EFFECT OF AlC ADDITION ON WEAR PROPERTIES OF UNALLOYED STEEL PRODUCED BY POWDER METALLURGY

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Salem Khalıfa Rhoma KHALIFA

ABSTRACT

M. Sc. Thesis

EFFECT OF AlC ADDITION ON WEAR PROPERTIES OF UNALLOYED STEEL PRODUCED BY POWDER METALLURGY

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Basically, the iron carbon alloy is called steel. When we group them according to their chemical composition, it is possible to divide them into two parts as "Non-Alloy Steels" and "Alloy Steels". Unalloyed steels consist of iron and carbon. In addition to alloy steel, iron and carbon, there are alloying elements such as nickel, molybdenum, vanadium, niobium, chromium, manganese, silicon, tungsten.

In this study, the wear behavior of Aluminum Carbide (AlC) added steel produced by Powder Metallurgy (TM) method was investigated. The chemical composition of the steel produced includes 4 different AlC ratios: 0.45% C and 0.2% - 0.5% - 1.0% - 2.0%. The hardness values of the samples were measured and the microstructure was examined. The wear test was carried out in a dry environment under loads of 30 and 60 N, with a slip of 25 meters and a stroke of 3 mm. After the wear test, the wear area loss was measured with the surface profilometer device. Wear surfaces were characterized by SEM and EDS analysis. As a result of the hardness test, the highest hardness value was seen in the steel sample with 2.0% AlC addition. In the same sample, the lowest wear volume was determined after the wear test.

Keywords **:** Powder metallurgy, aluminum carbide, hardness, wear, microstructure.

Science Code **:** 91421

ÖZET

Yüksek Lisans Tezi

TOZ METALÜRJİSİ ILE ÜRETİLEN ALAŞIMSIZ ÇELİĞE AlC İLAVESİNİN AŞINMA ÖZELLİKLERİNE ETKİSİ

Salem Khalıfa Rhoma KHALIFA

Karabük Üniversitesi
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Temel anlamda demir karbon alaşımına çelik denir. Kimyasal bileşimlerine göre guruplandırdığımızda "Alaşımsız Çelikler" ve "Alaşımlı Çelikler" olmak üzere iki kısıma ayırmamız mümkündür. Alaşımsız çelikler demir ve karbondan ibarettir. Alaşımlı çelik, demir ve karbonun yanı sıra nikel, molibden, vanadyum, niobyum, krom, mangan, silisyum, tungsten gibi alaşım elementleri bulunur.

Bu çalışmada, Toz Metalurjisi (TM) yöntemi ile üretilmiş Alüminyum Karbür (AlC) katkılı çeliğin aşınma davranışı incelenmiştir. Üretilen çeliğin kimyasal kompozisyonu % 0,45 C ve % 0,2 - % 0,5 - % 1,0 - % 2,0 olmak üzere 4 farklı oranda AlC içermektedir. Numunelerin sertlik değerleri ölçülmüş ve mikroyapısı incelenmiştir. Aşınma testi kuru ortamda 30 ve 60 N yük altında, 25 metre kayma ve 3 mm strok mesafesinde yapılmıştır. Aşınma testi sonrası yüzey profilometre cihazı ile aşınma alan kaybı ölçülmüştür. SEM ve EDS analizi ile aşınma yüzeyleri karakterize edilmiştir. Yapılan sertlik testi sonucunda en yüksek sertlik değeri % 2,0 AlC ilaveli çelik numunede görülmüştür. Yine aynı numunede aşınma testi sonrasında en düşük aşınma hacim miktarı tespit edilmiştir.

Anahtar Kelimeler : Toz metalurjisi, alüminyum karbür, sertlik, aşınma, mikroyapı.
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SYMBOLS AND ABBREVITIONS INDEX

SYMBOLS

min	: minute
mm	: millimeter
µm	: micrometer
MPa	: mega pascal
s	: second
T	: temperature
σy	: yield stress

ABBREVITIONS

AISI	: American Iron and Steel Institute
ASTM	: American Society for Testing and Materials
DIN	: Deutsches Institut für Normung
HV	: Vickers Hardness
SEM	: Scanning Electron Microscope
LSH	: Laser Surface Hardening

PART 1

INTRODUCTION

Powder metallurgy method is the process of mixing fine-grained metal powders, pressing them in the desired geometric form and sintering them at a temperature below the melting point. The pressing process is also called compacting. In the sintering performed after this process, strong bonds are formed as a result of partial melting between the metal powders with the effect of temperature and high pressure. The strength of the produced part is provided in this way.

Steels produced by powder metallurgical method have excellent surface quality. In addition, they are materials that are resistant to wear and can maintain their hardness at high temperatures, thanks to the alloying elements that form high hardness carbides such as W, Mo, V, Cr.

Compared to the cold work tool steels and high-speed steels produced by the conventional method, the properties such as hardness, abrasion resistance, high temperature resistance, surface quality of powder metal materials are higher even though they have similar chemical compositions.

In the powder metallurgy method, the desired properties are produced in a more comprehensive and controlled manner compared to the casting method. Based on this reason, our study is planned to be realized by powder metallurgy method. In our study, Graphite and Aluminum Carbide (AlC) were added to the Fe matrix. AlC was added at different determined proportions. The mixtures were mixed for one and a half hours using the Turbulo device. In order to bring these mixed powders into block form, 700 MPa was applied with one-way pressing. Block samples were sintered in an argon atmosphere at 1350°C. SEM and optical microscope device were used for microstructural properties and post-abrasion examinations. (SEM microstructure, SEM EDS). Hardness and wear tests were carried out to determine the mechanical properties.

When the production of the aforementioned steels is made by powder metallurgy method, good surface quality of the part, better quality and more precise production, production of some metals that are difficult to produce and process with other production methods will emerge. There will be a significant reduction in the production cost. It will facilitate the production of alloy steel materials in our domestic industry. However, it will become widespread. The main purpose of this study and research is to contribute to our country to have a say in production and export by providing domestic and national mass production of high-quality steels.

PART 2

STEELS

2.1. STEEL DEFINITION AND CLASSIFICATION

Iron-carbon alloys according to the carbon ratio they contain; It is examined in two main groups as “Steels” and “Cast Irons”. According to this category; Steel to alloys containing less than 2% carbon and alloys containing more than 2% carbon; It's called cast iron. There are different distinctions in the classification of steels. These; usage areas, chemical composition, applied heat treatment, forming method, microstructure and production method. It is of great value to take samples of the alloying elements they contain in the classification process of steels, since they can acquire various microstructures and properties depending on the thermal and mechanical processes they undergo until they reach the final product form. When we classify the steels according to their chemical composition, they are divided into two as “Non-Alloy Steels” and “Alloy Steels”.

The chemical property of the steel is suitable for welding processes. It is lighter and harder than iron. When heat treatment is applied, mechanical, electrical and physical properties can be gained. With different processes, its hardness and strength can increase at high temperatures. It can be brought into the desired shape by pressing, forging and rolling methods applied within a certain temperature. Steels with some properties have the ability to be cold formed. Metals can be coated with plastics.

When a hot steel is quenched suddenly and rapidly, its crystalline properties change and it hardens. To this process; It is called “quenching steel”. Stainless steels are heat and corrosion resistant, fully recyclable. It is easy to clean and manufacture. Alloy steels containing high carbon are referred to as "cast iron". As the carbon ratio increases, the tensile and yield strength of the steel increases, while reducing its formability and welding capabilities.

An alloy of carbon and iron is called steel, the word alloy steel is used to express that alloying elements other than carbon are present in the steel composition. Essentially, the carbon element, like other alloying elements, acts as an alloying element in the structure of the steel. Based on the traditional separation, steels are divided into two main sections as unalloyed steels and alloyed steels in the general separation.

2.1.1. Non-Alloy Steels

It is a type of steel that, unlike iron, contains only carbon by determining the basic alloying element, but can also contain elements at the level of 0.8% manganese, 0.1% titanium, 0.1% aluminum, 0.5% silicon and 0.25% copper in its composition. In another nomenclature, it is referred to as plain carbon steel.

