THE EFFECT OF CR ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF 316L STAINLESS STEEL USED AS IMPLANT MATERIAL PRODUCED BY POWDER METALLURGY

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I certify that in my opinion the thesis submitted by Fadhil Hussein OLEIWI titled "THE EFFECT OF CR ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF 316L STAINLESS STEEL USED AS IMPLANT MATERIAL PRODUCED BY POWDER METALLURGY" is fully adequate in scope and in quality as a thesis for the degree of Master of Science.

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

Fadhil Hussein OLEIWI

ABSTRACT

M. Sc. Thesis

THE EFFECT OF CR ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF 316L STAINLESS STEEL USED AS IMPLANT MATERIAL PRODUCED BY POWDER METALLURGY

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In this study, 316L stainless steel is produced with the addition of chromium in different compositions. The effect of chromium addition on the mechanical properties and microstructure was determined. To improve these mechanical properties and reduce the cost of the steel produced, powder metallurgy technology is used.

Chromium added 316L stainless steel has a high resistance to oxidation. This resistance increases with increasing chromium percentage (over 11%-12%). Corrosion resistance results from the formation of a self-healing negative chromium oxide layer on the surface of the stainless steel. Properties such as corrosion behavior of the material, how it interacts with the body, biocompatibility, good fatigue strength, increased ductility, low cost and mechanical workability make 316L stainless steel especially preferred for implant applications.

In this study, 316L stainless steel containing with Cr at different weight ratios (16.84, 17.14, 17.34.17.84 and 18.84) was mixed with powder metallurgy technique for 90 minutes. After mixing, all compositions were pressed unidirectional at a pressing pressure of 750MPa. The sintering process was applied for one hour in an argon atmosphere at 1200 °C. The microstructure and mechanical properties characterization of the produced parts were determined by tensile test, hardness test, optical microscope, SEM microstructure, SEM fracture surface, and EDS analysis.

Key Words: Powder Metallurgy; 316L Stainless Steels; Addition of Cr; Microstructure; Mechanical properties.

Science Code : 92503

ÖZET

Yüksek Lisans Tezi

TOZ METALURJİSİ İLE ÜRETİLEN İMPLANT MALZEMESİ OLARAK KULLANILAN 316L PASLANMAZ ÇELİKLERİN MİKRO YAPISI VE MEKANİK ÖZELLİKLERİ ÜZERİNE CR'NİN ETKİSİ

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Bu çalışmada farklı bileşimlerde krom ilavesi ile 316L paslanmaz çelik üretilmiştir. Krom ilavesinin mekanik özellikler ve mikro yapı üzerindeki etkisi belirlendi. Bu mekanik özellikleri iyileştirmek ve üretilen çeliğin maliyetini düşürmek için toz metalürjisi teknolojisi kullanılmaktadır.

Krom katkılı 316L paslanmaz çeliğin oksidasyona karşı direnci yüksektir. Bu direnç, krom yüzdesinin artmasıyla (geleneksel olarak %11-%12) artar. Korozyon direnci, paslanmaz çeliğin yüzeyinde kendi kendini onaran negatif bir krom oksit tabakasının oluşmasından kaynaklanır. Malzemenin korozyon davranışı, vücutla nasıl etkileşime girdiği, biyouyumluluğu, iyi yorulma dayanımı, artırılmış sünekliği, düşük maliyeti ve mekanik işlenebilirliği gibi özellikleri 316L paslanmaz çeliğini özellikle implant uygulamaları için tercih nedeni olmaktadır.

Bu çalışmada, ağırlık olarak farklı oranlarda (16.84,17.14,17.34.17.84 ve 18.84) Cr içeren 316L paslanmaz çelik, toz metalürjisi tekniği ile 90 dakika karıştırma işlemine tabi tutulmuştur. Karıştırma sonrasında tüm kompozisyonlar 750MPa presleme basıncında tek yönlü preslenmiştir. Sinterleme işlemi ise 1200 °C sıcaklıkta argon atmosferinde bir saat uygulanmıştır. Üretilen parçaların mikroyapı ve mekanik özellik karakterizasyonu çekme testi, sertlik testi, optik mikroskop, SEM mikroyapı, SEM kırık yüzey ve SEM, EDS analizleri ile belirlenmiştir.

Anahtar Kelimeler : Toz Metalurjisi; 316L Paslanmaz Çelikler; Cr ilavesi; Mikroyapı; Mekanik özellikler.
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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
Co : Cobalt
O : Oxygen
N : Nitrogen
Si : Silicon

ABBREVITIONS

PM : Powder Metallurgy
SEM : Scanning Electron Microscope
EDS : Energy Dispersive X-Ray Spectroscopy
FCC : Face Centered Cubic
SI : International System of Units
ISO : International Organization for Standardization
AISI : American Iron and Steel Institute

SCC : Stress Corrosion Crack
BCC : Body Centered Cubic
BCT : Body Centered Tetragonal
SCE : Standard Calomel Electrode
SHE : Standard Hydrogen Electrode
MRI : Magnetic Resonance Imaging
PH : Precipitation Hardening

PART 1

INTRODUCTION

Materials are the raw materials utilized to make anything in engineering. All the materials used in the body to treat or support tissues that do work in a biological environment and damaged organs are called biomaterials [1]. A lot of work is put into making sure the components of biomedical products adapt while interacting with biological systems and don't have any negative consequences. Materials utilized in the human body to assist or execute the tasks of organs or tissues that are in contact with body fluids continuously or at regular intervals and that should not cause cytotoxic reactions in the organism are known as biocompatible, reliable, and effective biomaterials, whose importance and application area are growing daily [2]. In generally, bio functioning, and biocompatibility are the two factors that determine how well a biological material performs. Bio functionality describes how well a gadget can carry out its intended function, whereas biocompatibility assesses how well a substance is compatible with the body [3]. Biomaterials are appealing because of a few key properties. Engineers are knowledgeable about characteristics the same as mechanical performance (tensile, deformation, bending, fatigue, toughness, elasticity, etc.), chemical stability (resistance to corrosion, etc.), and features such as biological properties, bioinertity, and biological properties that are of interest to more biological and medicine disciplines [4]. Among the minerals that are being studied in this research is chromium, which was identified by the French chemist Nicholas Louis Vauquelin at Paris in1798 [5], "that had been found in a Siberian gold mine in 1766 and was referred to as Siberian red lead. It is now known as crocoite and is a form of lead chromate, he named it chromium from the Greek word chroma meaning colour" [6]. There are various locations where this mineral may be found, such as South Africa, India, Kazakhstan, and Turkey. The metal chromium is rigid, brittle, and shiny. It may be highly polished and has a silver-gray color (Brandes, EA; Greenaway, HT; Stone, HEN (1956)). It does not tarnish in air. When Cr is present, a thin, layer of passive oxide which is chemically stable forms the exterior of the stainless steels. With oxygen present, the oxide film develops and repairs itself. The material's corrosion behavior, how it interacts with the body, and how much it is biocompatible are all governed by the physical-chemical characteristics of this passive layer. The corrosion of metal implants has been the subject of numerous in-vivo and in-vitro research. Most in vitro investigations, however, used mimicked bodily fluids like Ringer's or Hanks' solutions. According to reports, the quantity of chromium used in the protective coating procedures' produced coating directly influences how resistant stainless steel is to corrosion [8]. A significant change in the manufacture of steel was the realization that steel possibly created to be exceptionally unaffected by corrosion and discolored by using metalized chrome to create stainless steel. Together, chrome coating and stainless steel (chromium electroplating) account for 85% of industrial use. Steel is given chromium to make it more oxidation resistant. With the addition of additional chromium, this resistance grows. Stainless steels must contain at least 10.5% chromium (normally 10.9 or 12.1%). When compared to steels with a lesser amount of chromite, this provides a noticeable degree of general abrasion resistance. The creation of a passive self-healing the stainless steel's surface has a coating of chromium oxide, like before explained, is what gives the material its resistance to corrosion. The first orthopedic implants were created in the 1920s utilizing stainless steel, which offers superior corrosion resistance due to its higher than 12% chromium content [9]. Due to its good fatigue strength, increased ductility, low cost, and improved machinability, type (AISI 316L) is often employed in implantable devices, particularly orthopedic implants [10]. Stainless steels are recognized for their high corrosion resistance in many conditions [11]. However, excellent corrosion resistance of chromium and chromium alloys is shown in solutions of 30% nitric acid [12].

Through this study, it will be determined how the addition of Cr affects the microstructure and mechanical characteristics of 316L stainless steels. As a result, it is believed that the mechanical characteristics of these steels made using the novel powder metallurgy can be enhanced. As is well known, the costs of manufacturing will drop dramatically, it is depending on the number of samples if these steels are produced via powder metallurgy. It will be made sure that the created item has good surface quality, and it will be possible to make some metals that are challenging to produce and process using conventional techniques.

As a result, different compositions of 316L stainless steel with addition Cr will be developed. They will be created using sintering time during manufacture. By subjecting the generated components to tests such cutting, mounting, grinding, polishing, image take, SEM microstructure, SEM fraction surface, and EDS analysis, mechanical characteristics will be ascertained, microstructure examinations (SEM microstructure, EDS Spectral analysis) of the samples will be used to determine the ideal sintering temperature for 316L microstructure steels produced by powder metallurgy.

PART 2

STAINLESS STEEL

2.1. DESCRIPTION OF STAINLESS STEELS

A class of iron alloys known as stainless steel includes those with a minimum of 11.2 % chromium [13], a substance that gives the iron heat resistance and stops it from rusting [14]. The elements carbon (from 0.03% to higher than 1.1%), nitrogen, aluminum, silicon, sulfur, titanium, nickel, copper, selenium, niobium, and molybdenum are present in a variety of steel materials [15]. AISI three-digit codes are frequently used to identify certain varieties of stainless steel, like 340 stainless [16]. The alloy's chromium content, which creates a passive coating to shield protects the underlying material from corrosion attack and is capable of self-healing in the void of oxygen, is what gives stainless steel its resistance to aging. [8].

The following methods can be used to further boost corrosion resistance:

1. Up the chromium content to at least 11.2 % and add at least 8% nickel.
2. Involve molybdenum.
3. Nitrogen injection also boosts structural properties and cracking corrosion protection.

Since different environments require different amounts of chromium and molybdenum, there are several types of stainless steel available. Stainless steel is the perfect material for tools where both the strength of steel and corrosion resistance are desired because of its resistance to corrosion and staining, minimal maintenance requirements, and well-known shine. Stainless steel may also be rolled into a variety of forms. These can be used to surgical devices as well as other things. In the 1950s and 1960s, significant technological advancements made it possible to produce a wide variety of steel material. There are more types of steel is the Austenitic stainless steel is the more used of stainless steels [17]. They have a face-centered cubic crystal shape with an austenitic composition. This microstructure is created by melting steel with enough nickel, manganese, and nitrogen to preserve an austenitic microstructure at all temps, from the cold zone to the softening point. [18]. Since they have the identical microstructure at commonalty temps, austenitic stainless steels are not strengthened by thermal treatment.

2.2. CLASSIFICATION OF STAINLESS STEELS

Although the proportion of Cr is the same for all stainless steels, other alloying elements are frequently added to enhance their qualities. In contrast to other metals, Stainless steels are categorized based on their microstructure, or crystalline structure, which refers to the way in which the atoms that make up the steel's grains are arranged. Stainless steels can be divided into two classes based on their chemical makeup: Chromium and Chromium -Ni kinds. Consequently, there are several primary relatives that are mostly distinguished by their microstructure, including the as such duplex austenitic-ferritic, martensitic, ferritic, and austenitic-ferritic precipitation hardening families. Many of the steels' mechanical characteristics are stainless, including protraction, exertion strength, elastic modulus, ultimate tensile strength, rely on the kind of alloy (chemistry) as well as performing (microstructure) briefly described in table 2.1. The “Young's modulus” is an exemption, though, as it is rather extremely susceptible to the chemistry and microstructure of stainless-steel alloys [19].

Table 2.1. Properties of stainless steel [20].

AISI stainless steel	**Microstructure properties**	**Mechanical properties**			**Physical characteristics**
		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 Steel

The biggest and most diverse group of stainless steels, austenitic stainless steels account for nearly 70 % of all stainless industrial production. [21]. The steady austenite stage, which has a face-centered cubic (FCC) crystal structure, makes up the microstructure. In comparison to other varieties, austenitic stainless steels are more corrosion resistant, non-magnetic, with have superior weldability. A cold metal-forming procedure can also significantly strengthen them [22]. Since austenite is not magnetic, which is crucial given the extensive usage of magnetic resonance imaging (MRI) procedures, ferromagnetism is a preferable condition for orthopedic implant materials [23], among the key characteristics of a material for orthopedic applications is excellent corrosion resistance in bodily fluids. Austenitic stainless steels are a good option for orthopedic implants because to their properties. Conversely, austenitic stainless steels often exhibit lesser resistance to crevice corrosion and stress corrosion cracking (SCC) and increased vulnerability to corrosive environment and pitting corrosion [24].

2.2.2. Ferritic Stainless Steel

Similar to carbon steels, ferritic stainless-steel have a ferrite micro-structure that is a body-centered cubic (BCC) crystal structure at all temperatures. The Cr concentration in ferritic grades can range from 10.5% to 30.0%, while Carbon is maintained at least levels (0.08%). Although Chromium serves as the primary metallic component of an alloy in several ferritic stage, some of them also contain (Mo 4%), very few Ni, or not. These grades, such as austenitic stainless steels, are not heat treated and can’t be strengthened or toughened via heat treatment. When durability is not the major demand, ferritic grades are chosen because they are magnetic like carbon steel. However, ferritic stainless steels have a mediocre level of corrosion resistance, especially when stress and certain corrosive conditions are coupled, which results in a phenomenon known as stress corrosion cracking (SCC)[25]. Ferritic stainless steels are frequently used in car trim and exhaust units, and hot water tanks [26].

2.2.3. Duplex Stainless Steels

Duplex stainless steels have a collection of ferrite and austenite in their micro-structure (FCC, BCC). Even though producing a similar amount of austenitic and ferritic is the overall goal, the ratio is often 40/60 in commercial alloys. Due to this, duplex ferritic-austenitic steels incorporate several the best qualities of both classes. For example, they are tougher than ferritic steels but less tough than austenitic steels, and they are renitent to stress corrosion cracking (SCC), albeit not nearly as renitent as ferritic steels. Duplex stainless steels are magnetic and has an elastic limit. that is nearly double that of austenitic stainless steels. With about double the yield strength of austenitic stainless steels, duplex stainless steels are magnetic and have superior corrosion resistance. Additionally, they have a complex microstructure that offers superior austenitic stainless-steel kinds 304 and 316 resistance to corrosion protection and resistance to chloride stress corrosion [27]. These alloys may contain Mo (4.7%), while C is controlled at least amounts (0.03%). Duplex stainless steels are frequently used in heat exchangers, chemical industries, water filtration stations, maritime settings, and the paper manufacture [26]

2.2.4. Martensitic Stainless Steels

Martensitic stainless steels (MSS) are metal compounds of martensitic stainless steels, which are primarily composed of 11.5-18% Cr, 0.1-4.2 Ni, and 0.11-1.2% C. Sometime included are V, Mo, Nb, Al, and Cu. When cooled to a temperature that is adequate for martensitic transformation, these steels exhibit an austenitic structure at high temperatures. Martensitic stainless steels are composed of martensitic, undissolved carbides, and δ-ferrite, based on the content and activity history. The characteristics of martensitic stainless steels are influenced by the heat treatment process. To be certain that transition to homogenous martensitic structure and not of ferrite throughout the cooling phases, Martensitic stainless steels must undergo careful heat treatment due to their complex structure. This includes avoiding carbide precipitation and δ-ferrite production [28]. Chromium and carbon alloys with a body-centered tetragonal (BCT) structure make up martensitic stainless steels [29]. The alloy's carbon content affects the strength that may be achieved by heat treatment [30].

Martensitic stainless steels have a low ability to resist corrosion. By using cold working, these steels may be considerably toughened [31]. These alloys have good hardness and ductility characteristics when hardened and tempered. Surgery tools, creep-resistant steels, stainless tool steels, cutting blades, and propellers, and other applications that call for strong tensile strength, creep resistance, and fatigue strength qualities are some examples of where they are employed [26].

2.2.5. Precipitation Hardening Stainless Steels

Steels including chrome and nickel that may acquire extremely high tensile strengths are known as precipitation-hardening stainless steels (PH). The main advantages provided by this group of stainless-steel types are moderate corrosion resistance and simplicity of manufacture [26]. Utilizing precipitation hardening of crystalline phase, substantial strength is obtained in addition to the low mass percentage of C and the heavily alloyed structure [32]. Although precipitation hardening stainless steels may be precipitation hardened as well greater strengths than other martensitic types, their corrosion resistance is equivalent to that of austenitic steels [33]. Precipitation-hardening stainless steels have an austenitic or martensitic starting microstructure. Prior to precipitation hardening, austenitic grades undergo heat treatment to become martensitic grades. Precipitation hardening happens when the martensitic is heated to a point where hard inter - metallic substances form precipitates out of the array of crystals. Precipitation hardening stainless steels are often used in aviation and else high-tech sectors [26]. 17-4 PH, sometimes referred to as grade 630, is the most prevalent grade in this group. It has around 18% Cr, 5% Ni, 4.3% Cu, and 0.33% Nb in it [33].

