific applications. By adding a specific quantity of a second element, an alloy can have some of its properties significantly changed. The mechanical strength and corrosion resistance of pure iron are significantly enhanced by small quantities of other elements, as pure iron is not strong enough to be used as an active biomaterial. For instance, adding chromium (Cr) to composites encourages the development of a chromium oxide shield (passive film) [8]. Depending on the precise grade, stainless steels may also include some or all of the following alloying elements: silicon (Si), chromium (Cr), nickel (Ni), molybdenum (Mo), manganese (Mn), titanium (Ti), niobium (Nb), and vanadium [33].

Figure 2.1 illustrates the relationships between the composition and characteristics of the stainless-steel group of alloys. This figure highlights how varying the amounts and types of alloying elements can influence the mechanical and chemical properties of stainless steels, making them suitable for a wide range of industrial and biomedical applications.

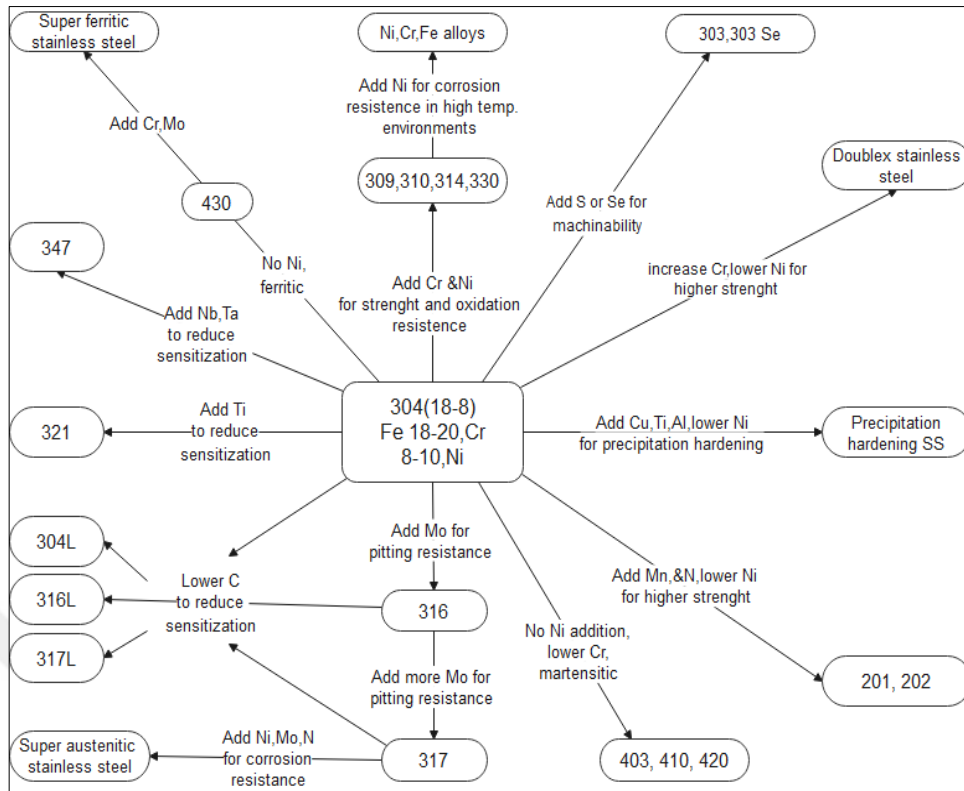


Figure 2.1. Correlations between composition and attributes of the stainless-steel group of alloys [34].

2.6.1. Molybdenum (Mo)

Together, molybdenum and chromium help stainless steels withstand corrosion from chlorides. When chromium concentration in stainless steel is around 18% in chloride-containing environments, molybdenum additions are almost three to four times more effective than chromium additions in preventing pitting and crevice corrosion [35]. Molybdenum causes ferrite, which increases the likelihood that stainless steels may produce undesirable intermetallic phases. As a result, it usually has a maximum of 5% in duplex stainless steels and less than 7% in austenitic stainless steels.

2.6.2. Titanium (Ti)

Titanium is the most often used stabilizing component for austenitic and ferritic stainless steels. Titanium also becomes more resistant to pitting corrosion. When dissolved in steel, titanium is thought to increase hardenability; nevertheless, titanium has a strong propensity to form carbides, which are often present in the structure of

steel as undissolved carbides, decreasing hardenability [33]. Stabilizing elements are added to FeCr (Mo) and FeCrNi (Mo) alloys to prevent sensitive intermetallic corrosion. Research on corrosion in Hanks' sol showed that 316L stainless steel treated with titanium resists corrosion better than unmodified 316L.

2.6.3. Chromium (Cr)

A steel needs a minimum of approximately 10.5% chromium in its stable chromium passive layer to be sufficiently protected against mild air corrosion. Stainless steel resists corrosion better at higher chromium concentrations [7]. Chromium stabilizes the BCC structure of iron, as it is a ferrite-producing element. To form an austenitic or duplex (austenitic-ferritic) structure with higher chromium concentrations, more nickel is required. Elevated chromium levels also promote the development of intermetallic phases. Austenitic stainless steels typically include at least 18% Cr, whereas second-generation duplex stainless steels generally contain at least 22% Cr. Chromium also increases resistance to oxidation at high temperatures.

This effect of chromium is crucial because it influences the formation and removal of oxide scale or heat tint resulting from heat treatment or welding. Dissolving and removing heat tint from duplex stainless steels is more challenging than from austenitic stainless steels. This increased difficulty is due to the enhanced stability and bonding strength of the oxide layer formed on duplex stainless steels, which requires more aggressive or prolonged chemical treatment to be effectively removed.

2.6.4. Manganese (Mn)

Manganese is usually added to stainless steels to improve their hot ductility. Manganese's effect on the ferrite-austenite equilibrium is temperature dependent; at low temperatures, it stabilizes austenite, while at high temperatures, it stabilizes ferritic. Austenitic and duplex stainless steels can contain high quantities of nitrogen because manganese increases the solubility of nitrogen. Because manganese creates austenite, it may also partially substitute nickel in stainless steel.

2.6.5. Carbon (C)

Carbon is a nonmetallic component included in all types of stainless steel and heat-resistant alloys. Carbon, acting as a powerful austenite stabilizer, enhances the strength of steel. Carbon is a significant alloying element in austenitic, ferritic, and duplex stainless steels. However, it has a strong tendency to combine with other alloying components to produce carbides. To minimize the occurrence of chromium carbide (Cr_{23}C_6) formation, carbon is maintained at a low level (usually between 0.003% C and 0.038% C in the lowest carbon range) by gradually cooling the material from high temperatures. This is done because the formation of (Cr_{23}C_6) removes chromium from the strong solution in the alloy. The mechanical characteristics of the microcrystals are weakened as a result of the introduction of Carbon, causing defects in their lattice structure [17].

2.6.6. Nickel (Ni)

Nickel acts as an austenite stabilizer in steel, inducing expansion of the austenite zone and compression of the ferrite region. When exposed to elevated temperatures, nickel exhibits enhanced resistance to both oxidation and corrosion. The particle size of nickel is altered to enhance its mechanical strength and durability. Additionally, it inhibits the development of scale on the substance's surface. It enhances the critical cooling rate, ductility, hardness, and fatigue resistance when combined with chromium. Since nickel has a lower diffusion coefficient than many other elements, it diffuses into iron at a slower rate [36].

2.6.7. Nitrogen (N)

Nitrogen enhances the resistance of austenitic and duplex stainless steels against fissure corrosion and cracking. Besides, it increases their tensile strength significantly, which makes it the strongest part of solid solutions overall [37]. Austenitic and duplex stainless steels with increasing nitrogen content are stronger and more durable. Duplex grades can be processed and produced because nitrogen slows down the development of intermetallic phases. Nitrogen is added to austenitic and duplex stainless steels,

which are highly corrosion-resistant and have significant amounts of chromium and molybdenum, to offset their tendency to create sigma phase. Nitrogen can partially replace nickel in austenitic stainless steels due to its strong austenite-forming potential. Duplex stainless steels routinely have their nickel content changed and have nitrogen added to them almost to their solubility limit in order to achieve the proper phase balance. The austenite formers, nickel, and nitrogen, are balanced with the ferrite formers, chromium, and molybdenum, to generate the duplex structure.

2.6.8. Silicon (Si)

Steels have a moderate capacity to harden due to their low silicon content. It also makes things more machinable [27]. Si can be added in small amounts to austenitic stainless steels that include Mo to improve their resistance to corrosion in sulfuric acid. Austenitic stainless steels with high concentrations of Si both reduce carburizing at high temperatures and improve resistance to oxidation [33].

2.6.9. Niobium (Nb)

Some stainless steels rely on niobium to inhibit interfacial corrosion. Since niobium is a relatively softly resistant metal, its popularity skyrocketed in the 1950s, when the space race began. The improved characteristics of carbon steel with a trace amount of niobium gave rise to the idea of a micro alloy. Niobium is mostly used to strengthen micro-alloyed steels by acting as an alloying agent. Among niobium's secondary applications is the construction of superalloys that can endure the high temperatures seen in the hot areas of aircraft engines. Nb-Ti alloys, which allowed for the construction of new kinds of magnets, are one example of an application that requires superconductivity and niobium. Niobium also improves the characteristics of austenitic stainless steels and increases their resistance to oxidation in ferritic stainless steels [33].

2.6.10. Vanadium (V)

By "precipitation-strengthening" the tiny carbonatite particles that are created during cooling or tempering, vanadium, a micro alloying element, is most frequently used to increase the strength and resistance of micro alloyed steels rather than their hardenability [38]. Some research suggest that vanadium has a critical role in increasing strength even when tensile strength remains constant. While some have shown that even small amounts of vanadium enhance steels very somewhat, others have suggested that adding vanadium increases impact toughness [39].

2.6.11. Aluminum (Al)

Significant aluminum additions improve oxidation resistance and are used in some heat-resistant grades for this purpose. In steels that are precipitation hardened by using aluminum to form intermetallic compounds, the strength of the steel increases as it ages [40]. This process, known as aging, results in a material that is not only more resistant to oxidation but also possesses enhanced mechanical properties, making it suitable for high-temperature and high-stress applications.

2.7. CORROSION OF MEDICAL IMPLANT

Implants in human bodies are still susceptible to corrosion today. It is commonly known that it produces non-biocompatible metal ions (debris) that lead to implant failure. Corrosion problems have been a common occurrence with medical implants. For example, it has been discovered that around 41% of implant failures combining Ti-6Al-4V and 316L were caused by corrosion. It's critical to comprehend how orthopedic implants, in particular, corrode, as do all implants utilized in the human body [41].

2.7.1. Corrosion

When exposed to the body's strong electrolytic environment, metallic devices constantly deteriorate due to electrochemical assault, a process called corrosion.

According to [42], implants are subjected to a harsh corrosion environment when exposed to blood and other bodily fluids that include water, salt, chlorine, plasma, amino acids, and salivary gland mucus. Anions such chloride, phosphate, and bicarbonate ions, cations like Na^+ , K^+ , Ca^{+2} , and Mg^{+2} , organic molecules with low molecular weight species mixed with polymeric components with relatively high molecular weight, and dissolved oxygen make up the aqueous dispersion in the human body [43]. By soaking up the byproducts of anodic or cathodic reactions, living molecules throw off the balance of the implant's corrosion processes. Protein binding and subsequent removal of transition metals from the implant surface has the potential to disrupt the double-layer surface equilibrium caused by the surface's electrons and any additional cations in the solution. On top of that, surface-digested proteins may block oxygen passage, which can lead to localized rusting. It seems that bacteria alter the corrosion-inhibiting properties of cathodic reaction-produced hydrogen by decomposing any hydrogen near the implant. This kind of action usually leads to worsening corrosion. Alterations to the pH level may also affect corrosion. After surgery, the pH level typically ranges from 5.2 to 5.7 close to the implant, although it can vary from 3.2 to 9.5 due to a variety of factors, including events, biological system imbalances caused by diseases, infections, and other conditions, and the body's normal pH level of 7.0. Clinical data suggests that metal ions are being leached from the implants, which is caused by corrosion, even though surface oxide coatings protect most of the components from environmental harm.

2.7.2. Medical Implant Corrosion Types

Both localized and systemic corrosion are possible. Corrosion is known to evenly deteriorate metal surfaces. Local corrosion, in contrast, affects a small area of the metal surface. The four most important forms of corrosion for medical implant metallic alloys are frictional, pitting, galvanic, and crevice corrosion, all of which are classified as local corrosion [44].

2.7.2.1. Galvanic Corrosion

Any pure metal or alloy will develop an electrode potential, known as E_{corr} , when exposed to an aggressive and corrosive environment. E_{corr} is the point at which the rates of oxidation and reduction are exactly equal. The electrochemical potential difference between two dissimilar metals is referred to as galvanic corrosion. In theory, when two distinct metals are used together, corrosion will begin rapidly: one metal will act as the cathode (the metal with the higher electrode potential) and the other as the anode.

As illustrated in Figure 2.2, the galvanic series is an ordered collection of corrosion potentials for pure metals and alloys in natural saltwater, experimentally measured. The saturated calomel electrode or the standard hydrogen electrode can be used as the standard electrode for evaluating corrosion potentials [45]. The galvanic series helps predict and understand the behavior of different metals in corrosive environments, aiding in the selection of compatible materials for various applications to minimize the risk of galvanic corrosion.

