

# INVESTIGATION OF THE EFFECT OF Cr AND AI ADDITION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF POWDER METAL 316L STAINLESS STEEL

# 2024 MASTER THESIS MECHANICAL ENGINEERING

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Abdalla A Ahmed ABODRABIKA

#### ABSTRACT

#### M. Sc. Thesis

## INVESTIGATION OF THE EFFECT OF Cr AND AI ADDITION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF POWDER METAL 316L STAINLESS STEEL

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Karabuk University Institute of Graduate Programs The Department of Mechanical Engineering

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The thesis focuses on improving the properties of 316L stainless steel by adding Chromium (Cr) and Aluminum (Al) using powder metallurgy (P/M) technology. This material is very important in various industries due to its excellent corrosion resistance and robust mechanical properties. However, there is a need to further develop these features to meet the demands of advanced industrial applications in sectors such as aerospace, automotive and biomedical. Features such as its corrosion behavior, how it interacts with the body, biocompatibility, good fatigue strength, increased ductility, low cost and mechanical machinability make 316L stainless steel especially preferred for implant applications. This study aims to fill the knowledge gap regarding how Cr and Al additions affect the microstructure and mechanical properties of 316L stainless steel processed through powder metallurgy. The research investigated the effect of varying Cr and Al amounts on the 316L stainless steel matrix. The process involves mixing chromium and aluminum powders with 316L stainless steel, pressing them into blocks and sintering them in an argon atmosphere. The resulting samples were subjected to comprehensive analysis. Tensile tests and stress-strain diagrams, density measurements, microstructure analysis, hardness testing and wear testing were used for characterization.

Key Words : Powder metallurgy; 316L stainless steel; Chromium; Aluminum; Mechanical properties, Microstructure, Wear.

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## ÖZET

#### Yüksek Lisans Tezi

## TOZ METAL 316L PASLANMAZ ÇELİĞE Cr VE AI İLAVELERİNİN MİKROYAPI VE MEKANİK ÖZELLİKLERİ ÜZERİNE ETKİSİNİN İNCELENMESİ

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Çalışma, toz metalurjisi (P/M) teknolojisi kullanılarak Krom (Cr) ve Alüminyum (Al) ilave edilerek 316L paslanmaz çeliğin özelliklerinin geliştirilmesine odaklanmaktadır. Bu malzeme, mükemmel korozyon direnci ve sağlam mekanik özellikleri nedeniyle çeşitli endüstrilerde çok önemlidir. Ancak havacılık, otomotiv ve biyomedikal gibi sektörlerdeki ileri endüstriyel uygulamaların taleplerini karşılamak için bu özelliklerin daha da geliştirilmesine ihtiyaç vardır. Korozyon davranışı, vücutla nasıl etkileşime girdiği, biyouyumluluğu, iyi yorulma dayanımı, artırılmış sünekliği, düşük maliyeti ve vii mekanik işlenebilirliği gibi özellikleri 316L paslanmaz çeliğini özellikle implant uygulamaları için tercih nedeni olmaktadır. Bu çalışma, Cr ve Al ilavelerinin toz metalurjisi yoluyla işlenmiş 316L paslanmaz çeliğin mikro yapısını ve mekanik özelliklerini nasıl etkilediğine ilişkin bilgi boşluğunu doldurmayı amaçlamaktadır. Araştırma, değişen Cr ve Al miktarlarının 316L paslanmaz çelik matris üzerindeki etkisini araştırılmıştır. İşlem, krom ve alüminyum tozlarının 316L paslanmaz çelikle karıştırılmasını, bloklar halinde preslenmesini ve argon atmosferinde sinterlenmesini içermektedir. Ortaya çıkan numuneler kapsamlı bir analize tabi tutulmuştur. Çekme testleri ile gerilim-gerinim diyagramları, yoğunluk ölçümleri, mikroyapı analizi, sertlik testi ve aşınma testi karakterizasyon için kullanılmıştır.

Anahtar Kelimeler : Toz metalurjisi, 316L paslanmaz çelik, krom, alüminyum, mekanik özellikler, mikroyapı, aşınma.

**Bilim Kodu** : 91437

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## SYMBOLS AND ABBREVITIONS INDEX

## SYMBOLS

- Cr : Chromium
- Ni : Nickel
- Mo : Molybdenum
- V : Vanadium
- Mn : Manganese
- Fe : Iron
- Ti : Titanium
- Al : Aluminum
- Nb : Niobium
- C : Carbon
- W : Tungsten
- Mg : Magnesium

## **ABBREVITIONS**

PM : Powder Metallurgy

#### PART 1

#### **INTRODUCTION**

Powder Metallurgy (P/M) technology stands at the forefront of contemporary material production, renowned for its ability to efficiently and economically massproduce small parts [1]. At its core, this technology involves the precision compression of metal-based powders into predefined shapes and sizes, which are then sintered to perfection in controlled environments-either in a vacuum or under a protective gas atmosphere at optimal temperatures. This versatile method leverages metal powders of varying sizes and grades, enabling the creation of parts that are not only durable but also exhibit high performance. The process of P/M is meticulously comprehensive, encompassing several critical stages. It begins with the careful preparation of metal powders, mixed in precise ratios, followed by the processes of shaping and pressing. The culmination of this process is the sintering phase, where heat plays a pivotal role in forging the final product. Every step in this sequence is governed by specific parameters, each influencing the characteristics of the end material, underscoring the precision and complexity of this method. In the realm of modern production technologies and industrial practices, P/M is highly esteemed for its harmonious integration of efficiency, energy conservation, and optimal utilization of raw materials. One of its most laudable attributes is its capability to produce highperformance components economically, with minimal wastage or even achieving near-zero loss in some instances. P/M's notable strengths—such as its manufacturability, cost-effectiveness, and the uniform quality of the products-have made it a popular choice, offering a compelling alternative to traditional manufacturing methods. Moreover, components fabricated through P/M technology are distinguished by their smoother, cleaner surfaces, often negating the need for any secondary finishing processes. This advantage is particularly beneficial in the production of components that are challenging to manufacture by conventional means. As technological advancements continue to escalate human needs and

demands, the significance of research and development in materials science becomes ever more pronounced. Within this sphere, the production of metal matrix composites holds a position of importance, further highlighting the indispensable role of Powder Metallurgy in modern manufacturing and technological innovation [1–6].

Steel is an alloy formed from the combination of the iron element and varying carbon amounts, typically between 0.2% to 2.1%. To alloy iron, elements like nickel, chromium, magnesium, vanadium, manganese, silicon, cobalt, niobium, and tungsten can be added. The addition of chromium and nickel gives steel its stainless characteristic. Stainless steel contains about 11% chromium. Altering the chemical composition of steel can yield alloys with different properties. Increasing chromium levels or adding nickel and molybdenum can enhance the corrosion resistance of stainless steel [2]. Additional elements like titanium, copper, molybdenum, niobium, nitrogen, sulfur, and silicon also positively impact the alloy. Stainless steels are preferred for their excellent mechanical properties, ability to maintain these properties at high and low temperatures, ease of processing, and resistance to wear and corrosion. Their superior wear and corrosion resistance compared to other steel types make them widely used in various fields, including the biomedical sector for medical, dental implants, surgical and laboratory instruments [7].

This thesis aims to investigate the effect of adding chromium (Cr) and aluminum (Al) to 316L stainless steel matrix using powder metallurgy (P/M) technology. The study aims to synthesize composites containing varying amounts of Cr and Al, evaluate its effect on the microstructure of 316L stainless steel, evaluate the mechanical properties of composite materials, and conduct comprehensive tests to understand the material. features. The scope includes powder metallurgy processes, material composition, microstructural analysis, mechanical testing and additional testing. The research aims to contribute valuable knowledge to materials science, especially on improving the properties of 316L stainless steel through alloying with Cr and Al and optimizing the production process for advanced industrial applications.

## PART 2

#### **BIOMATERIALS**

#### 2.1. DEFINITION AND HISTORY OF BIOMATERIALS

A biomaterial is a synthetic material that has been developed over time to function in close proximity to biological tissue or to replace parts of a living system. Materials, both natural and manmade, that come into contact with biological fluids such as blood or tissue and are meant to be used in prosthetic.Other definitions have also covered storage, therapeutic, and diagnostic applications that do not adversely affect the living body or any of its components [8]. They are defined as any compounds that, while executing their intended function, come into contact with the biological systems of humans or animals. Materials in the forms of gels, liquids, and solids can all be used to generate biomaterials. As biomaterials and their intended uses were established over time, this broad definition changed. It may also be further defined as new medical applications for biomaterials are developed [9]. As civilization has evolved, so too has the study of biomaterials, which now encompasses a wide range of materials at different length ranges, from nano to macro, with the obvious objective of extending and enhancing human existence. Silver was used as an antibacterial agent in different forms to prevent illness more than a millennium ago. Early stages of human civilization can also be observed via various surgical procedures but without a question, the most important advancements in the field of biomaterials happened between 1901 and 2000. Millions of patients have seen an improvement in their quality of life over the last seven decades thanks to artificial joints [10].