2.3. STEEL STRENGTH INCREASING MECHANISMS

Materials' metallurgical structures' behavior has a significant impact on how they behave mechanically. It might be argued that the mechanical characteristics of the material also depend on these circumstances since the metallurgical structure of a material changes because of the thermal, mechanical, and chemical processes that are applied to it. Strength is among the most crucial characteristics of a material.

Depending on strength, other qualities change. According to materials science, the resistance is best described as the substance's resistance to plastic deformation. Metals undergo plastic deformation due to the propagation of linear flaws. Therefore, mechanical characteristics like hardness, ductility, and strength account for the abundance of dislocations in metals' internal structures as well as how they interact with other flaws. Strength enhancing processes can be listed as follows.

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

To enhance some of the desirable qualities in micro alloyed steels and to make the most of the enhanced steels, they are essential to have a thorough understanding of the mechanical properties-microstructure interactions. The hardening processes employed in micro alloy steels, such as grain size hardening, solid melt hardening, precipitation hardening, strain hardening, and hardening processes, enhance the steels' tensile strength. While the grain reduction method enhances the toughness while enhancing the strength in the hardening procedures.

2.4. ADVANTAGES AND DISADVANTAGES OF STAINLESS STEEL

Stainless steel is commonly used in the fabrication of parts and components across many manufacturing sectors because of its many beneficial properties. Above all else, the chromium ingredient gives it a high level of corrosion resistance. When steel includes at about 10.5% chromium, it is extra corrosion-resistant than steels without chromium. Additional advantages for customers include its exceptional toughness and tenacity in both hot and cold environments, enhanced processability and simplicity of production, low upkeep needs, prolonged lifetime, attractive appearance, environmental sustainability, and nature of recycling [34]. Once utilized, no treatment, coating, or painting is necessary for stainless steel.

Several benefits of austenitic stainless steels include:

1. The face-centered cubic (FCC) geometry of austenitic stainless steels allows for exceptional machinability as well as a limited yield strength-to-tensile strength ratio.
2. Cold working and a process known as progressive strain aging can be used to enhance strength. Austenitic stainless steels are sufficiently to resist rust.
3. Most austenitic stainless steels are non-magnetic.

In terms of undesirable aspects:

When utilized as implantable, 316L stainless steel is still unimmune to limited kinds of corrosion as pitting, splits corrosion, and stress corrosion cracking [35], [36]. According to research's, 316L rusts in the body's milieu and generates iron, nickel, and chromium [37]. In addition, it has a nickel content of 11.0-16.0%, which, if discharged, can be harmful to humans. There is proof that nickel ions at high concentrations in tissues can cause cancer and the most common contact allergies when they come into touch with the skin [38],[39].

2.5. USAGE AREAS OF STAINLESS STEEL

A valuable alloy, alloy steel is used to produce a variety of items that are essential to our daily lives, including communication devices, steam boilers, automobiles, aircraft, dairy equipment, leather, paper and soap, small home appliances, nuclear engineering, and exhaust production devices.

Stainless steels are used to make medical devices and equipment for food preservation. They are also utilized in the medical industry to make scalpels, screws, prosthesis, needles, and hip and kneecaps. In storage containers created for food and drinks, oven molds, and coated pots, stainless steel plates are securely favored since they do not alter the characteristics of the food, such as color and fragrance.

2.6. ALLOYING ELEMENTS

Most metals are not utilized in their pure state; instead, their characteristics are altered by adding alloying components for certain uses. Some of an alloy's qualities can be substantially altered by adding a certain amount of a second element. Unmixed Fe is too pliable and non-sufficiently resistant to corrosion to be utilized as an active biomaterial, and modest amounts of additional components dramatically improve its mechanical strength and resistance to corrosion. For example, the inclusion of Cr promotes the formation of a shield of Cr oxide in the composites (passive film) [8]. Stainless steels also include some or all the following alloying components according on the exact grade: Secondary elements such as silicon (Si), Cr, Ni, Mo, Mn, Ti, Nb, and Vanadium are also present [26]. Relationships between the stainless-steel group of alloys' composition and properties show in figure 2.1.

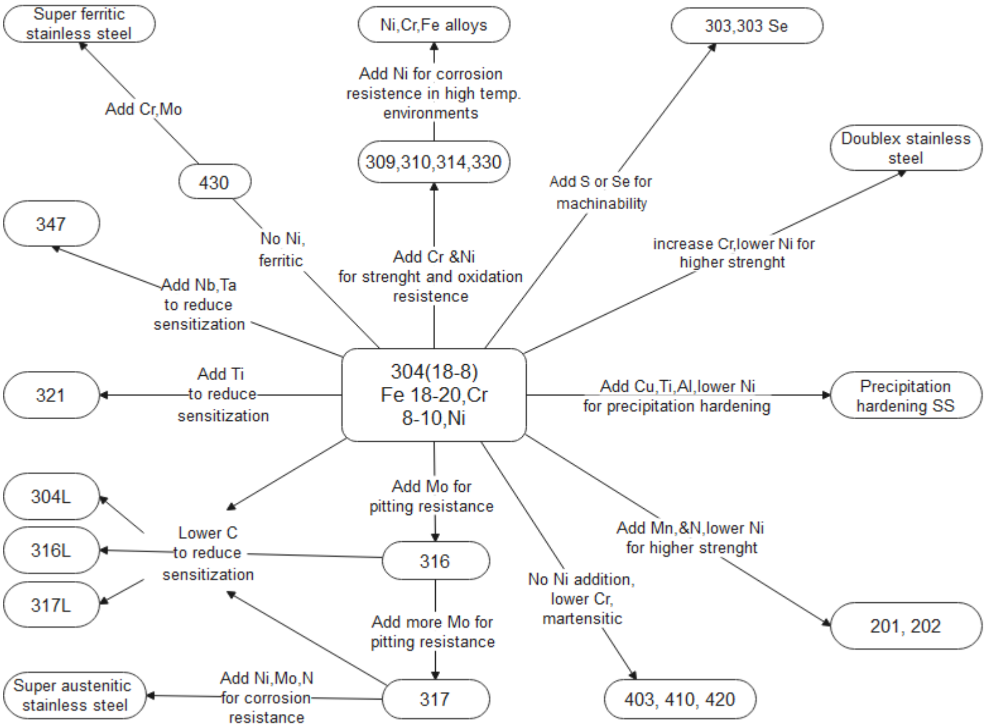


Figure 2.1. Relationships between the stainless-steel group of alloys' composition and properties [40].

2.6.1. Chromium (Cr)

Chromium for a steel to be adequately protected against moderate air corrosion, a steady chromium passive layer must have a minimum of roughly 10.5% chromium. With increased chromium concentration, stainless steel becomes more resistant to corrosion [9]. Because chromium is a ferrite forming, iron's body-centered cubic structure is stabilized when chromium is present. More nickel is required to generate an austenitic or duplex (austenitic-ferritic) form with greater chromium contents. Intermetallic phases are also encouraged to develop by increased amounts of chromium. Usually, austenitic stainless steels have at approximately 18% Cr, and commonly, second generation duplex stainless steels have at minimum 22% Cr. Additionally, chromium improves oxidation resistance at a high temp. This chromium impact is significant because it affects how oxide scale or heat tint that results from heat treatment or welding is formed and removed. Compared to austenitic stainless steels, it is more challenging to dissolve and remove heat tint from duplex stainless steels.

2.6.2. Molybdenum (Mo)

Chromium and molybdenum work together to help stainless steels resist chloride corrosion. In chloride-containing settings, if a stainless steel's chromium content is at approximately 18%, molybdenum increases become nearly three to four times as effective as chromium additions against pitting and crevice corrosion[41]. Due to its ability to create ferrite, molybdenum makes stainless steels more likely to generate unfavorable intermetallic phases. As a result, it is typically limited to less than 7 % in austenitic stainless steels and 5% in duplex stainless steels.

2.6.3. Manganese (Mn)

Stainless steels typically contain manganese to increase hot ductility. Temperature influences its impact on the ferrite-austenite equilibrium; at least temperatures, manganese stabilizes austenite; at elevated temperatures, it stabilizes ferritic. Due to manganese's ability to boost nitrogen's solubility, duplex and austenitic stainless steels can have significant nitrogen levels. Manganese may also replace part of the nickel in the stainless steel since it forms austenite.

2.6.4. Nickel (Ni)

Nickel acts as an austenite stabilizer in steel, causing the ferrite area to contract and the austenite zone to enlarge. Nickel becomes more resistant to corrosion and oxidation at high temperatures. The toughness and strength of nickel are increased by reducing the grain size. Additionally, it stops scale from growing on the material's surface. It enhances the critical cooling rate, ductility, hardness, and fatigue resistance when combined with chromium. Compared to many other elements, elemental nickel has a lower diffusion coefficient and diffuses into iron more slowly [42].

2.6.5. Carbon (C)

All grades of stainless steel and alloys designed to withstand heat contain the nonmetallic element carbon. Steel's strength is increased by Carbon, a powerful austenite stabilizer. Despite playing a significant alloying role in austenitic, ferritic, and duplex stainless steels, carbon has a strong propensity to producing carbides when combined with alloying substances. Accordingly, Carbon is maintained at a low rate (usually 0.003% C to 0.038% C in least-Carbon level) to lessen the likelihood of chromium carbide (Cr23C6) production by gradual cooling from high temp, as the creation of Cr23C6 eliminates the Chromium content from the sturdy solution in the alloy. Subsequently, the representation of Carbon results in imperfections in the lattice conditions of the microcrystals, which worsens their mechanical characteristics [21]. To achieve high strength and high hardness, carbon is purposefully added to martensitic stainless steels. Austenitic stainless steels are more resistant to creep when carbon is added. Therefore, because to its malleability and stainless qualities, steel with a high concentration of carbon is excellent for transient surgical devices (such as osteosynthesis plates and inside-the-medulla nails). However, it must be removed after a few years because to its low long-term corrosion resistance [43].

2.6.6. Nitrogen (N)

The ability of austenitic and duplex stainless steels to withstand cracking and fissure corrosion is improved by nitrogen. Additionally, it greatly boosts their tensile strength, making it the strongest component for solid solutions overall [44]. The nitrogen increased austenitic and duplex stainless steels offer greater durability in addition to greater strength. Nitrogen causes intermetallic phases to develop more slowly, allowing for the processing and production of duplex grades. To counteract their propensity to produce sigma phase, nitrogen is supplied to austenitic and duplex stainless steels with elevated concentrations of chromium and molybdenum that are extremely corrosion resistant. Since nitrogen has a significant austenite-forming ability, it can partially substitute nickel in austenitic stainless steels. To obtain the correct phase balance, nickel content is altered, and nitrogen is frequently added to duplex stainless steels almost to its solubility limit. The duplex structure is created by balancing the ferrite formers, chromium, and molybdenum, with the austenite molds, nickel, and nitrogen.

2.6.7. Niobium (Nb)

Niobium is an effective component in some stainless steels' interfacial corrosion prevention. Related to its characteristics to serve as a mild resistive metal, interest in niobium dramatically rose with the commencement of the space competition in the 1950s. The discovery of the enhanced qualities of a carbon steel with a little amount of niobium led to the development of the micro alloy idea. Niobium's most significant usage is as an alloying component to micro alloyed steels, where it is employed to reinforce the metal construction. The use of super alloys in the hot areas of aviation engines is a significant secondary application of niobium. Additionally, niobium is used to increase out resistance (ferritic) and for applications requiring superconductivity (Nb-Ti alloys), which allowed for the creation of new magnets. Niobium is ditto used as an austenitizing and ferreting component in stainless steels [26].

2.6.8. Silicon (Si)

Small levels of silicon give steels a moderate hardenability. Additionally, it enhances machinability [32]. Austenitic stainless steels with Mo can have a little quantity of Si added to them to increase their ability to resist corrosion in sulfuric acid. High Si concentration in austenitic stainless steels enhances oxidation resistance while as well preventing carburizing at high temperatures [26].

2.6.9. Vanadium (V)

Vanadium is a micro alloying element that is most often utilized to improve the strength and resilience as opposed to hardenability of micro alloyed steels; this is done by “precipitation-strengthening” the small carbonatite particles that are produced during cooling or tempering [45]. Vanadium is thought to have a crucial part in boosting strength, according to some studies, even while tensile strength stays unchanged. Some have proposed that the addition of Vanadium results in impact toughness, while others have determined that modest levels of Vanadium produce very minor improvements in steels [46].

2.6.10. Titanium (Ti)

For stainless steels, titanium is the most popular stabilizing ingredient (austenitic and ferritic). Titanium increases resistant to pitting corrosion as well. Thought to improve hardenability when dissolved in steel, titanium has a significant tendency to generate carbides, which can frequently be found in the structure of steel as undissolved carbides, reducing hardenability [26]. FeCr (Mo) and FeCrNi (Mo) alloys have stabilizing elements added to stop sensitivity intermetallic corrosion. Studies on corrosion in Hanks' sol revealed that titanium-modified 316L stainless steel has stronger resistance to corrosion than standard 316L.

2.6.11. Aluminum (Al)

Adding significant amounts of aluminum increases oxidation resistance. For this function, it is employed in certain heat-resistant grades. Utilizing aluminum to create intermetallic compounds in the precipitation hardening of steels boosts their strength after the steel is aged [47].

2.7. MEDICAL IMPLANT CORROSION

Corrosion continues to be a problem for implants in human bodies today. The fact that it results in the production of non-biocompatible metal ions (debris) that cause implant failure is well recognized [48]. Medical implants have frequently experienced corrosion issues. For instance, it's been found that corrosion was a contributing factor in about 41% of implant failures involving Ti-6Al-4V and 316L. Understanding how all implants used in the human body, especially orthopedic implants, corrode is crucial [49].

2.7.1. Corrosion

Whenever a metallic device is put into the body's aggressive electrolytic milieu, corrosion, the progressive destruction of materials by electrochemical assault, is of major concern. Blood and other bodily fluids that include components including water, sodium, chlorine, plasma, amino acids, and mucus in the like in saliva present a harsh corrosion environment for the implants [50]. Anions such chloride, phosphate, and bicarbonate ions, cations like Na+, K+, Ca2+, and Mg2+, organic compounds of low-molecular-weight species as well as comparatively high-molecular-weight polymeric components, and dissolved oxygen make up the aqueous dispersion in the human body [51]. By absorbing the byproducts of anodic or cathodic reaction, living molecules disturb the balance of the implant's corrosion processes. The equilibrium across the double layer of surface produced by the surface's electrons and additional cations in the solution can be disrupted by proteins attaching to transition metals and moving them far from the implant surface. Additionally, it has been shown that proteins that are digested on the surface restrict oxygen transport in some areas and lead to selective corrosion in those areas. By digesting the hydrogen present in the area around the implant, bacteria appear to alter the corrosion-inhibiting properties of hydrogen, which is created by cathodic response. This behavior tends to increase corrosion. Corrosion is affected by pH adjustments as well. Although the pH in the human body usually maintained at 7.0, it can change from 3.2 to 9.5 for a variety of reasons, including events, imbalances in the biological system caused by ailments, infections, and other causes, and post-surgery, the pH value close to the implant generally varies from 5.2 to 5.7. Even if most of the materials are protected from the environment's impact by surface oxide layers, Clinical data suggests that the implants are releasing metal ions, and the corrosion process is responsible for this leaching.

2.7.2. Corrosion Types of Medical Implants

In general, or locally, corrosion happens. The metal surface is known to disintegrate evenly in corrosion in general. Local corrosion, on the other hand, occurs only in a few places on the metal surface. Galvanic corrosion, pitting corrosion, crevice corrosion, and friction corrosion are the most pertinent forms of corrosion for the metallic alloys used in medical implants, and these types of corrosion are regarded as local corrosion [52].

2.7.2.1. Galvanic Corrosion

When any pure metal or alloy is placed in a corrosive and aggressive environment, its electrode potential will be formed as Ecorr. Ecorr is the point whenever the levels of oxidation and reduction are precisely equal. Galvanic corrosion is the electrochemical potential difference between two different metals. Theoretically, when two different metals are used, one will be the anode (the one with the lower electrode potential), the other the cathode, and corrosion will start quickly between the two. The galvanic series is an ordered list of experimentally measured corrosion potentials for both pure metals and alloys in natural seawater, as shown in Figure 2.1. When measuring corrosion potentials, the standard electrode can be “Standard Hydrogen Electrode” (SHE) or “Saturated Calomel Electrode” (SCE) [53].