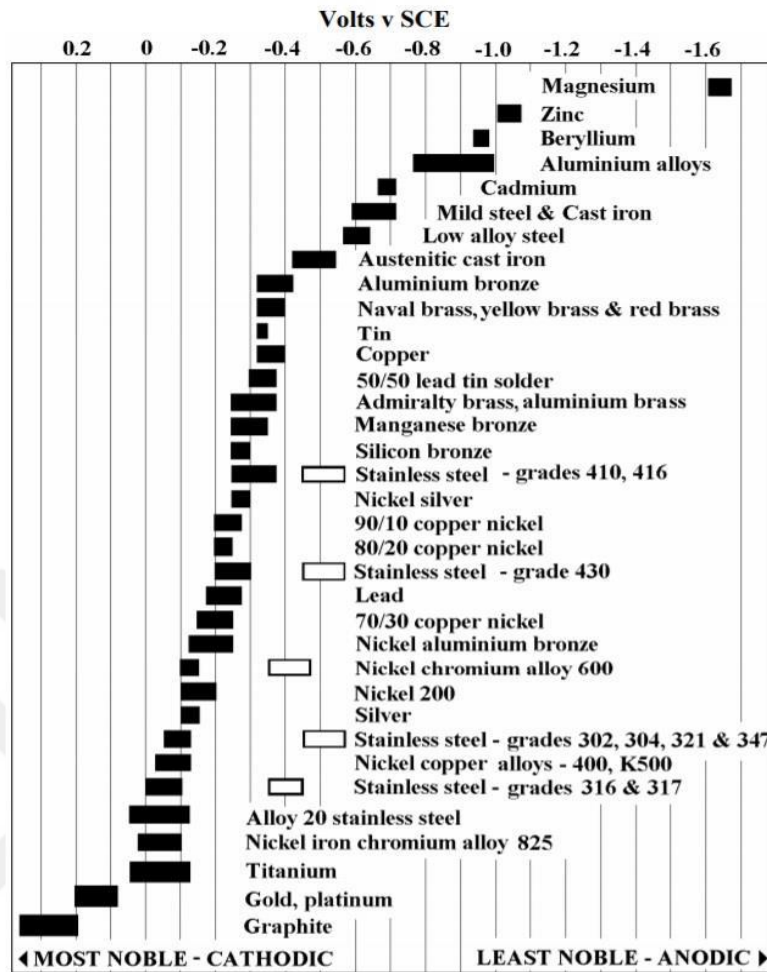


Figure 2.2. Galvanic series in sea water.

The galvanic series found experimentally in natural seawater is used to study the corrosion behavior of metal orthopedic implants used in human anatomy [46]. Simulated bodily fluids like Ringer's solution and diluted body fluids can be likened to seawater [47].

The kind of pure element or metal, the composition of the electrolyte, the thickness of the oxide film layer, and the amount of metals or alloys present all directly affect the electrode potential [45]. Galvanic corrosion is an issue because orthopedic implants commonly involve a range of metals.

2.7.2.2. Friction Corrosion

As a subtype of wear-induced corrosion, friction corrosion is known as tribocorrosion. A kind of corrosion known as friction corrosion is hastened by micromovements-induced wear. The process involves the oscillation of two surfaces that are in constant contact with one another, as is the case with the screw heads of medical implants and bone plates. Although friction may happen in the absence of a corrosive liquid, it is the minute relative motions between touching surfaces in a corrosive fluid that induce friction corrosion.

As a well-known corrosion inhibitor, an oxide layer develops on metals when they come into contact with moisture or humidity. But microwear debris disturbs the oxide coating on an implant, which throws off the electrochemical balance and brings about more corrosion. As long as an oxide layer is not formed, corrosion will continue. Orthopedic joints, such hip implants, are known to undergo this cycle, which includes micro-abrasion-related debris, oxide film breakdown, changes in environmental balance, and oxide film production [48].

Oxygen, proteins, wear, stresses applied to the implant, and integration with bone cells are just a few of the environmental factors that impact friction corrosion. These elements change depending on where you are in the body [48]. Improving implant longevity and function while decreasing the occurrence of failures owing to tribocorrosion requires a thorough understanding of these aspects.

2.7.2.3. Crevice Corrosion

Crevice corrosion is one kind of corrosion that is related to structural components. It occurs when there is little to no protection between a metal surface and the external environment. It happens in cracks that are fatigued as well as other fractures where oxygen's influence is diminished. occurs in places with little oxygen availability. For instance, the flexible connections in hip prosthesis that experience crevice corrosion in geometric regions [49]. It is brought on by tiny galvanic cells on the implant's surface as a result of its irregularity, which fractures the material as it ages. At the

onset of crevice corrosion, there is a reduction in oxygen on both the implant surface that is in contact with the electrolyte and the region inside the crack.

A large amount of oxygen is drawn out of the air by the exterior metallic portion of the implant that comes into touch with the electrolyte in order to keep the O_2 concentration constant. In contrast, because there is no oxygen circulation within the crack, the amount of O_2 diminishes. The metal portion on the outside of the crack serves as the cathode and the metal segment exposed to low oxygen concentration operates as the anode due to the difference in oxygen concentration between the two locations. When two metals are separated, the difference in electrode potential between them will eventually lead to corrosion [50].

Factors such as fissures, deep, narrow fractures, connections between portions of implant elements (e.g., between a plate and a screw head), or defects (e.g., fatigue fractures) are essential for the establishment of this process. Crevice corrosion attack is more prevalent in Type 316L stainless steel compared to other metallic implant materials [51]. Corrosion is a common problem with bone plates and stainless-steel screws, particularly at the points of contact with the counter sink holes. Crack extension may also occur due to crevice corrosion in the bone plate's countersink area; however, such produced crevice failures are very rare. Crevice corrosion concerns are often solved with the right device design and material selections.

2.7.2.4. Pitting Corrosion

Pitting is a severe kind of contained corrosion attack that results in significant damage and the discharge of large amounts of metal ions. The formation of minute cavities or gaps on a material's surface, known as pitting, is often concealed by a thin, robust, and long-lasting passive coating. These pits are believed to have formed as a result of the interaction of some hostile ions with the film at the film's weak or flawed regions. While the pits are usually invisible to the untrained eye, they can be detrimental enough to promote the growth of fatigue fractures or SCC, which might lead to catastrophic failure of the components during operation [52].

Pitting happens when the anodic deposit concentrates on a narrow area of the surface due to a break in the passive layer, changes in the scaling layers, inadequate shielding, accumulated metal in some locations, or any other abnormality. The cathodic region can be anywhere outside the pit. The significance of pitting is greatly affected by the sort of surface covering or coating that has evolved on the surface as a result of the material's interaction with the environment. The material is forced into a state of "passivity," which prevents surface-level degradation and all forms of corrosion. Consequently, the pit bases exhibit a strongly elevated corrosion current density. Due to the layer that covers it, H^+ ions or metal elements cannot move from the pit bottom [53]. The electrochemical potential between the metal around the pit and itself varies as oxygen is depleted. The emergence and disintegration of passivity as well as the pitting attack process are depicted in Figure 2.3. The metal ions precipitate at the top of the pit after it is initiated, often forming a film that covers the pit. The film repassivates, potentially restoring safeguard, by keeping oxygen and solution out of the pit [54].

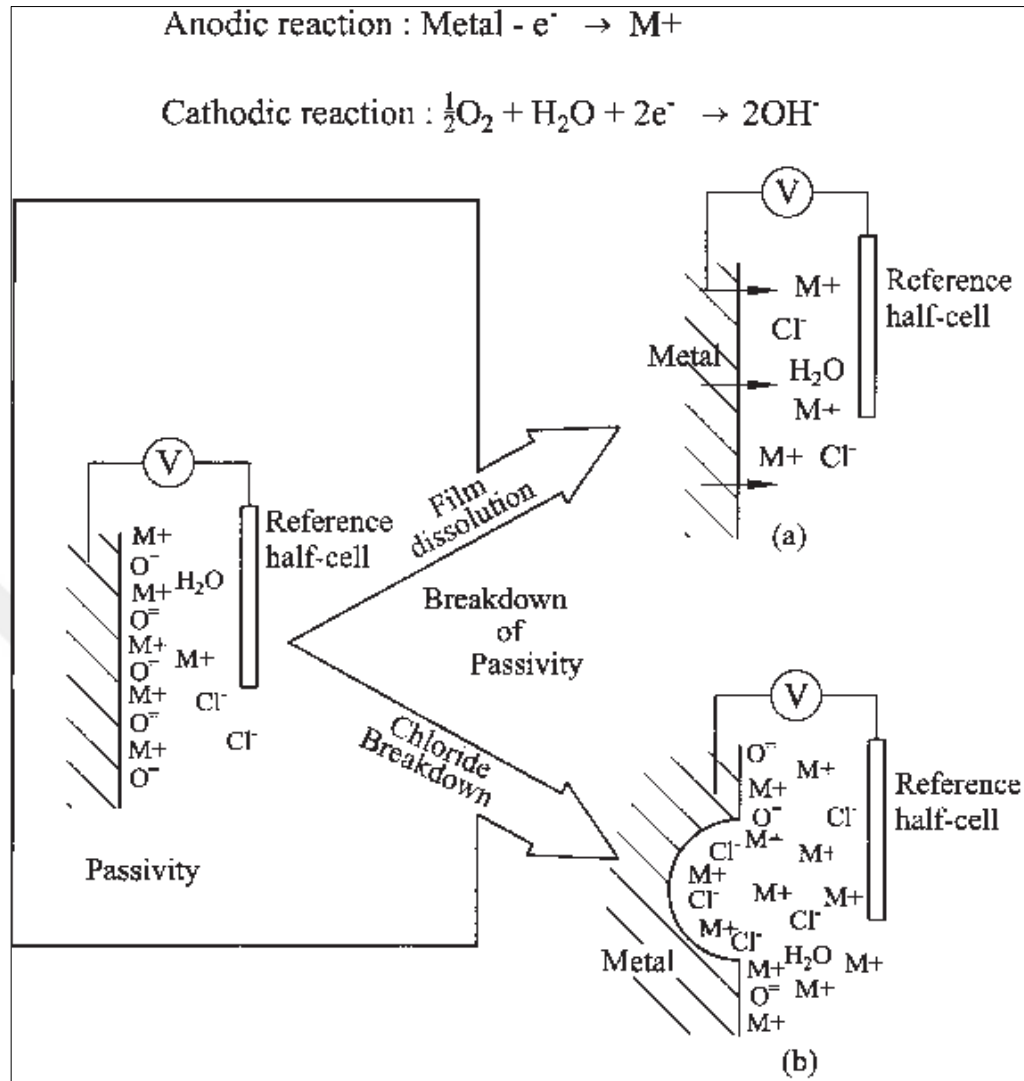


Figure 2.3. Passivity in metals broken down through general corrosion with film disintegration and pitting corrosion (a) general corrosion with film disintegration, and (b) pitting corrosion.

CHAPTER 3

POWDER METALLURGY

The method of manufacturing difficult-to-manufacture parts economically, more durable, with minimum tolerances, and more advantageously compared to various other manufacturing methods is called P/M. This production process aims to produce powders in desired shapes and sizes, combine the specified powders through the characterization process by performing different heat treatment and mechanical deformation stages, and transform them into applicable, high-performance engineering parts. P/M enables the production of parts close to the final shape and advanced materials that do not require secondary processing [55]. The most distinctive feature of the P/M method is that it is suitable for the economical production of high-quality and complex-shaped parts with minimal waste. In today's manufacturing technologies and industry, efficiency, energy, and raw materials are the most important concepts. P/M is a production method that is a good combination of these three. The properties of the materials produced through P/M technology vary depending on the Powder's shape, size, content, compression pressure, and sintering temperature. Part manufacturing using this technology is widely preferred today, and over time, it has become an alternative to known classical manufacturing techniques. In addition, P/M allows the production of parts with good strength through mechanical alloying from insoluble elements in the liquid phase. P/M technology can solve problems to a large extent as it provides superior features such as manufacturability, economy, and homogeneity, and thus it is highly preferred. Since the manufacturing cost is not high and the performance of the manufactured part is high, it is used in various fields. In prostheses produced using P/M technology, it is also possible to develop features such as functionality expected from biomaterials, biocompatibility, biocompatibility, and resistance to abrasion and corrosion. Parts produced with this technology have smoother surfaces than other production methods and generally do not require a secondary process. This method is preferred for producing the manufactured part more

economically and with the desired composition and for manufacturing parts that are difficult to machine [56,57]. P/M covers the following essential steps:

- **Generation of Powder:** The process begins with creating a powdered substance or a mixture of powders containing the necessary constituents in proportions.
- **Mixing and Blending:** The powders thoroughly mix and blend to achieve the desired characteristics and properties.
- **Shaping:** The powders are formed and manipulated into the desired shapes and dimensions while reinforcing the sections.
- **Sintering:** The compacts formed from the powders are subjected to high temperatures during sintering, which enhances their strength.
- These steps are essential in producing metal parts using P/M, allowing for precise control over the final product's properties and performance.