#### **2.2. TYPES OF BIOMATERIALS**

Among the main categories of biomaterials that are widely used in biomedical applications are metals, polymers, ceramics, and composites. There are four main classifications for biomaterials [11].

- 1) Metal
- 2) Polymer
- 3) Ceramics
- 4) Composites

#### **2.3. METAL AS A BIOMATERIALS**

"Vanadium steel" was the first metallic alloy created specifically for human use; it was used to make screws and bone fracture plates, also known as Sherman plates. The body can only withstand very modest amounts of the majority of metals, including iron (Fe), cobalt (Co), chromium (Cr), titanium (Ti), tantalum (Ta), nickel (Ni), molybdenum (Mo), niobium (Nb), and tungsten (W), that are used to make alloys for implant manufacturing. The body cannot tolerate high concentrations of some metallic elements, yet occasionally they are required for the synthesis of vitamin B-12 (Co) or red blood cells (Fe) in their naturally occurring forms [12].

#### 2.4. STAINLESS STEELS

Stainless steels are iron-based alloys that have at least 11% chromium (Cr). Include additional alloying elements such as titanium, molybdenum, nickel, silicon, aluminum, sulfur, copper, nitrogen, niobium, and selenium in addition to chromium in order to improve specific qualities [13]. When "ordinary" steels are exposed to these corrosive processes, the term "stainless" describes the lack of staining, rusting, or corrosion [14]. These alloys are named after their "stainless" quality, which is also what makes them desirable. In most cases, the stainless characteristic is achieved via the surface formation of a coating of chromium oxide, or Cr2O3. This thick, hard, sticky, and self-healing coating of oxide stops the underlying alloy from oxidizing

any further [15]. If cutting scratches or damages this layer, it rapidly recovers in the presence of oxygen. Despite being extremely thin (around 5 m), this layer is strong, nonporous, and offers corrosion resistance adequate for the service environment. These alloys oxidize at almost zero rate in air at ambient temperature [16].

#### 2.4.1. Types Of Stainless Steel

There are five primary categories of stainless steel, each with a unique feature and a variety of corrosion resistance levels.

<b>AISI stainless</b>	Microstructure	Mechanical properties			<b>Physical characteristics</b>
steel	properties	Elastic	Yield	Protraction -	
		strength	strength	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	688-910	409-912	15-45	High strength and heat-
	&ferrite				intolerant
Precipitation	Austenite	890-995	271-992	10-32	high strength heat-treated
hard enable	&martensitic				hardening

Table 2.1. Properties of stainless steel [17].

#### 2.4.1.1. Austenitic Stainless Steels

The most common forms of austenitic stainless steel have additions of 8 to 11% nickel and 17 to 18% chromium. While structural carbon steels have a body-centered cubic (bcc) atomic (crystal) structure, The atomic structure of austenitic stainless steels is face-centered cubic (fcc). Therefore, austenitic stainless steels have high ductility, are easy to cold shape, and are simple to weld in addition to their resistance to corrosion. They also have significantly greater resilience over a larger temperature range. Rather than being heated, they are reinforced via cold working. Corrosion resistance can be further enhanced by increasing the quantity of chromium in them and by adding nitrogen, molybdenum, and chromium [16,18].

#### 2.4.1.2. Ferritic Stainless Steels

They have between 10.5% and 18% chromium, either entirely without nickel or in very modest amounts. Like structural carbon steels, they feature a body-centered cubic (bcc) atomic structure. Compared to austenitic stainless steels, they frequently have lower formability, ductility, and welding capabilities. They can be used in a variety of corrosive environments and, in contrast to austenitic, cannot be reinforced by heat treatment. They can withstand stress corrosion cracking quite well, and the molybdenum addition can make them even more resistant to corrosion [16].

#### 2.4.1.3. Duplex Stainless Steels

Duplex stainless steels have a combination of austenite (fcc) and ferrite (bcc) in their microstructure. In addition to the primary alloying elements of chromium (20–26%) and nickel (1-2%), molybdenum, tungsten, copper, silicon, and nitrogen may be added to control structural balance and provide particular corrosion-resistance qualities [13,16]. When comparable alloying components are used, the corrosion resistance of duplex stainless steels is comparable to that of austenitic stainless steels. Duplex stainless steels have greater yield and tensile strengths, nevertheless. In terms of toughness, duplex stainless steels fall between austenitic and ferritic stainless steels. Duplex stainless steels are stronger than austenitic, but because of their increased ductility, they are less formable [13,16].

#### 2.4.1.4. Martensitic Stainless Steels

Although martensitic stainless steels have a body-centered cubic structure (bcc) akin to structural carbon steels, they can be heat-treated to reinforce it due to their higher carbon content. Tensile strength and moderate corrosion resistance are the outcomes of martensitic stainless steel's hardening and tempering processes. Among the many applications that often call for wear and abrasion are cutlery and medical instruments, industrial knives, wear plates, and turbine blades. in contrast to the stainless steels that are ferritic, austenitic, and duplex. They are less ductile and more notch-sensitive [16].

#### 2.4.1.5. Precipitation Hardening Stainless Steels

They are divided into three microstructure families, martensitic, semi-austenitic, and austenitic, depending on the kind. Their corrosion resistance is typically comparable to austenitic stainless steels with 18% chromium and 8% nickel and better than martensitic stainless steels. The aerospace industry is where they are most commonly utilized for tension bars, shafts, bolts, and other products that need to be extremely strong with only a minimal bit of corrosion resistance [16].

#### 2.4.2. Steel Strength Increasing Mechanisms

The mechanical qualities of materials—that is, how they react to forces applied to them—are largely determined by the behavior of their metallurgical structures. It can be argued that these conditions also affect the material's mechanical properties since they change the metallurgical structure of the material and the heat and mechanical processes that are applied to it. Strength is one of the material's most important properties. Other characteristics of the material may vary depending on its strength. Mechanical resistance can be explained by the material's resistance to plastic deformation when subjected to a mechanical force. If a linear defect is greater than the defect, metals may become plastically deformed. The mechanical characteristics of metals control the quantity of dislocations—tiny pieces of metal that have been dragged out of their usual position—and how they interact with other flaws [19]. The following is a list of mechanisms that increase strength:

- 1) Precipitation Hardening (Aging).
- 2) Hardening by Reducing Grain Size.
- 3) Cold Process.
- 4) Alloy Hardening.
- 5) Hardening by Martensitic Transformation.
- 6) Deformation Aging.
- 7) Hardening by Dispersion.

#### **2.5. ALLOYING ELEMENTS**

When iron (Fe) and carbon (C) come into contact, steel is produced, which has between 0.2% and 2.1% carbon (C). Among the other elements that make up this alloy are vanadium (V), tungsten (W), magnesium (Mg), chromium (Cr), cobalt (Co), molybdenum (Mo), manganese (Mn), and nickel (Ni). When these components are introduced to the substance, the steel hardens or transforms into a stainless form [19].



Figure 2.1. Relationships between the stainless-steel group of alloys' composition and properties [20].

#### 2.5.1. Carbon And Graphite

There are many different forms of the element carbon in nature, which is found in the sixth row of the periodic chart. Its atomic weight is six. It has a gray or black crystal structure with a hexagonal or cubic shape. The study of carbon and its subatomic particles is known as organic chemistry [21]. The increased carbon ratio causes decreased toughness and weldability but increases perlite structures. It does, however, reinforce the yield.

The increased carbon ratio causes decreased toughness and weldability but increases perlite structures. It does, however, reinforce the yield. Furthermore, the presence of martensite and bainitic structures becomes more pronounced with increased carbon content. Carbon can only be utilized up to 0.2% in micro-alloyed steels under hot rolling conditions. However, the carbon ratio is higher than 0.25 percent since the controlled cooling mechanism approach is the only way used to make forged parts, which are commonly used in the automobile sector [22].

The word "graphite" is derived from the Greek word "Graphein," which means writing. Graphite is a substance that has been around for ages. The first pencil composed of graphite was made in England in the fifteenth century. It was not until the 18th century that graphite was discovered to be one of the carbon allotropic materials [23,24]. When thermodynamics is considered, it is found that under atmospheric pressure, graphite has a more stable structure than carbon. The diamond changes into a graphite structure above 1500 °C [25].

Graphite is among the softest materials. It is a special kind of carbon that is mostly used for lubrication. In its native condition, graphite is found in nature. To make it, petroleum coke is processed in furnaces devoid of oxygen. In nature, graphite comes in two varieties: alpha and beta. Alpha and Beta are identical in their physical properties, although having different crystal shapes. It is utilized in the production of steel as well as lubrication. There are two types of carbon in its atomic structure: diamond and graphite [25].

In general, iron is alloyed with carbon. Sometimes, different elements can be used as alloying components, such as manganese, magnesium, and chromium. Changing the ratio of alloying components in the steel composite results in changes to its ductility, hardness, and stress point [26].