Carbon is the most valuable alloying element in unalloyed steels. Therefore, according to the carbon ratio they contain; They are categorized as “Low Carbon Steels”, “Medium Carbon Steels” and “High Carbon Steels”.

At high temperatures, the material softens and its yield limits decrease, and it is exposed to plastic deformation at low stresses. Permanent deformations that occur slowly in long-term stresses cause the material to break after a certain period of time (Tükel, 1979).

2.1.2. Alloy Steels

While iron and carbon alloys contain only iron and carbon, alloy steels contain other alloying elements such as chromium, manganese, silicon, nickel, molybdenum in addition to iron and carbon. In addition, alloyed steels are classified as low alloyed and high alloyed steels according to the ratio of alloying elements. Low-alloyed steels containing less than 5% alloying elements are generally used in the production of machine parts and high-strength structural elements.

One of the changes of alloying elements is the transformation temperatures in the iron-carbon thermal equilibrium diagram. Since almost all of the elements are austenite at room temperature, it affects face-centered cubic crystals. Elements that dissolve in each other form the face-centered lattice structure. Thus, the alloying elements transform from the gamma phase to the ferrite phase in the opposite direction. Alloying elements will fix the austenite and expand the available temperature range.

High alloy steels; steels with a total of alloying elements greater than 5%. If unalloyed and low-alloyed steels do not contain the desired properties or are insufficient, high-alloyed steels are preferred. Stainless steels and tool steels can be given as examples (Tekin, 1986).

Low alloy steels have basically similar behavior to unalloyed steels and the most important feature is their superior hardenability. The deficiency in the use of unalloyed steels and the situations that cause low alloy steels to be preferred are as follows. The impact resistance of unalloyed steels is very low at low temperature values, the depth of hardness formed by quenching, and the oxidation and corrosion resistance of unalloyed steels are low (Tekin, 1986).

Based on these disadvantages of unalloyed steels, steels that are produced and contain nickel, manganese, molybdenum, chromium and tungsten as basic alloying elements in the mixture are low alloy steels. In addition to these steels, alloying elements such as aluminum, vanadium, boron, niobium, titanium, lead, copper and cobalt can be added.

2.2. DEVELOPMENT OF STEELS

Steel is an alloy that has been used in the oldest periods of human history and continues today. Although it was known in ancient times, the use of steel was limited to weapons and similar war materials, since a comprehensive production and production level was not applied. In the following years, with the development of technology, when the exact time was determined, raw iron production and production started in England in the 18th century. After this stage and history, steel and iron structures have started to be made in the world, albeit a little. We can say that the first steel structures were bridges.

One of the most important structures built using steel and iron is the Eiffel Tower. The tower, which was finalized in 1889, is sufficient to present the use of iron and steel as an example over the years.

It is of great importance among the categories of materials needed and used in engineering applications. In particular, it can be said that the time after the second world war passed as the beginning of speed. The need for steel in many sectors is increasing day by day. With the increase in strength and weight ratio, as a result of the development of lighter and thinner steels, the unit cost in operating and production expenses has been reduced (Erden, 2017).

2.3. STRENGTH INCREASING MECHANISMS OF STEEL

The mechanical properties of materials are highly dependent on the behavior of their metallurgical structures. Since the metallurgical structure changes with the thermal and mechanical processes applied to the material together with the chemical composition, it can be said that the mechanical properties of the material also depend on these conditions. One of the most important material properties is strength. Other properties vary depending on strength. resistance in materials science; can be explained by the resistance of the material to plastic deformation. Plastic deformation of metals is formed by the progression of linear defects, which we call dislocation as the essence of the narrative, in the crystal. Therefore, mechanical properties such as hardness, strength, ductility; explains the density of dislocations in the internal structure of metals and their interaction with other defects. Strength-enhancing processes can be listed as follows:

- Deformation Aging
- Hardening by Reducing Grain Size
- Cold Working
- Hardening by Martensitic Transformation

- Precipitation Hardening (Aging)
- Alloy Hardening
- Hardening by Dispersion

It is necessary to know the mechanical properties-microstructure relations in detail in order to improve some of the desired properties in microalloyed steels and to make the most of the improved steels. Hardening mechanisms such as grain size hardening, solid melt hardening, precipitation hardening, strain hardening and hardening mechanisms used in microalloy steels increase the strength of the steels. While the grain reduction mechanism increases the strength in these hardening mechanisms, it also improves the toughness (Kim, 1983).

2.4. ADVANTAGES AND DISADVANTAGES OF ALLOY STEELS

Compared to plain carbon steels, alloy steels have superior hardness, strength, wear resistance, toughness, hardenability, hot hardness. In order to gain these advantages, heat treatment may be required. It has high strength. The ratio of its own weight to the load it carries is very small; therefore, the overall weight of the structures decreases.

Strong carbide-forming elements such as V, Ti and Nb make carbide. Alloying elements also affect the eutectoid temperature. Mn and Ni reduce the eutectoid temperature. That's why they are known as austenite builders. Carbide-forming elements raise the eutectoid temperature and are known as ferrite-formers. It is more costly than unalloyed steels.

2.5. USAGE AREAS OF ALLOY STEELS

It is a valuable alloy that contributes to a large part of our lives, from the construction sector to the healthcare field, from the materials used to the technological materials.

Stainless steel, which does not cause chemical changes in the human body and the foods it uses; It also includes materials used in the health sector such as hips and knee caps, screws, prostheses, needles and scalpels. Stainless steel, which does not spoil the color and smell of the food; Plates, oven molds, coated pots are safely preferred in storage containers produced for food and beverages.

2.6. ALLOYING ELEMENTS AND SOME ADDITIVES

Alloying elements play an important role in performing thermomechanical processes. While Mo, Cr and Mn elements act especially thanks to their hardening, there are different mechanisms in Nb, V and Ti microalloying elements. The toughness and strength increase here is extended by hardening and grain refinement methods. But grain refinement; It contributes to the increase of toughness and strength at the same time (Taş, 2012). The alloy that contains 0.2%-2.1% carbon (C) in its composition and is formed by the effect of the carbon-iron (Fe) mixture is called steel. Some of the other elements that make up this alloy are; vanadium (V), tungsten (W), magnesium (Mg), chromium (Cr), cobalt (Co), molybdenum (Mo), manganese (Mn), nickel (Ni). When these elements are included in the material, the steel turns into stainless form or becomes harder.

2.6.1. Carbon and Graphite

Carbon, which is in the sixth row of the periodic table, is an element that occurs in different formations in nature. Its atomic number is 6. Its crystal structure is hexagonal or cubic, black or gray in color. The branch of science that studies carbon and its components is called organic chemistry (Pierson, 1993).

The high carbon ratio causes an increase in the perlite structures and a decrease in weldability and toughness. However, it increases the yield strength. In addition, the use of carbon at high rates causes bainitic and martensite structures to be prominent. The maximum use of carbon in microalloyed steels is 0.2% under hot rolling conditions. However, since the forged parts, which are frequently used in the automotive industry, are produced only with the controlled cooling mechanism method, the carbon ratio is above 0.25% (Karabulut, 2004).

When the etymology of graphite is examined, it is understood that it comes from the name "Graphein", which means writing in Greek. The usage areas of graphite material cover a wide period of time. The first pencil production from graphite material was carried out in England in the 15th century. By the 18th century, it was found as a result of research that carbon is an allotrope.

When examined in the thermodynamic field, it was seen that graphite has a more stable structure than carbon at atmospheric pressure. Above 1500 °C, diamond turns into graphite (Mcenaney and Timothy, 1999).

Graphite, which is known to be one of the softest materials, is a different formation of carbon and is especially used for lubrication. Graphite is found in nature in a natural form. However, they are obtained by processing petroleum coke (petroleum coke) in oxygen-free furnaces in order to be faster and more economical when used for commercial purposes. Graphite is found in nature in Beta and Alpha form. Beta and Alpha have the same physical features. But they have different crystal structures. Artificially manufactured graphite is known as Alpha type graphite. Besides its use as a lubricant, most of its use is in steel production. Carbon has two allotropes in its atomic arrangement; graphite and diamond (Mcenaney and Timothy, 1999).