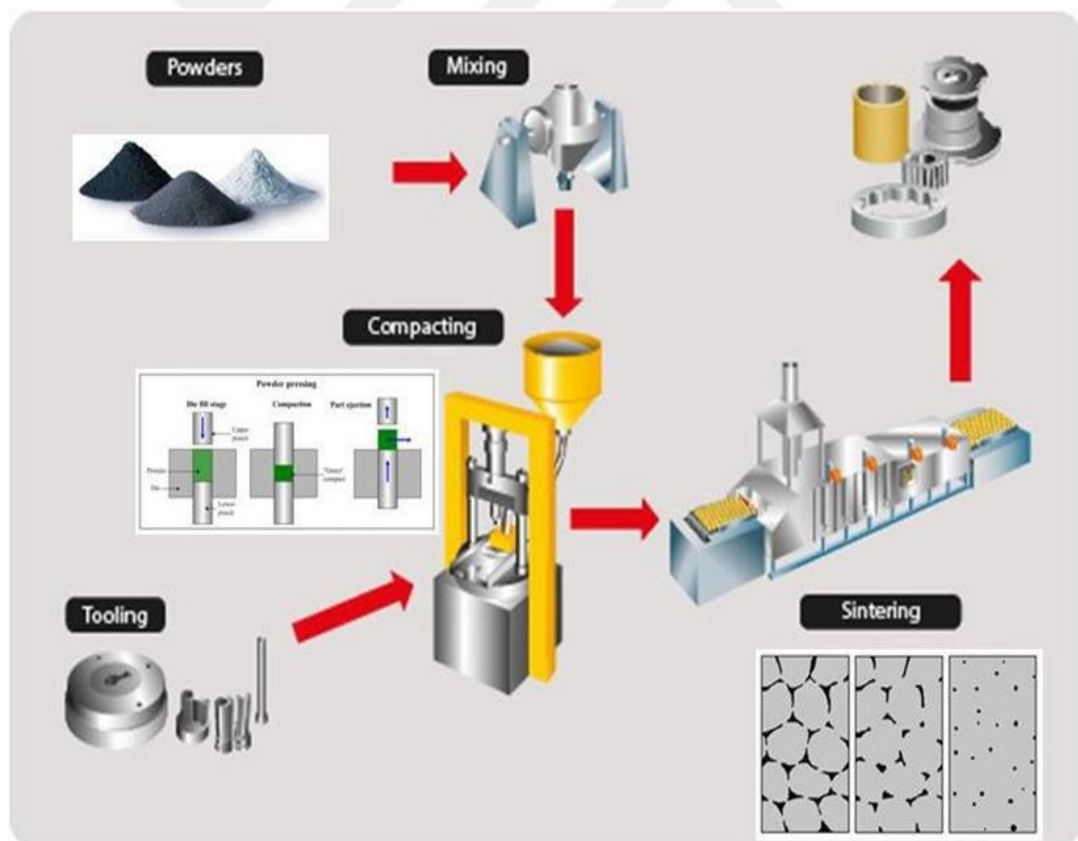


Figure 3.1. P/M process steps [57].

3.1. POWDER METALLURGY USAGE AREAS

Materials with hardness and high melting points can be shaped by applying the P/M technique. The application area of P/M is very wide. In medicine, prostheses, orthopedic implants, solders, and electrodes in welding, tungsten is used in P/M technology to make light bulb wires and tungsten lamp filaments, resistance, contact, etc., in the electrical and electronic field. Manufacturing of parts, metal filters that must have a porous structure, manufacturing of self-lubricating friction bearings by impregnating the spaces between the dust particles with oil, manufacturing of bullets and gun parts in the military field, production of artificial magnets by shaping cobalt, nickel and aluminum powders, engine parts, gears in the automotive field. This method is carried out to produce impellers and parts that must have high strength and thus be resistant to wear (e.g., brake discs). Since P/M can produce solid and resistant parts at high temperatures, various parts used as cutting tools (hard metal inserts, milling cutters, etc.) can also be manufactured using this method. Abrasive sandpaper production is carried out entirely by this method. In addition, this method is used in the production of ceramic materials as well as metals [55,58].

3.2. METHODS OF POWDER PRODUCTION

Three processes can create metallic powders: electrolytic deposition, chemical reduction, and atomization. Melted metal is atomized into tiny droplets, which harden into powdered particles. Metal ions are transformed into solid particles by chemical reduction, and metal powders are deposited onto a substrate via electrolytic deposition. Each approach has different benefits and uses, as shown in Figure 3.2. Every technique has benefits and uses of its own.

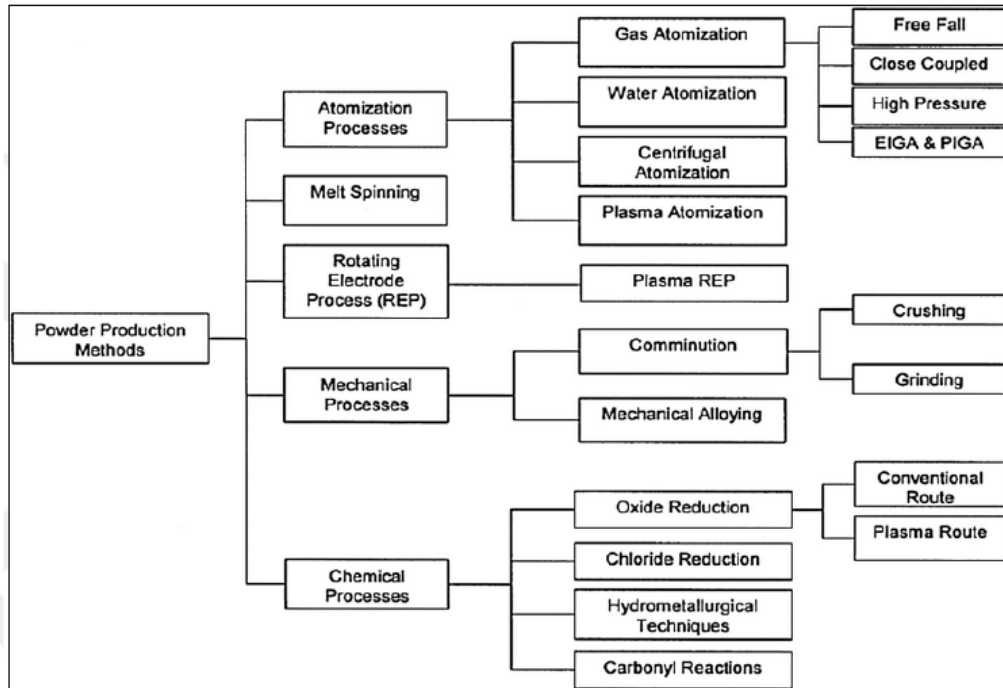


Figure 3.2. Methods of powder production [59].

3.3. ADVANTAGES AND DISADVANTAGES OF POWDER METALLURGY

The advantages can be listed as follows:

- Parts produced in P/M can be manufactured in final shape or close to their final shape.
- Parts that are not easy to machine with various other production methods can be formed using P/M.
- Since production work is carried out using molds, labor and time can be saved.
- Since enough Powder is used in the molds in production, material savings can be made compared to machining.
- Accuracy in dimensions can be ensured during production. The reason for this is that expansion does not occur in P/M, as in other casting processes performed with molds.
- Some alloys produced using P/M cannot be produced by other methods.
- It is better than other production methods regarding shape and size control.
- Corrosion and wear resistance in parts produced with P/M are at good levels.

- Since the powders are blended in the desired composition, producing materials with various properties is easy.
- In this method, porous samples can be produced by leaving the desired space between the powder particles, which can also be used to produce porous metal parts.
- Since scrap parts are generally used to produce Powder in the P/M method, the production cost decreases in this method.
- The P/M method is an economical and automatable method for mass production.

The disadvantages can be listed as follows:

- In the P/M method, the wear levels of the molds used in production must be high. This is because metal powders have corrosive properties. This desired feature in the molds increases the cost of production.
- Metal powders are expensive, and metal powder production is difficult.
- The cost of the powders and other materials used may be high.
- Strength is low in metal materials because the bonds they contain are not strong.
- Metal powders are difficult to transport and store, so care must be taken.
- Changes in density that may occur in part during production can sometimes be a disadvantage [60]

3.4. MATERIAL CHARACTERIZATION

3.4.1. Samples of Powder

There are several strategies you might use to complete the challenging and time-consuming powder sample procedure. Taking little samples from different places, combining them, and applying them is standard procedure. The overall form of the particles is coherent, and given the conditions, there is a strong chance that they will adhere to one another. Aggregation of particles is a potential outcome. An overabundance of aggregation might be caused by surface wetness. Weak forces hold

together particles known as clinging agglomerates and are susceptible to destruction by tiny shear pressures. Most of the time, mechanical and ultrasonic agitation techniques influence particle dispersion and subsequent attribute measurement. The flocculation formation is usually propagated by mechanical mixing or ultrasonic agitation [61].

3.4.2. Measuring Particle Size

Characterizing the size and form of particles is important for many different businesses, but when particles are not perfectly spherical, it becomes more difficult to evaluate these qualities. Particle geometry and characteristic diameter are among the crucial factors to consider. Particle size can be ascertained using techniques such as laser diffraction, dynamic light scattering, sieve analysis, and direct imaging approaches. Sieve analysis is a tried-and-true method for classifying particle size distributions and dividing particles into various size fractions. For particles between 30 μm and 125 μm , dry sieving is the main method; for particles between 20 μm and 3 mm, wet sieving is the method. Cumulative distribution of particle sizes can be achieved by precisely calculating and quantifying the mass percentage of particles in each sieve [62,63].

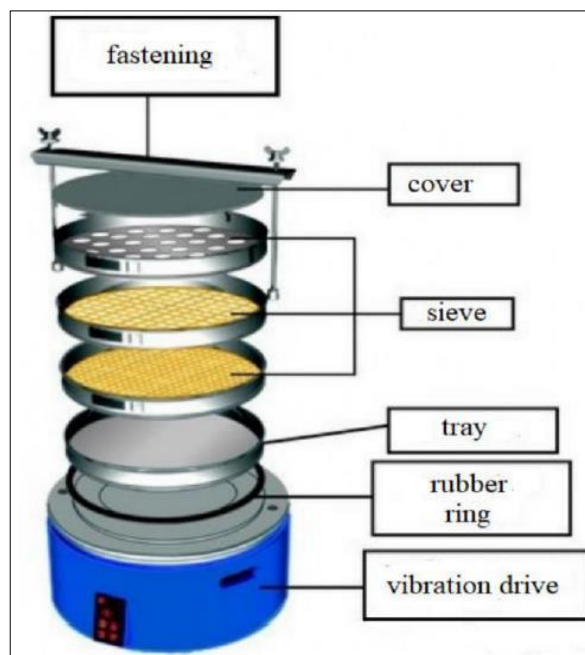


Figure 3.3. Test procedure sieve analysis [64].

3.5. POWDER METALLURGY PROCESSING STEPS

3.5.1. Powder Mixing

In the P/M method, the powder mixing or blending process is required to adjust the specific size distribution for the particles, to bring together the desired powders for forming various alloys by the sintering process, to add lubricant for the pressing process and to form the powder-binder composition in the shaping process. During transportation, vibrations and powders separate according to their sizes, and this separation causes negativities in the pressing and sintering processes. The main reason for mixing powders is to prevent this dimensional separation. There are three reasons for this decomposition in powders. These are the size of the powder particle, its density, and the shape differences of the powders. The P/M method mixing step has parameters such as material, shape or size of the part, properties of the mixer used for mixing, blending speed, or time. These parameters affect the operation performed. In addition, humidity, an environmental factor, is also effective in the mixing process step. Some precautions, such as re-mixing the dry Powder after transportation and avoiding vibrating the dry Powder, allow us to minimize the problems in this process step [55].

3.5.2. Powder Pressing

The next process step after mixing the powders is shaping the powder mixture, pressing, compacting, or briquette. The most well-known shaping process is to fill the powder composition into a mold and shape it using a hydraulic or mechanical press with the help of a punch. The piece formed after this process has a density and strength but is raw; that is, it has not been sintered yet. The produced raw part will obtain its main density and strength in the sintering process, which is the progressive P/M process step.

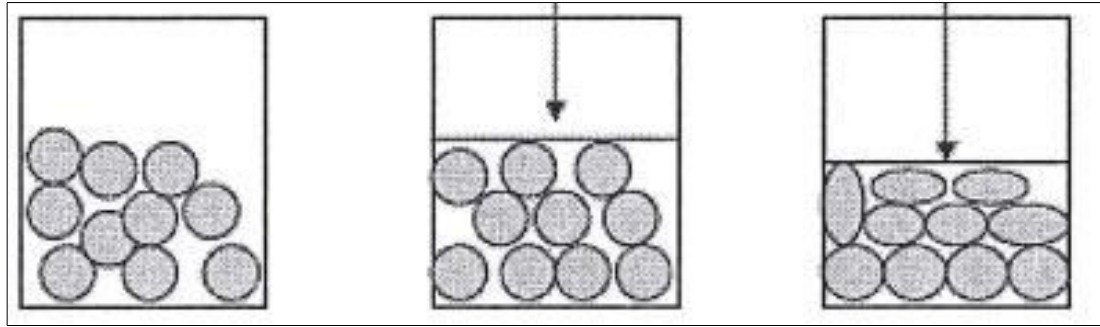


Figure 3.4. One-way pressing [55].

The molds used in the process are designed according to the Powder, press, and size planned for production. The purpose of the compression process is to give the powder composition the desired size or shape and to provide the desired porosity in the structure of the part. As pressure is applied to the compressed part, the pores in the structure decrease, dust particles settle and form bonds, the part begins to resist condensation, and its hardness increases. The pressure must be increased to continue the compression process. Hardness is an important factor in the compression process and is inversely proportional to raw density. The number of contacts before the powders are pressed is very small. Thanks to the applied pressure, the contact points in the powder composition are deformed, allowing the powder particles to merge and become denser. The raw strength obtained after the pressing process depends on the coordination number between the powder particles, contact size, partial density, and contact quality. The partial density mentioned here eliminates the differences specific to the materials in engineering materials with high density and low density [65].

Compression consists of powder composition, mold, lubricant, press, and properties. The molds used here are composite materials containing hard metals for long life and are important for production in the desired shape, size, and density. Molds are production materials designed to be minimally worn and long-lasting. Presses are very diverse and their dimensions are tonnage. Tonnage is the force required for pressing. Parts are produced in tonnage presses according to their sizes. Large tonnage presses are preferred for large parts, and small ones are preferred for small parts. Press types include hydraulic, mechanical, computer numerically controlled (CNC), hybrid, rotary, and anvil-type presses [55].

The piece formed after pressing is fixed in the mold. Abrasions may occur inside the mold due to density differences caused by friction during both the pressing process and the removal of the part from the mold. As the pressure applied increases, the removal of the part becomes more difficult. Another is the growth and expansion in the part dimensions, known as spring back, which occurs during the removal of the part from the mold. Springback causes cracks to form in the produced part. To avoid all these negative situations, we use lubricants. Some lubricants can be mixed into polymer powder or applied to the mold walls. Lubricants are generally stearic acid-based, but there are also animal-derived lubricants [55,65].