#### 2.5.2. Hydroxyapatite

Apatite refers to a class of constituents with structural similarities. The phosphate mineral family known as hydroxyapatite, fluorapatite, and chlorapatite is made up of phosphate minerals and contains OH-, F-, and Cl-ions, respectively. This group's member, hydroxyapatite, has the formula Ca<sub>10</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>. Because of its extremely close calcium phosphate ratio (Ca/P) to that of actual bone and teeth, hydroxyapatite is bioactive and frequently used as an implant material in medical settings [27]. The main inorganic component of the hard tissues of a living organism is hydroxyapatite, a bioceramic composed of calcium phosphate. It is extensively used in many different medical contexts, such as controlled drug release systems, dental implants, and filling materials [28,29]. Owing to its striking similarity to the inorganic mineral components of teeth and bone, it possesses exceptional biocompatibility and distinct bioactivity. They are safe and have no effect on the immune system when incorporated into the bones of people and animals [30]. An additional use for hydroxyapatite bioceramics is in ocular implants. Hydroxyapatite is a great biomaterial for ocular implants because of its non-toxic and biocompatible qualities [28,29]. Because of its structural similarity to the hard tissue in the living body and its bioactive quality, hydroxyapatite can act as a natural glue between the hard tissue and the implant. The binding of hard tissue by hydroxyapatite promotes bone formation in diseased or damaged areas while also preventing the immune system's adverse consequences, such as the formation of fibrous tissue on implant surfaces. The implant adheres to the tissue and keeps it from slipping because the bioactive hydroxyapatite binds to the tissues. Furthermore, the implant is not recognized by the body as a foreign substance. It has been found that bone proteins and other regional development factors have a considerable affinity for HDA. When osteoblasts, the cells that create bone, attach to hydroxyapatite, the substance instantly promotes the growth of collagen and other bone components. The way that hydroxyapatite

stimulates bone formation depends on a number of factors, including the form and size of the pores, the crystal structure, and the chemical composition (Ca/P ratio). As bone fillers, hydroxyapatite comes in a variety of forms, such as powder and porous blocks. The porous structure acts as a network of channels, allowing new cells to proliferate inside of the pores. Enhancing the tissue-implant contact not only prevents implant slippage but also promotes better integration by facilitating the flow of blood and other essential bodily fluids into the tissue. It is recognized as a source of phosphate and calcium as well [31-33].

### 2.5.3. Aluminium Oxide

The mixture of aluminum and oxygen known as aluminum oxide, or alumina, has the chemical formula  $Al_2O_3$  [34]. Aluminum oxide is one of the most important ceramic materials and finds applications in metallurgy, electronics, optics, and nanotechnology, among other domains [35].

Aluminum oxide's outstanding mechanical, thermal, chemical, and physical properties provide it several benefits over other ceramic materials [36]. Al<sub>2</sub>O<sub>3</sub> has gained importance recently for a variety of applications because of its hardness, chemical inertness, high melting point, non-volatility, high refractoriness, and resistance to oxidation and corrosion [34,35].

Every year, more than 45 million tons of Al<sub>2</sub>O<sub>3</sub> are produced for industrial use, and its uses are numerous. Due to its high melting point and hardness, Al<sub>2</sub>O<sub>3</sub> can be used as an effective abrasive and as a potential refractory material [36]. High hardness, high stability, high insulation, and transparency are just a few of the excellent properties that make aluminum oxide ideal for a variety of application domains. It also has a wide range of uses, such as fire retardants, insulators, catalysts, composite materials, and surface protective coatings [37]. Because it serves as a catalyst and a catalytic support in a variety of chemical processes, aluminum oxide is important. Its homogenous size distribution, wide surface area, and outstanding textural properties fall into the mesoporous range. Furthermore, aluminum oxide's inherent acid-base characteristics make it helpful in a number of operations [38].

#### 2.6. MEDICAL IMPLANT CORROSION

The human body still suffers from corrosive implants. This corrosion releases metal ions, and because these ions are not biocompatible, they lead to implant failure [39]. The rusting of medical implants is a constant problem. About 41% of 316L and Ti-6Al-4V medical implant failures are attributed to corrosion [40,41]. Consequently, it's critical to comprehend the various forms of corrosion that impact implants in humans, especially those related to orthopedics [42].

#### 2.6.1. Corrosion

Metal deteriorates through a process called corrosion when it is exposed to harsh and caustic conditions. The metal corrodes as a result of this unfavorable chemical reaction [42]. The human body is thought to be a corrosive and aggressive environment for metal implants. Blood and other body fluids contain water, plasma, amino acids, anions such as phosphate, bicarbonate, and chloride, and cations such as calcium, magnesium, sodium, and potassium. For metal implants, all of them create a corrosive environment [40,41]. Therefore, the metal implant materials need to have a high level of corrosion resistance in order to preserve biocompatibility.

#### PART 3

#### THEORETICAL BACKGROUND

#### **3.1. 316L STAINLESS STEEL**

316L stainless steel is an austenitic chromium-nickel alloy with a low carbon content, enhancing its corrosion resistance. Its chemical composition includes chromium, nickel, molybdenum, and iron, making it a preferred material in various industries. 316L's high chromium and molybdenum content provides excellent resistance to various chemicals and corrosive environments. Despite its high ductility, it maintains strong tensile and yield strength, making it suitable for demanding applications. Its thermal stability remains stable even at elevated temperatures. 316L is widely used in the chemical, pharmaceutical, marine, and food processing industries due to its corrosion-resistant nature. It is also a popular material in biomedical implants, architectural structures, and automotive parts due to its biocompatibility and aesthetic appearance [43,44].

#### 3.1.1. Basic Composition of Stainless Steel

Stainless steel is an iron-based alloy with a minimum of 10.5% chromium, which forms a passive layer of chromium oxide to prevent surface corrosion and internal structure damage. Other elements like nickel, molybdenum, titanium, and manganese enhance its properties.

#### 3.1.2. Classification of Stainless Steel

Austenitic stainless steels are non-magnetic steels with high chromium and nickel content, known for their formability and corrosion resistance. Ferritic steels have moderate chromium content, good ductility, and resistance to corrosion and oxidation. They are magnetic and less formable than austenitic steels. Martensitic stainless steels can be hardened by heat treatment and have moderate chromium and low nickel content. Duplex stainless steels, a combination of austenitic and ferritic steels, offer superior strength and corrosion resistance compared to single-phase alloys. In additive manufacturing, the phase composition of austenitic stainless steels can vary, with the presence of ferrite and other phases influencing their properties [45].

#### 3.1.3. Properties of 316L Stainless Steel

316L stainless steel is a variant of the broader group of 316 stainless steels, known for its lower carbon content and unique composition. Its composition includes 16-18% chromium, 10-14% nickel, and 2-3% molybdenum, which contribute to its corrosion resistance and mechanical properties. The alloy's austenitic microstructure, a face-centered cubic crystal structure, maintains excellent toughness and ductility across various temperatures. The mechanical properties of 316L stainless steel include a good balance between strength and ductility, with a tensile strength in the range of 485-690 MPa. Its hardness is moderate, typically less than martensitic or hardened steels but sufficient for many applications. It also maintains excellent toughness, even at cryogenic temperatures.

The high chromium and molybdenum content provides excellent overall resistance to corrosive environments, including acids, bases, and chlorides. 316L is particularly noted for its resistance to pitting and crevice corrosion in chloride environments, a critical property for marine and chemical processing applications. The low carbon content significantly reduces the risk of intergranular corrosion, a common issue in higher carbon stainless steels, especially after welding or high-temperature exposure. Thermal properties of 316L stainless steel include a relatively high coefficient of thermal expansion but moderate thermal conductivity, important in applications involving temperature variations. The alloy's low carbon content enhances its

weldability, making it less susceptible to welding-related corrosion issues. Additionally, its excellent formability allows it to be easily shaped and fabricated into various components.

#### 3.1.4. Chromium in Stainless Steel

Chromium plays a crucial role in stainless steel, enhancing its corrosion resistance, mechanical properties, and microstructure. It forms a passive layer on the steel's surface, which is self-healing if damaged. The minimum chromium content is typically 10.5%, but higher concentrations can improve corrosion resistance. Chromium also strengthens the steel through solid solution strengthening, preventing the movement of dislocations. It also increases hardness and wear resistance, making stainless steel suitable for critical applications. Chromium also influences phase composition, stabilizing the austenitic phase at room temperature. The balance between chromium and nickel is essential for maintaining the desired austenitic microstructure in stainless steels like 316L. Chromium can alter the temperatures at which phase transformations occur in steel, affecting heat treatment processes and the microstructure. Chromium can influence grain size, leading to improved toughness and strength. Higher carbon steels can form chromium carbides, which can reduce corrosion resistance. However, in low carbon variants like 316L, this risk is minimized. Chromium can also modify the type and distribution of non-metallic inclusions within the steel matrix, impacting properties like toughness, ductility, and the steel's response to welding [46].