Graphite is a material with good thermal conductivity and electrical properties. It also has the ability to maintain its form at high temperatures. It shows its refractory properties well at high temperature values such as 3650°C. Graphite is a functional material that has been used in many applications that have benefited humanity since its discovery.

Although synthetic applications of graphite are still of great interest today, natural graphite is preferred in some applications. Natural graphite is divided into three types. It is crystalline (vein), amorphous and granular (flake). These names were chosen because they give information about their forms (Pierson, 1993).

When the production process of graphite was evaluated intensively, it is observed that it started with the interest in carbon production technologies in the 19th century.

Synthetic graphite, which is created for use in industrial furnaces in the form of electrical resistance material, reveals rapid developments in the next stages. Graphitization processes of carbon materials are obtained by applying heat treatment method at 2600-3300°C in the production of special form graphites. In the graphitization process, the carbon clusters turn into three-dimensional graphite. Graphite materials with different crystalline structures are obtained based on additives, raw materials and processing parameters (Speight, 2015).

The distances between dislocations and the crystal structure of graphite change with temperature. The distance between these layers decreases as the heat treatment temperature rises. The alloying element of the iron element is generally Carbon. Sometimes, different elements such as Chromium, Magnesium, Tungsten and Magnesium can be used to alloy the iron element. It examines the varying amounts of alloying elements in the steel, as well as the existing forms (dissolved elements, precipitation phase) in the formed steel, such as ductility, hardness and stress point. Steels with high carbon content are stronger and harder than iron, but less ductile.

2.6.2. Nickel

Nickel widens the austenite area, is austenite stabilizer, and narrows the ferrite zone in iron chromium carbon alloys. Increases resistance against oxidation and corrosion at high temperatures. Nickel, which has a grain reduction effect, increases the strength and toughness of the material. In addition, it contains the feature of preventing the scale area that will form on the surface of the material. As a result of its use with chromium, it increases the ductility, hardness and high fatigue resistance of the alloy. It also reduces the critical cooldown rate. In some studies in the literature, an increase in the amount of perlite was observed by adding Ni.

For example, in their research by Frederick and Kalathur, it was stated that with the increase of Ni ratio in Fe-C-Ni alloys, the amount of carbon in the perlite decreased, while the amount of perlite in the microstructure increased and the amount of ferrite decreased. As a result, it was observed that the ductility of the material increased while the strength of the material decreased (Kalathur and Frederick., 2007).

It maintains its strength at high temperature values, and its toughness and ductility at temperatures below zero. It can be easily processed both hot and cold. Therefore, it is easy to machine and weld. When examined chemically, it is non-reactive, insoluble in hot or cold ammonia and water. Also, it is unaffected by alkalis and concentrated nitric acid. It dissolves in hydrochloric acid, diluted nitric acid and sulfuric acid (Aşkun and Hasırcı, 2007).

Nickel, which is an element that provides convenience in important areas of use in human life, has been valuable for over 200 years in modern technology in the materials it contains. Nickel and its alloys are used in many sectors and fields. Stainless steels are used in many products such as rechargeable batteries, coins, special alloys, magnets, valuables, electric guitar strings, surgical cables. Nickel in all these areas; It has intensive use due to its strength, high ductility, electrical and thermal conductivity and corrosion resistance properties.

High corrosion resistance, which is one of the important features of nickel, is the main feature that makes it have a wide usage area. Therefore, stainless steels containing 7% Nickel are used in the oil, gas, power industry, marine applications and the chemical industry. Kitchen materials, which are frequently used in homes, are one of the widest uses of stainless steel and indirectly nickel. In addition, electrolytic coating of nickel is also an important area of use. Nickel is a metal that has gained its character in the historical literature. Although one of the Swedish scientists, Cronstedt, discovered nickel in 1751, it is known that nickel alloys have been used for much longer (Rosenberg, 1968).

2.6.3. Molybdenum

It is an element with molybdenum carbide and ferrite-forming properties. It reaches into the solid phase at high temperature, and precipitates in different forms depending on the molybdenum and carbon content on slow cooling. It has a strength-enhancing feature in stainless steels (Çamlıdere, 1999).

When molybdenum, which is a carbide and nitride forming element, is present in low alloy steels at a rate of 0.15-0.30% together with Ni, it increases the hardenability of the steel, its resistance to the drawing process and heat. In addition, it improves wear resistance (Topbaş, 1998).

Molybdenum obtained by acid-base reactions is not found in nature. It includes features such as high wear resistance, temperature resistance and low coefficient of friction. It has a melting temperature of 2610°C and a density of 10.22 g/cm3. It is highly sensitive to impurities at the grain boundaries. It may show intergranular fracture even in completely recrystallized medium. Some of the usage areas; steel alloying, dentistry, electrical and electronics, spacecraft and aircraft construction, nuclear energy applications. Since molybdenum and its alloys start to form oxidation in the oxidizing zone above 500°C, it is necessary to protect the Molybdenum by covering it with an anti-oxidation layer while working in this environment. Molybdenum transforms into MoO_3 at approximately 700°C in atmospheres containing oxygen. This oxide has the appearance of a white smoke and is odorless. Due to evaporation, a protective oxide layer cannot form on the surface of Molybdenum and the metal loses weight continuously (Dokumacı and Önay, 2008).

2.6.4. Tungsten

It is a strong carbide builder. These carbides formed become very hard and improve toughness. Increases wear resistance at high temperatures and creep. It is preferably used in hot work die steels, high speed steels, highest hardness diamond steels and creep resistant steels. It is often used in high-speed steels at rates up to 20%. It increases the wear resistance in the W_2C_6 type complex carbides it creates.

Among the reasons why tungsten metal finds many practical applications in industry, the most important one is; This metal has low evaporation pressure and high melting temperature. Thanks to this feature, it easily covers a wide area of use in high vacuum applications.

The thermal expansion of tungsten is also very low with rising temperature. The electrical resistance of Tungsten, which is used as a heating material in the production of high temperature furnaces, also contributes. Although the tungsten element includes a high absorption capacity against radioactive emissions with its high density, it creates a disadvantage in absorption applications due to the very difficult processing of pure tungsten in machines. For this reason, tungsten alloys containing nickel-copper or nickel-iron binder phase with high tungsten mixture are preferred for absorption of gamma and x-ray rays, considering their easy mechanical processing (Ovalı, 2013).

2.6.5. Vanadium

It is the microalloy element with the highest solubility in steel. Vanadium nitride is less soluble than Vanadium carbide. Thanks to the increase in the nitrogen ratio in the material, the dissolution and precipitation efficiency of vanadium can be highly controlled. Vanadium precipitates are less potent than Titanium. The recrystallization delay is low enough. The high solubility of Vanadium leads to a favorable precipitate hardening of the ferrite after normalizing annealing (Hannane, 1989).

2.6.6. Niobium

Niobium is known as one of the most effective microalloys. It forms carbide and nitride. The NbC alloy is practically formed below 1000°C. It prevents the recrystallization of austenite and allows the formation of small ferrite grains.

It has the feature of increasing the secondary hardening temperature. It narrows the austenite zone. It can be added as an additive in place of other materials used in high-speed steels.

In the austenite zone, Niobium plays an important role in providing low temperature toughness with small precipitate formations. The niobium inland is delimited; because in this process the niobium carbonitrides remain partially undissolved in austenitization. Niobium also plays a role in increasing toughness and strength. It is achieved by delaying recrystallization and reducing grain size. The highest grain reduction effect and property is in Niobium element. The limit rate of the active amount is 0.04% Nb. This amount shows that the toughness of the Nb-alloy has reached its maximum level. However, it has a precipitation hardening effect even at low levels (Çeviker, 1991).

2.6.7. Phosphorus (P)

A strong solid solution hardening forms in ferrite. The brittleness caused by segregation at the austenite and grain boundaries is caused by the use of phosphorus at values greater than 0.05%. It is used together with copper to provide corrosion resistance to a great extent (Topateş, 1995; Karabulut, 2004).