3.5.2.1. Single Action Die Compaction

In traditional powder compression processes, uniaxial compression is commonly employed. The top pressure plate begins the compression as soon as the powder mixture is introduced into the mold. Utilizing the bottom piston facilitates the easy removal of the final product from the mold. To expedite the extraction of components, lubricant is applied to the mold walls, aiding in the compression process. During single-action compaction, as the material is compressed, its density increases, typically leading to a rise in the applied pressure. This increase in density might be attributed to a reduction in the number of voids in the powders used, resulting in a decrease in volume while maintaining constant mass. Although this method is useful in certain applications, it is not ideal for producing metal components with high length-to-width ratios and complex designs, as it may not achieve the necessary density.

3.5.2.2. Double Action Die Compaction

Punches in compression processes possess a significant amount of energy. A punch's impact intensity can vary depending on whether its trajectory is uniform or variable. The distribution of raw density in the Powder exhibits considerable variability, influenced by factors such as the frictional forces generated by the Powder, punches, particles, and mold surface. To address these issues, some practitioners use lubricants to reduce friction or employ specific compression techniques to mitigate these effects.

Achieving complete density using the single-action compaction method is challenging. However, the double action compaction method, where both upper and lower punches compress the Powder, increases friction between the powder particles and the mold wall, overcoming the spring force of the material. This technique allows the mold to apply consistent pressure on the Powder with both punches as they descend to the lowest position. After compression, the component is ejected from the mold by the upward movement of the bottom punch. This results in a more uniform density distribution compared to components produced by single-action compaction. Typically, the lowest density value is found in the central part of the compacted area, following a symmetrical distribution pattern [66].

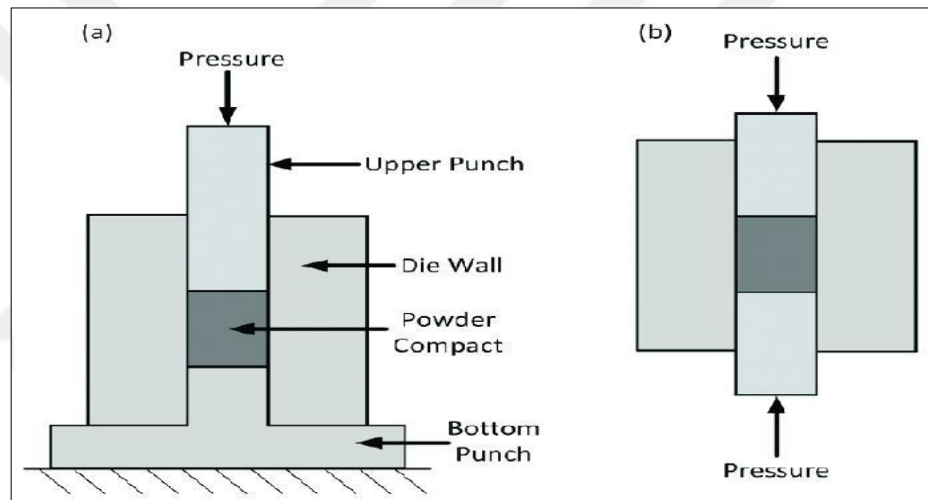


Figure 3.5. Graphic diagrams of single-action die compaction and double-action die compaction [67].

3.5.3. Isostatic Pressing

The process of pressing metal particles under hydraulic (fluid) pressure is known as isostatic pressing. In isostatic pressing, compressible powder particles are placed in a mold made of a flexible, sealed material, and each point of the mold is subjected to a high-pressure force. The parts made by the isostatic pressing technique have the same density throughout, regardless of the part's dimensions, such as its diameter and length. The homogeneous density arrangement is guaranteed by the uniform force applied to all surfaces and the absence of friction on the mold wall. It is a practical and necessary

method for producing objects with complex shapes. Several application sectors exist for both hot and cold isostatic pressing [68].

3.5.3.1. Cold Isostatic Pressing

The pressing process is typically carried out at room temperature using either cold water or oil. These features and methods give rise to the name "cold isostatic pressing" for this process. In an oil-prepared pressing section Figure 3.6, the powders are placed into a rubber mold and pressed using the cold isostatic pressing technique. Because there is oil in the mold, every sample point has the same density and gets the same pressure value from the pressure piston when pressure is applied. While 1400 MPa compression pressures can be attained, the procedure usually occurs at less than 350 MPa. For the cold isostatic process, flexible materials such as rubber, polyvinyl chloride, or elastomers are commonly used to produce the mold. Because flexible molds are used, this approach works well for creating parts with complex shapes. The cost of mold is greatly decreased when rubber is used. Tool steel billets and super alloy aircraft turbines are prime examples of this pressing process [69].

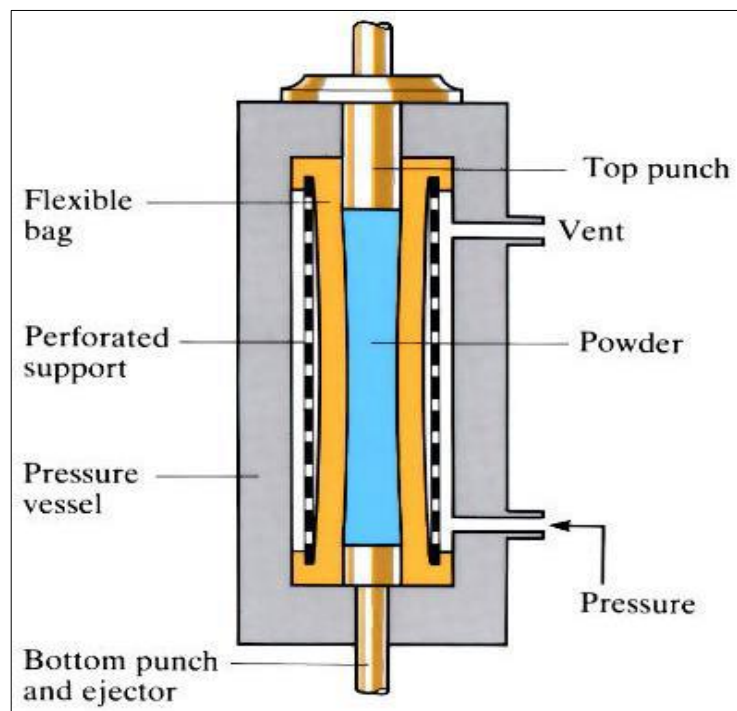


Figure 3.6. Cold isostatic pressing [69].

3.5.3.2. Hot Isostatic Pressing

Pressure is applied to the statically or dynamically heated Powder along a single axis from one or more locations in opposing directions. Pressing hot metal into a compressed box is one of the most traditional ways of compacting powder metal. Please see Figure 3.7. Although hot pressing yields products with superior qualities, there are several disadvantages. These include the gap between the box walls and the moving bushings, die wear, tool wear, sample loss, the boiling of the liquid metal from the punch surfaces to the die walls, and the ensuing deterioration of the squeezed surfaces [70].

Hot pressing requires less energy, less time, and colder temperatures than traditional cold pressing techniques. This method also produces densities that are higher than those of cold pressing. Hot pressing is a method used to compress the molten metal. Additionally, rich approaches are provided. The physical properties of the metal determine the method of selection. Every hot-pressing technique has some traits in common [71]. The hot isostatic pressing technique may provide products with high metallurgical bonding, corrosion resistance, outstanding surface quality, density close to the theoretical density, and very good mechanical properties. The aviation industry favors employing this method to create components.

In contrast to the cold isostatic pressing method, the hot isostatic pressing method crushes powder particles in a chamber filled with an inert gas, such as argon, instead of a liquid. This method raises the temperature value to 1700°C by applying pressure between 150 and 350 MPa.

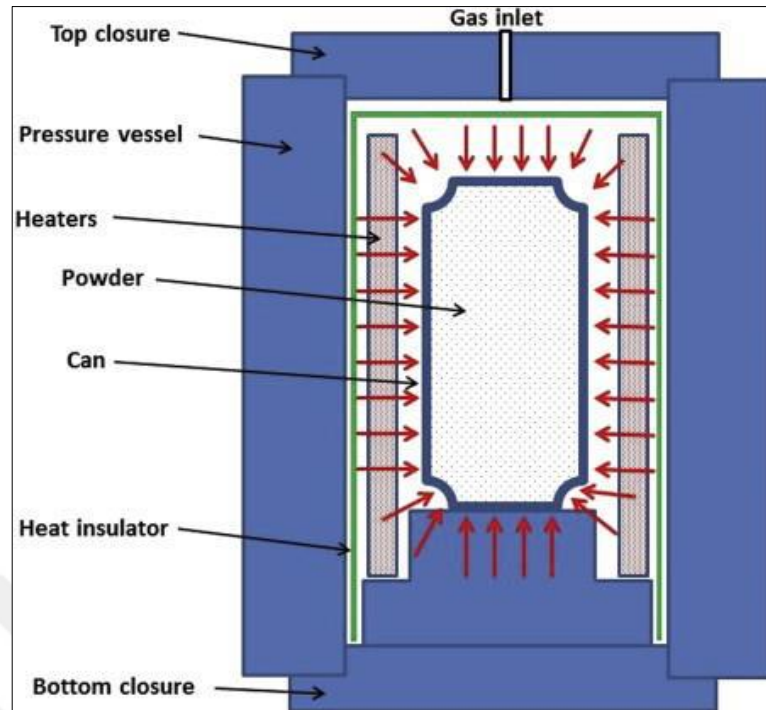


Figure 3.7. Hot isostatic pressing [72].

3.5.4. Sintering

The sintering process, the last of the P/M method process steps and the most complex but basic is the part where the part with temporary strength is transformed into engineering products with very good performance in the desired quality and size. The sintering process is also simply called the heating process or baking process.

Sintering binds the particles together, causing a significant increase in strength and improvement of properties. In addition to forming inter-particle bonds, they have secondary purposes, such as oxidation removal. Dimensional changes that occur during the sintering process are caused by heating, which creates movement of atoms. The energy required to move atoms is called "activation energy". In powder manufacturing, surface area or surface energy is created by transferring energy to the material. This superficial area or energy created through the sintering process is removed. Reductions in the particle's geometry are inversely proportional to the surface energy per unit volume. Therefore, small-size particles sinter faster [55,73].

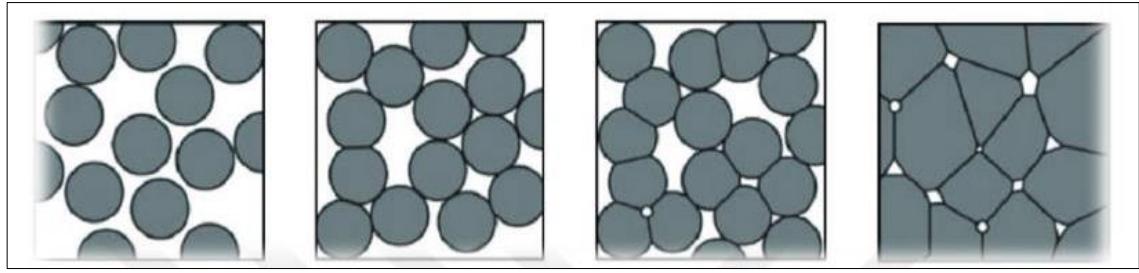


Figure 3.8. Sintering process [74].

When we examine Figure 3.8, we see that in the sintering process, first-point bonds are seen between the powder particles. The contact points of the bonding particles grow, and a neck is formed. Grain boundaries occur in the neck-formed parts. In the process, the number of pores between the particles gradually decreases. Grain boundaries enlarge, creating a more homogeneous structure in the material [75].

Sintering applications are carried out under a special protective atmosphere or vacuum. The characteristics of the atmosphere preferred here are as follows:

- Possible oxidation, etc., between the part and its surroundings. to prevent or reduce reactions.
- To clean the part used in the sinter from absorbed wastes, oxide films, and foreign substances.
- To provide one or more elements that will accelerate the sintering process by forming an alloy with the part used in the sinter.

3.5.4.1. Sintering of Liquid Phase

During sintering, metal powder particles transform instead of melting when exposed to temperatures below their melting point. Utilizing a combination of powders in the sintering process proves advantageous, allowing the procedure to be executed at a temperature below the fusion point of the mixture's constituents. Typically, materials are not subjected to sintering temperatures exceeding their low melting points. As mentioned in reference [76], this method is known as liquid phase sintering.

Throughout the sintering process, a coherent mass of powdered particles and a liquid phase form. Liquid phase sintering accelerates both the initiation of particle aggregation and the overall sintering rate [77]. Capillary forces generated by the liquid phase draw the powder particles together, facilitating a self-sustaining condensation process independent of external pressure. Subsequently, a fluid phase allows for reorganization by reducing inter-particle friction. This results in noticeable changes in the pore structure and component characteristics, including antiparticle bonding, flexibility, strength, conductivity, and corrosion resistance.

Solid state-controlled sintering is a significant advancement in techniques, showing notable improvements over traditional liquid-stage sintering methods. In this stage, the condensation process slows down due to the formation of a skeletal framework [78]. Typically, sintering compacts involve using single-phase particle structures within a temperature range of about 80% of the material's melting point. Indicators of changes during sintering include dimensional shifts and microstructural alterations, which are telltale signs of transformations in the component's mechanical and physical properties. Decreased strength can be attributed to changes in the raw materials' microstructure.

In the first stage of sintering, the contact area between powder particles significantly increases, leading to a notable reduction in the distance between their axes and the smoothing of pre-existing pores. This reduces the component's size and increases its density. The particles gradually coalesce in the subsequent phase. Over time, pores naturally develop and diminish in size as materials move through the framework. Grain growth is observed as a final stage [79].