#### **3.1.5.** Aluminum in Stainless Steel

Aluminum plays a crucial role in steel, enhancing its oxidation resistance, hightemperature stability, and grain refinement. It forms a stable, protective aluminum oxide layer on the steel surface, which prevents oxygen diffusion and protects against oxidation and scaling. This layer maintains its protective properties at higher temperatures, making Al-alloyed steels suitable for high thermal exposure applications [47].

Aluminum also improves toughness and ductility by refining the grain structure, leading to more uniform and homogeneous properties. This uniformity is beneficial for consistent mechanical properties across the material. Aluminum's influence on

mechanical properties includes solid solution strengthening, which enhances the strength of the steel by hindering dislocation movement. It also indirectly contributes to improved hardness through its role in grain refinement.

The grain refinement effect leads to increased ductility and toughness, particularly at lower temperatures. Aluminum can also improve steel's resistance to stress corrosion cracking, especially in corrosive environments. The addition of aluminum can affect the thermal expansion coefficient and thermal conductivity of steel, which is important in applications involving thermal cycling or high temperatures.

### 3.2. POWDER PRODUCTION TECHNIQUES FOR STAINLESS STEEL

Stainless steel powder production involves various techniques, including gas and water atomization, electrolysis, and reduction. The choice of atomization medium significantly impacts the powder's characteristics, with gas producing a spherical shape and smooth surface, while water produces irregular shapes and rougher surfaces. Other methods include electrolysis or reduction of metal oxides, but are less common due to cost or complexity [48].

Sintering is the process of heating compacted metal powders below their melting point, leading to bonding of particles and densifying the powder compact. The process typically goes through stages including initial particle bonding, pore size reduction, and grain growth, eventually leading to a dense, solid material. Alloying elements like chromium and carbon need careful control during sintering to prevent oxidation and maintain the alloy's integrity. Aluminum can also affect sintering behavior by influencing diffusion processes and the formation of oxides, which can affect the mechanical properties and corrosion resistance of the final product [49].

Controlled atmospheres, such as hydrogen, vacuum, or inert gases, help maintain the alloy's integrity. Post-sintering heat treatments can be applied to optimize the properties of sintered stainless steel, including solution annealing, aging, or stress relieving.

The microstructure of stainless steel is significantly influenced by the sintering process, with the size, shape, and distribution of pores and grain size being critical factors. High density is essential for excellent mechanical properties in sintered stainless steel, and optimizing the sintering process and parameters is crucial for achieving desired properties.

#### **3.3. THEORETICAL MODELS AND COMPUTATIONAL APPROACHES**

Thermodynamic models, such as CALPHAD (Calculation of Phase Diagrams), are used to predict phase equilibrium and phase transformations in alloys, which are crucial for understanding the effects of alloying elements like Cr and Al in steel. Kinetic models, like the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model, predict the kinetics of phase transformations, helping to understand the rate at which elements like chromium and aluminum influence the microstructure during heat treatment processes. Mechanical property models, such as Dislocation Theory Models, help to understand how alloying elements affect mechanical properties like strength and hardness [50].

Finite Element Modeling (FEM) is used to simulate the mechanical behavior of alloyed steels under various loading conditions, providing insights into the macroscopic effects of alloying on stress distribution and deformation. Simulation techniques for Cr and Al in 316L stainless steel include Molecular Dynamics (MD) simulations, Density Functional Theory (DFT), computational thermodynamics, Monte Carlo simulations, and process simulations [51].

These methods provide a comprehensive toolset for predicting and understanding the behavior of alloying elements in steel, essential for designing new alloys with tailored properties, optimizing processing conditions, and predicting material performance in various applications. By combining theoretical models and computational approaches, researchers can develop new steel alloys with tailored properties, optimize processing conditions, and predict material performance in various applications.

#### PART 4

#### **POWDER METALLURGY**

### 4.1. OVERVIEW

Powder metallurgy is a manufacturing method that allows for the economical production of durable parts with minimal tolerance compared to various other manufacturing methods. This technique involves producing powders of desired shapes and sizes, characterizing these powders through a process that includes different stages of thermal treatment and mechanical deformation, and combining them to transform into applicable, high-performance engineering parts. Powder metallurgy enables near-net-shape manufacturing of parts without needing secondary processes and allows for the production of advanced materials [52,53].

The most prominent feature of powder metallurgy is its suitability for economically producing high-quality, complex-shaped parts with minimal waste. In today's manufacturing technologies and industry, efficiency, energy, and raw materials are the most important concepts. Powder metallurgy is a production method that combines these three aspects effectively. The properties of materials produced through powder metallurgy technology depend on the shape structure, size and composition of the powders, compaction pressure, and sintering temperature. This technology is widely used today as an alternative to classic manufacturing techniques and is preferred for its ability to produce parts with good strength from elements that are insoluble in the liquid phase through mechanical alloying. The use of powder metallurgy technology solves many problems due to its superior features such as manufacturability, economy, and homogeneity. It is used in various fields due to the low manufacturing cost and high performance of the produced parts. Prostheses manufactured using powder metallurgy technology can also enhance properties like

functionality, biocompatibility, resistance to wear and corrosion. Parts produced with this technology usually have a smoother surface compared to those produced by other methods and often do not require secondary processing. This method is preferred for the economic production of parts with desired compositions and for manufacturing parts that are difficult to process [53, 54].

#### 4.2. APPLICATIONS OF POWDER METALLURGY

Materials that are hard and have a high melting point can be shaped using the powder metallurgy technique. The application area of powder metallurgy is very wide. In medicine, it is used for prostheses, orthopedic implants, solders and electrodes in welding, and in the production of bulb wires and tungsten lamp filaments using tungsten with T/M technology. In the electrical and electronics field, it is used for the manufacturing of parts such as resistors and contacts. It is also used in the production of metal filters that require a porous structure, the manufacturing of self-lubricating friction bearings by impregnating oil into the spaces between powder particles, the production of bullet and weapon parts in the military field, the shaping of cobalt, nickel, and aluminum powders to produce artificial magnets, and in the automotive industry for engine parts, gear wheels, and parts that need high resistance to wear (for example, brake discs). The T/M production method allows for the creation of strong, durable parts at high temperatures, making it suitable for the manufacturing of various parts used as cutting tools (like hard metal tips and milling cutters). The production of abrasive grinding wheels is entirely achieved by this method. Additionally, this method is also used in the production of ceramic materials.

#### 4.3. BENEFITS AND DRAWBACKS OF POWDER METALLURGY

Powder metallurgy (T/M) offers several advantages, including the ability to produce parts in their final shape or close to it, the ability to shape parts that are difficult to process using other methods, labor and time savings due to mold production, and better shape and size control compared to other manufacturing methods. It also provides good levels of corrosion and wear resistance in parts produced by T/M. The production process is easier to produce materials with various properties, and porous metal parts can be produced by leaving the desired space between powder particles. T/M production costs are lower due to scrap parts being used, and it can be automated for economic and mass production [55].

Also, powder metallurgy production has several disadvantages, including high wear levels in molds due to the abrasive nature of metal powders, high production costs due to the high cost of metal powders, lower strength in metal materials due to weak bonds, difficult transportation and storage, and potential changes in density during production. These factors contribute to the high cost and difficulty of metal powder production.

#### 4.4. POWDER METALLURGY PROCESSING STEPS



The processing step of powder metallurgy as shown in Figure 4.1, after choose the powders the mixing step and compaction and finally sintering.

Figure 4.1 Powder metallurgy processing steps.

### 4.4.1. Powder Mixing

In the powder metallurgy process, the mixing or blending of powders is necessary to achieve a specific particle size distribution, to create various alloys during the sintering process by combining desired powders, to add lubricants for the pressing process, and to form a powder-binder mixture for the shaping operation. During transportation, vibrations can cause the powders to segregate by size, and this segregation can lead to issues in pressing and sintering operations. The primary
purpose of mixing powders is to prevent this size segregation. There are three main reasons for this segregation in powders: the size of the powder particles, their density, and the shape differences of the powders. The mixing stage in the powder metallurgy method is influenced by parameters such as the material, the shape or size of the part, the characteristics of the mixer used for blending, the blending speed, or duration. These parameters affect the mixing process. Additionally, environmental factors like humidity also play a role in the mixing stage. Precautions such as re-mixing the dry powder after transportation and avoiding the vibration of the dry powder can help minimize problems in this stage of the process [52, 55].

#### 4.4.2. Pressing

The step following the mixing of powders is the shaping of the powder mixture, which means pressing, compacting, or briquetting. The most well-known shaping process involves filling a die with the powder composition and then forming it using a punch with the assistance of either a hydraulic or mechanical press. The part formed after this process has a certain density and strength but is still green, meaning it has not yet been sintered. The green part will obtain its final density and strength in the subsequent powder metallurgy process step known as sintering [52].