The element that increases the strength of ferrite the most is Phosphorus. Based on this, it has the effect of increasing the hardness and strength of the steel, and decreasing the ductility and impact resistance in the forming direction, even if it is added at low rates. These effects are greater for high carbon tempered steels. It plays a role in determining the quality in the foreground, as it improves the corrosion resistance of steel and is present in the steel together with sulfur (Topbaş, 1998).

Phosphorus, which is between 0.07 and 0.12% in some steels, improves the cutting property; However, since it reduces the ductile properties of steels in high phosphorus addition, it causes the steel to crack or break during cold forming (Savaşkan, 2000).

2.6.8. Manganese (Mn)

It improves the quality of Manganese steel castings with a good oxygen absorber property. The maximum manganese ratio used in steels varies between 1.3-1.7% (Topateş, 1995; Karabulut, 2004).

2.6.9. Aluminum

Aluminum, which is a highly effective deoxidizer and nitride former, improves the toughness of steel at low temperatures. It is an economical grain reducer. It creates high quality wear resistance and surface hardness, especially when added to nitride steels. Dissolution occurs above 1000˚C.

It includes a long period of time in order that the dissolution and subsequent formation lattice is hexagonal tight-packed. For example, in case of rapid heating of steel, AlN can stay in the structure formed above 1000˚C and prevent grain growth during dissolution. It contributes to the strength and toughness of the steel by preventing grain growth during rolling and forging operations in AlN normalization steels (Gladman, 1997).

Aluminum is the third most abundant element on earth after oxygen and silicon. Despite this, its production in the industrial field was realized in 1886 with the use of the electrolysis method. In the metal market, it ranks second after iron and steel. Thanks to the sufficient and superior properties of aluminum, which has been used frequently at the beginning of the twentieth century, its use in the industry is increasing day by day.

Aluminum is used in many areas such as space shuttle, vehicles, aircraft, packaging and packaging, communication, thermal insulation, decoration and many more. It has a valuable quality because it is environmentally friendly and recyclable from the production level to the consumption movement.

2.6.10. Titanium

Titanium is an allotropic material. At room temperature, titanium displays a tight-packed hexagonal structure, and at high temperatures, it exhibits a volume-centered cubic structure. The α phase in the hexagonal structure, which is around 885ºC, transforms into the β phase in the volume-centered cubic structure. This temperature is called the “β transformation temperature” for pure titanium. While this transformation temperature increases with the effect of intermediate elements such as oxygen and nitrogen, which stabilize the A phase, the temperature rises or decreases with the effect of the alloying elements involved (Matthew and Donachie, 1988; Barksdale, 1968).

The effectiveness of titanium is lower than that of the element niobium. Since it increases proportionally with its alloying and strength-enhancing effect, high strength increases are aimed with the titanium element. Titanium microalloyed steels also tend towards recrystallization inertia. Titanium nitride is thermodynamically and physically similar to niobiumcarbonitride. Titanium nitride prevents grain coarsening during welding in the heat-affected zone. The grain reduction feature of titanium element is between vanadium and niobium. The sediment hardening characteristic feature provides the appearance of vanadium (Hannane, 1989).

It is a strong carbide and nitride builder such as W, V, Mo and Nb. It creates a balance in the structure of austenitic steels and refines the grain sizes in steels in general. However, it is used as a deoxidant (oxidizer) with aluminum in planned poured steels (Topbaş, 1998).

2.6.11. Silicon

Silicon is the most abundant element in the world after oxygen, which makes up 27.7% of the earth's crust. It is also seen in plant tissues, animal skeletons, and the wall structures of diatom cells living in the sea. Meteors and stars contain a large amount of the element silicon. It is usually seen in the form of silicon dioxide (SiO2), known as quartz or silica, which is formed by compounding with oxygen.

It is one of the basic elements used as an oxygen remover in the steel production phase. The ratio of silicon used in steel varies depending on the production style.

2.6.12. Aluminium Carbide

Aluminum carbide, Al4C3, is an important compound in aluminum metal technology and can combine high thermal conductivity and high electrical resistance at room temperature.

It is a starting material for producing ceramics with a diamond-related structure (Al_2O_3–Al_4C_3–AlN). In addition, Al_4C_3 is an additive that strengthens composite materials and alloys.

It is known that aluminum activates the growth of large colorless diamond crystals at high pressures and temperatures in the Fe-C system. Very recently, an important role of aluminum carbide in diamond crystallization from Al-C melt was reported by Petrusha et al. (Gilman, 1983, Petrusha, 2004, Grogen et al.)

PART 3

POWDER METALLURGY

3.1. DEFINITION OF POWDER METALLURGY

Powder metallurgy is the science of production that aims and ensures the production of products with the expected quality using specially manufactured powders. It extends to the action of mixing unalloyed or alloyed powders using determined proportions, compressing the homogeneously mixed powders with the help of a suitable mold, and then sintering the powders in a controlled atmosphere in order to gain their metallurgical properties. The production of homogeneous parts ensures that their chemical, physical and mechanical properties can be controlled (Panda and Dobransky, 2018).

History BC Powder metallurgy dating back to 3000 BC has provided valuable benefits for most of human history. The ancient Egyptians obtained sponge iron by reducing iron oxide. M.S. By 400 B.C., Delhi Column weighing 6.5 tons was obtained with powder metallurgy resembling today's method in India. When it comes to 1826, the platinum money produced in Russia can be said to be the first industrial application of powder metallurgy. In 1892, W.H. Wolaston is known as the person who started the first official powder metallurgy application by obtaining platinum by powder metallurgy method.

Hot and cold pressing in the powder metallurgy process was developed to replace traditional casting and machining production methods and entered the literature as a new part production technique in this field with the Second World War.

The casting method is highly economical in the production of parts and components of the machine, which is made of bulky, large surface area and alloy materials with certain geometries. However, when mass production is taken into account, as the part sizes decrease, the number of production per unit time increases, and the use of new alloys developed to provide services in up-to-date technology, the Powder Metallurgy (T/M) production method is ahead of machining and casting production methods (Beddow, 1978). Another feature of powder metallurgy is that it is a metal forming technique. In addition, powder metallurgy is a material production method.

8000 alloys have emerged from 86 elements in the periodic table, which are considered metals. Essentially, it is possible to produce around 1025 alloys from the 86 elements mentioned, with different mixtures such as binary, triple and quaternary. The only method that will make this possible is the powder metallurgy method. When analyzed considering today's time, the preference for part production with the powder metallurgy method will become one of the most valuable and productive features with the technological advantages that develop and renew itself increasingly on the basis of the cost part. The parts planned to be produced by powder metallurgy method will have the feature of being used immediately after they are produced, or they can be left for secondary processes upon request. The production of materials that cannot be produced with the classical production and casting method can be realized with the powder metallurgy method (Siyonr, 2007).

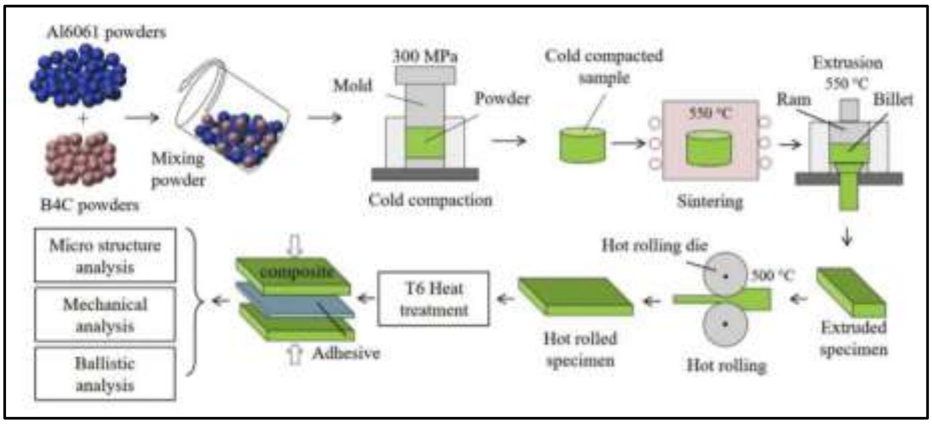


Figure 3.1. Powder metallurgy production scheme of a material (Karakoç et all 2018).