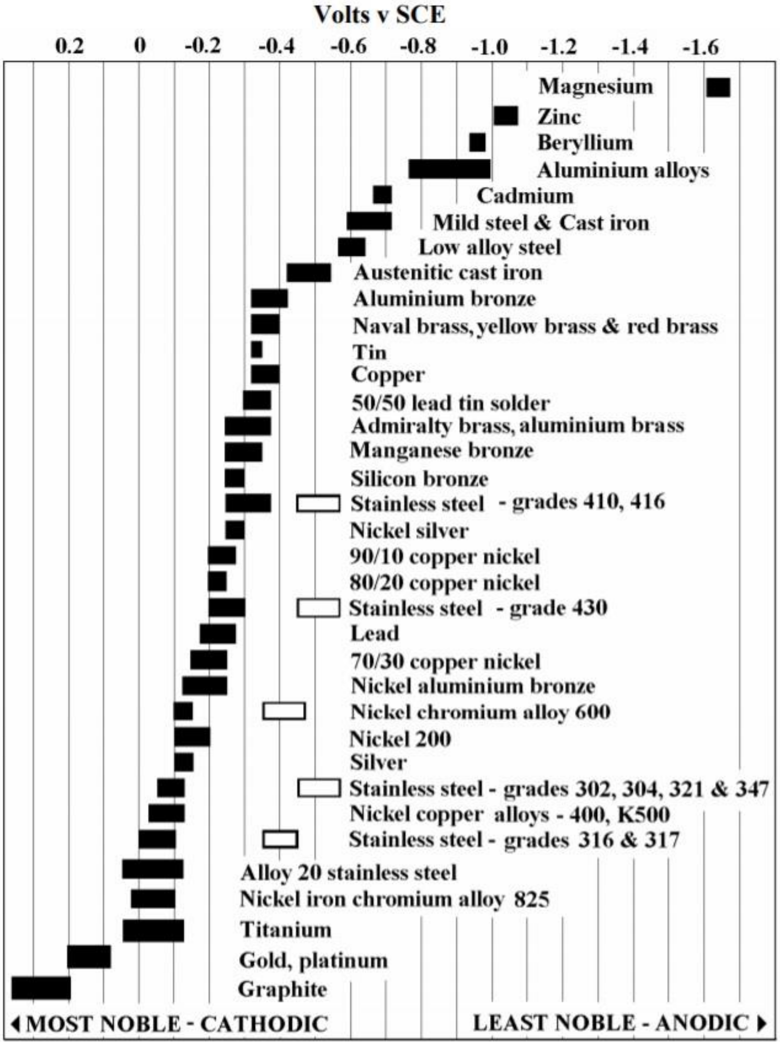


Figure 2.2. Galvanic series in sea water .

Metal orthopedic implants utilized in the human body are studied for their corrosion behavior using the galvanic series discovered experimentally in natural sea water [54]. Seawater can be compared to diluted body fluids and Simulated Body Fluids (SBF), such as Ringer's solution [55].

The electrode potential directly depends based on the kind pure element or metal, the electrolyte's makeup, the width of the oxide film coat, and the quantity of metals or alloys found [53]. Because orthopedic implants frequently employ a variety of metals, galvanic corrosion is a major problem.

2.7.2.2. Friction Corrosion

The phrase "tribocorrosion" refers to corrosion that is aided by wear. In terms of terminology, tribocorrosion includes friction corrosion as a subtype. Friction corrosion is a kind of corrosion promoted by micro-movement caused wear. When two opposed interfaces, such as bone plates and the screw heads of medical implants, rub against one other continually and oscillatory in the bodily environment, friction corrosion results. In a corrosive media, it results from minute relative motions between the contacting surfaces. Friction can happen even in the absence of a corrosive liquid. As is known, when a metal is exposed to an aqueous or humid environment, an oxide film forms on it. This layer tries to protect the metal from corrosion. Micro-wear debris disrupts the oxide film on the implant, altering the electrochemical balance and the implant will corrode again. Corrosion will continue until new oxide film is formed. This cycle (debris due to micro-abrasion, deterioration of the oxide film, changing the balance in the environment and formation of a new oxide film) usually occurs in orthopedic joints such as hip implants [56].

Friction corrosion is impacted by several factors of environmental aspects in the body, including oxygen, proteins present, wear, stresses placed on the implant, and integration with bone cells. These elements differ in various bodily regions [56].

2.7.2.3. Crevice Corrosion

A type of corrosion connected to structural elements is crevice corrosion. It happens when a metal surface is only slightly protected from the outside world. It occurs in fatigue cracks and other cracks where the effect of oxygen is reduced. Occurs in areas where oxygen is not available in abundance. For example, crevice corrosion that happens in geometric spaces like the hip prostheses' flexible connectors [57]. It is caused by small galvanic cells on the surface of the implant due to its inhomogeneity, which causes cracks when the material is eroded. Both the implant surface in contact with the electrolyte and the area inside the crack experience oxygen decrease at the start of crevice corrosion. However, the exterior metallic portion of the implant that is in contact with the electrolyte draws plenty of oxygen from the atmosphere to keep the

O2 content stable. On the other hand, because there is no oxygen circulation in the fracture, the amount of O2 falls. The metal portion exposed to low oxygen concentration serves as the anode while the exterior metal portion serves as the cathode due to the difference in oxygen concentration within and outside the crack. Corrosion of the broken metals will begin because of the electrode potential variation between the inner and exterior metal [58]

The presence of a fissure, a deep, narrow fracture, a junction between pieces of an implant parts, such as between a plate and a screw head, or flaws like fatigue cracks are fundamental conditions for the occurrence of this process. Comparing Type 316L stainless steel to the other regularly utilized metallic implant materials, crevice corrosion attack is especially vulnerable to Kind 316L stainless steel [59]. Corrosion of stainless-steel bone plates and screws, particularly where the screw heads meet counter sink holes, is a regular occurrence. Although these kinds of crevice- artificial failures have very rarely been seen, the inclusion of crevice corrosion in the bone plate's countersink region can also cause crack expansion. Crevice corrosion issues are frequently resolved by using the right device design and material selection.

2.7.2.4. Pitting Corrosion

Pitting is a acute type of confined corrosion assault that outcome in considerable volumes of metal ions being released and major damage. Pitting is the development of tiny the presence of voids or gaps on a material's surface that is normally covered by the existence of a slender passive coating that is self-healing, strong, and persistent. The interaction of some hostile ions with the film at the film's weak or faulty points is what is thought to have caused these pits to develop. In certain instances, the pits may be apparent to the unaided eye, but in most situations, they are unseen and harmful to the point where they may enable the development of stress corrosion cracking (SCC) or fatigue fractures, which may cause the components to fail catastrophically while in use [60].

The kind of a surface covering or coating that has developed on the surface because of the material's contact with the conditions has a substantial impact on how important pitting is. As a result, the material is pushed into a condition of "passivity," which protects it from all types of corrosion by delaying the surface-level deterioration process. Pitting occurs when a breach in the passive layer causes the anodic deposit to concentrate on a small area of the surface, abnormalities in the surface brought on by changes in the scaling layers, insufficient shielding, and the metal itself that accumulate at specific regions, etc. Outside of the pit, the cathodic reign may be located wherever. Consequently, the base of the pits has a high corrosion current density. The film covering the superior of the pit prevents the movement of metal element or H+ ions from the bottom [61]. Oxygen depletion causes a variant in electrochemical potential between the pit and the metal around it. Figure 2.3. illustrates the development and dissolution of passivity as well as the process of pitting assault. Once the pit is started, the metal ions precipitate at the top and frequently create a film that covers the pit. The film prevents oxygen and solution from entering the pit and repassivates, which might restore safeguard [62].

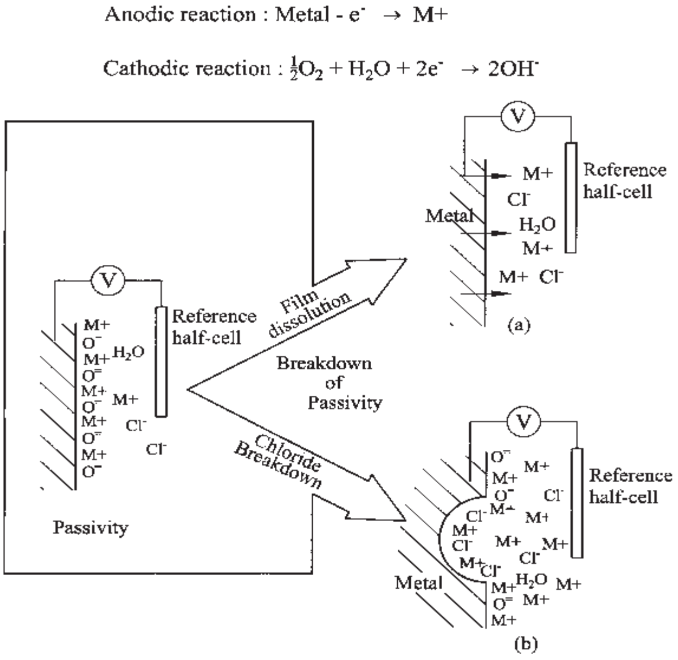


Figure 2.3. There are two main mechanisms by which passivity breaks down in metals: (a) general corrosion with film disintegration, and (b) pitting corrosion.

PART 3

POWDER METALLURGY

Powder metallurgy (P/M) is the process of mechanically and physio-chemically dissolving metals and metallic alloys to make workpieces under pressure and heat without melting the powders [63]. In this process, mixtures of pure metals in powder form, carbon, ceramic, and plastic components are put under pressure after mixing. It is then put through a procedure known as "sintering," which is carried out at a temperature that is lower than the metal's melting point powders utilized, to forge a solid link between the contact surfaces of the particles and to offer the appropriate qualities [64].

The basic steps involved in the production process are given below, Figure 1:

1. Making a powder or mixture of powders with the required content.
2. Complete mixing and blending are done to give the powder the special characteristics.
3. Molding the powders into the required form and size while strengthening the portions.
4. Sintering, which involves heating compacts at a high temperature to give them strength.

The most unique manufacturing method among the numerous metalworking technologies is powder metallurgy (P/M). The use of powder metallurgy is efficient because it allows for the cost-effective manufacture of intricate and high-quality components. P/M is the initial pressing of metal powders with various sizes, shapes, and packing characteristics, followed by the combination of the particles by sintering and creation of robust, delicate, and high-performance components. With these advantages, P/M effectively employs automated processes with low energy use, high material utilization, and cheap cost, eliminating current worries about efficiency, energy, and raw materials [65]. Powder metallurgy is ideal for producing tiny, intricate, and precise-dimensional objects. Little to no tangible loss occurs. Although the tolerances acquired are discretionary, this process yields smooth surfaces without material loss. In [64].

P/M is therefore continually changing and displacing conventional metal forming techniques. P/M has a variety of uses. Examples of products made from metal powders include tungsten lamp filaments, dental equipment, gear wheels, non-lubricated bearings, surgical devices, workplace machine parts, high temperature filters, airplane brake pads, electrical connectors, and nuclear fuel components, battery components, and jet engine parts. Metal powders are also used to make explosives, welding electrodes, rocket fuel, printing inks, soldering tools, catalysts, porous concrete, printed circuit boards, enriched wheat, and other products. However, according to [64], the production of cutting tools for machining, electronic components, magnetic materials, and high technology composites has grown quickly in the aircraft sector.

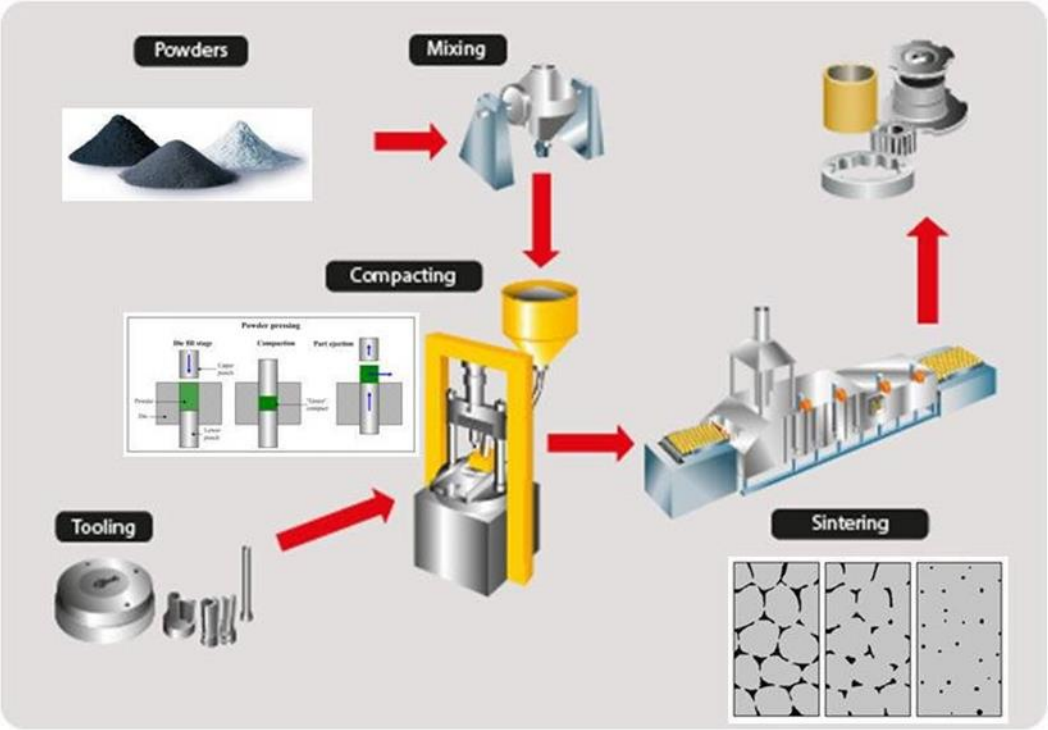


Figure 3.1. Powder metallurgy stages [66].

3.1. POWDER PRODUCTION

Most materials can be ground into powder. The metal particles utilized in P/M are less than 200 µm and continue to grow smaller due to technical advancements. The size, shape, microstructure, and chemical characteristics of the powder generated are always influenced by the process used to make it. Powders with certain qualities are produced using a variety of manufacturing techniques. Manufacturing processes for P/M and metal powder have a close relationship. The techniques employed have an impact on the powder's physical characteristics. According to the intended part output, the right powder choice should be determined in this respect [67]. The powder's size, shape, and surface morphology reveal what production-related variables were in control when the metal and alloy were created. Dimension is a crucial feature in powder metallurgy that is influenced by the product's final usage.

Most metal powders manufactured now adays range in size from 10 to 500 µm. In P/M processes, the size and form of the powder are crucial for both fluidity and compressibility. Powder production methods are mainly divided into many groups:

1. Solid State Reduction
2. Atomization
3. Electrolytic Methods
4. Chemical Methods
5. Mechanical Methods (Grinding method)

Mechanical, chemical, and electrolytic procedures were once the most widely utilized techniques for producing powder [68]. According to a 2006 study by Gazi University, more than 60% of powders used in industry today are generated via the atomization process. For instance, gas atomization is used to produce spherical powder, water atomization is used to produce irregular powder, and inert gas or oil is used to produce particles without oxide.

3.1.1. Solid State Reduction

In the process of solid-state reduction, the metal ore is broken and crushed, typically combined with carbon, and then treated in a continuous furnace. The powder is reduced in oxygen and carbon in the furnace, creating a “cake of sponge metal” that is then shattered, cleaned of any non-metallic substance, and sieved to create powder. The production procedure does not involve any refining; thus, the powder's excellent quality is solely reliant on the quality of the crude ingredients. The unusual and non-uniform sponge-such as particles are soft and readily compressible, and they produce pact with kindly green strength (pre-sinter strength) [69].

3.1.2. Atomization

Atomization is a technique that may be used to turn any molten metal into powder. In this procedure, slushy metal is separated to minute droplets and instantly frozen to prevent the droplets from coming into touch with one other or with a solid surface. As seen in Figure 3.2, a thin flow of molten metal is typically broken up by being struck by high-energy jets of liquid or gas. This process is used to generate brass, alloy steels, bronze, iron, copper, and metals with minimal melting points like lead, zinc, tin, cadmium, and aluminum [70]. In theory, it may be applied to and utilized with any metal that can be smelted.