The molds used in the process are designed according to the powder, press, and the size considered for production. The purpose of the compaction process is to give the powder mixture the desired size or shape and to achieve the required level of porosity in the structure of the part. As pressure is applied during the compaction of the part, the pores within the structure decrease, the powder particles settle and form bonds, the part begins to densify, resist, and its hardness increases. An increase in pressure is necessary to continue the compaction process. Hardness is an important factor in compaction and is inversely proportional to the green density. Before pressing, the number of contact points between the powder sis very few. The pressure applied causes deformation at the contact points in the powder composition, leading to further integration and densification of the powder particles. The green strength obtained after the pressing process is dependent on the number of coordination between the powder particles, the size of the contact area, the partial

density, and the quality of the contact. The mentioned partial density is used to eliminate differences specific to materials with high and low densities in engineering materials [53].

Amount of powder needed will be based on the bulk density of the powder and the amount of material in the final part. Bulk density is discussed in the previous section, it density of the loose powder by itself. Bulk density is important when measuring powder quantities. The effects of additives such as lubricants must always be calculated. For example, a green compact has a certain amount of lubricants and binders in it that add extra material. During sintering, these lubricants and binders are burned off. Their material is no longer in the part after sintering and this must be a consideration.

To begin the manufacturing process, a certain amount of powder is filled into a die. Rate of die filling is based largely on the flowability of the powder. Powders that flow readily can be poured at higher rates. Pouring can be an automated process.

Once the die is filled, a punch moves towards the powder. The punch applies pressure to the powder, compacting it to the correct geometry. A simple illustration of the pressing process is shown in figure.

The compaction process generally consists of the powder composition, mold, lubricant, press, and their characteristics. The molds used here are composite materials containing hard metals to ensure longevity and are crucial for producing the desired shape, size, and density. Molds are designed to minimize wear and prolong their service life. Presses, on the other hand, come in various types and are measured in tonnage, which is the force required for pressing. Parts are produced in presses with tonnage appropriate to their sizes; large tonnage presses are used for large parts, while smaller ones are preferred for small parts. Types of presses include hydraulic, mechanical, computer-controlled (CNC), hybrid, rotary, and anvil presses.

After pressing, the part is fixed in the mold. During both the pressing process and the removal of the part from the mold, wear can occur inside the mold due to density differences caused by friction. As the applied pressure increases, it becomes more

difficult to remove the part. Another issue is the growth or expansion of the part dimensions during removal from the mold, a phenomenon known as springback. Springback can lead to cracks in the produced part. To avoid all these adverse conditions, lubricants are used. There are lubricants that are mixed with the powder polymer structure or applied to the mold walls. Lubricants are usually based on stearic acid, but there are also lubricants derived from animal sources.

## 4.4.3. Sintering

The final and arguably the most complex yet fundamental step of the powder metallurgy process is sintering. This step transforms the temporarily strong shaped part into engineering products with desirable quality and size that perform very well. The sintering process is also simply referred to as the heating or furnace process.

The sintering application significantly increases the strength of the part by bonding the particles together and also improves other properties. Besides the primary goal of particle bonding, sintering also serves secondary purposes such as oxide removal. The dimensional changes that occur during the sintering process are caused by the heat that enables the movement of atoms. The energy required for the movement of atoms is referred to as "activation energy". In powder manufacturing, energy is transferred to the material to create surface area or surface energy. The sintering process removes this created surface area or energy. The shrinkage in the particle's geometry is inversely proportional to the surface energy per unit volume. Therefore, smaller particles sinter more rapidly [56].

#### 4.4.4. Other Production Techniques

In certain circumstances, the chemical compound's thermal decomposition is employed. This can be seen in the dissociation of nickel carbonyl, for instance. The carbonyl method was initially developed with the goal of purifying nickel. Using this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The carbonyl reaction occurs as a gas.Using this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The carbonyl reaction occurs as a gas. As temperature rises and pressure drops, it breaks down. Powders of carbonyl iron are used when a high degree of purity is needed. The current need for tiny powders for the injection molding technique has accelerated the carbonyl processUsing this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The carbonyl reaction occurs as a gas. As temperature rises and pressure drops, it breaks down. Powders of carbonyl iron are used when a high degree of purity is needed. The current need for tiny powders for the injection molding technique has accelerated the carbonyl process.Using this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The current need for tiny powders for the injection molding technique has accelerated the carbonyl process.Using this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The carbonyl reaction occurs as a gas. As temperature rises and pressure drops, it breaks down. Powders of carbonyl process.Using this process, carbon monoxide and raw metal combine under pressure to create carbonyl. The carbonyl reaction occurs as a gas. As temperature rises and pressure drops, it breaks down. Powders made of carbonyl iron are used when a high level of purity is needed. The current need for tiny powders for the injection molding technique has accelerated the carbonyl process.

#### 4.5. PHYSICAL PROPERTIES OF POWDERS

The terms compressibility, density, and fluency are used to characterize the size and form of the grains. Grain size in metal powders is mostly evaluated using the sieve analysis method. Grain size can only be determined by computing the particle sizes. The usual grain size can be stated, despite the fact that it is difficult to state that the entire powder is the same. The size analysis is defined by the percentage and average grain size of the powder in each range. The form of the powder is a major factor in determining the particle size. For spherical powders, knowing the diameter at center suffices, but knowing the size of the powder particles in the plane direction and their flake thickness are also important. Starting with the surface area, one can determine the particle size for powders in different forms. The surface to sphere ratio is taken into consideration while calculating grain size [52].

One of the most crucial properties of powders is the grain shape, which has a substantial impact on the apparent density, fluidity, compressibility, and raw strength of powder particles. The apparent density of a powder that is randomly selected is the main factor influencing the size of the cavities in the mold made to press the powders. The calculated volume of the non-adherent metallic powder is completely poured

into the known container, and the resulting mass is measured to determine the apparent density. The apparent density is then calculated using the mass to volume ratio. The powder is added to the inclined plates, filling the container's hollow. Particles of iron powder are visible in a range of apparent densities [53].

All substances found in nature can be ground into powder after processing. There are numerous techniques for preparing metal powder particles. However, depending on the ingredient, different techniques are employed to make the powders. Particles of typical dimensions up to a few microns are referred to as particulate powder. As technology develops, dust particles get smaller and smaller. The powder might have a spherical or dendritic geometric shape, depending on the production processes used.

#### 4.6. POWDER METALLURGY USAGE AREAS

Powder metalworking is used in various fields. These consist of copper alloys, magnetic alloys, aluminum and titanium alloys, cermets, tool steels, stainless steels, and nuclear materials. Tungsten and molybdenum are examples of refractory materials, together with super alloys, are used to create wear-resistant components. In particular, the car industry is prominent. In addition, circuit boards, nuclear power fuel elements, electrical contacts, aircraft brake linings, welding electrodes, catalysts, and soldering tools gear wheels, non-lubricated bearings, tungsten lamp filaments, orthopedic equipment, and high temperature filters. Applications for paints, explosives, welding electrodes, and dentistry are among them [54].

#### 4.7. ADVANTAGES OF POWDER METALLURGY

The ease and affordability with which different alloys, which are difficult to shape by casting and machining, may be turned into products is the significance of powder metallurgy. The powder metallurgy approach has advantages and disadvantages that can be compared to other production approaches.

#### Advantages:

1) It is easy to manufacture in intricately formed, accurate pieces.

- 2) The high rate of production means that labor is not needed.
- 3) The materials that are produced have excellent mechanical and physical properties.
- 4) The manufactured goods exhibit strong tensile and machineability characteristics along with tiny particle sizes.
- 5) Materials with different qualities that do not dissolve in one another can be created by combining them.
- 6) Parts made with powder metallurgy usually don't require any additional steps, such as machining.
- 7) During the powder metallurgy process, very little material is lost. When considering
- 8) The material loss caused by casting and machining procedures, material savings are substantial.
- 9) It provides an affordable manufacturing method due to the increased output, decreased material waste, and decreased labour.
- 10) There are no melting losses.
- After the sintering process, the item is ready for use, and other steps are mainly superfluous.

## Disadvantages:

- 1) The molds used in production come at a significant cost.
- 2) The pores in the microstructure may result in lower mechanical characteristics when compared to other methods.

There are difficulties in producing pieces with a high thickness to diameter ratio. The sizes of particles that can be utilized to produce uniform densities are limited, though. Up to 20 kg of powder-metallurgy components can be generated. In terms of cost, materials produced as ingots are less expensive than metal powder particles [55].

#### 4.8. CHARACTERIZATION OF POWDERS

#### 4.8.1. Powder Sampling

There are several ways to go about powder sampling. The most widely utilized technique is to combine and apply little samples from numerous locations. The particles have a strong propensity to adhere to one another and a coherent overall structure. Particle agglomeration is eventually possible. Surface wetness may be the cause of high agglomeration. Furthermore, a collection of particles adhering to weak forces that are susceptible to destruction by minute shear pressures is known as a clinging agglomeration. Surfactant liquids, mechanical and ultrasonic agitation techniques, and the majority of particles all actively contribute to the dispersion of particles and the subsequent determination of their properties [56].