3.2. POWDER PRODUCTION PROCESSES

The methods used in the production of metal powders determine many properties of powders. The geometric form of the powder can be very different, from complex shape to spherical shape, depending on the production method. The surface condition of the dust particle also varies according to the production method. Many of the materials can be pulverized by a technique suited to their properties. Among the powder production techniques, there are those that are used commercially. These;

- Mechanical (Grinding)
- Chemical
- Electrolysis
- Atomization
- Other Production Techniques (Sarıtaş, 1994).

3.3. PHYSICAL PROPERTIES OF POWDERS

Grain size and shape are shown as fluency, density and compressibility. Grain size in metal powders is mostly determined by sieve analysis method. Particle size is important in particle size can be calculated. It cannot be said that the dimensions of all the powder are the same, but the average particle size can be mentioned. By size analysis, the % amount and average particle size of the powder in each range is determined. The form of the powder has an important place in determining the particle size. When spherical powders are centered, it is sufficient to know only the diameter, while the flake thickness of the flake powder particles and the size in the plane direction should be calculated. For powders in various forms, the particle size is calculated by going from the surface area. The grain size is determined by considering the surface sphere (Bocchini et al., 1991).

The grain shape, which is considered as one of the most important features of powders; It is an important factor affecting the properties of powder particles such as apparent density, fluidity, compressibility and raw strength.

The apparent density of a randomly selected powder is the most important factor determining the dimensions of the cavities in the mold designed for pressing the powders. For the determination of the apparent density, the determined volume of metallic powder, which is not adhered to each other, is completely filled into the known container and the resulting mass is measured. The apparent density is then calculated by the ratio of mass to volume. The void filling of the container is achieved by dropping the dust on the inclined plates. Apparent densities of iron powder particles can be observed, however they show differences (TS., 1985).

All materials in nature can be pulverized after processing. There are many techniques in the industry for the preparation of metal powder particles. However, the techniques chosen to produce the powders vary according to the nature of the material. Particles with an average size of up to a few microns are defined as particulate dust. Along with the developing technology, the dust size is getting smaller day by day. The geometric form of the powder varies from spherical to dendritic form depending on the production techniques.

3.4. POWDER METALLURGY USAGE AREAS

Powder metallurgy is used in various fields. These; Manufacture of wear-resistant parts produced using super alloys, refractory materials such as tungsten and molybdenum, tool steels, stainless steels, copper alloys, magnetic alloys, aluminum and titanium alloys, cermets and nuclear materials. In particular, the automotive industry is at the forefront. In addition, jet engine parts, tungsten lamp filaments, orthopedic equipment, gear wheels, non-lubricated bearings, high temperature filters, electrical contacts, aircraft brake pads, welding electrodes, catalysts, soldering tools, high temperature filters, nuclear power fuel elements, circuit boards There are application areas such as dentistry, paints, explosives, welding electrodes (Akoral, 2003).

3.5. ADVANTAGES OF POWDER METALLURGY

The importance of powder metallurgy stems from the fact that different alloys, which are difficult to form by machining and casting, can be turned into products in an economical way and easily. When the powder metallurgy method is compared with other production methods, advantages and disadvantages emerge.

Advantages:

- It can be easily produced in parts with complex forms and precision.
- The production rate is high, therefore the need for labor is low.
- The materials produced have high mechanical and physical properties.
- The particle size of the produced parts is small, the tensile strength and machinability are high.
- Production can be made by combining materials with different characteristics that do not dissolve in each other.
- Parts produced with powder metallurgy generally do not require additional processes such as machining.
- The loss in the material produced by powder metallurgy method is low. Considering the material loss caused by machining and casting method, material savings occur to a large extent.
- It provides an economical production due to the acceleration of production, waste of material and less labor.
- There are no melting losses.
- Materials with high hardness and wear resistance can be produced.
- Suitable for mass production and mass production.
- After the sintering process, the part is ready for use and secondary operations are largely unnecessary.

Disadvantages:

- The cost of the molds required for production is high.

- Due to the presence of pores in the microstructure, lower mechanical properties may occasionally occur compared to other methods.
- Difficulties are encountered in the production of parts with high thickness and diameter ratios. There are also limitations on particle sizes to produce homogeneous densities. Powder metallurgy parts up to 20 kg can be produced.
- The cost of metal powder particles is more expensive than materials produced in ingot form (Demir, 1993).

3.6. CHARACTERIZATION OF DUSTS IN POWDER METALLURGY

3.6.1. Dust Sampling

Dust sampling, a grueling and time-consuming task, can be accomplished using a variety of methods. The commonly used method is to blend and apply small samples from many different points. The general form of the particles is cohesive, the tendency to stick to each other due to the situation is high. Eventually agglomeration of particles is possible. High agglomeration may occur due to surface moisture. In addition, clinging agglomerates are characterized as a set of particles clinging to weak forces that can be destroyed by small shear stresses. For most particles, mechanical and ultrasonic agitation methods, surfactant liquids play an active role in dispersing the particles and subsequently determining the properties. Mechanical mixing or ultrasonic agitation are frequently preferred methods of dispersing the flocculation formation. (Sarıtaş et al., 2007; Matik, 2010; Karabulut, 2011).

3.6.2. Particle Size Measurement

Particle size calculation and detailed information are necessary for researchers doing research on powder. Determining the particle size is not a simple task unless the particle is in spherical form. In order to calculate an odd number of particle size, it is mostly based on the geometrical structure of the particle and the diameter feature is used (Kousaka, 1997).

The size of the powder particles is determined by sieve or other methods. Not all dust particles have the same size. The particle size is determined using the average particle size property. If the particle geometry of the powder is intricate, particle size measurement techniques also vary.

If dust particles are found mixed with powders of different sizes, four to five dimensional measurements are required. If its complex structure is dense, the particle size can be determined based on the surface area. The grain sizes of the dust particles are generally determined by the sieve analysis measurement method. The size of the sieve is calculated by the large hole in its structure. The calculation process is determined by the mesh method. Today, since the metric system has been passed, the size of the sieves is now calculated by writing in microns. The sieve method does not measure the actual size of the powder particle, it only categorizes it as greater or smaller than a certain value (German, 2007).

3.6.3. Mixing of Powders

Since powder mixtures are generally used in the powder metallurgy method, it is an appropriate orientation to mix the powders effectively before they are subjected to the compacting technique. The homogeneity of the powder particles is the main purpose of the mixing process. Homogeneous mixing of powder particles of different sizes, forms and densities increases the performance of the part planned to be produced. When there are no standard dispersions in the powder mixture, blending should be performed before the mixing process. Blending is recommended in order to improve pressing and sintering properties and to produce a uniform size distribution. Some factors affect the mixing and blending process. These factors are;

- Powder volume in the mixer
- Physical characteristics of powders
- Mixing speed
- Humidity and atmospheric conditions
- Mixing time
- Mixer dimensions

- Rotation speed.

3.6.4. Pressing of Powders

When the metal powders to be used are formed by means of a mold, it is desired that the powder fills the mold cavity very well, yet completely. When the powders are cold pressed in the mold, it is considered to reach the theoretical density as much as possible. Although the same pressing pressure is applied, the density reached as a result of pressing in each metal powder is different according to the theoretical density of the material. The factors that depend on this situation are the shape of the powder, the grain size and surface, the type of material (production methods, specific surface) and the pretreatment applied to the powder.

The softer the structure of the material, the higher the compressibility feature. Pressability is closely related to the friction of powder grains with the mold and between itself.

3.6.4.1. One Way Pressing

Conventional powder compaction processes are carried out unidirectionally. Immediately after the powder mixture is filled into the mold, pressing processes are applied by means of the upper pressure plate. The task of using the lower piston is to scrape the part to be formed from the mold. In order to facilitate the removal of the part from the mold and to provide the compression feature, lubrication is applied to the mold walls. As the amount of pressure applied in the unidirectional pressing process is increased, the density of the compressed material also increases. The reason for this is the decrease in the pores among the powders used, as well as the fact that the mass remains the same and the volume decreases. In this method, due to the simplicity of the mold form, although it is a suitable method by taking the cost into the center, it is not preferred because it cannot reach the expected density in intricately shaped parts and metallic parts with a high length/width ($L/D \geq 2.5$) ratio.