3.5.4.2. Sintering of Solid State

Solid-state sintering refers to the transformative process when a single-element mixture is subjected to sintering. This process changes the mixture's dimensions, morphology, and mechanical and physical properties. Reactive power aids the sintering process by reducing the system's free energy. Consequently, there is an observed increase in dust and grain accumulation, along with a reduction in the total

surface area of the grain boundaries. The intensification of these boundaries becomes more pronounced as their degree of curvature increases. Atomic and boundary movements are primarily governed by temperature, with a significant exponential increase in atomic emission as temperature rises. Due to the dynamic movement in the grain boundary area, larger grains form while smaller particles gradually diminish.

The solid-state sintering process is driven by the mobility of materials and the dispersion of grains. Sintering at elevated temperatures is advantageous due to the increased propensity for material diffusion. The material's structural integrity remains intact during the sintering process. Including a solid phase within a material facilitates two intrinsic phenomena: diffusion and density augmentation. The difference in free energy between the surface and neck regions of the grain provides the essential impetus for solid-state sintering within the material's internal framework. The process begins with point contact between grains, leading to grain displacement and increased inter-grain interaction. Over time, the contacting particles develop necks, also known as sintering bridges. Despite their proximity, there remains a distinct separation between the dust particles in this area. As the first stage nears completion, the particle centers with neck development gradually draw closer, forming volumetrically shrunken grains [80].

The intermediate sintering stage is characterized by the growth of inter-particle necks and the reshaping of particles beyond a certain neck width threshold. Both inter- and intra-particle spaces reduce during this stage. As void ratios decrease, the material undergoes physiological contraction. Grain formation occurs as particles aggregate towards the end of the intermediate stage [81].

3.6. CHARACTERISTICS OF MATERIALS FABRICATED THROUGH POWDER METALLURGY TECHNIQUES

3.6.1. Mechanical Characteristics

When a single element is sintered into a mixture, the solid-state sintering process alters the mechanical and physical properties of the component and its dimensions. A critical

aspect of sintering is reactive power, which reduces the system's available energy. The rise in dust and grains and a decrease in the total grain boundary area can be attributed to free energy dissipation. There's an acceleration in grain boundaries alongside an increase in curvature radii. Temperature predominantly governs the mobility of atoms and boundaries, with the rate of atomic emission rising sharply as temperature increases. This dynamic movement in the grain boundary area leads to the formation of larger grains while smaller particles disappear simultaneously. The solid-state sintering process is driven by material displacement and grain diffusion. Heating the material to high temperatures is one of the most effective methods to ensure consistent diffusion during sintering. It is crucial to note that the material's internal structure remains unchanged post-sintering. A solid phase within the material facilitates two inherent processes: diffusion and an increase in density. Considering the free energy difference between the grain surface and neck areas is essential to understanding the consolidation process within the material's internal structure. The process of solid-state sintering initiates when grains come into proximity. The first stage is marked by grain mobility, followed by a second phase where increased and more pronounced reciprocal contact occurs.

Over time, particles nearby often develop sintering bridges or necks. Despite their proximity, dust particles in this area maintain their distinct spatial arrangement. At the end of the first stage, particle centers progressively move closer due to neck formation, resulting in compressed grains with reduced volume.

During the intermediate sintering process, the gaps between particles become noticeably larger. Particle shape changes only occur if the neck width exceeds a certain threshold. There is a discernible reduction both within and between particles. As void ratios decrease, the material's physical structure contracts. Grain development takes place at the culmination of the intermediate stage.

3.6.2. Microstructural Features

The microstructure of a material is defined by its geometric configuration, the arrangement of its constituent elements, and the chemical and structural characteristics

of these components. This microstructure encompasses various flaws and phases that constitute the material. Significantly, a material's microstructure profoundly influences its properties. Understanding the unique aspects of a material's microstructure and how various factors relate to it is crucial when determining how to utilize the ore. In materials science, establishing these connections involves one analytical approach, while discerning the material's microstructure requires a different perspective.

3.6.3. Surface Related Properties

The surface of a material is susceptible to oxidation and corrosion. In materials produced by P/M, especially those with a high porosity rate, fluid formation within the pores and its subsequent accumulation can accelerate the corrosion process. Based on our observations, it can be inferred that materials sintered under optimal conditions demonstrate the greatest resistance to environmental stressors [82].

CHAPTER 4

MATERIALS AND METHODS

The investigation has developed innovative composite materials by incorporating stainless steel 316L with additions of titanium (Ti) and molybdenum (Mo). All samples were fabricated using P/M techniques. The sizes and purity levels of the powders used are detailed in Table 4.1.

Table 4.1. Sizes purities and density of the powders.

| | Elemental powders | Size (μm) | Purity % | Density (g/cm^3) | Supplied Company |
|---|----------------------|---------------------------|-------------|--------------------------------|---------------------|
| 1 | SS316L | <149 | 99.9 | 7.95 | Höganäs, USA |
| 2 | Ti | <45 | 96.5 | 4.54 | Aldrich, Germany |
| 3 | Mo | <150 | 99.9 | 10.28 | Aldrich, Germany |

The size of particles in powders impacts their flowability, packing density, and sintering behavior. Titanium (Ti) has the smallest particle size (<45 μm), resulting in better sintering characteristics and surface finish in additive manufacturing processes. High purity levels (99.9% for SS316L and Mo) are critical for applications where material properties cannot be compromised by impurities. Titanium (96.5%) has lower purity due to refining difficulties or acceptable impurities. Density values show differences in material properties, with Molybdenum (Mo) having the highest density (10.28 g/cm^3) for high mass and thermal stability, and Titanium (Ti) suitable for lightweight applications like aerospace components. Suppliers like Höganäs and Aldrich ensure powders meet quality and consistency standards for research and industrial applications.

Figure 4.1 illustrates the step-by-step process employed in the fabrication of these samples

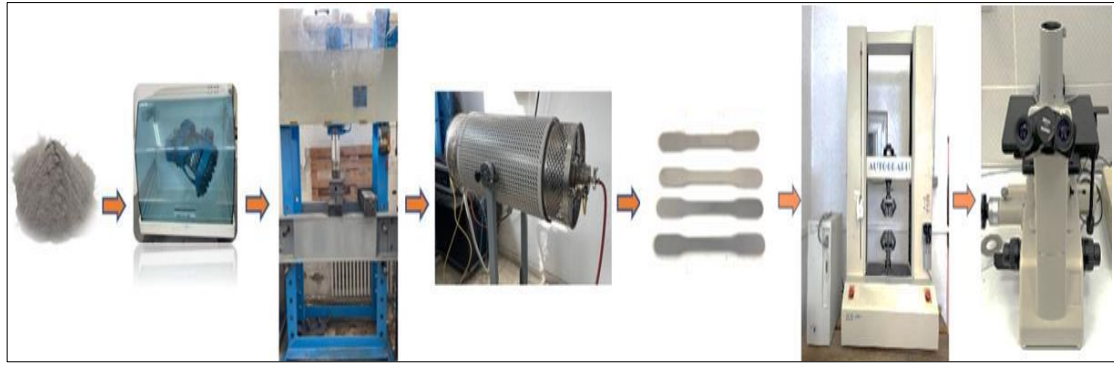


Figure 4.1. The sequence of operations of this study.

By following these steps, we can develop a thorough understanding of a material's properties and performance. Each step is crucial for ensuring the quality and reliability of the final product, from initial powder preparation to detailed microscopic analysis. This comprehensive approach enables the optimization of materials for specific applications, leading to improved products and innovations in various industries.

Table 4.2 lists the different compositions of 316L stainless steel with varying amounts of molybdenum (Mo) and titanium (Ti), all subjected to the same thermal treatment profile (1310°C for 6 hours in a neutral environment).

Table 4.2. Chemical compositions of powder metal steels.

| Composition | |
|-------------------------|----------------|
| 316L | 1310°C - 6h-Ne |
| 316L+0.1Mo | 1310°C - 6h-Ne |
| 316L+0.2Mo | 1310°C - 6h-Ne |
| 316L+0.1Ti | 1310°C - 6h-Ne |
| 316L+0.2Ti | 1310°C - 6h-Ne |
| 316L+0.1Ti+0.2Mo | 1310°C - 6h-Ne |
| 316L+0.1Mo+0.2Ti | 1310°C - 6h-Ne |
| 316L+0.2Mo+0.2Ti | 1310°C - 6h-Ne |

By analyzing the results of mechanical testing and microscopic analysis for these different compositions, it is possible to optimize the material for specific uses, leading to better performance and longer lifespan in practical applications.

4.1. MIXING

The preparation of the powders begins with precise weighing using a RADWAG AS-60-220 C/2 precision scale, known for its 0.0001 g measurement accuracy as shown in Figure 4.2. This high level of precision is crucial for ensuring that each component of the powder mixture is measured accurately, thereby maintaining consistency in the experimental results. Accurate weighing helps minimize variations, ensuring that the proportions of each element in the powder mixture are correct.



Figure 4.2. RADWAG AS-60-220 C/2 weigh device.

Following the precise weighing, the powders are blended using a TURBULA T2F device from Willy A. Bachofen AG, Muttensz, Switzerland as shown in Figure 4.3. This blending device operates on the basis of three-dimensional motion and is used to meticulously mix the powder compositions over a period of one hour. The TURBULA T2F ensures that the powder particles are uniformly mixed, which is crucial for achieving homogeneity in the final product. Homogeneous mixing ensures that the desired enhancements in mechanical and chemical properties, such as those provided

by alloying elements like molybdenum (Mo) and titanium (Ti), are uniformly distributed throughout the material.



Figure 4.3. Mixing TURBULA T2F device.

The thorough blending process facilitated by the TURBULA T2F also plays a key role in process efficiency and property optimization. By ensuring a uniform distribution of the alloying elements within the base powder, the blending process helps achieve consistent properties in the final sintered product. This level of thoroughness is essential for subsequent steps such as compaction and sintering, which further enhance the material's properties. The meticulous preparation and blending of the powders lay a strong foundation for the entire material preparation process, ensuring reliability and reproducibility in the results.

4.2. PRESSING

As required by the ASTM E8M standards [83], the powders were meticulously crushed and formed into the required shape for the tensile test specimens. This critical step was achieved using a robust 100-ton hydraulic press manufactured by Hidrolikсан as shown in Figure 4.4. The pressing process was executed with an impressive precision, setting the pressure to a remarkable 750 MPa. This high level of pressure ensures that

the powders are compacted to the desired density and structural integrity, which is essential for accurate and reliable tensile testing.



Figure 4.4. Hydraulic pressing (Hidroliksan machine).

4.3. SINTERING

Following the pressing process, the material was subjected to sintering in an argon environment, utilizing the device shown in Figure 4.5. The sintering process involved heating the material at a controlled rate of 5°C per minute until reaching a peak temperature of 1310°C , maintained for a duration of 6 hours. This careful control of the heating rate ensures uniform temperature distribution and optimal bonding between powder particles.

In the subsequent phase of the experiment, the samples were cooled at a controlled rate of 5°C per minute from the peak temperature down to room temperature. This controlled cooling rate is crucial to prevent thermal shocks and to maintain the material's structural integrity and desired properties. The entire sintering process, both heating and cooling, plays a vital role in achieving the final microstructure and mechanical properties of the material, making it suitable for its intended application.



Figure 4.5. Sintering device.

After sintering, the samples are shown in Figure 4.6.



Figure 4.6. Produced samples after sintering.

4.4. MEASUREMENT OF DENSITY

Using the Radwag density kit, as shown in Figure 4.7, the densities of the specimens were determined in accordance with the Archimedes principle-based ASTM B 328-96 standard [84]. This method involves measuring the weight of the specimen in air and then in a fluid, typically water, to calculate the density based on the displacement of the fluid. The Radwag density kit ensures precise and accurate measurements, essential for verifying the material's properties and ensuring consistency with the required standards.



Figure 4.7. The Radwag density kit.

This approach is critical for applications where material density directly influences performance and quality.

4.5. TENSILE TEST

The tensile strength of the sintered samples was evaluated using a 50 kN SHIMADZU tensile testing machine (Shimadzu, Tokyo, Japan) as shown in Figure 4.8. The testing was conducted at a controlled crosshead speed of 1 mm/min. This precise setup ensures

accurate measurement of the tensile properties, providing critical data on the mechanical performance of the samples.



Figure 4.8. SHIMADZU tensile test device.

For every test, a stress-strain graph was acquired. By closely examining these graphs, we were able to precisely measure the samples' yield strength (0.2%), tensile strength, and strain values. This investigation also allowed us to ascertain the degree of variation in mechanical qualities brought about by modifications in the chemical composition.

4.6. MEASUREMENTS OPTICAL

The cutting samples were molded in cold molding using epoxy and silicone mold as shown in Figure 4.9.

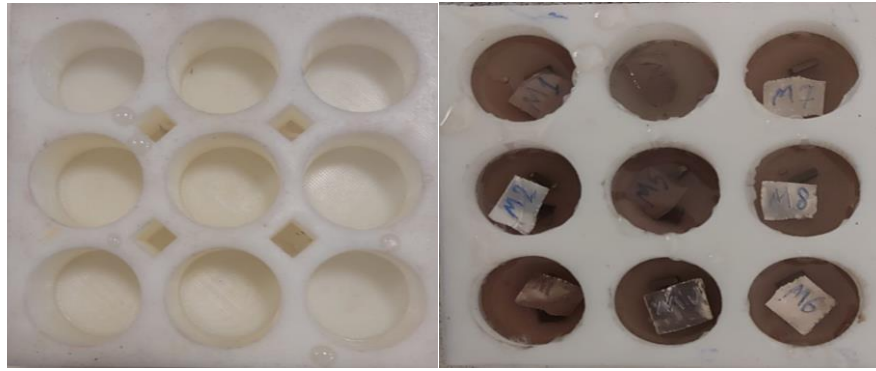


Figure 4.9. Cold molding mold.