#### 4.8.2. Particle Size Measurement

For researchers dealing with powder, calculations of particle size and comprehensive information are required. Unless the particle is spherical in shape, determining the size of the particle is a difficult undertaking. The diameter feature can be used to calculate particle size, which is mostly based on the geometrical structure of the particle. Sieving or other techniques are used to measure the size of the powder particles. The size of powder particles varies. The average particle size property is used to calculate the particle size. Particle size measurement methods also differ if the powder's particle shape is complicated [57].

It is necessary to take four or five-dimensional measurements if powder particles are discovered combined with powders of various sizes. The surface area can be used to calculate the particle size if the complicated structure is dense. The sieve analysis measurement method is usually used to determine the grain sizes of the powder particles. The huge hole in the sieve's structure serves as a calculator for its size. The mesh approach determines the computation procedure. The size of the sieves is expressed in microns per SI and the sieve method merely classifies the powder particle as bigger than or smaller than a given value; it does not measure the actual size of the particle [58].

#### 4.8.3. Powder Mixing

It is crucial to thoroughly mix the powders prior to applying the compaction technique since powder combinations are typically utilized in the powder metallurgy procedure. The primary goal of the mixing procedure is to ensure that the powder particles are uniform. The performance of the finished product is increased when powder particles with varying sizes, shapes, and densities are mixed homogeneously.

Blending ought to happen before combining if the powder combination doesn't contain any typical dispersions. It is advised to blend to create a homogeneous size distribution and to enhance pressing and sintering qualities. A few variables influence the process of blending and mixing. These elements are:

- 1) Physical characteristics of powders.
- 2) Mixer dimensions.
- 3) Mixing speed.
- 4) Mixing time.
- 5) Powder volume in the mixer.
- 6) Humidity and atmospheric conditions.
- 7) Rotation speed.

There are several techniques for making powders. The two primary categories of these techniques are physio-chemical and mechanical. Several characteristics of the powder particles are revealed by the methods utilized in the manufacture of metal powders. The desired amounts of powder are added to the process in the mixing mills to make it acceptable for the pressing process, or the powder particles created with the required specifications can be weighed using precision scales and the pressing process can begin immediately. The quality of metal powders determines the production procedures used. One of the most crucial aspects of production technique is economy.

#### **4.8.4.** Powder Compaction (Pressing)

A dense filling of the mold is the intended result when creating metal powders using a mold. Attaining the maximum theoretical density is thought to occur when the powders are cold-pressed within the mold. Despite applying the same pressing pressure, the final density attained in each metal powder varies based on the material's theoretical density. The form of the powder, its surface and grain size, the kind of material (manufacturing processes, particular surface), and the pretreatment done to the powder are the variables that rely on this condition [57].

Steel molds that have been specifically prepared are typically used for pressing at room temperature. Cemented carbide-based and heat-hardened tool steel is employed in the mold-making process. The amount of pores varies with the pressing pressure and reduces with an increase in the pressure ratio. On the other hand, the surface area and the quantity of powder in contact with one another grow with increasing pressing pressure. Its density falls with an increase in the number of pores. The number of pores affects the tensile strength of the items made using the powder metallurgy procedure. Tensile strength ratios rise in tandem with density ratios [58].

From the outside, powder compression appears to be a straightforward process. Conversely, though, it's a multifaceted, intricate process. When compression starts, there is more friction than any other force acting on the inside surface of the mold. But when it approaches the mold's center, it seems to be decreasing gradually. Much of the forming energy is lost as a result of the friction between the powder and the mold surface. Furthermore, frictions occur between the powder and the balls, between the contacting particles, and between the balls and the mold surface. It is crucial that there be friction between the product and the mold's inner surface when the product is being taken out of the mold. For these reasons, a lubrication system that is efficient in the forming and demoulding processes must not interfere with the sintering process [7]. The vibration of the mold increases the density of the powders that are added to it before they are pressed. The powder's shape and distribution plan have an impact on the increased density that is achieved with vibration. Compared to powders with smooth and spherical surfaces, this rise is significantly greater in powders with irregular shapes. This can be explained by the low relative densities of irregularly shaped powders with limited powder size distribution and the high density ratios based on spherical-shaped powders. Relative density is the ratio of the raw density to the theoretical density [58].

There are two types of pressing that are frequently discussed in press discussions: hot pressing and cold pressing. Heat is applied after pressing in cold pressing operations, whereas pressure and heat are applied simultaneously in hot pressing methods. When it comes to giving the product good mechanical qualities and precise measurements, cold or hot isostatic pressing techniques are far better than pressing using rigid molds.Since the pressure applied to the powder masses is distributed uniformly, high wet density and high wet strength values can be achieved even at low pressure when using the aforementioned pressing methods. Tensile strength and fatigue strength are two rather strong mechanical qualities of the materials made using the hot isostatic pressing process. The following headings provide explanations of hot and cold isostatic pressing [59].

#### 4.8.4.1. Single-action Die Compaction

Traditional powder compression techniques are carried out in a uniaxial manner. The upper pressure plate applies pressing actions as soon as the powder combination is inserted into the mold, as seen in Figure 4.2. The bottom piston is used to scrape the finished product out of the mold. Lubrication is given to the mold walls to produce compression and to make it easier to remove the object from the mold. The density of the compressed material increases in tandem with the pressure applied during the single action compaction process. This is caused by the used powders' decreasing porosity as well as the fact that the volume reduces but the mass stays constant. This process has advantages, but it is not recommended since it cannot achieve the

necessary density in metallic parts with a high length/width ratio and intricately. formed parts.



Figure 4.2. Single action die compaction [60].

### 4.8.4.2. Double-action Die Compaction

As seen in Figure 4.3, pressure is applied to the die by upper and lower punches. The two punches have movement. Punches can exert varying degrees of pressure and movement. The friction between powder particles, punches, the mold surface, and the particles can cause large variations in the raw density distribution. Some attempts are made to reduce these properties by reducing friction by using a lubricant or by using suitable compression methods.

Using the single action compaction approach, complete density cannot be achieved. Due to the compression of the powder by the upper and lower punches in double action compaction, the friction force between the powder particles and the mold wall is greater than the spring force of the flexible material, such as the spring on the mold base. This causes the mold to move toward the lower region, where the lower punch can equally press the powder with the upper punch on the powder's base surface. When the compression process is finished, the bottom punch moves in the direction of the upper section, removing the resultant part from the mold. Consequently, the density distribution is found to be more homogeneous than the portions generated from single action compaction. The middle of the compressed section has the lowest density rating. This density has a symmetrical distribution [61].



Figure 4.3. Double action die compaction [60].

#### 4.8.4.3. Cold Isostatic Pressing

Pressures are delivered uniformly in all directions using this procedure. It is possible to use cold isostatic compression (Figure 4.4) on stepped and complex parts with significant aspect-to-diameter ratios. An elastic container that is closed and sealed and serves as a mold is filled with the powder. Since the air in the soft mold will escape during compression, it is removed in advance. Liquid is pumped into the pressure vessel to submerge the mold inside. The fluid is subjected to high pressure, leaving the mold under hydrostatic pressure. The flexible mold is taken out of the item when the pressing process is finished, and it is often not used again. The powder metal components made with this technique can have a more uniform density distribution [62].



Figure 4.4. Cold isostatic pressing [63].

#### 4.8.4.4. Hot Isostatic Pressing

Powder metals are atomized with inert gas to obtain the material blocks needed to produce powder metallurgy parts using the hot isostatic compression process. The only ways to make metals that are extremely difficult to produce using traditional production processes, have extremely unique qualities, and cannot be combined in any other way are by mixing metals in powder form or by using several layers of powdered metal. Capsules containing metal particles are placed within a heated isostatic compression furnace (autoclave). Here, the compressed inert gas exposes the capsules to high pressure and high temperature. While the exact temperature and pressure vary depending on the kind of material, they are often 1400°C and 2000 bar, respectively. Under isostatic gas pressure and high temperature, the particles inside the enclosed capsule combine to form a totally dense structure (Figure 4.5). The crushed component that results has a fully non-porous structure since the crevices between the powder particles have been completely eliminated. Parts produced by hot isostatically compressed powder metallurgy are a very cost-effective technique for applications involving high temperatures, high mechanical stress, high corrosion stress, and high wear [62].



Figure 4.5. Hot isostatic pressing [64].

## 4.9 MACHINING

## 4.9.1. Chip Removal Mechanics and Chip Formation

Since the machining process is actually quite complex and three-dimensional, the machining mechanics are described using the two-dimensional orthogonal model shown in Figure 4.6. This model is essential to the understanding of the machining process because it provides an accurate and thorough description of the mechanics of machining. The cutting edge of a wedge-shaped tool used in the orthogonal cutting model is perpendicular to the cutting direction. The shear deformation along the shear plane, which is aligned at an angle with the material's surface as the tool is

driven into the workpiece, produces the chip; this angle is known as the shear angle. Along the shear plane, plastic deformation takes place [65].