3.6.4.2. Bidirectional Pressing

Pressure is applied by the punch up and down the die. Both staples are dynamic. Staples can apply different or equal amounts of movement and pressure. The resulting pieces fall into the first and second grade category. A raw homogeneous density cannot be achieved. The raw density distribution can vary significantly due to friction between particles, punches and powder particles and the die surface. These various properties are tried to be reduced by reducing friction by means of lubricant or by using suitable compression methods.

It is not possible to reach full density through the unidirectional compression method. Dust in bidirectional compression; Because it is compressed by the upper and lower punches, the friction force formed between the wall of the mold and the powder particles exceeds the spring force of the flexible material such as the spring on the mold base, allowing the mold to move towards the lower region, allowing the lower punch to apply equal pressure to the powder surface of the powder with the upper punch. After the compression process is completed, the TM part is stripped from the mold by the movement of the lower punch towards the upper section. As a result, it turns out that the density distribution is more homogeneous than the parts obtained by unidirectional pressing. The lowest density value is found at the midpoint of the compressed part. The distribution of this density is symmetrical compared to the horizontal and vertical axes passing through the midpoint (Hiçyılmaz, 1999).

3.6.4.3. Isostatic Pressing

Isostatic pressing is the pressing of metal powders under a hydraulic (fluid) pressure. Isostatic pressing is the process of forming the compressible powder particles, which are placed in a mold made of a sealed and flexible material, by compressing them with the high pressure force applied to each point of the mold. The density of the parts obtained by isostatic pressing technique is equal in every part of the part, regardless of its dimensions such as diameter and length. The homogeneous density arrangement is ensured due to the homogeneous effect on all surfaces by means of pressure and the absence of frictions on the mold wall. It is a suitable and necessary method for the production of parts in intricate form. There are two different application areas: hot isostatic pressing and cold isostatic pressing (Hammes and Binder, 2014).

3.6.5. Sintering of Powders

The sintering technique is expressed as a baking process applied at high temperatures in a controlled atmosphere in order to give strength to the pressed powder particles. The mechanical bonds formed in the part formed as a result of shaping the particles by pressing in the mold are transformed into metallic bonds through the sintering process, thereby gaining strength to the part. The strength ratio of the part before and after the sintering process is quite different.

While the sintering technique is carried out at a temperature below the absolute melting temperature of the metal in systems with a single component; In systems with more than one component, it is mostly done above the melting temperature of the components with the lowest melting temperature.

While the sintering technique is carried out between 70-80% of the melting temperature of the base material; For some refractory materials, the melting temperature can be increased to 90% (Ataş, 2003).

The sintering technique can be expressed as a material transport system activated by the thermal method, which causes the reduction of the surface area of the powders in its region, the enlargement of the contact points in the particles, and accordingly the change of the pore form and the decrease of the pore volume. The device that controls the temperature and time during the sintering cycle is also called the sintering furnace. In addition to this feature, it provides the possibility of heat treatment to the part after sintering by keeping the atmosphere as well as removing lubricants and binders (Randall, 2007).

After the pressing process, the spherical powder particles are in point contact. During the sintering process, welds are formed between the particles in contact with each other and the bonds are strengthened. First of all, the size of the particles grows, then shrinkage of the pores occurs with the advancing sintering time. Later, closures occur in the pore channels and turn into a closed pore form (Akoral, 2003).

During the sintering method, a decrease in the high surface energies of the powder particles occurs, resulting in perfectly formed sizes in the particles. As a result, the porosity value in the internal structure decreases to zero. Small powder particles are easily sintered because the surface energy falling according to the volume ratio is found to be directly proportional to the inverse of the diameter of the powder particles (Boz, 2003).

While transparency, thermal conductivity, density and strength values increase in ceramic materials, strength and density increase in polymers. In metals, conductivity and strength increase. The sintering method is used for porous bronzes and alloys similar to their properties, between 600°C and 800°C, metal alloys in the iron group between 1000°C and 1300°C, hard alloys between 1400°C and 1600°C. Sintering time and temperature vary according to the type of material. While less than half an hour is sufficient for diamond alloy materials, a sintering method lasting more than one hour is applied for hard alloys. There is an inverse relationship between sintering time and temperature. When the sintering temperature is high, the time is short, and as the temperature decreases, the applied time increases (Gryczka and Humbeeck, 2008).

As the sintering temperature increases, the properties of the material such as strength, electrical conductivity, density and ductility increase. In the initial stage of sintering, the powder particles are in point contact. The sintering process applied to the materials is carried out in three stages as initial, intermediate and final.

3.7. ANALYSIS OF MATERIALS PRODUCED BY THE METHOD OF POWDER METALLURGY

3.7.1. Mechanical Properties

The choice of material depends on the mechanical properties of that material for a particular application. It is important to familiarize yourself with the standard techniques used to calculate these properties and to evaluate the results from these experiments depending on different parameters. The potential of a material to withstand static loads is determined by the compression and tensile technique. Information about the resistance to plastic deformations can be obtained by means of static tests. The impact process technique is used to determine the resistance of the material against the stresses under the impact. By performing these experiments at different temperatures, the brittle-ductile transition temperatures of the material are calculated. With the fatigue process technique, the behavior of the material under repeated stresses and variability and its useful life under these influences can be calculated. Creep tests are applied to see the manifestation of the behavior of the material under long-term loads at high temperatures (Odabaşı, 2017).

3.7.2. Microstructural Features

The microstructure of a material, the structural and chemical nature of its geometric formation, is expressed by the arrangement of the components that make up the material, and to this extent includes the imperfections it contains and the component phases of the material. The microstructure is sufficient to significantly determine the properties of a material. In addition, in order to use the ore, it is necessary not only to learn the factors that contribute to the determination of the microstructure of the material, but also to understand the relationships between the microstructure and properties. It is necessary to determine the relations in materials science from one part and the microstructure of the material from another aspect (Odabaşı, 2017).

3.7.3. Surface Related Properties

Events such as oxidation and corrosion are among the surface properties of the material. If the porosity rate in the structure of the materials produced by the powder metallurgy method is high, corrosion occurs faster due to the fluid formed in the pores and subsequently accumulated. It has been observed that the materials produced under suitable sintering conditions have a high degree of environmental resistance.

PART 4

EXPERIMENTAL METHOD

4.1. INTRODUCTION

In this study, alloys in desired ratios were combined by powder metallurgy method and turned into samples. The processing steps carried out are explained in section 4.2, mixing of powders, pressing and sintering process in section 4.3, optical microscope image analysis in section 4.4, hardness measurements in section 4.5, wear test in section 4.6, SEM and EDS analysis in section 4.7. Optical and SEM microscope examinations, hardness measurement and wear tests were carried out in Karabuk University, Iron and Steel Institute, Margem laboratories.

4.2. EXPERIMENTAL PROCESSING STEPS

The processing steps performed in this experimental study are given in Figure 4.1. Within the scope of this thesis, some information about the production of ready-made powder metal steels will also be given. The production of the supplied steel was as follows. The powders are supplied, mixed, then pressed and then sintered. Finally, it includes mechanical tests and microstructural examinations.

4.3. MIXING, PRESSING AND SINTERING OF THE SUPPLIED STEEL SAMPLE POWDERS

Before the mixing process, the powder particles were weighed with a precision of 0.0001 with a precision balance at the rates given in Table 4.1. The powder mixtures obtained as a result of weighing were mixed with a triaxial mixer for 1 hour without a ball in order to form a homogeneous structure.