Before using the optical microscope, the sample surfaces underwent a detailed preparation process. This included polishing with a range of grinding papers of varying mesh sizes, from 400 to 7000 meshes, progressing from coarse to fine. Afterward, a $0.3\ \mu\text{m}$ Al_2O_3 solution was employed for the final polishing.

The samples were then etched for a duration of 3 seconds in a solution composed of 2% nitric acid and 98% ethyl alcohol. Finally, each sample was thoroughly cleaned with distilled water and ethyl alcohol, followed by a meticulous drying process using hot air. Figure 4.10 shows the grinding and polishing device.



Figure 4.10. Grinding and polishing device

The optical microscopy analysis was conducted using the highly accurate and excellent Nikon ECLIPSE L150 microscope, manufactured in Melville, New York, USA, as shown in Figure 4.11. The mean linear intercept method was employed to calculate the grain sizes of the composites [85].



Figure 4.11. Nikon ECLIPSE L150 microscope.

Additionally, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) were utilized for further analysis. SEM provided detailed images of the sample surfaces, allowing for a closer examination of the microstructural features. EDX analysis complemented this by identifying and quantifying the elemental composition of the samples.

4.7. CORROSION TEST

In order to ensure the samples' conductivity with the corrosion unit, copper wire was connected to them before they were sliced to the proper size as shown in Figure 4.12. Subsequently, the samples were cold molded with epoxy resin to provide insulation. This preparation step was crucial for maintaining the electrical conductivity required

for corrosion testing while also ensuring that the samples were adequately insulated to prevent any unwanted interference during the tests.

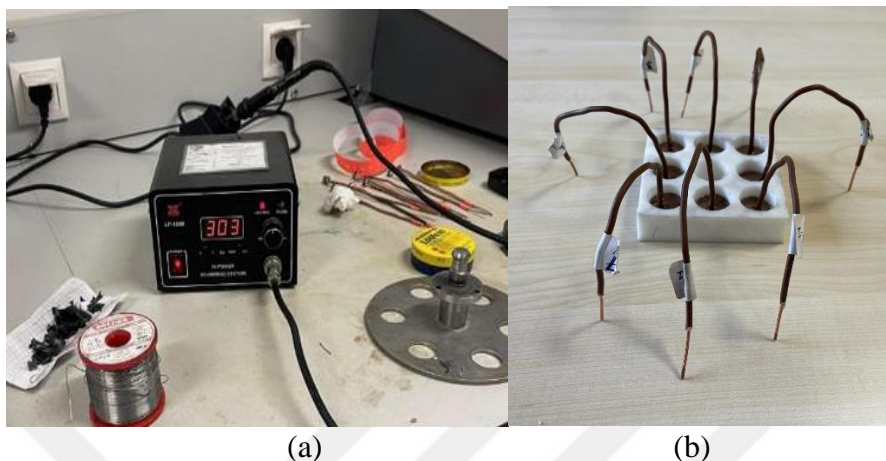


Figure 4.12. a) Soldering station device. b) Cold molding for corrosion samples.

The conductivity of samples was checked via voltmeter device. After that variety of abrasive paper meshes were used to prepare the samples (400, 600, 800, 1000, 1500, 2000 and 2500 meshes, coarse to fine).

As shown in Table 4.3, the SBF were synthesized with the chemical composition at pH 7.4 and $T = 36.6\text{ }^{\circ}\text{C}$. Add small paragraph about prepare SBF

Table 4.3. Chemical composition of simulated body fluid (SBF) [86].

| Reagent | Amount |
|---|--------------|
| NaCl (g) | 8.035 |
| NaHCO ₃ (g) | 0.355 |
| KCl(g) | 0.225 |
| K ₂ HPO ₄ .3H ₂ O(g) | 0.231 |
| MgCl ₂ .6H ₂ O(g) | 0.311 |
| CaCl ₂ (g) | 0.292 |
| Na ₂ SO ₄ (g) | 0.072 |
| Tris (hydroxymethyl) aminomethane(g) | 6.118 |
| 1M HCl (ml) | 40 (balance) |

After cleaning the test surface, the samples were washed with ethyl alcohol and distilled water. To prepare for corrosion testing, a large piece of adhesive tape with a 0.25 cm² diameter hole was applied to the specimen's surface. This step aimed to minimize potential negative effects from the epoxy bonding areas.

The corrosion tests for all specimens were conducted in a controlled environment. The potentiodynamic polarization experiments were performed in a simulated body fluid (SBF) solution at a temperature of 37°C, as shown in Figure 4.13. The solution was prepared using a hot plate stirrer to ensure thorough mixing and accurate temperature control.



Figure 4.13. Prepared the (SBF) solution.

The testing setup included a computer-operated Gamry model PC4/300 mA potentiostat/galvanostat for DC105 corrosion research. The electrochemical cell, depicted in Figure 4.14a, was configured with three electrodes: a saturated calomel reference electrode (Ag/AgCl), a graphite counter electrode, and the working electrode. To minimize power loss, the capillary lugging was carefully positioned as close to the working electrode as possible.

The Tafel corrosion setup, shown in Figure 4.14b, was used to perform the polarization measurements. This meticulous configuration ensured accurate and reliable measurement of the corrosion properties of the specimens. The data obtained from these tests provided valuable insights into the effects of different material compositions and surface preparations on corrosion resistance.

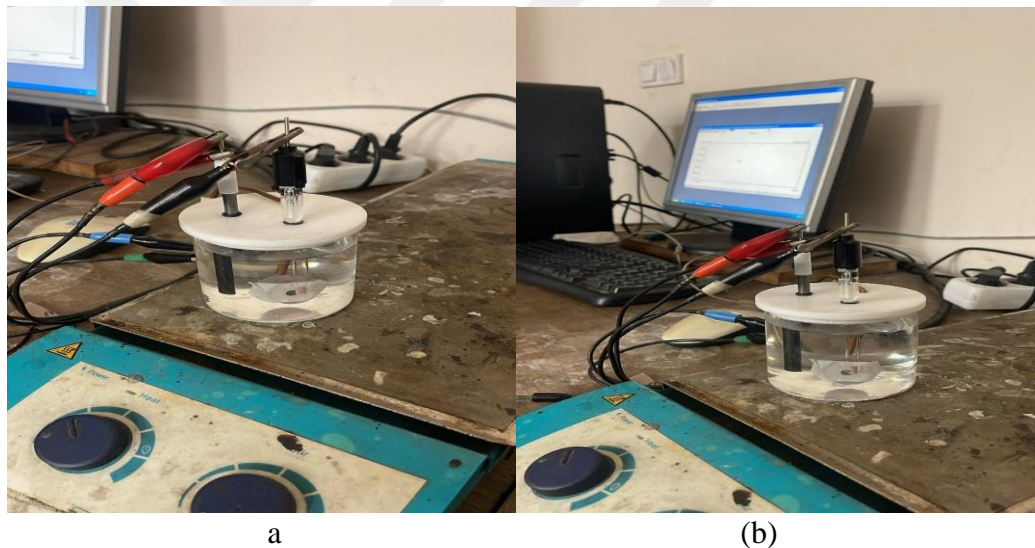


Figure 4.14. a) Three-electrode electrochemical cell, b) Tafel corrosion set.

4.8. WEAR TEST

A variety of abrasive paper meshes were used to prepare the samples for the wear test (400, 600, 800, 1000, 1500, 2000, and 2500 meshes, coarse to fine).

The tribometer gadget, 4D-ECN type, was employed for wear tests. The wear test was performed with a stroke of 10 mm, a total of 100 meters of sliding distance for each

load, and a sliding speed of 40 mm/s in an SBF solution at 37 °C under 20N and 40N loads as shown in Figure 4.15. For the wear test, 6mm-diameter alumina balls were utilized.

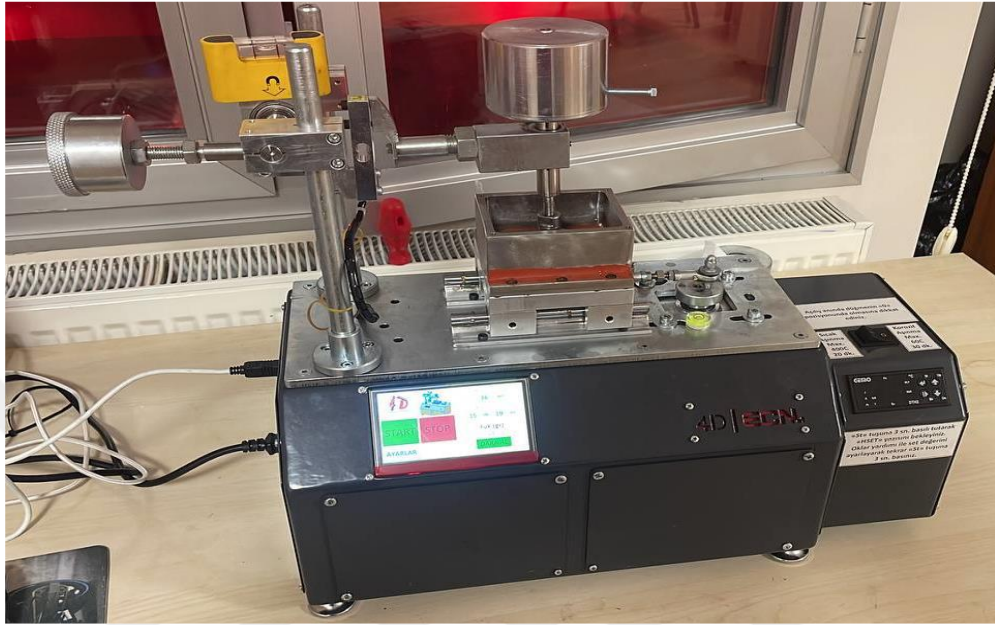


Figure 4.15. Wear test device.

After the wear test, the surface profiles of the samples were determined using a profilometer (Mitutoyo SJ-410, Tokyo, Japan) to investigate the depth of the wear marks on the surface of the alloy steel as shown in Figure 4.16. This step provided precise measurements of the wear depths, which are critical for assessing the extent of material loss due to wear.



Figure 4.16. Mitutoyo SJ-410.

Subsequently, SEM images were taken to examine the wear lines in detail using the Zeiss Gemini Ultra55 Plus (Figure 4.17). SEM provided high-resolution images of the wear surfaces, revealing the microstructural changes and wear patterns that occurred during the testing.



Figure 4.17. Zeiss Gemini Ultra55 Plus.

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1. MICROSTRUCTURE

Microstructure images of the samples produced with the PM technique were taken at 750 MPa pressure and sintered in an argon atmosphere at 1310°C for 6 hours. Figure 5.1. To Figure 6.6. It shows optical microscope images of samples produced by the PM method before and after adding alloying elements (Ti and Mo).

When 0.1% Mo, 0.2% Mo, 0.1% Ti, 0.2% Ti, 0.1%Ti + 0.2%Mo, 0.2% Mo + 0.1% Ti and 0.2% Mo + 0.2% Ti are added to 316L Stainless steel, respectively, the grain size in the microstructure increases. It has been observed that the grain size decreases depending on the amount of addition, and the grain size decrease gradually increases with double additions to see the synergistic effect.

Ti has a high affinity for carbon. Therefore, the formation of TiC (titanium carbide) can be observed with the addition of Ti. These carbides often precipitate at grain boundaries, and these precipitations can limit grain growth. Titanium can react with nickel to form intermetallic phases. These phases are generally hard and brittle, which can reduce the ductility of the material. With the addition of Ti, the precipitation hardening mechanism can be activated. In this case, the hardness and strength of the material may increase. Ti can precipitate at grain boundaries and inhibit grain growth. This can reduce the grain size of the material and increase microstructural homogeneity [87].

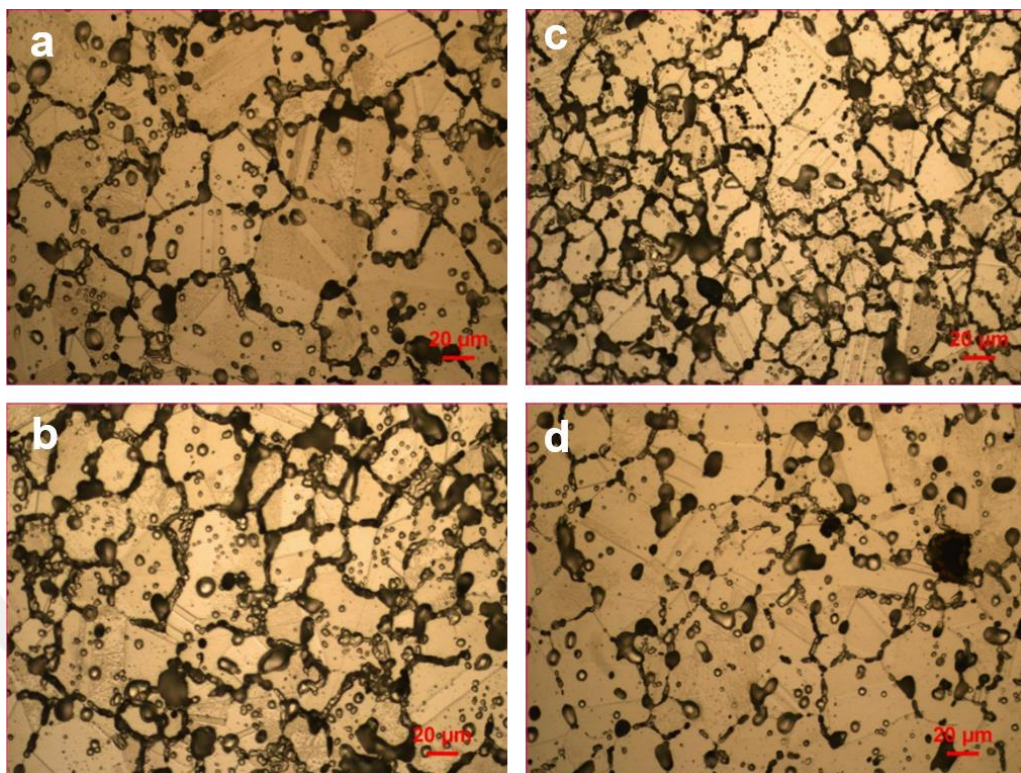


Figure 5.1. a) 316L, b) 316L-0.1Mo, c) 316L-0.2Mo and d) 316L-0.1Ti.