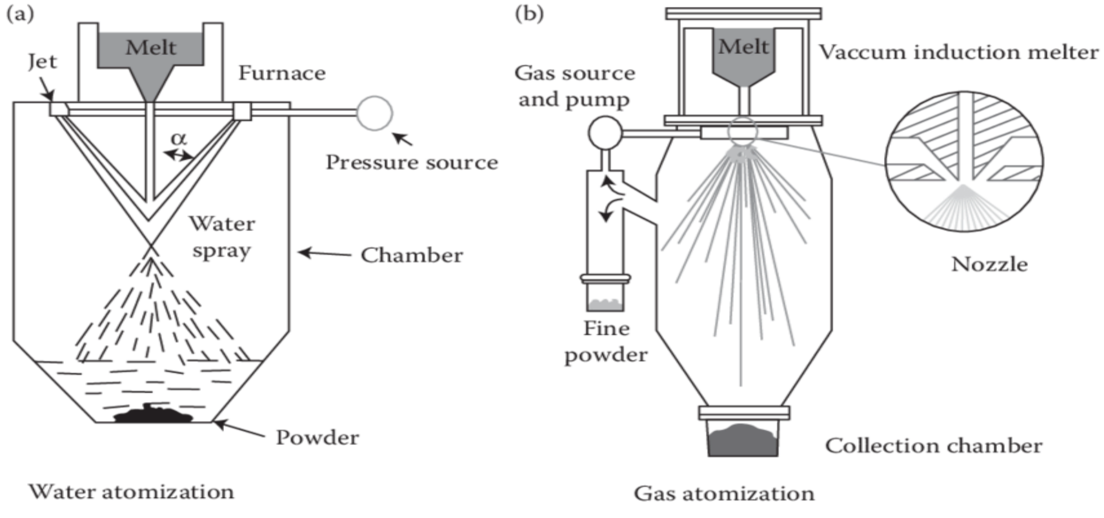


Figure 3.2. a.Water atomization process and b.Gas atomization process [71].

The alloys are beneficial when produced as powder particles because they are entirely alloyed when in the molten state. As a result, the chemical make-up of the powder particles will be 1:1. One benefit of the atomization approach is that it may be employed quickly to melt alloy powders since all the grains in the powder are the same size [72]. Below are some of the techniques used in the atomization process:

1. Gas atomization technique.
2. Water atomization technique.
3. Vacuum atomization technique.
4. Centrifugal atomization.

The atomization process is used to create more than 75% of metal powders. All fusible metals and alloys may be turned into powders using atomization processes. The water atomization technique creates 80% of the powder particles created by the atomization technique [73].

3.1.3. Electrolysis

This process is often used to create high purity metal powders that can be easily crushed and sintered [74]. This process yields powders that are 99.99% pure or more [75]. This technique uses the substance to be pulverized as the anode and places it in the electrolytic bath. The anode disintegrates in the electrolytic bath when voltage is applied and accumulates on the cathode. Following removal, the cathode is cleaned. Scraping is used to get rid of the dust on the cathode. It is powdered to a fine consistency. An improved grain compaction is ensured by annealing the powders in a reducing environment to remove any oxide. The powder produced by the electrolytic process is dendritic, spongy, and porous [76]. The composition, temperature, circulation rate, current density, size and form of the anode and cathode, electrode spacing, and rate of dust clearance all affect the powder's unique qualities. This process is the main one used to make copper powder. By using this technique, iron, nickel, cobalt, and many other metal powders are generated [77].

3.1.4. Chemical Reduction

The process of making iron powders frequently involves chemical reduction. This technique involves crushing the metal ore, mixing it with coke, and then putting the composite through a continuous furnace where reduction takes place. The continuous furnace is seen in Figure 3.3. The result is sponging iron that is produced as a cake. After that, non-metallic elements are removed from sponge iron before it is crushed and sieved. Purity of the powders affects the finished products' materials. Smooth and supple particles have a spongy, uneven shape.

They are easily compressible, and the resulting samples are robust. Refractory metals are created in a similar way by reducing their oxides with hydrogen [78].

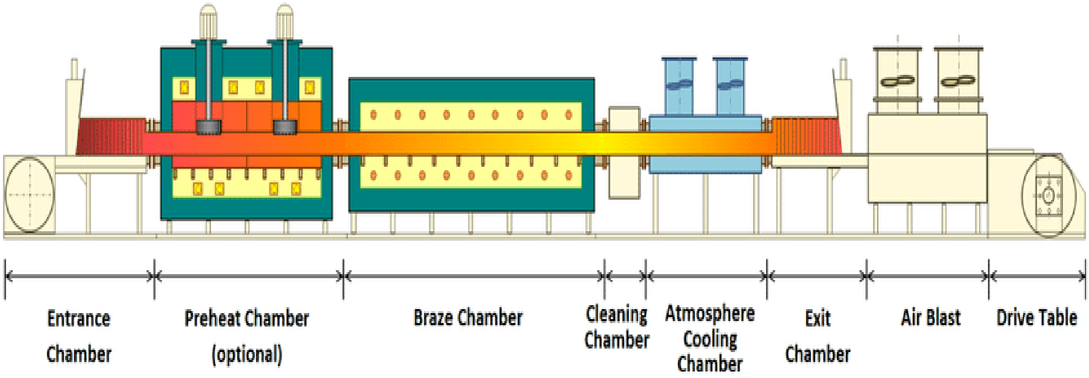


Figure 3.3. Continuous furnace [79].

3.1.5. Grinding Method

Grinding is a mechanical process mostly done in ball mills. Using this technique brittle material powders are produced; the fundamental idea is to make sure that a hard item strikes the substance you want to crush. The metal to be ground is put into a container with big, hard, wear-resistant balls. The material to be ground is separated into extremely tiny, large-diameter particles, which are then put to the grinding container as seen in Figure 3.4. If the material being milled is fragile, the impact of the balls will reduce it to very fine particles. The ductile particles that make up the milled material are flattened by deformation because of impact. The size of the balls to be used and the quantity of material to be ground are crucial factors in creating a homogenous mixture. The material to be ground should make up around 25% of the container volume, while the volume of the balls should be roughly 50% of the container content. In ball mills, brittle minerals including iron-chromium, iron alloys, and iron-silicon are mechanically processed [80].

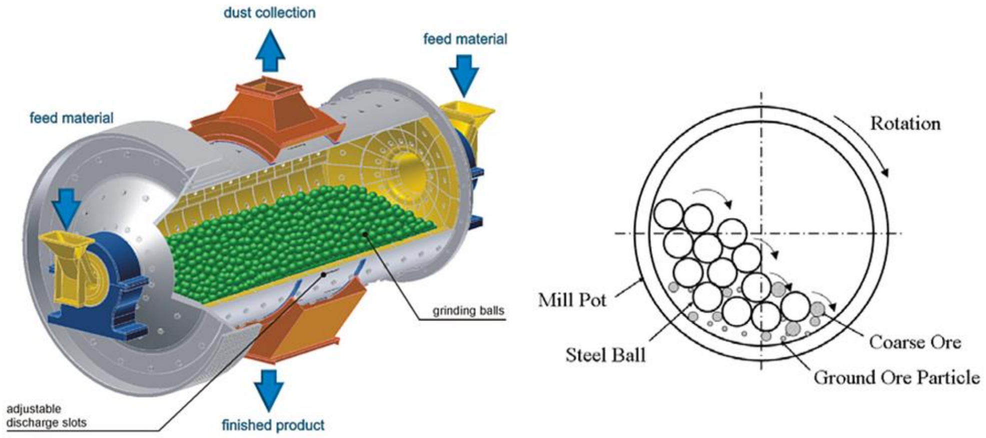


Figure 3.4. Grinding process [81].

3.2. ADVANTAGES AND DISADVANTAGES OF POWDER METALLURGY

Powder metallurgy method, is like other methods, has its pros and cons. In this section, the most important advantages and disadvantages of powder mining compared to other methods will be reviewed.

3.2.1. Advantages

1. The materials produced have high mechanical and physical properties.
2. Materials with different characteristics that do not dissolve in each other can be produced by combining them.
3. The particle size of the produced parts is small, the tensile strength and machinability are high.
4. Parts produced with powder metallurgy generally do not require additional processes such as machining.
5. There are no melting losses.
6. Materials with high wear resistance and high hardness can be produced.

7. After the sintering process, the part is ready for use and there is no need for secondary operations.
8. It can be easily produced in parts with complex forms and precision.

3.2.2. Disadvantages

1. The cost of metal powder particles is more expensive than materials used in casting methods.
2. It is Difficult to produce parts with high thickness and diameter ratio. There are also limitations on particle sizes to produce homogeneous densities. Powder metallurgy can produce parts up to 20 kg.
3. Lower mechanical properties may occur from time to time due to the existence of pores in the microstructure as compared to other approaches.
4. The cost of the molds required for production is high.

3.3. CHARACTERIZATION OF POWDERS

3.3.1. Powder Sampling

You can use several techniques to complete the arduous and time-consuming operation of powder sampling. Taking tiny samples from a variety of sites, blending them, and applying them is a popular technique. The particles have a cohesive overall form, and because of the circumstances, there is a significant probability for them to cling together. The possibility of particle aggregation eventually exists. Due to surface dampness, high aggregation may happen. Additionally, clinging agglomerates are defined as a collection of particles adhering to weak forces that are susceptible to destruction by minor shear pressures. Most of the time, the dispersion of the particles and subsequent determination of their properties are influenced by mechanical and ultrasonic agitation techniques. It is commonly chosen to disperse the flocculation formation using mechanical mixing or ultrasonic agitation [82].

3.3.2. Particle Size Measurement

In a variety of industries, including food, construction, biopharma, and pharmaceuticals, the study of particles and the characterization of their size and form are crucial. Particle size determination is not an easy procedure unless the particle is spherical. The diameter characteristic and the geometrical makeup of the particle are the main factors utilized to compute an odd number of particle sizes [83]. Particle size may be determined using a variety of techniques, the most popular being sieve analysis, laser diffraction, dynamic light scattering, and direct imaging approaches. When analyzing the same samples, the outcomes of various approaches frequently do not correspond well with one another. This can be explained by the fact that each method for determining particle sizes uses different underlying measuring concepts.

The oldest and most widely used technique for describing particle size distributions is sieve analysis. By putting the material through several sieves with various mesh sizes, the procedure separates small particles from more coarse particles by basically fractionating the particles within certain sieve bin sizes see Figure 3.5. To create a cumulative distribution, the mass fraction of the particles is weighed and quantified. The sieve technique just categorizes powder particles as larger or less than a given value; it does not actually measure the size of the powder particles [84]. Wet sieving and dry sieving are the two different types of sieve analyses that may be done. Dry sieving is ideal for particles from 30 µm up to 125 mm, while wet sieving is suitable for particle sizes from 20 µm up to 3 mm.

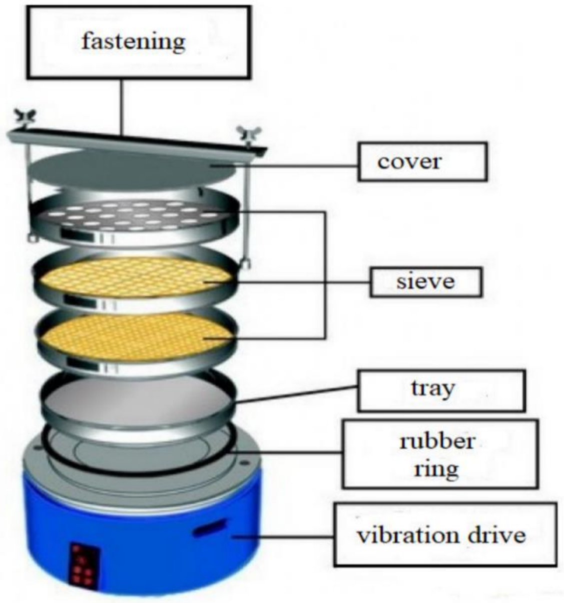


Figure 3.5. Sieve analysis test procedure [85].

3.4. PRODUCTION PROCESSER

3.4.1. Powder Mixing

Since powder mixes are frequently employed in the powder metallurgy method, it is crucial to thoroughly mix the powders before applying the compaction process. The primary goal of the mixing procedure is to ensure that the powder particle uniformity [86]. The performance of the finished product is improved by homogeneously blending powder particles of various sizes, shapes, and densities. The powder combination should be blended before it is mixed if there are no conventional dispersions in it. For better pressing and sintering qualities as well as a consistent size distribution, blending is advised. The process of mixing and blending is impacted by a few factors. These factors are:

1. Physical characteristics of powders.
2. Mixer dimensions.
3. Mixing and rotation speed.
4. Mixing time.
5. Powder volume in the mixer.

6. Humidity and atmospheric conditions.

Powders can be made using a variety of techniques. Mechanical and physio-chemical are the two primary divisions of these techniques. The methods used to create metal powders reveal a variety of powder particle characteristics. Using precision scales, the powder particles generated to the specified requirements can be weighed before the pressing process is initiated, or the desired powder quantities can be added to the process in the mixing mills to prepare them for the pressing process. The effectiveness of production methods depends on the caliber of the metal powders. One of the most crucial elements in manufacturing methodology is economy.

3.4.2. Powder Compaction (Pressing)

It's important that the metal powders fill the mold completely when they're created using a mold. The theoretical density is thought to be as close as feasible to being achieved when the powders are cold pressed in the mold. While using the same pressing pressure, each metal powder achieves a variable density after pressing, depending on the material's theoretical density. The powder's form, grain size and surface, material type, and pretreatment the powder has received are the variables that determine this state [72].

In most cases, pressing is carried out at room temperature using specifically designed steel molds. Tool steel that has been heat-hardened and based on cemented carbide is utilized in the mold-making process. The number of pores varies with the pressing pressure and gets smaller as the pressure ratio rises. However, when the pressing pressure increases, more powder comes into touch with each other and has a larger surface area. Its density diminishes with an increase in the number of pores. Depending on how many pores are present, the pieces made using the powder metallurgy approach have varying tensile strengths. The tensile strength ratio also rises in proportion to the density ratio.

When viewed from the outside, compressing powders appears straightforward. However, it is a complicated procedure with numerous variables. When compression starts, there is a higher degree of friction on the inner surface of the mold than any other force. But as it gets closer to the mold's center, it seems to progressively start to shrink. Significant losses in forming energy occur because of the friction between the mold surface and the powder. Furthermore, there are frictions between the contacting particles, the balls and the mold surface, and the powder and the balls. The amount of friction that develops between the product and the mold's inner surface when it is being withdrawn from the mold is crucial. To be effective in the forming and demolding process, a lubrication system that won't interfere with the sintering process is required [87].

The vibration of the mold causes the powders that are added to it before pressing to become denser. The powder's distribution pattern and form both affect the density that is obtained during vibration. Comparing powders with smooth and spherical surfaces to powders with irregular shapes, this rise is significantly bigger in the latter. This may be explained by the low relative densities of irregularly shaped particles with a limited powder size distribution and the high-density ratios based on spherical shaped powders. Relative density is an expression for the ratio of the raw density to the approximated density [88].

There are two forms of pressing, known as cold pressing and hot pressing, when discussing the pressing procedure in general. While heat and pressure are provided simultaneously in hot pressing operations, heat is added after pressing in cold pressing methods. When it comes to giving the finished product strong mechanical qualities and measurement precision, cold or hot isostatic pressing procedures are noticeably better than pressing with rigid molds. Due to the uniform distribution of pressure on the powder masses in the pressing techniques, high wet density and strength values may be attained even at low pressure. The materials generated by the hot isostatic pressing process have rather good mechanical qualities, such as tensile strength and fatigue strength. Under the following categories, cold and hot isostatic pressing are described [88].

3.4.2.1. Single action Die Compaction

Uni-axial compression is used in traditional powder compression procedures. The upper pressure plate starts pressing as soon as the powder combination is put into the mold, as seen in Figure 3.6.a. Scraping the finished item out of the mold is the goal of employing the lower piston. Lubrication is given to the mold walls to make the component removal process easier and to create compression. The single action compaction process results in a material that is denser as more pressure is applied, increasing the total amount of pressure applied. The decrease in the number of pores in the powders utilized, together with the fact that the mass is constant, but the volume is decreasing, are the causes of this. Despite the method's benefits, it is not favored since it is unable to achieve the desired density in elaborately formed components and metallic parts with a high length to width ratio.

3.4.2.2. Double Action Die Compaction

Upper and lower punches press on the die to exert pressure as shown in figure 3.6.b. They are both dynamic punches. The movement and pressure that a punch can exert might be different or equal. Because of friction created by the particles, punches, powder, and mold surface, the raw density distribution might vary greatly. Some efforts are made to lessen these characteristics by decreasing friction by using a lubricant or by adopting the appropriate compression techniques.

The single action compaction approach does not allow for complete density to be achieved. Since the powder is compressed by both the upper and lower punches in double action compaction, the friction force among the mold's wall and the powder particles is greater than the flexible material's spring force. This allows the mold to move towards the lower region, where the lower punch can equally press the powder with the upper punch. The resultant portion is removed from the mold by the movement of the bottom punch toward the higher section when the compression process is complete. As a result, the density distribution is found to be more homogenous than the components produced by single action compaction. The middle of the compressed area is where the density value is lowest. This density is distributed in a symmetrical manner [89].