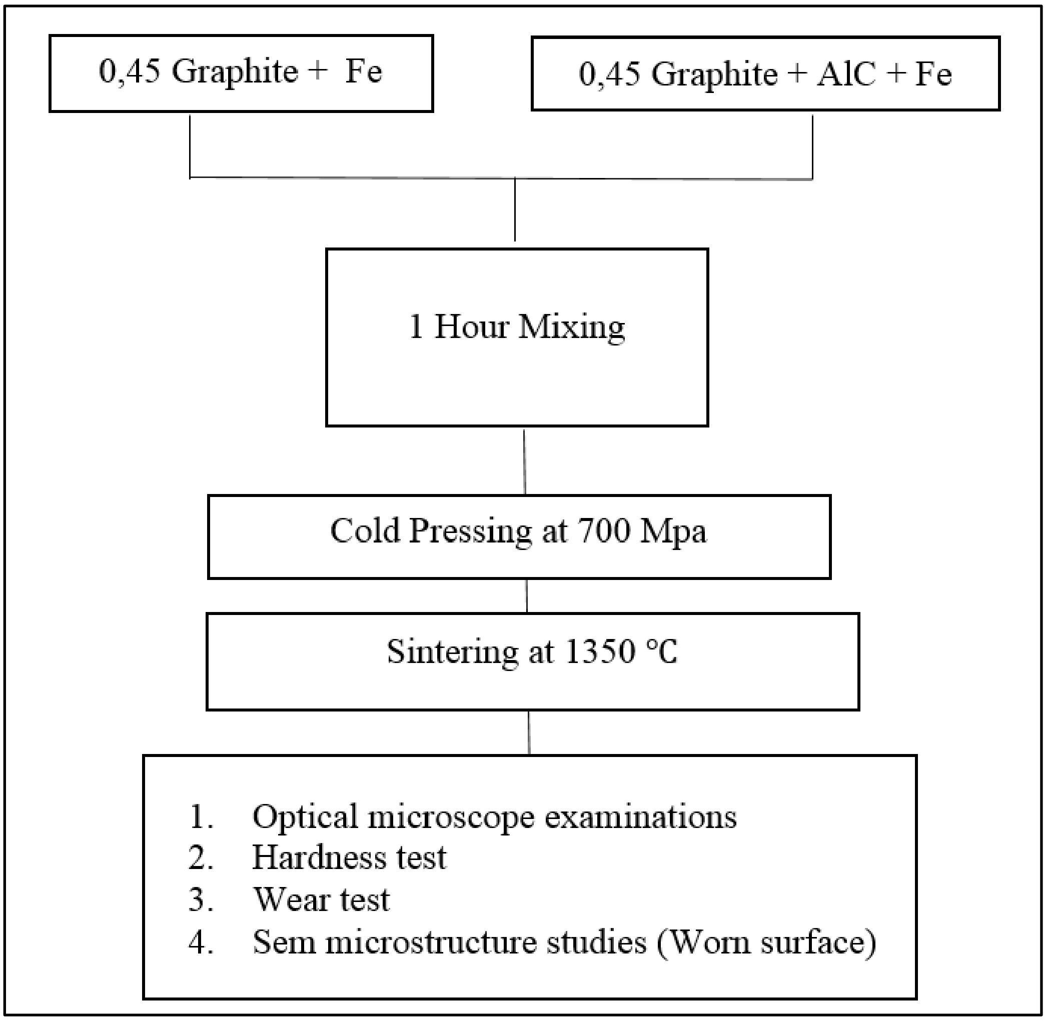


Figure 4.1. Process steps followed in the experimental study.

Table 4.1. Powders and properties.

	Elemental Powders	Powder Size (µm)	% Purity Value	Supply Company
1	Iron (Fe)	<150 µm	99,9	Höganäs
2	Carbon (C)	10-20 µm	96,5	Höganäs
3	Aluminum Carbide (AlC)	~325 mesh	95	Aldrich

Table 4.2. Chemical compositions of alloyed PM steels.

Compositions	Carbon (%wt.)	AlC (%wt.)	Iron (%wt.)
Fe+0,45 Carbon (Composition 1)	0.45	-	The rest
Fe+0,45 Carbon +0,2AlC (Composition 2)	0.45	0,2	The rest
Fe+0,45 Carbon +0,5AlC (Composition 3)	0.45	0,5	The rest
Fe+0,45 Carbon +1AlC (Composition 4)	0.45	1	The rest
Fe+0,45 Carbon +2AlC (Composition 5)	0.45	2	The rest

The pressing process of the prepared powders was carried out by subjecting them to the cold process. The samples, which were subjected to cold pressing process, were made in the form of wear samples in a one-way, 700 MPa pressing pressure, with a mold manufactured according to ASTM (E8M) powder metal material standards, in a 96-ton capacity Hydraulic press.

The powders were cold pressed to Ø12x7 mm for experimental studies. Figure 4.2 shows the mold used in cold pressing. Hidroliksan brand press which is given in Figure 4.3 in Karabük University of Margem laboratory.

Figure 4.2. Metal mold.

Figure 4.3. Hydraulic press machine.

The sintering process for alloys was carried out at a heating rate of 5 °C/min, at 1350 °C and under vacuum of 10^{-6} mbar for two hours with Protherm Brand which is given in Figure 4.4.

Figure 4.4. Image of vacuum heat treatment furnace with sintering operations.

4.4. OPTICAL MICROSCOPE EXAMINATIONS

Microstructure examinations were carried out with a Nikon Epiphot 200 optical microscope with a magnification of X200-X500. Images of different sizes were taken from different parts of each sample and care was taken to ensure that these images were representative of the whole microstructure. The investigations were carried out at Karabuk University, Iron and Steel Institute, MARGEM Laboratories.

4.4.1. Preparation of Samples Used in Metallographic Studies

In this study, an optical microscope was used to examine the microstructure of samples sintered in argon atmosphere. Alloyed and unalloyed sintered samples with different compositions were sanded with 200, 400, 600, 800, 1000, 1200, 2500 mesh water sandpaper, respectively, until the surface roughness was removed. These surfaces were polished with 6 μm, 3 μm and 1 μm diamond pastes, respectively, and made ready for etching. Before etching, a 30-minute cleaning process was carried out with an ultrasonic cleaner to remove the chemical residues used in polishing the samples.

4.4.2. Preparing the Etchers and Performing the Etching Process

Nital solution obtained by mixing 2% nitric acid into ethanol was used for the samples that were polished for metallographic examinations and made ready for etching. Etching was carried out by immersing the samples in Nital solution and keeping them for 8-10 seconds. After the etching process was completed, the etched surfaces were cleaned and dried with alcohol and then made ready for examination under an optical microscope.

Figure 4.5. NIKON inverted metallurgical microscope.

4.5. HARDNESS TEST

Hardness measurements were made for all samples. Hardness measurements were measured on samples prepared for microstructure examination. Microhardness measurements of the samples were made in SHİMADZU brand hardness measuring device. Hardness measurements were carried out by applying a load of HV0.5 (500gr). 5 hardness measurements were taken from each sample and the average of these measurements was the sample hardness value.

4.6. WEAR TEST

The analyzes of the wear test were carried out at the MARGEM Laboratories at the Iron and Steel Institute of Karabuk University. The samples sintered at 1350°C were sanded up to 1000 mesh sanding number and the Reciprocating Wear Test was applied on the UTS-10 Tribometer test device, which performs back-and-forth type wear test. AISI 52100 material steel ball was used for this application. The stroke distance was determined as 3 mm. The applied load is 30 and 60 N, and the sliding distance is 25 meters. After the wear test, the wear volumes were measured from the worn surfaces with a surface profilometer device. Then, the wear surfaces of the powder metallurgy steel samples were investigated by SEM.

Figure 4.6. Image of microhardness tester for hardness measurements.

Figure 4.7. Wear test machine (Tribometer).

Figure 4.8. Surface roughness measurement machine.

4.6. SCANNING ELECTRON MICROSCOPE (SEM + EDS) ANALYSIS

After the wear test, SEM images and EDS analyzes were taken from the wear surface. SEM studies were carried out at Karabuk University, Iron and Steel Institute, MARGEM Laboratories. Images were taken at various magnifications from different parts of the samples obtained. Special attention was paid to ensure that these images were representative of the microstructure. The SEM device used in the experimental study is shown in Figure 4.9.