Figure 4.6. Orthogonal cut model [66].

In the manufacturing process, plastic deformation is essential to the production of chips. The first deformation zone is the area in the shear plane where the regional deformation of the workpiece occurs in a very small area in front of the cutting tool. The plastic deformation zone in front of the cutting tool edge is the primary zone of deformation, and the deformation zone on the back surface is the secondary zone of distortion. By plastically deforming the workpiece in the primary deformation zone, the stress created in the workpiece as a result of the relative movement between the cutting tool's tip and the workpiece leads to chip formation. As the tool moves across the rear surface, the layer that was removed from the workpiece experiences a second deformation zone. The tertiary (third) deformation zone is the area created by the frictional force exerted by the chipped surface coming into contact with the cutting tool's lateral surface. The zone where the workpiece's surface quality is impacted is known as the third deformation zone. The process of chip creation is depicted in Figure 4.7 [65].



Figure 4.7. Chip formation [65].

#### 4.9.2. Shear and Cutting Forces

It is well known that the cutting performance, the quality of the machined surface, and the part's unit cost are all significantly impacted by the cutting forces produced during chip removal. In order to create sturdy, vibration-free cutting machines, cutting forces are also taken into account during the design of cutting machine elements. It is also used to figure out how much energy the cutting forces that arise during the turning process. An essential phase of the chip generation process is represented by these forces exerted on the cutting instrument. When cutting metal, the primary cutting force (Fc), which acts in the direction of the cutting speed, is crucial in determining the amount of power needed. About half of the cutting force is the feed force (Ff), which is the force operating in the direction of the cutting tool. The last element is the radial (passive) force (Fr), which makes up around 50% of the feed force and acts perpendicular to the machined surface [65].



Figure 4.8. Cutting forces in machining process [65].

All of the active forces need to be accurately measured in order to produce a machining solution that is high-quality, safe, and affordable. Because of this, the cutting forces are ascertained using dynamometer measurements, and mathematical models are derived from these results [65].

#### 4.9.3. Heat and Temperature

Near the tool tip, almost all of the power needed to cut metal is transformed into heat. As a result, temperature and heat creation in the cutting zone play crucial roles in chip removal. It is crucial to analyze the heat produced in relation to the effectiveness of the cutting tool and the quality of the workpiece [65].

Tool life is significantly impacted by the heat produced during metal cutting operations. Ideally, chipping reduces the majority of the heat created in the cutting zone. The workpiece material, the machining parameters, the cutting tool material, and the geometry all affect how much heat is reduced. The cutting process is less efficient when there is significant heat leakage to the workpiece or cutting tool [65]. It is well known that the heat produced during the cutting process is greatly

influenced by the cutting speed. The energy per unit of time rises with increasing cutting speed, which raises heat and, consequently, temperature. One of the key elements affecting cutting speed and tool life is high temperatures. In this regard, makers of cutting tools have concentrated on enhancing their capacity to hold onto their characteristics at elevated temperatures [65].

#### 4.9.4. Surface Quality

When creating an item utilizing machining techniques, surface quality and dimensional accuracy are crucial factors to take into account. Here, it is explained how the part's processing quality, size and form tolerances, and surface treatment marks will be decided. Surface quality is especially crucial in applications that aim to achieve wear life, fatigue strength, and corrosion resistance. Furthermore, surface quality influences a number of functional aspects of a component, including wear, light reflection, heat conduction, coating, and resistance life, all of which contribute to friction in a part when it is working. To achieve the required quality during a part's manufacturing phase, it is crucial to ascertain the optimal levels of parameters like depth of cut, cutting speed, feed rate, and cutting tool geometry, all of which directly affect surface roughness during machining [65].

### PART 5

#### **MATERIALS AND METHODS**

In this investigation, samples were created by combining alloys in the necessary ratios using the powder metallurgy process. The purpose of preparing these samples in various Cr and Al ratios was to investigate the impact of microstructural and mechanical characteristics. Section 4.1 details the process steps used in this study; Section 4.1.1 deals with the mixing of powders and pressing process; Section 4.1.2 deals with the sintering process; Section 4.2 describes the tensile test sampling; and Section 4.3 deals with optical microscopy and SEM image analysis. Tensile testing, SEM experiments, and optical microscope analyses were conducted in the MARGEM facilities at Karabuk University, the Iron and Steel Institute, MARGEM laboratories.

#### 5.1. EXPERIMENTAL PROCESSING STEPS

In this study, the appropriate compositions of 316L stainless steel will be produced using powder metallurgy (PM), and the effects of different concentrations of Cr and Al micro-alloying elements on the mechanical properties and microstructure will be examined. Furthermore, wear behaviors will also be examined.

As seen in Figure 5.1, the process steps for this experimental study involved obtaining, mixing, pressing, and sintering the powders. It also includes microstructure analysis and mechanical assessments.



Figure 5.1. Process steps in experimental study and Microstructure analyzing.

## 5.1.1. Mixing and Pressing the Powders

In this investigation, powder metallurgy (PM) was used to create PM steel samples with chromium in the appropriate compositions. Prior to mixing, the powder particles were weighed with an accuracy of 0.0001 gr using a precision balance of the RADWAG AS-60-220 C/2. Powder mixes obtained by weighing were mixed for 1.5 hours without a ball in a Turbula brand triaxial mixer in order to generate a uniform structure. Erden discovered that combining the resulting powder mixes for 90 minutes produced the highest yield and tensile strength ratings throughout his inquiry into mixing duration. Similar to Erden's study, which discovered that extending the mixing period beyond 90 minutes had almost little effect on the mechanical features, this investigation set its mixing time at 90 minutes.

Table 5.1 Chemical compositions of powder metal steels.

-	
316L	1200°C – 2Hr
316L+0.1Cr	1200°C – 2Hr
316L+0.2Cr	1200°C – 2Hr
316L+0.1Cr+0.75Al	1200°C – 2Hr
316L+0.2Cr+1.5Al	1200°C – 2Hr

Composition

The tensile test specimen was compressed into a complete form using a mold that was created in accordance with ASTM (E8M) powder metal material standards, Resulting in a complete specimen.

Figure 5.2 displays the press used in the compaction process, the powder mixing device used in the mixing process, and the precision balance used in the powder weighing. The final sample is displayed in Figure 5.3.



Figure 5.2. Images of a) Precision balance, b) Hydraulic press machine, and c) Powder mixing device used in experimental studies.



Figure 5.3. Image of cold pressed drawing specimens.

## 5.1.2. Sintering Processes

The samples were prepared and processed at the necessary rate, and then they were sintered under argon atmosphere. The Protherm PTF 16/75/610 model atmosphere-controlled tube furnace, which has a maximum temperature of 1600 °C and is located in the Manufacturing Engineering, Mechanical Metallurgy laboratory of Karabuk University, was utilized for the sintering process. The sintering process starts at the sample's sintering temperature and proceeds at a rate of 5 °C per minute. completed by heating quickly. For one hour, the alloy listed in Table 5.1 was maintained at 1250°C. Return to room temperature at a rate of 5 °C per minute after two hours of cooling down. Figure 5.4 depicts the atmosphere-controlled furnace.



Figure 5.4. Atmosphere-controlled tube furnace.

#### 5.2. TENSILE TEST AND HARDENESS MEASUREMENT

Using a PRESI MECAPOL P262 brand tool, the surfaces of the sintered tensile specimens were ground to make them suitable for hardness measurements. In Vickers hardness tests, an HV (0.5) load was applied, and the average of each sample's five hardness measurement findings was used to calculate the hardness value.

A SHIMADZU tensile tester equipment was used to apply tensile tests to samples at a tensile speed of 1 mm/min. As a result, after the testing, stress-strain graphs were drawn. The samples' tensile strength, yield strength (0.2%), and percent elongation values were computed in order to examine the material's behavior in these graphs. Tensile testers and hardness testing tools are shown in Figure 5.5.



Figure 5.5. SHIMADZU tensile test and hardness measurement device [107].

### **5.3. DENSITY MEASUREMENT**

The density of P/M steel samples was measured using the Archimets principle utilizing distilled water, as illustrated in Fig. 5.6, using a RADWAG AS-60-220 C/2 precision balance with RADWAG AS density measurement kit.



Figure 5.6. Density measurement device.

The porosity value was calculated using equation 5.1's procedure.

Porosity (%) = 
$$\frac{\rho \text{ theoretical} - \rho \text{ measured}}{\rho \text{ theoretical}} \times 100$$
 (5.1)

Where:  $\rho$  theoretical is theoretical density (g/cm<sup>3</sup>)  $\rho$  measured is measured density (g/cm<sup>3</sup>)

## 5.4. OPTICAL MICROSCOPE EXAMINATIONS

Powder metallurgy-produced stainless-steel samples were mounted for microstructural examination. Next, the device (Fig. 5.7) was used to grind the surfaces of the prepared samples in the mechanical laboratory of Karabuk University's Faculty of Technology. SiC abrasives with grit numbers of 240, 320, 400, 600, 800, 1000, 1200, 2500, 5000, and 7000 were used to grind them under water. Following a water wash, the ground samples were sprayed with alcohol to dry their surfaces.