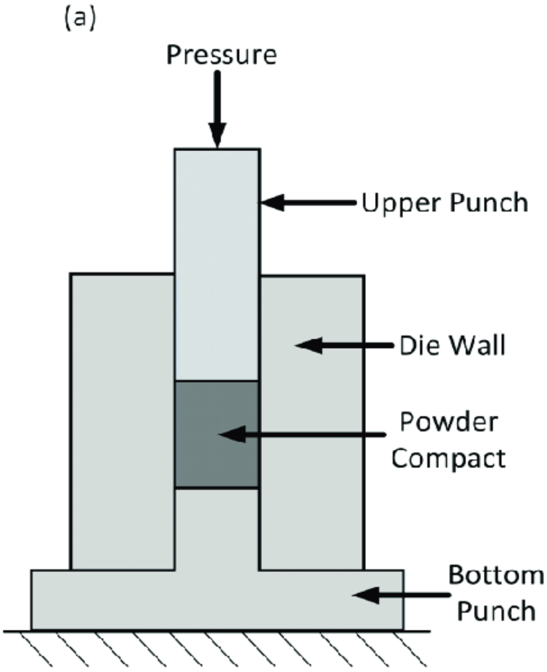


Figure 3.6. Graphical diagrams of (a) single-action die compaction and (b) double-action die compaction [90].

3.4.3. Isostatic Pressing

Isostatic pressing is the pressing of metal powders under a hydraulic (fluid) pressure. The procedure of isostatic pressing involves applying high pressure force to each point of the mold to compressible powder particles that have been deposited in a mold constructed of a sealed, flexible material. Regardless of the part's dimensions, such as its diameter and length, the components produced by the isostatic pressing process have a same density throughout. The homogenous impact of pressure on all surfaces and the lack of friction on the mold wall ensure the homogeneous density arrangement. It is a useful and essential approach for the creation of intricately shaped pieces. Cold isostatic pressing and hot isostatic pressing each have diverse application fields [91].

3.4.3.1. Cold Isostatic Pressing

The pressing procedure is often done using oil or cold water and at room temperature. This technique is known as cold isostatic pressing because of these characteristics and approaches. The powders are inserted in a rubber mold and pressed using the cold isostatic pressing technique in an oil-prepared pressing section figure 3.7. Each point of the mold receives the same pressure value from the pressure piston when pressure is applied, and each point of the sample has an identical density because of the oil in the mold. Although compression pressures of up to 1400 MPa are capable of being reached, the operation is typically conducted at pressures lower than 350 MPa. Flexible materials like rubber, polyvinyl chloride, or elastomers are frequently utilized to create the mold for the cold isostatic procedure. It is an appropriate technique for producing parts with complicated forms due to the use of flexible molds. The usage of rubber results in very reduced mold costs. Super alloy aviation turbines and tool steel billets are excellent examples of this pressing technique [92].

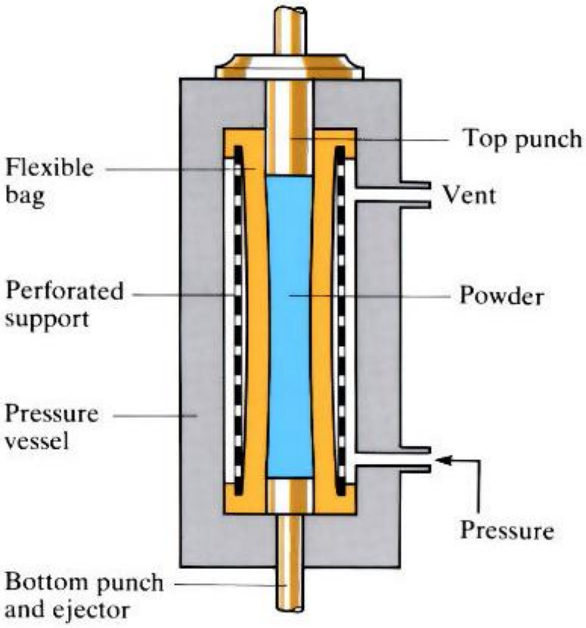


Figure 3.7. Cold isostatic pressing.

3.4.3.2. Hot Isostatic Pressing

The statically or dynamically heated powder is subjected to pressure coming from one or more areas in opposing directions along a single axis. One of the older methods of compacting powder metal is known as hot pressing the metal inside a box that has been compressed. Refer to Figure 3.8. Hot pressing produces goods with excellent characteristics, however there are a lot of drawbacks. These include the boiling of the liquid metal from the punch surfaces to the die walls and the resulting degradation of the squeezed surfaces, tool wear, die wear, sample loss, the space between the box walls and the moving bushings, and die wear [93].

Hot pressing needs less energy, less time, and lower temperatures than conventional cold pressing procedures. In comparison to cold pressing, this technique also achieves greater densities. The molten metal is crushed using a technique called hot pressing. Rich techniques are also given. The metal's physical characteristics determine the technique of choosing. There are characteristics shared by all hot-pressing methods [94]. Products with excellent surface quality, density that is near to the theoretical density, high metallurgical bonding, corrosion resistance, and extremely good mechanical qualities may be produced using the hot isostatic pressing process. The aviation sector prefers to produce parts using this technique.

The hot isostatic pressing technique differs from the cold isostatic pressing technique in that it presses powder particles in a chamber with an inert gas, such as argon, rather than a liquid. By pressing with a pressure of 150–350 MPa, the temperature value in this approach increases to 1700°C.

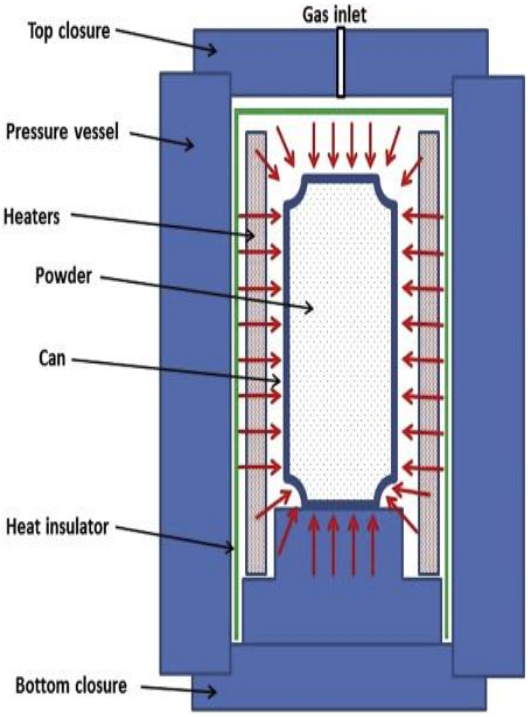


Figure 3.8. Hot isostatic pressing [95].

3.4.4. Sintering of Powders

The sintering process is described as a high temperature, carefully regulated curing process used to strengthen the crushed powder particles. With the help of the sintering process, the mechanical bonds that were created when the particles were pressed and shaped in the mold are converted into metallic bonds, giving the object further strength. There is a significant difference in the part's strength ratio before and after the sintering process. In systems with a single component, the sintering process is used at a temperature least the absolute melting level of the metal; however, in systems with several components, it is often done at a temperature above the melting point of the component with the lowest melting point. For some refractory materials, the sintering process is used between 70 and 80% of the basic material's melting temperature, however the melting temperature can be raised to 90% [96].

The sintering process can be described as a material transport system that is activated by heat and results in the reduction of the powders' surface area, the expansion of their contact points, and, ultimately, the modification of the shape and volume of their pores.

Sintering furnace is another name for the machine that regulates time and temperature during the sintering cycle. Additionally, by maintaining the environment and removing lubricants and binders, it gives the part the option of heat treatment after sintering. Following the pressing procedure, the spherical powder particles are in point contact, and during the sintering procedure, welds are created between the particles in contact with one another and the bonds are reinforced, as illustrated in figure 3.9. The size of the particles first increases, then as the sintering period progresses, the pores then begin to contract. Later, the pore channels shut and transform into a closed pore shape.

A decrease in the high surface energy of the powder particle during the sintering process leads to the particles having a precise size. This causes the interior structure's porosity value to drop to zero. Because the surface energy dropping according to the volume ratio is determined to be precisely proportional to the inverse of the powder particles' diameter, small powder particles are readily sintered. Ceramic materials gain in transparency, thermal conductivity, density, and strength, whereas polymers gain in density and strength. Conductivity and strength both rise in metals. Alloys that have similar qualities to porous bronzes are sintered between 600°C and 800°C, iron-group metal alloys between 1000°C - 1300°C, and hard alloys between 1400°C and 1600°C. According to the type of material, sintering temperature and time might differ. Hard alloys need a sintering process that takes more than an hour, whereas diamond alloy materials may be processed in less than a half-hour. Temperature and sintering time have an inverse relationship. When the temperature is high for sintering, the duration is short, and as the temperature drops, the applied time lengthens [97].As the sintering temperature increases, the properties of the material such as strength, electrical conductivity, density, and ductility increase.

The powder particles are in point contact during the earliest stages of sintering. The materials are subjected to three phases of sintering: initial, middle, and final. The sintering process uses reactive power since the system's internal energy is reduced. The reduction in surface area caused by the expansion of the particle contacts areas, the decrease in pore volume or the transformation of the pores into spherical form, and the elimination of the difference caused by the dissolution of the solid phase in the liquid phase in systems with multiple components are the factors supporting this decrease [98]. When using the sintering procedure, certain atomic transport orientations that occur with temperature inside the microstructure cause events including dimensional alterations, size and shape modification of pores, and grain development. Atomic transport directions: condensation, evaporation, plastic flow, grain boundary diffusion, surface, and volume diffusion. The surface diffusion has no effect on dimensions. Different environments are employed for the sintering process. Below are products that represent these:

1. Nitride atmospheres: Ammonia
2. Atmospheres containing vacuum, helium, argon, and nitrogen.
3. Reducing atmospheres: H, Methane CH_4 and ammonia, CO_2
4. Oxidizing atmospheres: Air, CO_2, and H_2O.

The sintering process is generally performed in two different ways, and these techniques will be explained below.

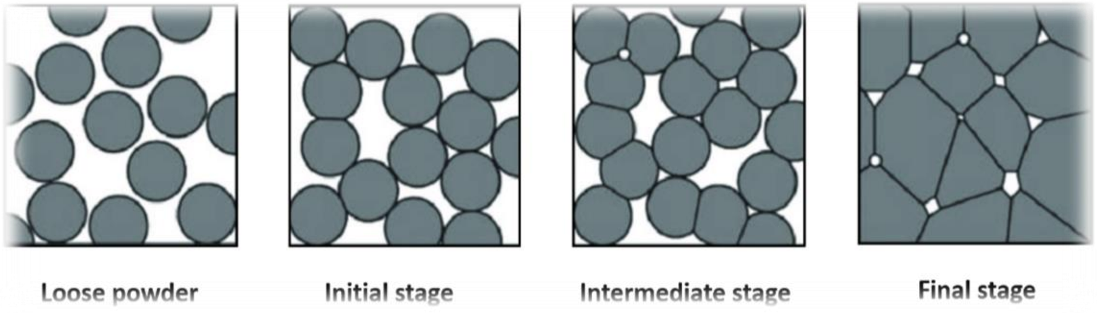


Figure 3.9. Sintering process [99].

3.4.4.1. Liquid Phase Sintering

Sintering is a heating process that takes the place of melting and occurs below the melting point of the metal powder particle. When using a mixture of powders, the sintering process is conducted at a temperature that is lower than the melting point of the powder that has the greatest melting point among the mixture. Rarely, the sintering process is conducted above the material's low melting temperature or melting point. Liquid phase sintering is the term used to describe the sintering method above [100].

The liquid stage sintering process, the liquid stage and a solid powder cluster coexist. Both the binding formation between the particles and the sintering speed are accelerated by liquid phase sintering [101]. The powder particles attract one another and form a serial condensation without pressure with the help of the capillary attraction forces produced by the liquid phase condition. By lowering particle-to-particle friction, the ensuing liquid phase quickens the rearrangement. Part characteristics and pore structure, including interparticle bonding, ductility, strength, magnetic permeability, conductivity, and corrosion resistance, undergo significant modifications.

Solid state-controlled sintering is the next step in the liquid stage sintering process. The presence of skeletal structure at this stage causes condensation to slow down [102]. The melting temperature of the material is selected by selecting a temperature range of roughly 80% at the sintering process temperature to sinter compacts made up of single-phase particle formations. Dimensional variations and changes in the microstructure during sintering show variations and transformations in the mechanical and physical characteristics of the component. These changes in the microstructure are preformed and subsequently provide raw materials with low strength.

The contact area between the powder particles increases, the structure's existing pores round out, and the separation between the particle axes reduces during the first stage of sintering. The component experiences the shrinking phenomena as a result, and the density rises. The dust particles eventually combine with one another in the next stage. Pores eventually develop in isolation. With the movement of materials through the structure, the pores' size reduces. Later, grain growth is seen [103].

3.4.4.2. Solid State Sintering

Solid state sintering refers to the modifications made to the part's mechanical and physical characteristics, as well as its size, during the sintering of mixtures created as a result of the sintering of a single element. Reactive power lowers the system's free energy, allowing the sintering process to proceed. The increase in dust and these grains, as well as the decrease in the overall grain boundary region, are caused by the loss in free energy. As the degree of curvature of the grain boundaries rises, the pace at which the boundary moves increases. Temperature is the primary factor influencing how the boundaries and atoms move. With the increase of this temperature, the atomic emission increases rapidly. This movement of the grain boundary region ensures the disappearance of small particles and the emergence of large grains. The material is transported, and the grains diffuse to create the solid-state sintering process. Because the material diffuses more readily at high temperatures, a high temperature is preferred for sintering. The internal structure of the material does not become liquid during the sintering process. The solid phase facilitates internal changes in the material, such as diffusion and density rise. The driving force needed for solid state sintering is revealed in the internal structure of the material by the difference in free energy between the grain's surface and the neck area. The point contact of the grains with one another initiates the process of solid-state sintering. Beginning with the movement of the grains, the contact of the grains with one another rises. With increasing passage of time, it becomes possible to see the development of necks, or sintering bridges, between the particles in contact. Even though the dust particles in this area are in touch, they are still in a separated posture. As the centers of the particles with neck development approach one another in the later phases of the first section, volumetric shrunken grains begin to form [104].

An increase in the size of the necks produced between the particles takes place during the intermediate stage of sintering. The width created in the neck must be greater than a specific width value for the particle shapes to change. Both the inter- and intra-particle spaces show reductions. Shrinkage happens in the material's physiology because of the decline in void ratios. Grain growth happens as the particles agglomerate near the conclusion of the intermediate segment[105] .

3.5. ANALYSIS OF MATERIALS PRODUCED BY POWDER METALLURGY METHOD

3.5.1. Mechanical Properties

Choosing a material for a certain application depends on the material's mechanical characteristics. It's crucial to become familiar with the accepted methods for calculating these attributes and assessing the outcomes of these trials considering various inputs. The compression and tensile technique are used to assess a material's capacity to endure static stresses. Static testing can be used to learn more about a material's resistance to plastic deformations. The resistance of the material to stresses under impact is ascertained using the impact process technique.

The material's brittle-ductile transition temps are determined by doing these tests at various temperatures. The behavior of the material under repeated loads and variations, as well as its useful life under these effects, may be predicted using the fatigue process approach. Creep tests are used to observe the material's behavior under sustained loads at high temperatures.

3.5.2. Microstructural Features

The arrangement of the components that make up a material, the structural and chemical character of its geometric creation expresses the microstructure, which to some extent includes the flaws and component phases of the material. The qualities of a material can be strongly determined by the microstructure. Additionally, understanding the connections between the microstructure and characteristics is important to use the ore in addition to learning the elements that affect the material's microstructure. The microstructure of the material must be determined from one aspect while the relationships in materials science must be determined from another .

3.5.3. Surface Related Properties

The material's surface characteristics include things like oxidation and corrosion. The fluid that forms in the pores and then accumulates causes corrosion to happen more quickly if the structure of the materials produced by the powder metallurgy process has a high porosity rate. According to observations, materials made under ideal sintering circumstances have a high level of environmental resilience.

PART 4

MATERIALS AND METHODS

In this study, alloys in required ratios were combined by powder metallurgy method and create samples. These samples were prepared in different Cr ratios to examine the effect of mechanical and microstructural properties. The process steps performed in this study are in section 4.1, the mixing of powders and pressing process are in section 4.1.1, The sintering process is in section 4.1.2, the tensile test sampling is described in section 4.2, optical microscopy and SEM image analysis are described in section 4.3 Tensile tests, Optical microscope examinations and SEM experiment was carried out in Karabuk University, Iron and Steel Institute, MARGEM laboratories.

4.1. EXPERIMENTAL PROCESSING STEPS

In this experimental work, the process steps included obtaining, mixing, pressing, and sintering the powders, as shown in Figure 4.1. It also comprises mechanical evaluations and microstructure analyses.