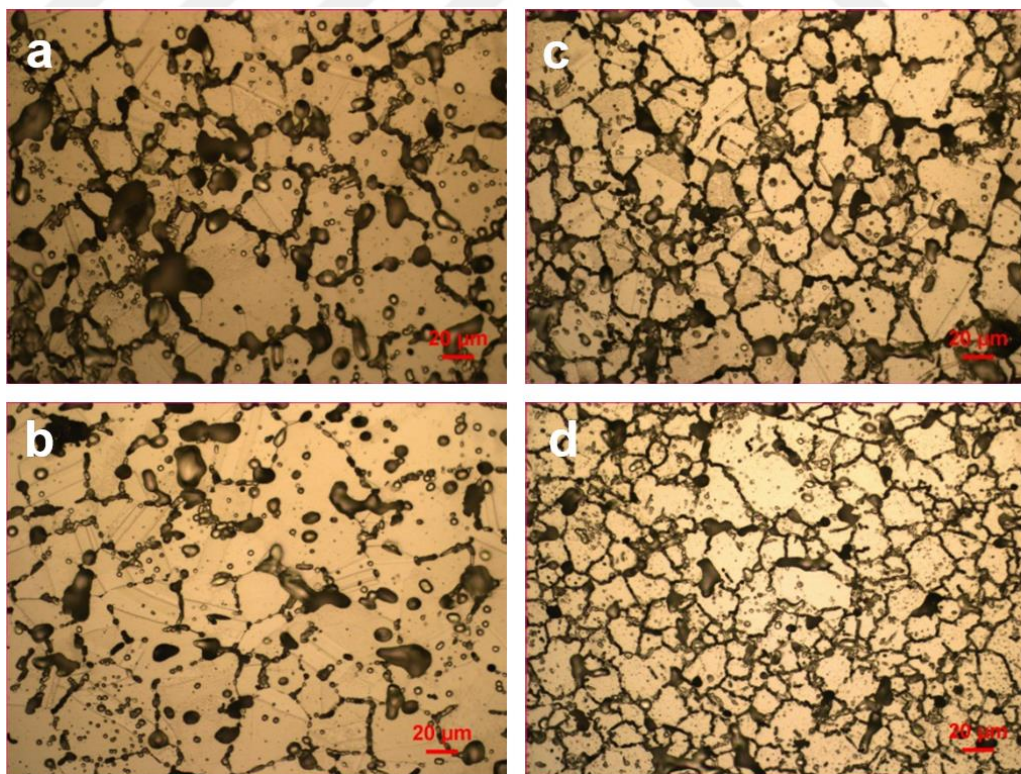


Figure 5.2. a) 316L-0.2Ti b) 316L-0.1Ti-0.2Mo, c) 316L-0.2Ti-0.1Mo and d) 316L-0.2Ti-0.2Mo

5.2. MECHANICAL PROPERTIES

Tensile tests were used to ascertain the resulting alloys' mechanical characteristics. Based on the information gathered, Table 6.1 presents the findings of the density measurement and the tensile test.

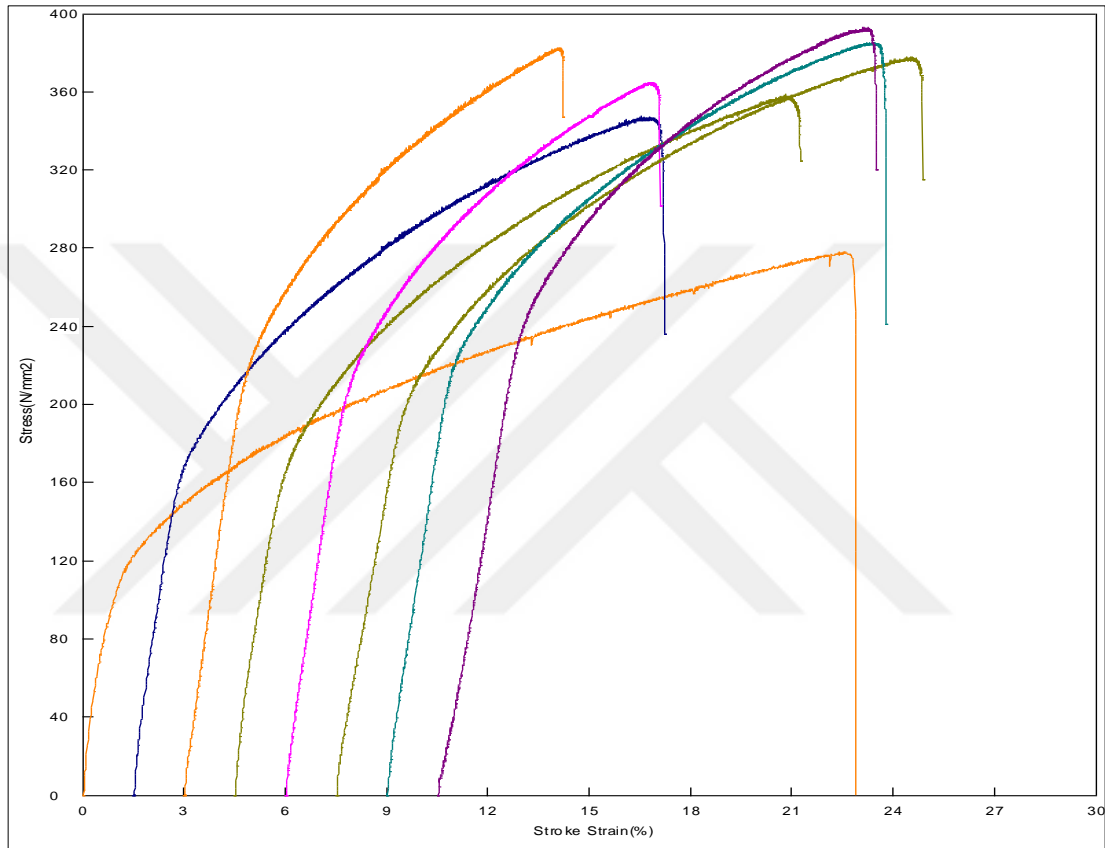


Figure 5.3. Tensile test results of PM steel samples.

When Figure 5.3 and Table 5.1 are examined, the graph and maximum tensile and elongation values obtained as a result of the drawing of the produced powder metal samples are seen. When Table 5.1 is examined, it is seen that 316L stainless steel has a maximum tensile strength of 279 MPa and a total elongation value of 26%. When 0.1% molybdenum is added to 316L stainless steel, the tensile strength increases to 348 MPa, and when 0.2% molybdenum is added, it increases to 383 MPa. The effect on tensile strength was not as effective as the addition of Titanium as the addition of Molybdenum. Considering the synergistic effect of Titanium Molybdenum elements, the highest tensile strength value of 394 MPa is read in the 316L+0.2 Ti+0.2Mo alloy.

Table 5.1. Mechanical properties of PM steel samples.

| Number | Alloys | Ultimate Tensile Stress (MPa) | Elongation (%) |
|--------|------------------|-------------------------------|----------------|
| 1 | 316L | 279 | 26 |
| 2 | 316L-0.1Mo | 348 | 16 |
| 3 | 316L-0.2Mo | 383 | 12 |
| 4 | 316L-0.1Ti | 360 | 15 |
| 5 | 316L-0.2Ti | 365 | 11 |
| 6 | 316L-0.1Ti-0.2Mo | 379 | 16 |
| 7 | 316L-0.1Mo-0.2Ti | 386 | 15 |
| 8 | 316L-0.2Mo-0.2Ti | 394 | 13 |

Ti reacts with carbon in steel to form titanium carbide (TiC). These carbides precipitate at grain boundaries and within the matrix. TiC particles increase the hardness and tensile strength of steel by making dislocation movement more difficult. Ti precipitates at grain boundaries, limiting grain growth. A smaller grain structure generally provides higher strength. This is known as the Hall-Petch effect and increases tensile strength. The addition of Ti can activate the precipitation hardening mechanism. This mechanism increases the hardness and strength of the material through the precipitation of carbides within the matrix.

Mo atoms increase the hardness of the material by preventing dislocation movement. This results in an increase in tensile strength. Mo increases strength by making it harder for dislocations to slide. Mo reacts with carbon to form molybdenum carbide (MoC). These carbides are hard phases that increase the hardness and tensile strength of steel. Mo precipitates at grain boundaries and inhibits grain growth. This keeps the grains smaller and increases the tensile strength.

Both Ti and Mo form carbide (carbide) precipitations in steel. These carbides increase tensile strength by making dislocation movement more difficult and forming hard particles within the matrix. Both Ti and Mo precipitate at grain boundaries, inhibiting grain growth. Smaller grains increase tensile strength. Ti and Mo additions increase the strength of the material by activating the precipitation hardening mechanism. This mechanism occurs through the precipitation of carbides within the matrix. The addition of Ti and Mo to 316L stainless steel can increase the tensile strength by various

mechanisms. Ti addition increases strength through carbide precipitations, grain boundary strengthening and precipitation hardening, while Mo addition increases tensile strength by making dislocation movement more difficult and inhibiting grain growth. The combination of these two elements can significantly improve the tensile strength of 316L stainless steel, making it usable in more demanding mechanical applications. However, determining the optimal alloy composition is critical to achieving the desired mechanical properties [88].

5.3. CORROSION TEST AND RESULTS

Three electrodes were used in the electrochemical cell for these experiments: the working electrode, a graphite counter electrode, and a saturated calomel reference electrode (Ag/AgCl). To guarantee that there was as little power loss in the configuration as feasible, the capillary lugging was carefully placed in relation to the working electrode.

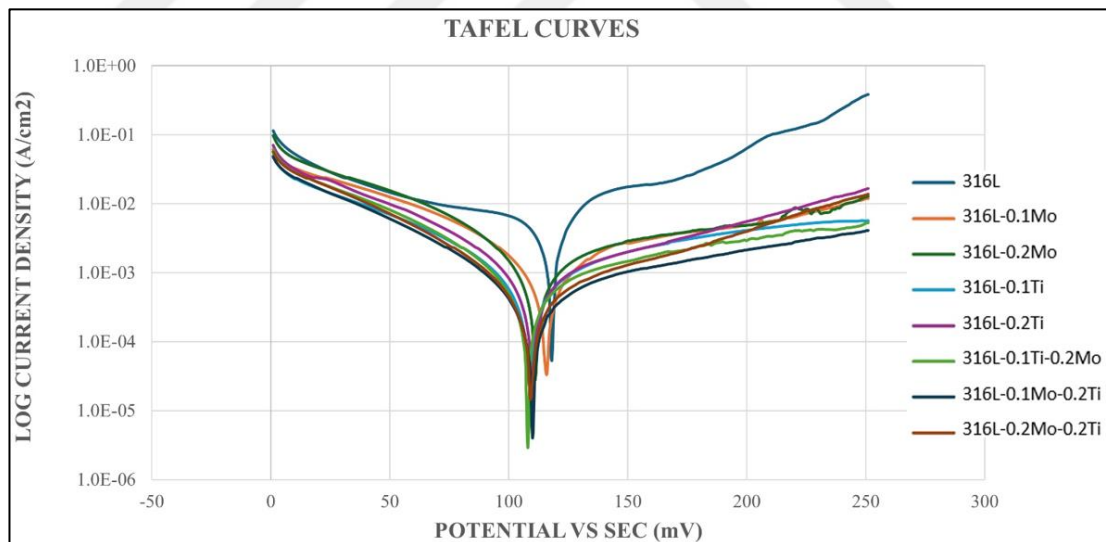


Figure 5.4. Tafel curves of samples.

Molybdenum added to 316L stainless steel increases resistance to pitting corrosion, especially in environments containing chloride ions. This type of corrosion causes localized damage to the surface of the steel, creating small pits that can deepen over time. Molybdenum helps prevent the formation of these pits. Molybdenum also

provides resistance to intergranular corrosion. This is a type of corrosion that occurs along the grain boundaries of the steel and is especially common in heat-treated or welded areas. Molybdenum prevents intergranular corrosion by reducing carbide precipitation at the grain boundaries. Molybdenum increases the oxidation resistance of steel at high temperatures. This allows the steel to be used for longer periods at higher temperatures. Thanks to these contributions of molybdenum, 316L stainless steel is widely used in various fields such as chemistry, petrochemistry, marine and medical devices.

Titanium bonds with carbon to form titanium carbide (TiC). This prevents carbon from bonding with chromium and reduces the formation of chromium carbide. This reduces the risk of vehicle corrosion and increases the high temperature resistance of the steel. Titanium helps protect the grain boundaries of the steel, preventing vehicle corrosion and increasing the overall corrosion resistance of the material. This property is especially important in welded areas. Titanium also forms a protective oxide layer on the surface, which increases the oxidation resistance of the steel. This improves the performance of the steel, especially at high temperatures [89].

Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) analyses of corroded surfaces after molybdenum (Mo) and titanium (Ti) were added to TM 316L stainless steel provide various important information. These analyses help to evaluate the microstructure and chemical composition of the surface.

In the SEM examination performed after corrosion, pitting or oxide layers are observed on the surface. Since the molybdenum addition provides protection against pitting corrosion in particular, pitting formation is seen to be at a minimum level in the 316L+0.2Mo+0.2Ti sample in SEM images.