Figure 5.7. Polisher used for grinding.

An optical microscope of the Nikon Epiphot 200 brand with an X500–X1000 magnification capability was used for microstructure investigations in the Metallography lab of Karabuk University's Technology Faculty (Fig. 5.8). Care was taken to ensure that the photos accurately depicted the entire microstructure, and they were taken at different magnifications from different points inside each sample.



Figure 5.8. Optical microscope devices.

## 5.5. 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).

4D-ECN model tribometer device was used for wear tests. The wear test was carried out at 37 °C in SBF solution under 10N and 20N loads, with stroke of 15 mm, a total of 100 meters of sliding distance for each load, and 0.01 m/s sliding speed. Alumina balls with 6mm diameter were used for wear test. After the wear test, worn surface was measure with the surface profilometer.



Figure 5.9. Wear test specimen, Al<sub>2</sub>O<sub>3</sub> ball and wear area loss measurement.



Figure 5.10. Wear test device.

Reagent	Amount
NaCl (g)	8.036
NaHCO3(g)	0.353
KCl(g)	0.225
K2HPO4.3H2O(g)	0.230
MgCl2.6H2O(g)	0.311
CaCl2(g)	0.293
Na2SO4(g)	0.072
Tris (hydroxymethyl) aminomethane(g)	6.063
1M HCl (ml)	40

Table 5.2. Chemical composition of simulated body fluid (SBF).

## PART 6

## **RESULTS AND DISCUSSION**

# 6.1. MICROSTRUCTURE RESULTS



Figure 6.1. The optical micrographs a)316L, b) 316L+0.1Cr, c) 316L+0.2Cr, d)316L+0.1Cr+0.75Al and e) 316L+0.2Cr+1.5Al (500x).

The microstructure images of the samples produced via the PM technique were taken at a pressure of 750 MPa and sintered in an atmosphere of argon at a temperature of 1250 °C for two hours. Figure 6.1. show the optical microscope images of the samples produced by the PM method before and after the alloying elements (Cr and Al) were added.

It can be seen from the microstructure images that when 0.2 Cr is added to 316L stainless steel, the grain size in the microstructure decreases, and when Al is added as an alloying element in addition to the Cr element, the grain size increases.

Microstructure images of the samples produced with the PM technique were taken at 750 MPa pressure and sintered for two hours at 1250 °C in an argon atmosphere. Figure 6.1 shows optical microscope images of samples produced by the PM method before and after the addition of alloying elements (Cr and Al). It can be seen from the microscopic images that when 0.2Cr% is added to 316L stainless steel, the grain size in the microstructure decreases, and when 0.2 Cr + 1.5Al is added as an alloying element, the grain size increases. Additionally, when Figure 6.1, which shows the optical microscope images taken from the entire 316 L composition produced, is examined, it is seen that structures consistent with similar findings in the literature are formed.

With the optical light microscope images obtained, the grain size differences and pore formations of the microstructures became clearly evident with the addition of different amounts of chromium and Al compared to the original alloy 316L. When the microstructures are examined, an increasing amount of porosity is observed with the addition of Al. It is understood from the literature that this increased porosity also manifests itself negatively in mechanical properties [68].

### **6.2. MECHANICAL PROPERTIES RESULTS**

Figure 6.2. It shows the elongation stress curve of the produced compositions. It is also clearly stated which line belongs to which alloy. When the curve is examined, it is seen that the highest stress value is in the 316L+0.2Cr sample.





Figure 6.2. Difference of stress–strain curves of the 316L stainless steels with Cr and Al Additions.

The data obtained from the mechanical and density test results are given in Table 6.1. When the table is examined, it is seen that the maximum tensile value of the powder metal 316L stainless steel produced is 338 MPa, and when 0.1% Cr is added to 316L, this value increases to 360 MPa. When 0.2% Cr is added, we read the value of 379 MPa, which is the highest tensile value obtained in the alloys. A decrease was observed in the 316L+0.1+0.75A1 alloy, which was added to see the effect of Al, and the reading value became 263 MPa. In the 316L+0.2+1.5A1 alloy, where the amount of Al is slightly increased, the tensile value of 140 MPa is observed, which is the lowest value seen in the table. It is thought that the amount of porosity in the 316L+0.2+1.5A1 alloy, as seen in the microstructure test results, is a factor in not obtaining this result.

Samples	Composition	Experimental Densities (g/cm3)	Porosity (%)	Ultimate Tensile Stress (MPa)	Elongation (%)	Hardness 0.5Hv
1	316L	7.06	9,02	338	27	200
2	316L+0,1Cr	7.17	8,53	360	25	211
3	316L+0,2Cr	7.27	8,42	379	17	220
4	316L+0,1Cr+0,75A1	6.96	10,6	263	5	188
5	316L+0,2Cr+1,5Al	6.85	11,4	140	2,1	179

Table 6.1. Mechanical and density properties of the alloys.



Figure 6.3. Maximum tensile and elongation values of the examined alloys.



Figure 6.4. Hardness values of the examined alloys (Hv0.5).

The data obtained from the hardness test results are given in Figure 6.4. When the figure is examined, it is seen that the hardness value of the powder metal 316L stainless steel produced is 200 Hv0.5, and when 0.1% Cr is added to 316L, this value increases to 211 Hv0.5. When 0.2% Cr is added, we read the value of 220 Hv0.5, which is the highest hardness obtained in the alloys. In the 316L+0.1Cr+0.75Al alloy added to see the Al effect, a decrease was observed and the reading value became 188 Hv0.5. In the 316L+0.2Cr+1.5Al alloy, where the amount of Al is slightly increased, the tensile value of 140 Hv0.5, which is the lowest value seen in the table, is observed. As seen in the microstructure test results, it is thought that the amount of pores in the 316L+0.2Cr+1.5Al alloy is a factor in not obtaining this result.

## 6.3. WEAR TEST RESULTS

316L matrix samples, which were also used as biomaterial in the study, were subjected to corrosion testing in body fluid. The wear test was carried out under 10 and 20 N loads, at a wear distance of 100 meters and at a sliding speed of 0.048 m/s. A 6 mm diameter  $Al_2O_3$  ball, which is also used as a biomaterial, was used as the abrasive. The data obtained in the wear test gave results parallel to the tensile and hardness results. Figures 6.5- 6.6 show the wear test results under 10N and 20N load.

After the wear test, the area loss obtained along the 15 mm trace length was measured with the surface profilometer device. When the measurements were examined, it was seen that the highest area loss was in the 316L+0.2Cr+1.5Al sample at 20N load. The 316L+0.2Cr sample, which gave the highest hardness and tensile results, had the lowest area loss.



Figure 6.5. Area loss of wear test specimens under 10N load.



Figure 6.6. Area loss of wear test specimens under 20N load.

In Figure 6.7, the volume losses obtained by multiplying the area losses of the wear traces obtained at a wear distance of 100 meters by the trace length of 15 mm in the wear test conducted under 10N and 20N loads are given. When the results are examined, it is seen that parallel results are obtained considering the microstructure, max tensile and hardness results. It is seen that the highest volume loss is in the sample with 316L+0.2Cr+1.5Al alloy in the wear test performed under 20N load.



Figure 6.7. Volume loss of wear test samples under 10N and 20N load.

#### PART 7

#### CONCLUSION

In this study, the effect of adding Cr and Al elements to 316L stainless steel alloy produced by powder metallurgy method was investigated for two hours under cold pressure (750 MPa) and sintering temperature (1250 °C) in argon atmosphere and the following results were obtained:

- With the addition of varying amounts of chromium and Al in comparison to the original alloy 316L, the grain size variations and pore forms of the microstructures became readily obvious with the images captured by an optical light microscope. Analysis of the microstructures shows that the addition of Al results in an increased degree of porosity. The research indicates that the mechanical characteristics are negatively impacted by this increased porosity.
- The maximum tensile value of the generated powder metal 316L stainless steel is 338 MPa when the mechanical and density test results are analysed; this value rises to 360 MPa when 0.1% Cr is added to 316L. The greatest tensile value found in alloys is 379 MPa, which is attained when 0.2% Cr is added. The 316L+0.1+0.75Al alloy that was introduced to observe the Al effect showed a decrease; the reading value increased to 263 MPa. The lowest tensile value in the table, 140 MPa, is found in the 316L+0.2+1.5Al alloy, where the amount of Al has been significantly increased. It is believed that the amount of pores in the 316L+0.2+1.5Al alloy is a reason in not attaining this result, as demonstrated in the microstructure test findings.
- A surface profilometer was used to measure the area loss along the 15 mm trace length following the wear test. Upon analysis of the results, it was shown that the 316L+0.2Cr+1.5Al sample at 20N load had the largest area
loss. The sample with the highest hardness and tensile values, 316L+0.2Cr, also has the lowest area loss.

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## RESUME

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