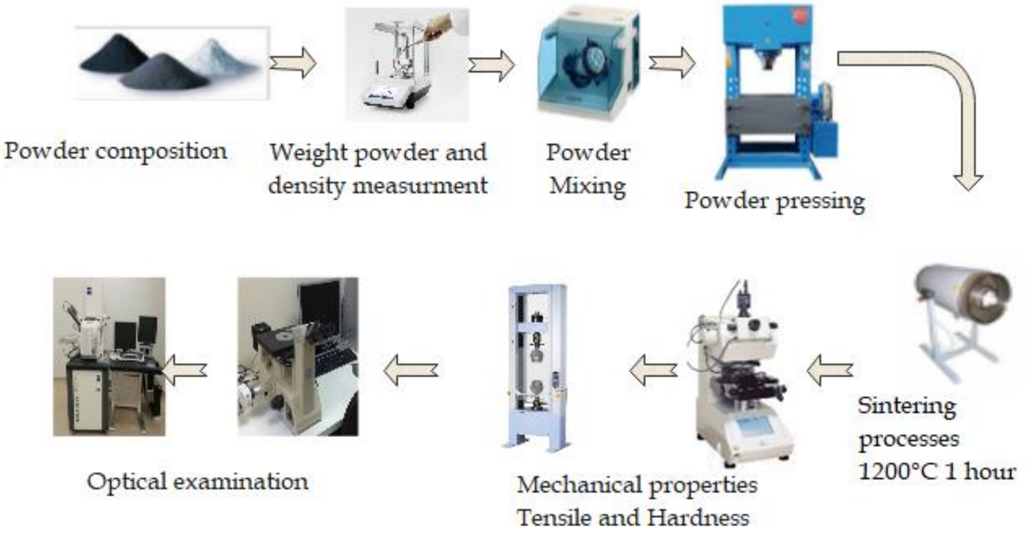


Figure 4.1. Process steps in experimental study and Microstructure analyzing.

4.1.1. Mixing and Pressing the Powders

In this study, PM steel samples containing chromium were produced in desired compositions by powder metallurgy (PM) method. The purities, density and sizes of Cr and 316L powders are given in Table 4.1. Prior to mixing, the powder particles were weighed using a precision balance of the RADWAG AS-60-220 C/2 brand at the rates listed in Table 4.2 with an accuracy of 0.0001. To create a homogenous structure, powder mixes acquired by weighing were combined in a triaxial mixer of the Turbula brand for 1.5 hours without a ball. In his investigation on mixing duration, Erden found that mixing the produced powder mixes for 90 minutes resulted in the greatest yield and tensile strength values [106]. This research's mixing time was similarly set at 90 minutes in accordance with Erden's study, which found that prolonging the mixing time past 90 minutes had nearly no impact on the mechanical characteristics.

Table 4.1. Powders and their properties used in the study.

Id. No	**Elemental Powders**	**Powder size (µm)**	**Density (g/cm³)**	**Purity (%)**
1	Cr	<45	7.14	99.8
2	316L	<150	7.95	99.9

Table 4.2. Chemical compositions of the samples prepared for the experiment.

Alloys	**C**	**Mn**	**P**	**S**	**Si**	**Cr**	**Ni**	**Mo**
316L (included 16.84Cr)	0.03	0.17	0.019	0.01	0.98	16.84	12.98	2.23
316L (included 17.14Cr)	0.03	0.17	0.019	0.01	0.98	17.14	12.98	2.23
316L (included 17.34Cr)	0.03	0.17	0.019	0.01	0.98	17.34	12.98	2.23
316L (included 17.84Cr)	0.03	0.17	0.019	0.01	0.98	17.84	12.98	2.23
316L (included 18.84Cr)	0.03	0.17	0.019	0.01	0.98	18.84	12.98	2.23

The unidirectional pressing of the powder particles acquired after pretreatment was done in a 100 Ton HIDROSAN press using 750 MPa pressing pressure to create tensile specimens. A mold made in compliance with ASTM (E8M) powder metal material requirements was used to compress the dimensions of the tensile test specimen, resulting in a complete specimen.

The precision balance used in the weighing of the powders, the powder mixing device used in the mixing process and the press used in the compaction process are shown in Figure 4.2. The resulting sample is shown in Figure 4.3.

Figure 4.2. Images of a) Precision balance, b) Hydraulic press machine, and c) Powder mixing device used in experimental studies.

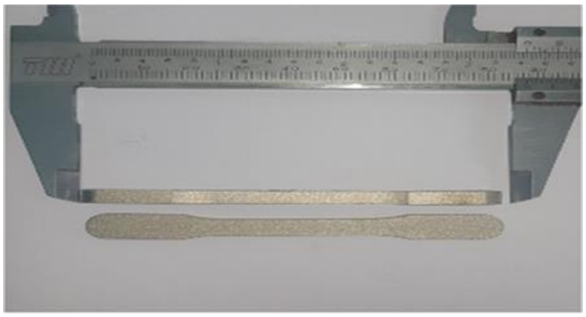


Figure 4.3. Image of cold pressed drawing specimens.

4.1.2. Sintering Processes

The sintering processes of the samples, which were prepared and processed at the desired rate, were carried in argon atmosphere. For the sintering process, Protherm PTF 16/75/610 model atmosphere-controlled tube furnace with a maximum temperature of 1600 °C used in Karabuk University, Faculty of Technology, Manufacturing Engineering, Mechanical Metallurgy laboratory was used. The beginning of the sintering process is 5 °C/min. to the sintering temperature of the samples. Accomplished by rapid heating. Alloy shown in Table 4.1 were kept at 1200°C for one hours. After two hours, return to room temperature at 5 °C/min. rapidly cooled. The atmosphere-controlled furnace is shown in Figure 4.4.

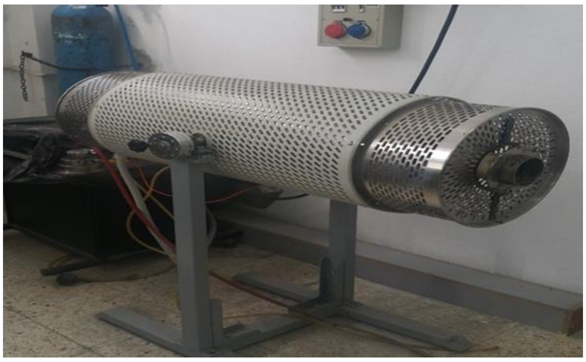


Figure 4.4. Atmosphere-controlled tube furnace.

4.2. TENSILE TEST AND HARDENESS MEASUREMENT

The surfaces of the sintered tensile specimens were grinding with a PRESI MECAPOL P262 brand device and made suitable for hardness measurement. In Vickers hardness tests, HV (0.5) load was applied, and the hardness value was determined by taking the average of 5 hardness measurement results for each sample.

Tensile tests were applied to samples at a tensile speed of 1 mm/min using a SHIMADZU tensile tester machine. Consequently, graphs of stress-strain were plotted following the tests. Tensile strength, yield strength (0.2%), and percent elongation values of the samples were calculated to study the behavior of the material in these graphs. Figure 4.5 showing tensile tester and hardness measurement devices.

Figure 4.5. SHIMADZU tensile test and hardness measurement device [107].

4.3.1. Density Measurement

For density measurements of P/M steel samples, it was determined by using RADWAG AS-60-220 C/2 precision balance with RADWAG AS density measurement kit and Archimets principle using distilled water show in Fig 4.6.

Figure 4.6. Density measurement device.

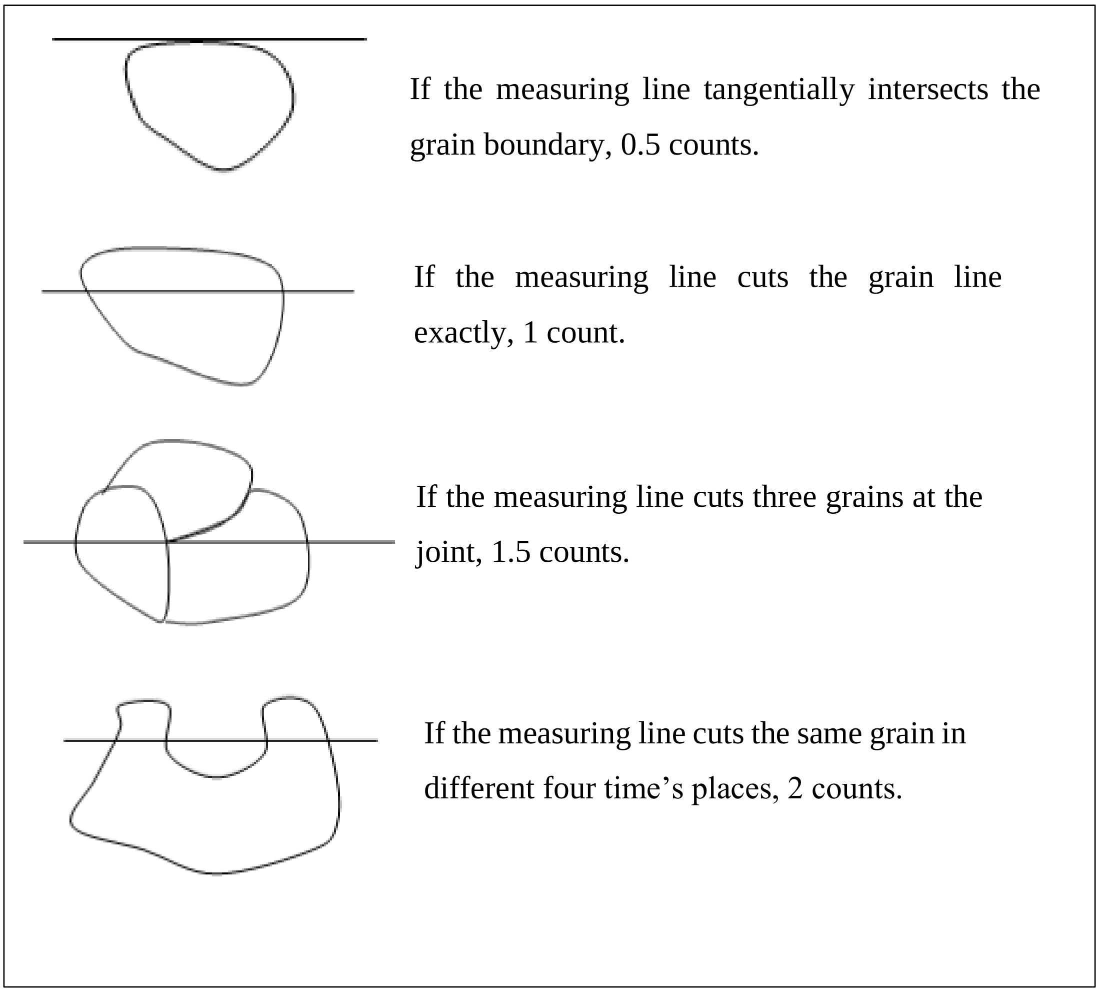
The method in equation 4.1 was used to determine the porosity value.

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

Where: ρ theoretical is theoretical density (g/cm³)
ρ measured is measured density (g/cm³)

4.3.2. Grain Size Measurement

The size of the grains can be measured by the mean linear intersection method. In this method, the measurement line is drawn to form an angle of 45° to the rolling direction, and the grain boundaries that the line cuts are counted. The total length of the measuring line is divided by the number of grain boundaries. The points to be considered in calculating the grain size are given in Figure 4.7.



If the measuring line tangentially intersects the grain boundary, 0.5 counts.

If the measuring line cuts the grain line exactly, 1 count.

If the measuring line cuts three grains at the joint, 1.5 counts.

If the measuring line cuts the same grain in different four time’s places, 2 counts.

Figure 4.7. Points to be considered when calculating grain size [108].

Equation 4.1 given below is used to measure grain size.

$$G = \frac{L}{n}$$

Where:

G= Grain size.
L= Total length of measuring line.
n= indicates the number of grain boundaries cut by the measuring line.

4.4. OPTICAL MICROSCOPE AND SEM EXAMINATIONS

Stainless steel samples produced by powder metallurgy were mounted for microstructural analysis. Then, in the mechanical laboratory of Karabuk University's Faculty of Technology, the surfaces of the prepared samples were grinded using the PRESI MECAPOL P262 device Fig 4.8. These grinded was done underwater using SiC abrasives with grit numbers 240, 320, 400, 600, 800, 1000, 1200, 2500, 5000, and 7000, respectively. The grinded samples were washed with water, then their surfaces were sprayed with alcohol to dry them.

Figure 4.8. Polisher used for grinding.

In the Metallography lab of Karabuk University's Technology Faculty, microstructure analyses were conducted using an optical microscope of the Nikon Epiphot 200 brand with an X500–X1000 magnification capability Fig 4.9.a. Photos were captured at various magnifications from various locations inside each sample, and extra care was made to ensure that these images accurately depict the whole microstructure.

While Microstructural studies were then carried out using optical and field emission scanning electron microscopy (FESEM) by a Zeiss Ultra Plus machine (Carl Zeiss SMT GmbH, Oberkochen, Germany) Fig 4.9.b, equipped with a secondary electron (SE2) and energy dispersive X-ray spectroscopy (EDS) analysis system.

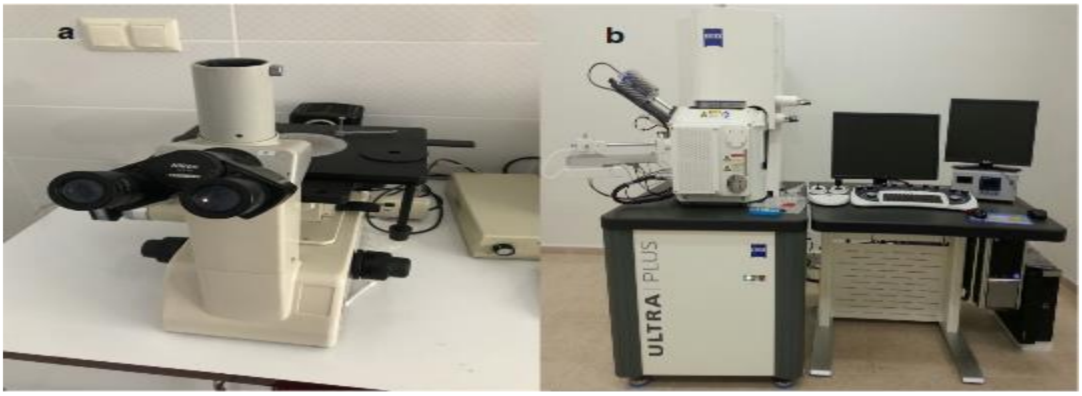


Figure 4.9. Optical microscope and SEM examinations devices.

CHAPTER 5

RESULTS AND DISCUSSION

5.1. MICROSTRUCTURE AND MECHANICAL PROPERTIES

5.1.1. Tensile Tests and Hardness Measurement

The tensile graphs obtained after the tensile test of the samples are shown in Figure 5.1.

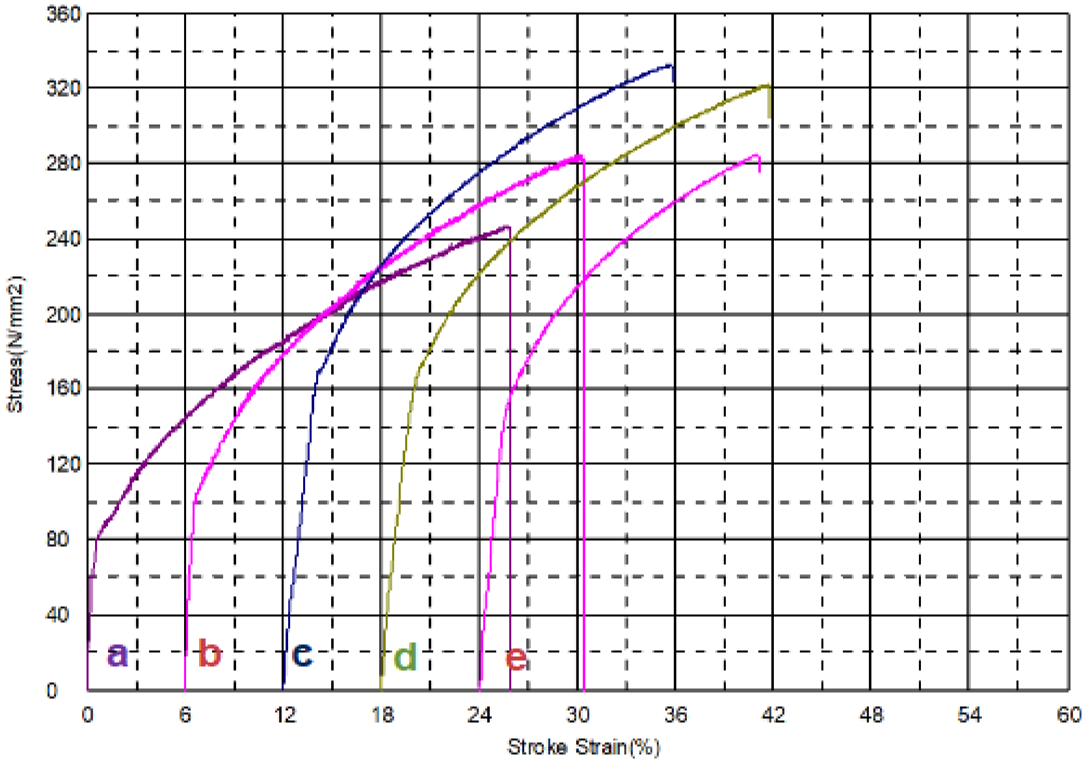


Figure 5.1. Difference of stress–strain curves of the unalloyed and alloyed 316Lstainless steels with Cr a) 316L (included 16.84Cr), b) 316L (included 17.14Cr), c) 316L (included 17.34Cr), d) 316L (included 17.84Cr), e) 316L (included 18.84Cr).