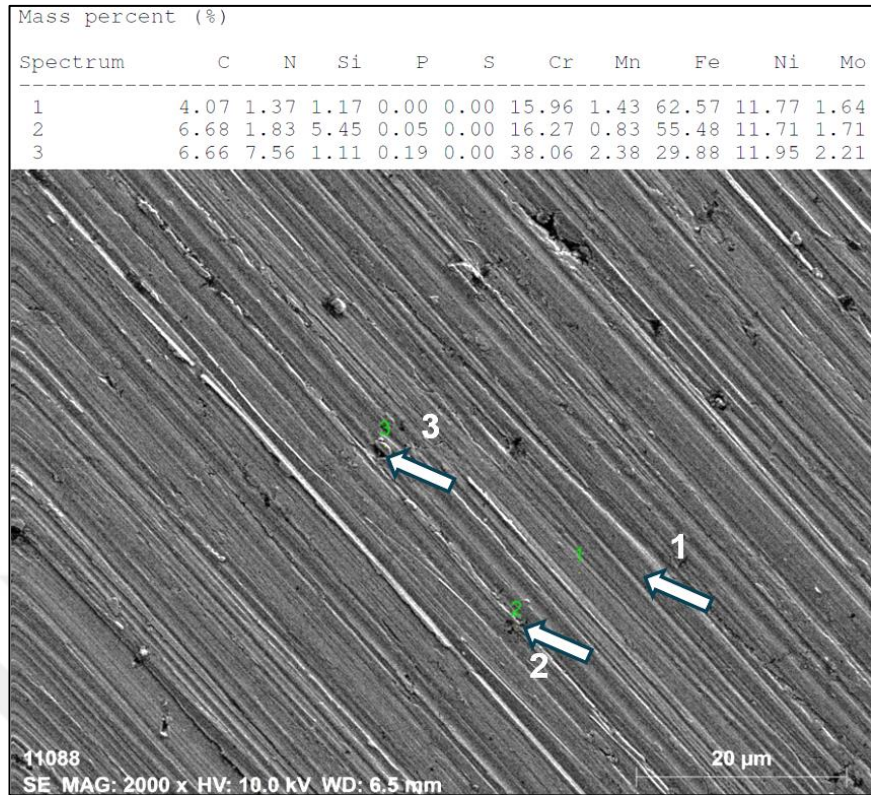


Figure 5.5. SEM image and EDX results of the 316L sample after corrosion test.

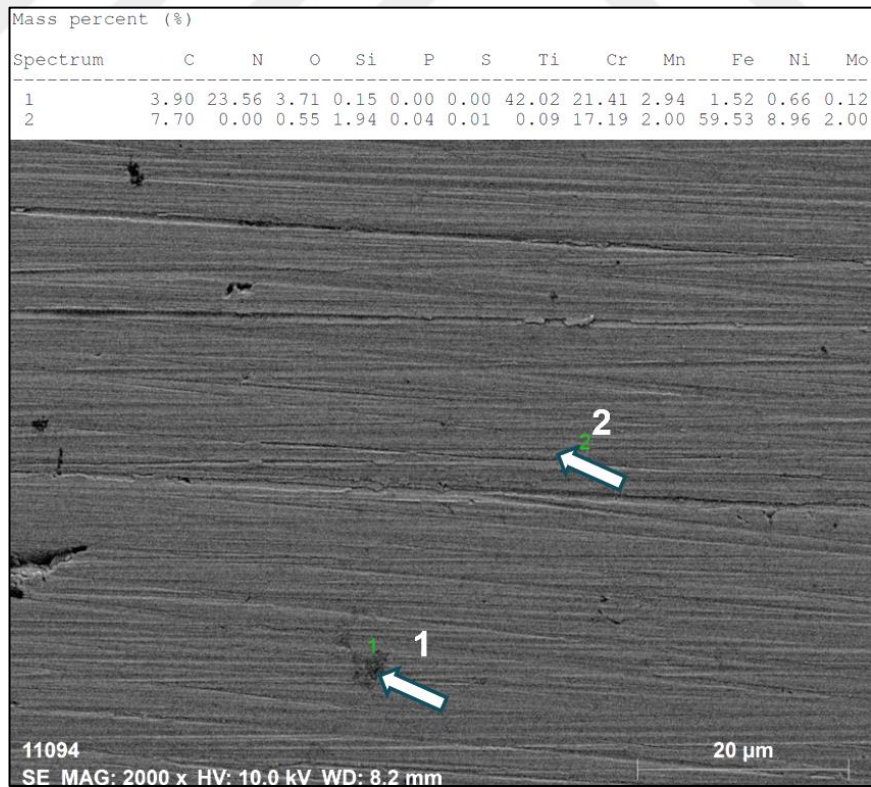


Figure 5.6. SEM image and EDX results of the 316L+0.2Mo+0.2Ti samples after corrosion test.

It can be interpreted that the presence of titanium contributes to the smoothness and uniformity of the surface, together with the reduction of carbide precipitates around the grain boundaries.

EDX analysis determines the distribution and density of elements on the surface. The presence of molybdenum and titanium on the surface after corrosion indicates that it causes a chemical change. EDX results show that additional elements contribute to the protection of the surface in the 316L+0.2Mo+0.2Ti sample compared to the 316L sample. The presence of titanium can be observed as titanium oxide (TiO_2) in the EDX analysis. This oxide is thought to provide an additional layer of protection against corrosion by passivating the surface.

5.4. WEAR TEST AND RESULTS

Abrasion tests were performed on 316L matrix samples, which are also utilised as biomaterials in the study, using bodily fluid. The wear test was conducted at a sliding speed of 0.048 m/sc, a wear distance of 100 m, with loads of 10, 20, and 40 N. An abrasive was made out of a 6 mm diameter ball of Al_2O_3 , which is also used as a biomaterial. The wear test data produced findings that were in line with the tensile and hardness data. The wear test results under a 40N load are displayed in Figure 6.9. Following the wear test, the area loss detected by the surface profilometer device was multiplied by the 10-mm trace length to get the overall volume loss. Upon analysis of the findings, sample 316L had the largest volume loss at 40N load. The sample with the highest tensile findings, 316L+0.2Ti+0.2Mo, also had the lowest volume loss.

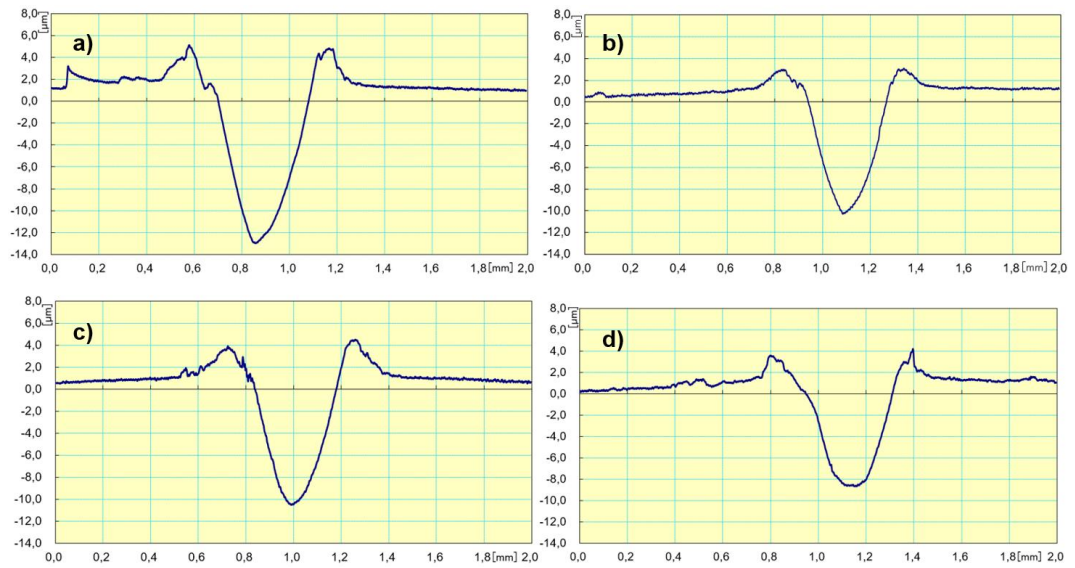


Figure 5.7. Area loss measurements of the samples after the wear test (a) 316L, b) 316L-0.2Mo, c) 316L-0.2Ti and d) 316L-0.2Ti-0.2Mo.

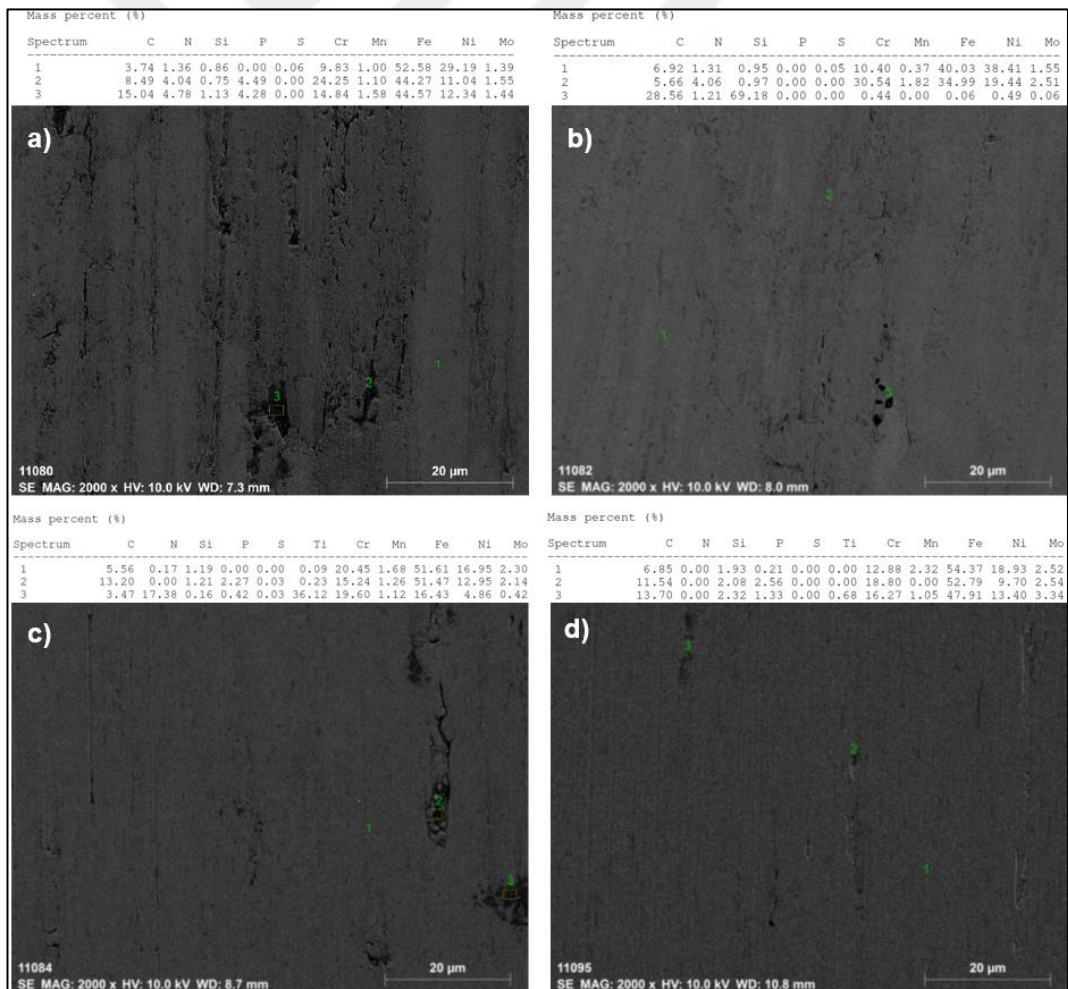


Figure 5.8. Surface SEM images after wear test. (a) 316L, b) 316L-0.2Mo, c) 316L-0.2Ti and d) 316L-0.2Ti-0.2Mo.

The effect of molybdenum (Mo) and titanium (Ti) addition to PM 316L stainless steel on SEM (Scanning Electron Microscope) images of worn surfaces has increased the wear resistance of the material and caused significant changes in the structure of wear marks, deformations and oxide layers on the surface.

Molybdenum addition increases the hardness and wear resistance of 316L stainless steel. It can be said that this leads to less wear marks and microcracks on the surface in SEM images. It has also been reported in the literature that the surface is more uniform and smooth compared to steels without molybdenum after wear. It can be said that molybdenum reduces the number and size of microcracks and pitting on the surface. A significant decrease in the number of pitting and cracks on the worn surface was observed in SEM images.

Titanium promotes the formation of TiC (titanium carbide) on the surface, which increases the surface hardness and improves wear resistance. In SEM images, fine and uniform carbide precipitates formed by the addition of titanium can be observed. These precipitates can contribute to a more durable surface and a reduction in wear marks.

The addition of molybdenum and titanium significantly increased the wear resistance and surface stability of PM 316L stainless steel. SEM images provided direct observation of these improvements. Less deformation, pitting, microcracks and more uniform oxide layers were observed on the worn surface. This makes the material more durable and long-lasting, especially when used in corrosive environments.

CHAPTER 6

CONCLUSION

In this study, the effect of adding Ti and Mo elements to 316L stainless steel alloy produced by powder metallurgy method was investigated for 6 hours under cold pressure (750 MPa) and sintering temperature (1310 °C) in argon atmosphere and the following results were obtained:

- From the microscopic images, it is seen that the grain size in the microstructure decreases when 0.2 Ti and 0.2Mo are added to 316L stainless steel.
- When Ti and Mo (0.2% by weight) are added to the original stainless steel alloy without using additives, the tensile and yield strength of the steel alloy increases due to the precipitation of the elements and it is thought that these precipitations prevent grain growth.
- It was observed that better wear and corrosion performance was obtained compared to other alloys when 0.2%Mo+0.2%Ti was added to 316L stainless steel.

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RESUME

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