Table 5.1. Mechanical properties of the 316L PM steel

Alloys	**Yield strength (MPa)**	**Ultimate tensile strength (MPa)**	**Elongation (%)**	**Hardness ($Hv_{0.5}$)**
316L (included 16.84Cr)	88	247	25.79	107
316L (included 17.14Cr)	103	284	24.14	114
316L (included 17.34Cr)	169	333	23.71	141
316L (included 17.84Cr)	163	322	23.65	133
316L (included 18.84Cr)	145	286	16.98	119

When 17.14 Cr is added, the elongation of the yield point achieves its maximum value, and when the amount of Cr is increased to 18.84, it begins to shorten. Lower mobility and a longer molding point are caused by the free carbon atoms in the solid solution diffusing into ferrite dislocations because of the Cottrell environment of disturbances surrounding the carbon atoms [109]. The elongation of the yield's point reduced when chrome weight percentage reached 17.84 or 18.84. In addition, elongation ratio, tensile strength, yield strength and hardness values were obtained after this test for all samples are given in Table 5.1.

When comparing 316L alloy and other alloys, although the amounts of carbon are similar, the effect of adding Cr in proportions of 17.14 and 17.34 by weight in other alloys appears clearly with an increase in yield and tensile strength. Precipitation and grain size reduction of alloying elements such as carbide, nitride, and carbonitride are the main reasons for the increase in mechanical properties [110]. As a matter of fact, [111], [112], [113] produced P/M steels by adding alloying elements such as Cr, Mo, and Ni in their studies. When examining the results, it was observed that the yield, elongation, and hardness values of the produced materials with the addition of the alloy element increased. The reason is that precipitates formed from some alloying elements such as MoC (N) and CrC (N) formed during and after sintering prevent the austenite grain size from growing, and that these deposits are affected by the mechanisms of precipitation hardening, dispersion hardening and an increase in strength have been reported.

In addition, the hardness results appear in parallel with the results of the tensile test. The hardness results obtained are 107, 114, 141, 133 and 119, respectively. An increase in the hardness of the third alloy by 32% was determined when 17.34 Cr was added compared to the original alloy.

However, upon addition of 17.84 Cr, a decrease in strength was observed. This is believed to be caused by the heavy accumulation of precipitates such as CrC at the grain boundaries and thus the loss of grain purifying properties [114]. The accumulation of sediment at the grain boundaries caused an increase in the number of pores. Thus, the large sediment formed cannot sufficiently inhibit the grain growth, causing the growth of the average grain size. This situation can be associated with a general decrease in density when 18.84 Cr is added showed in Table 5.2. It can be seen in Table 5.2 that the relative density of PM steel is generally about 89.8%. During the sintering process, the density of PM steel is increased by making sure that the powder particles are connected to each other at a certain temperature and time. As a result, PM steel acquires a more homogeneous structure [115], [116]. But, in 316L steels with chromium added in different percentages, a significant decrease in density was observed at 17.34 Cr is added, and it gradually increased with the increase in the percentage of chromium. Some properties such as strength, ductility, and conductivity are related to density, namely porosity and pore structure. The reason for the decrease in strength in Alloy 4 and Alloy 5 is thought to be the formation of precipitates such as CrC, CrN and CrCN, which occur intensively at the grain boundaries. In addition, the presence of precipitates such as CrC in steel increases precipitation hardening, which makes the material brittle and leads to a decrease in strength.

Table 5.2. % Density, % Porosity and Grain size values of P/M steel samples.

Alloys	**Theoretical density (g/cm³)**	**Measured density (g/cm³)**	**Density (%)**	**Porosity (%)**	**Grain size (µm)**
316L(included 16.84Cr)	7.95	7.3152	92.02	7.98	20.92
316L(included 17.14Cr)	7.9476	7.2948	91.79	8.21	20
316L(included 17.34Cr)	7.9459	7.0478	88.70	11.30	16.79
316L(included 17.84Cr)	7.9419	7.0264	88.47	11.53	17.89
316L(included 18.84Cr)	7.9338	6.9887	88.09	11.91	19.42

When the table is examined, it is seen that the addition of Cr to 17.34 by weight reduces the grain size, but the grain size tends to increase with the addition of these alloying elements. For example, while the average grain size of alloy 316L included 16.84 Cr is 20.92 µm, 316L compositions which is the basic alloy are higher than of alloy 2,3,4, and 5, respectively. One of the prominent features of alloying elements is that they prevent grain growth during austenitization or sintering with the carbides and nitrides they form. The formation of small precipitates during austenitization makes it difficult for austenite grains to grow [117], [118] . The effect of alloying elements in solid solution on the recrystallization of austenite is quite weak. Inhibition of grain boundary movement by precipitated particles is more effective than with dissolved atoms.

On the other side, when 18.84 by weight of Cr was added in the table, an increase in average grain size occurred. This is due to the dense accumulation of precipitates such as large MoC (N) and CrC (N) at the grain boundaries, thus a loss of grain refinement property. In addition, the accumulation of precipitates at the grain boundary causes an increase in the number of pores. As the large-sized precipitates formed in this way cannot completely prevent grain growth, it causes an increase in the average grain size [119], [120].

There are studies in the literature that support this. As a matter of fact showed that the average particle size increased slightly with the increase of Cr value from 17.34 to 18.84 by weight they have observed. The reason for this was expressed as the excessive formation of large CrC precipitates at grain boundaries [121].

5.2. MICROSTRUCTURE RESULTS AND EVALUATION

5.2.1. Optical Microscope Images

Pictures of the microstructures of the sintered samples under argon atmosphere for one hour at 1200 °C are shown in Figure 5.2.

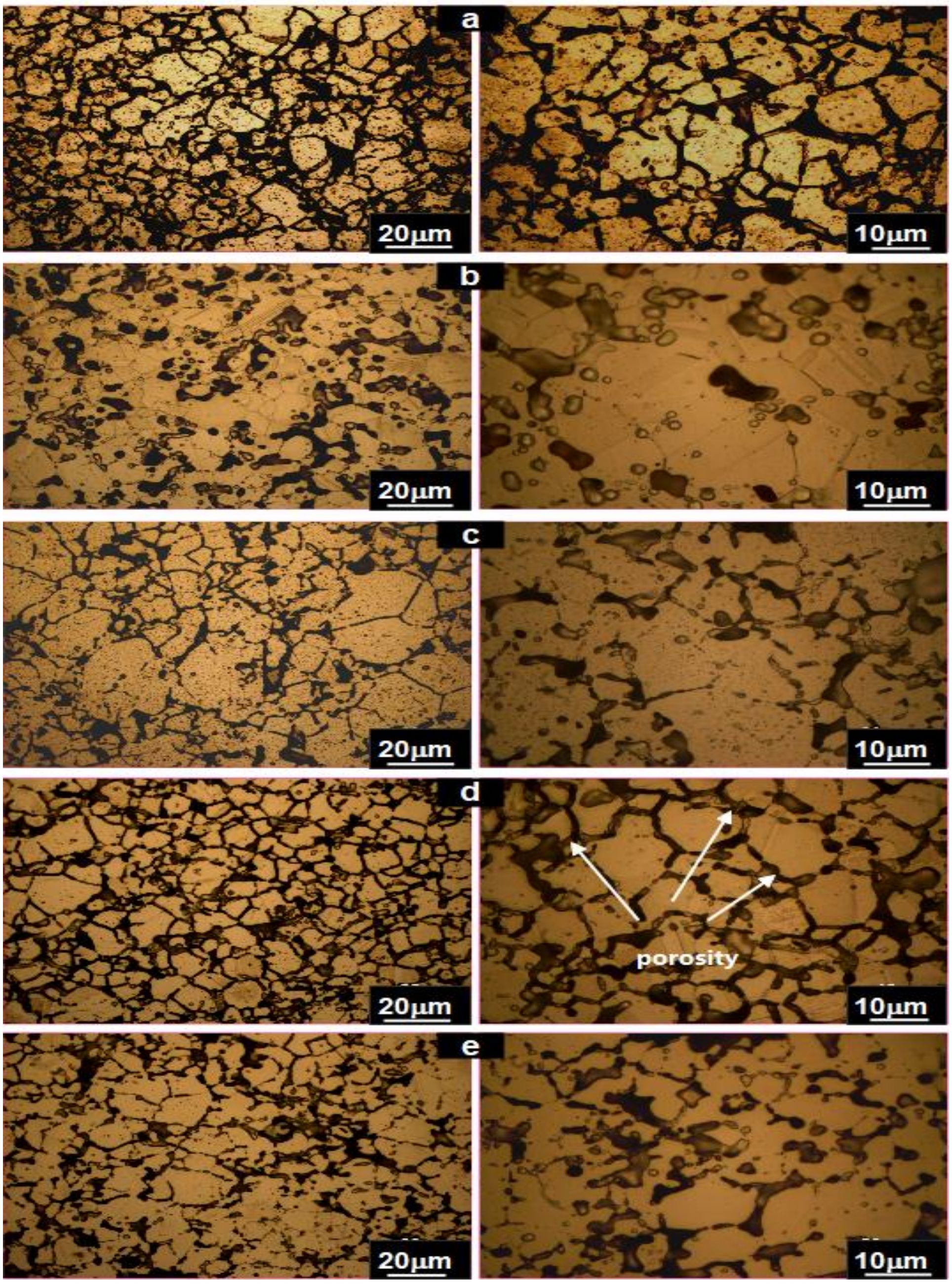


Figure 5.2. Microstructure images of PM 316L stainless steels with different chromium ratios (500X-1000X): a) 316L (included 16.84Cr), b) 316L (included 17.14Cr), c) 316L (included 17.34Cr), d) 316L (included 17.84Cr), and e) 316L (included 18.84Cr).

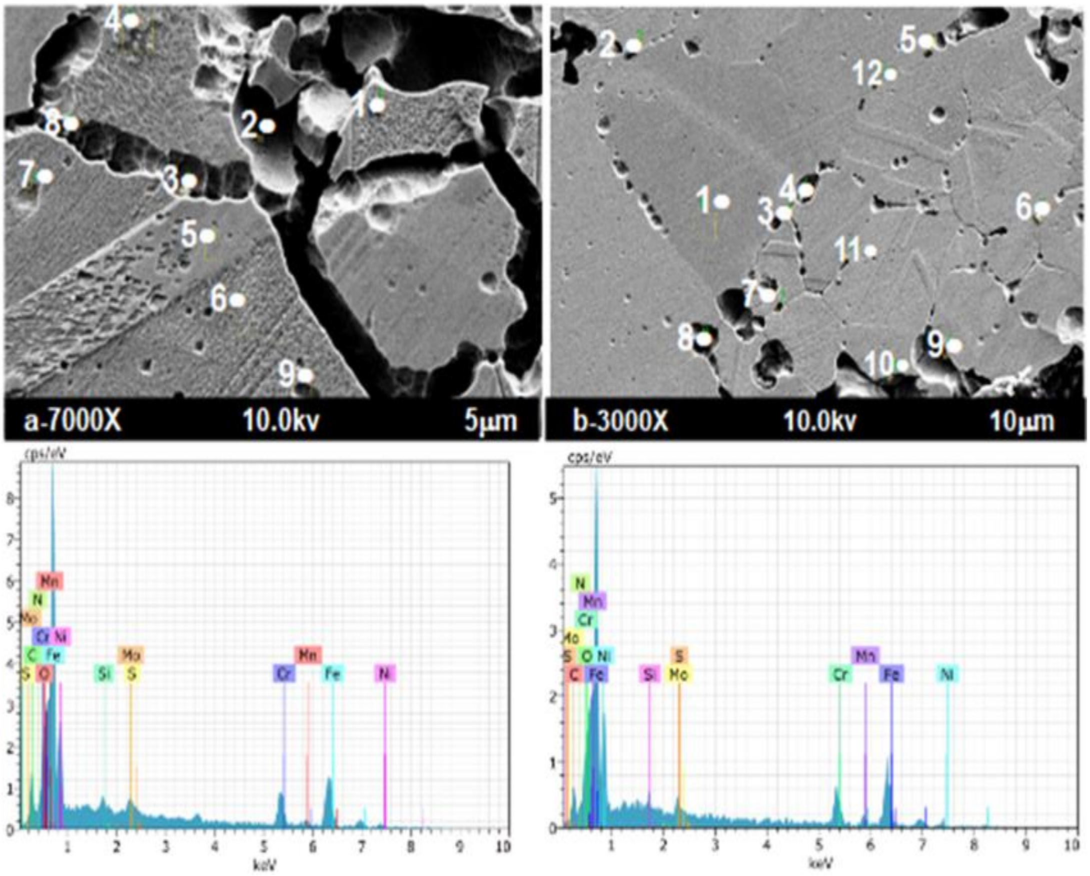
Through the resulting light microscope images, it was noticed that the structure was more accurate with the addition of chromium in different ratios compared to the original alloy 316 L, and the presence of partially closed pores at the borders of the grains and inside the grains.

Although it is reported in most sources that the porous structure negatively affects the strength, it has also been reported that the small spherical size of the pores does not affect the strength [122]

5.2.2. SEM Microstructure and EDS Analysis

The data of SEM point EDS and line EDS analysis of 316L steel samples produced by P/M method, sintered at 1200 ºC with 17.84 Cr added and 18.84 Cr added, are shown in Figure 5.3.

The SEM picture of the 316L included 17.84 Cr steel sample shown in Fig. 5.3 revealed that precipitates such as nitrides, carbides or carbides were found. Additionally, it is known from the EDS point analysis data that the iron and carbon content of the Fe_3C deposits had a role in their formation. Through SEM and EDS analyses, it was discovered that the presence of chromium elements in the steel caused CrC(N) precipitates to created when the 316L stainless steel with various relative weights of Cr was analyzed. In addition to improving the material's strength by precipitation hardening, it has been observed that these deposits restrict the formation of austenite grains [123]



Spectrum	C	O	Cr	Mn	Fe	Ni	Mo
1	6.43	2.23	13.92	1.67	54.63	16.47	2.53
2	6.04	1.06	15.29	1.11	53.17	11.96	2.45
3	31.05	9.78	10.42	1.44	33.81	9.80	1.57
4	13.27	4.45	13.22	0.52	46.16	11.16	2.42
5	3.39	0.81	15.96	1.04	55.29	18.96	2.22
6	4.84	0.96	16.88	1.12	53.28	19.12	1.98
7	7.10	1.27	13.82	1.58	53.54	18.41	1.89
8	11.13	1.56	16.54	1.85	57.65	9.34	1.35
9	14.53	2.85	13.39	0.98	50.36	12.85	1.80

Spe	C	N	O	Cr	Mn	Fe	Ni	Mo
1	4.38	2.18	1.30	14.86	3.60	52.39	18.27	2.08
2	13.64	3.84	48.56	8.40	0.98	12.78	3.59	0.58
3	4.58	1.49	7.98	15.41	0.38	55.93	10.73	1.62
4	9.55	2.97	25.42	10.90	1.05	31.48	9.60	0.65
5	5.12	1.70	6.34	15.72	1.60	46.65	16.19	1.51
6	4.90	1.43	9.41	16.53	0.00	42.78	17.23	1.48
7	4.88	1.92	2.05	13.51	2.90	52.74	18.34	1.48
8	11.58	2.50	40.07	7.37	1.52	18.49	4.96	0.31
9	4.45	1.43	2.40	17.56	1.82	52.71	17.78	1.18
10	3.60	0.00	4.00	25.17	3.39	58.43	4.72	0.36
11	3.91	1.31	7.66	14.85	1.45	48.11	15.71	2.21
12	5.05	2.11	1.55	16.42	1.14	52.17	17.90	2.33

Figure 5.3. SEM point and EDS results from a-316L (included 17.84Cr) alloy, b-316L (included 18.84Cr) alloy.

The results of the SEM microstructure and EDS analysis of the annealing samples produced by the PM method are shown in Fig. 5.4. The presence of C, Fe, Mo, Cr and N in the steel and the formation of nitrides, carbides, or carbide precipitates such as CrC(N), MoC(N), CrMoC(N) and Fe_3C were determined because of SEM microstructure and EDS analysis. These deposits have been shown to limit the growth of austenite grains while also increasing the strength of the material through precipitation hardening [124].

The effect of the alloying elements in the solution on the recrystallization of austenite is relatively weak. The influence of precipitated particles on the movement of grain boundaries is much greater than that of solute atoms [125]. In the results of EDS analysis and SEM microstructure, it was determined that the C, Mo, and N elements were in composite form and in the form of precipitated particles, as shown in Fig. 5.4 and Fig.5.5. For example, SEM and EDS analysis taken from the microstructure support this. As indicated by the EDS line taken from steel containing 17.84 Cr by total weight, the base material phase was found to be rich in iron, but the round-shaped precipitate was rich in nickel and chromium. In addition, a sharp increase in the amount of carbon and chromium was observed when the analysis line cut from the matrix through the precipitate. When it crosses the precipitate analysis line, there is an increase in iron and a decrease in chromium and molybdenum. When the EDS line result is evaluated, this precipitate is considered as MoCrC(N) precipitate. When EDS line analysis results obtained in this study are compared with previous literature studies, they show that precipitates such as MoC(N), CrC(N) and MoCrC(N) occur in PM steel alloys. For example, the reported that the addition of Cr increases the ultimate tensile strength and yield strength of PM steels due to the formation of CrC(N) and MoC precipitates [126]. On the other side, it was reported that the effect of the alloying elements in the solution on the recrystallization of austenite is very weak. The influence of precipitated particles on grain boundary motion is much greater than that of solute atoms [125]

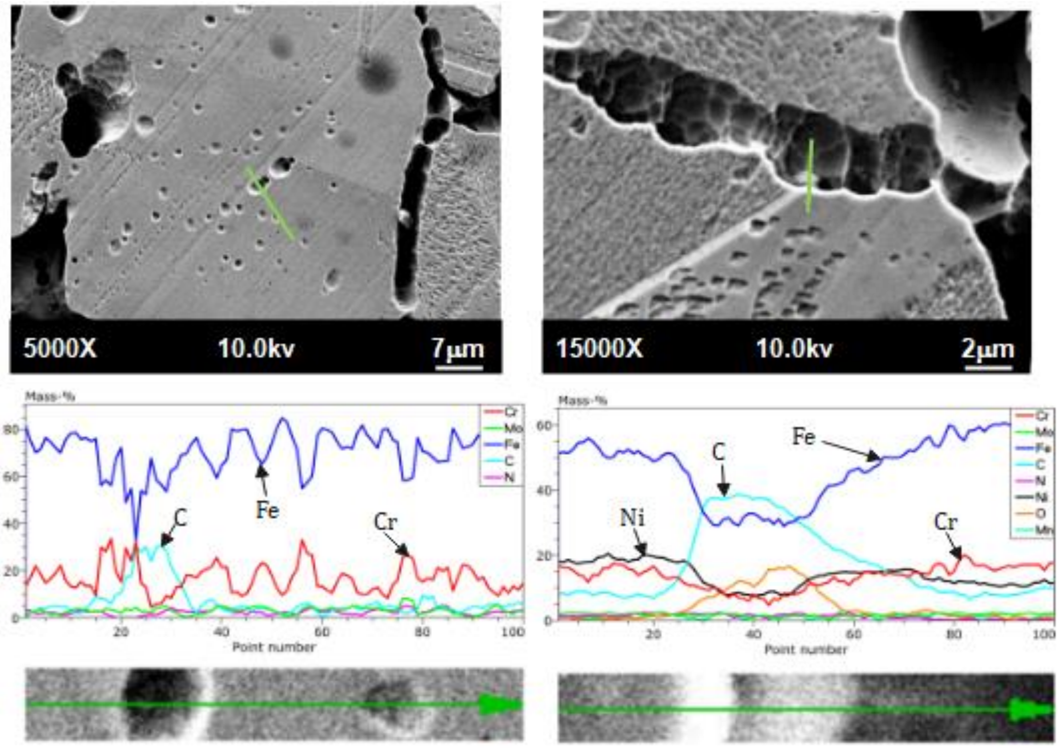


Figure 5.4. SEM line and EDS results of 316L (included 17.84Cr) product by powder metallurgy methods.

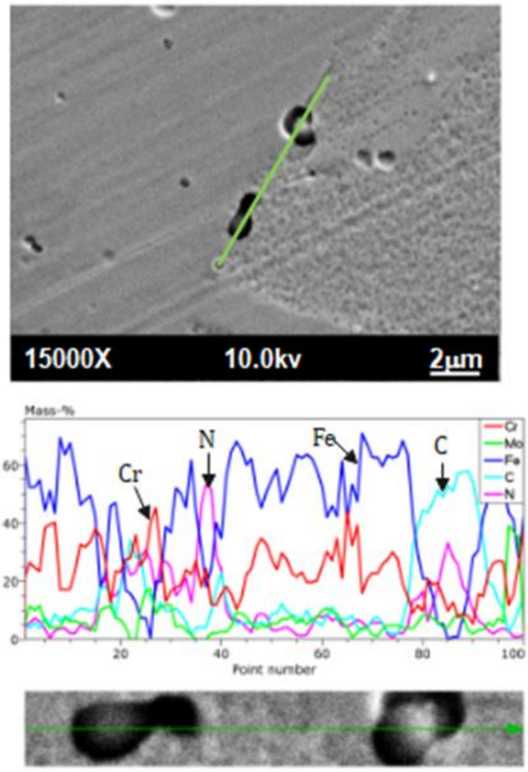


Figure 5.5. SEM line and EDS results of 316L (included 18.84Cr) product by powder metallurgy methods.

On examination of Fig. 5.8, it was observed that the fractured surfaces showed partially brittle behavior (cleavage planes) and partially ductile behavior (honeycomb structure). Moreover, it was evident that all the cracked surfaces had pores. This indicates that the fracture occurs by fusion and propagation of small pores. However, cleavage levels, which are an indicator of brittle fracture, are seen to be highest in 316L stainless steel containing 18.84 Cr by weight, moderate in chromium steels containing 17.34–17.84 Cr, and lowest in chromium steels with 16.84 Cr . The samples also have large gaps. The appearance of these gaps indicates that during the tensile test, precipitates such as MoC(N), and CrC(N) were separated from the surface. Chandramouli and Shanmugasundaram bonded separating nitrides, carbides, and carbides from the surface during the tensile test [127]. To summarize, it was observed that when Cr was increased to 17.34 by weight. The tensile strength, yield strength and hardness increased, and then chromium increased from 17.84 – 18.84 by weight tensile strength, yield strength and hardness decreased, and the elongation values decreased with the increase of chromium content, but the amount of chromium added by 18.84 wt. Fig 5.6, Fig 5.7. It has high mechanical properties compared to the basic 316L alloy except for the elongation, and the values obtained due to the tensile test are consistent with the images of the fracture surface.

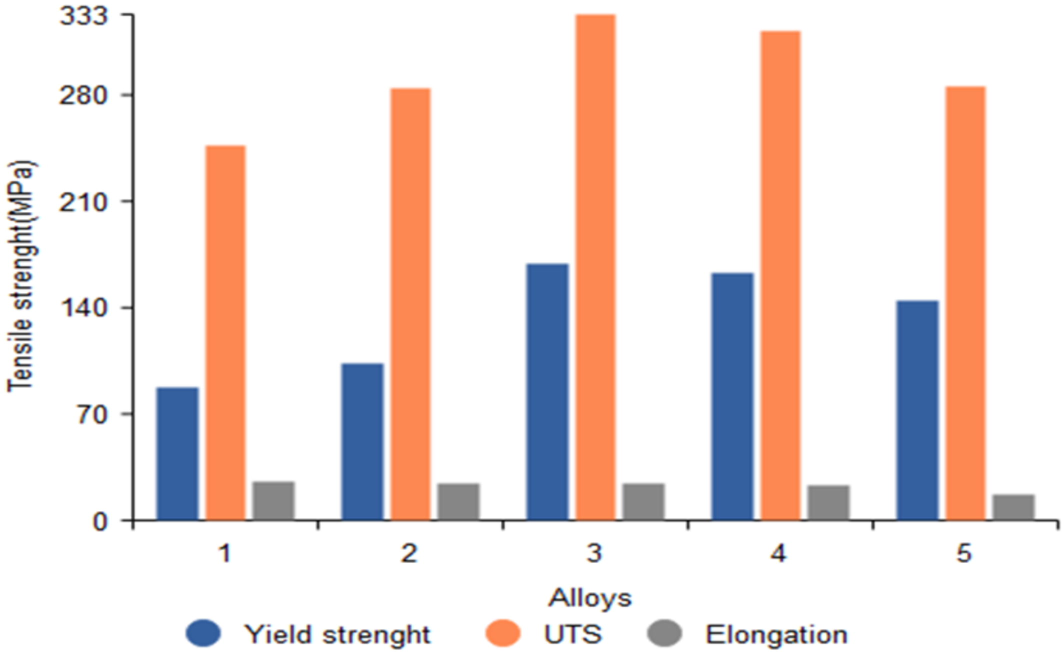


Figure 5.6. Tensile tests chart for 316L steel with Cr.

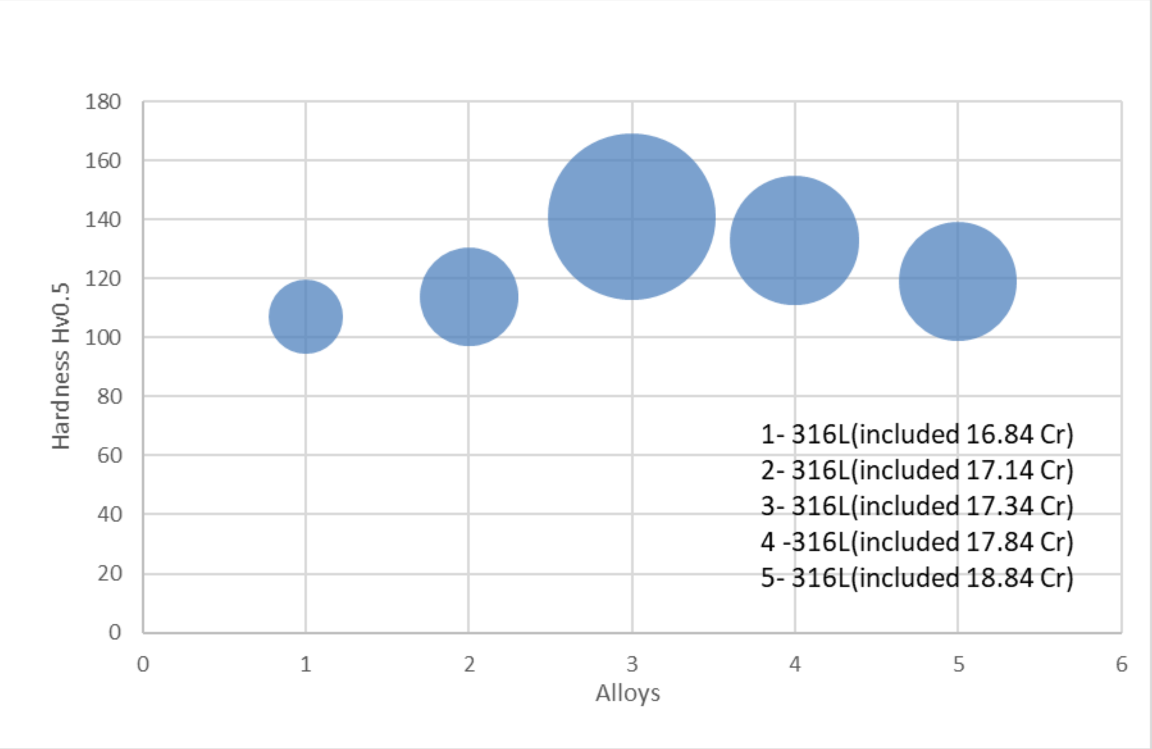


Figure 5.7. Hardness measurment chart for 316L steel with Cr.

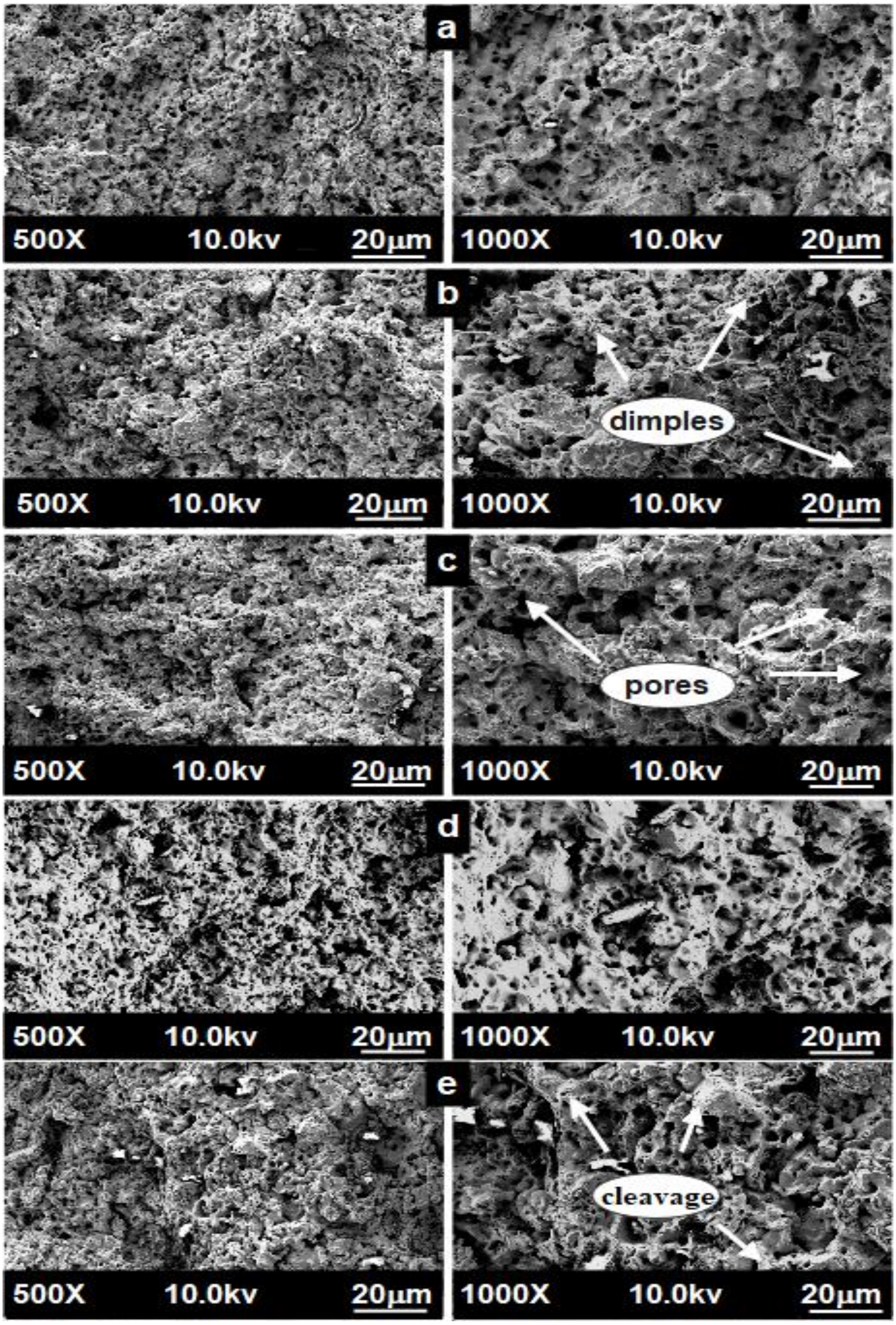


Figure 5.8. Fractured surface image of samples sintered at 1200ºC at (500X-1000X) a) 316L (included 16.84Cr), b) 316L (included 17.14Cr), c) 316L (included 17.34Cr), d) 316L (included 17.84Cr), and e) 316L (included 18.84Cr).

PART 6

CONCLUSION

In this study, 316L P / M steel contains different proportions of Cr by powder metallurgy by pressing cold after mixing metal powders, followed by sintering at 1200 °C under the atmosphere of argon. The mechanical properties were distinguished in terms of tensile, yield strength, the values of elongation and the values of hardness, considering the images of microscopic structure, the results obtained from this study are as follows:

1. The 316L steel, which is added to different proportions of Cr, showed smaller grains in all alloys compared to the basic 316L steel. This is since carbides and nitrides formed by alloy elements prevent grain growth.
2. 316L steel to which Cr is added can be produced by the powder metallurgy. Solid solution hardening, precipitation hardening and grain size reduction during sintering or during cooling after sintering increase the strength of the steel.
3. EDS analyzes of PM steel are revealed that the Mn, C, Si, Cr and Mo and the sediments that were formed by these elements such as MoC (N), CrC (N) and CrMoC (N) are found in a steel matrix compound.
4. It was observed that 316L P/M alloy steel mixed with 17.34 Cr by weight is superior in yield strength, tensile strength and hardness compared to 316L P/M steel to which Cr is added in different proportions. This is because alloying elements have strong carbide-forming properties. It was observed that precipitates such as CrCN, MoCN, and MoCrCN accumulate significantly at the pore and grain boundaries.
5. There is a decrease in the yield strength, tensile strength, and hardness values when the Cr ratio increases from 17.34 to 18.84 in terms of weight. Generally,

compared to basic 316L steel, an increase in the strength values of all the Cr added P/M steels were observed.

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RESUME

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