Figure 4.9. SEM device used in the experimental study.

PART 5

EXPERIMENTAL RESULTS AND DISCUSSION

5.1. INTRODUCTION

In the experimental results and discussion section, the experimental results of the steel samples produced in different compositions are shown. It also includes the evaluation of the results. In Section 5.2, the results of the optical microstructure of the samples are examined. Hardness results are evaluated in Section 5.3, and wear graphs, weight losses and worn surface SEM images are evaluated in Section 5.4.

5.2. OPTICAL MICROSTRUCTURE RESULTS AND EVALUATION

Microstructure examinations were carried out with a Nikon Epiphot 200 optical microscope with a magnification of X200-X500. Images of different sizes were taken from different parts of each sample and care was taken to ensure that these images were representative of the whole microstructure. The optical microstructure results taken at 200X and 500X magnification are shown in Figure 4.10.

Alloyed steel with the desired composition was produced by adding AlC at different rates into the Fe matrix by powder metallurgy method. The microstructure and mechanical properties of the alloy steels produced with the addition of AlC from 0.2% to 2% were compared. Figure 5.1 shows the microstructure of steels with different ratios of AlC added.

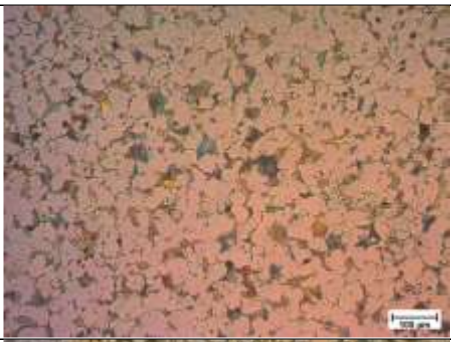
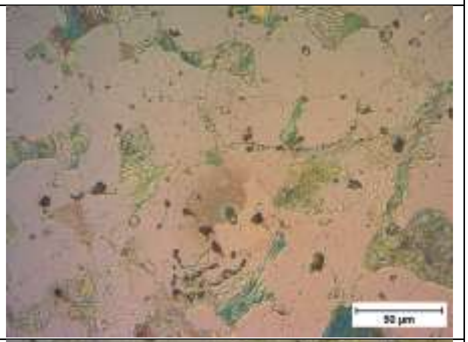
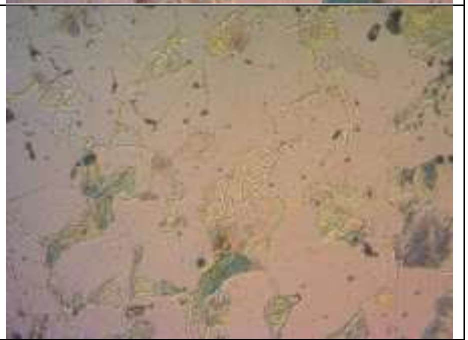
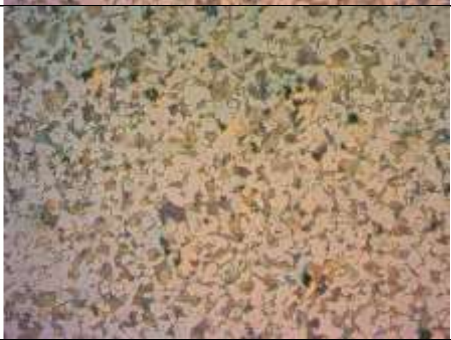
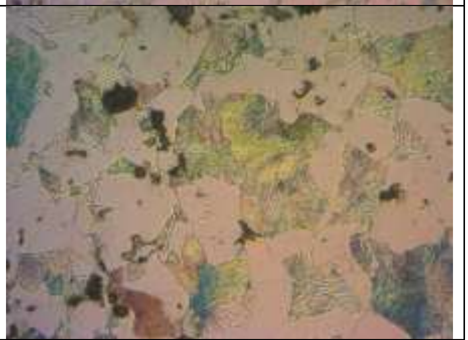
	200X	500X
Fe+0,45C +0 AlC		
Fe+0,45C +0,2AlC		
Fe+0,45C +0,5AlC		
Fe+0,45C +1AlC		
Fe+0,45C +2AlC		

Figure 5.1. Microstructure pictures of steels containing different ratios of AlC.

As seen in Figure 5.1, all structures consist of ferrite and pearlite phases. When the microstructure pictures were examined, it was determined that there were partially unclosed pores at the grain boundaries. Although it is stated in the literature that porosity affects the strength negatively, it has been stated that the pores are very small and spherical in shape, but do not reduce the strength (Sarıtaş et al., 2007).

It is seen in Figure 5.1 that the average grain size decreases with the addition of AlC. This situation arises when AlC precipitates formed during sintering prevent the growth of austenite grains (Ollilainen et al., 2003). One of the properties of microalloying 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 inhibits the growth of austenite grains and causes the formation of small ferrite grains during cooling (Xiang-done et al., 2013; Bakkali et al., 2008; Gladman, 1997).

5.3. EVALUATION OF HARDNESS TEST RESULTS

In Table 5.1 the hardness results of the steel to which AlC is added at different rates are given. As discussed in the previous section, AlC added steels have been found to exhibit superior mechanical properties. The highest strength value was obtained in steel containing 2% Al. This change in strength values is a result of the formation of AlC precipitates and their dispersion in the matrix of different sizes (Erden et al., 2014).

Table 5.1. Hardness results of steel with different ratios of AlC added.

Composition	**Average Hardness HV (0.5)**
Fe+0,45C +0 AlC	98
Fe+0,45C+0,2 AlC	114
Fe+0,45C+0,5 AlC	127
Fe+0,45C+1 AlC	141
Fe+0,45C+2 AlC	155

5.4. WEAR TEST RESULTS AND EVALUATION

In the wear test performed with the UTS-10 Tribometer abrasion tester, the stroke distance was determined as 3 mm, the applied load as 30 and 60 N, and the sliding distance as 25 meters. After the wear test, the wear volumes were measured from the worn surfaces with a surface profilometer device. Then, the wear surfaces of the powder metallurgy steel samples were investigated by SEM. The values measured with the surface profilometer device after the wear test are given in the Figures and Tables below.

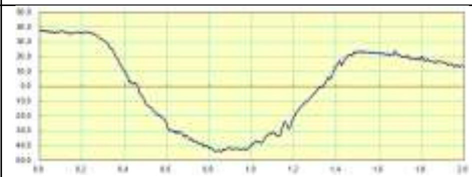
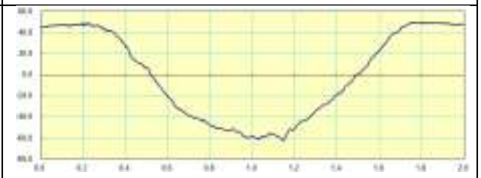
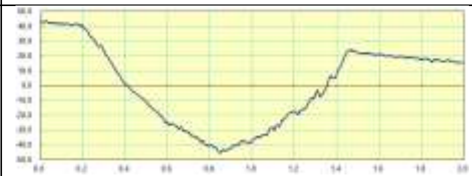
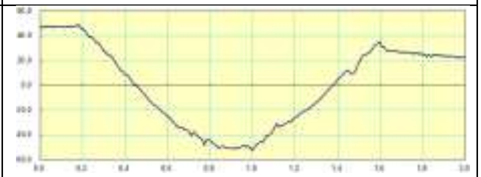
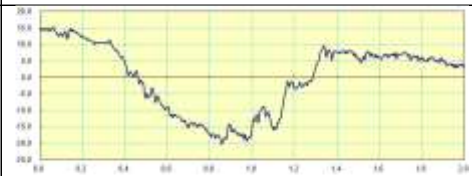
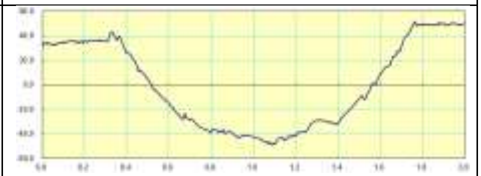
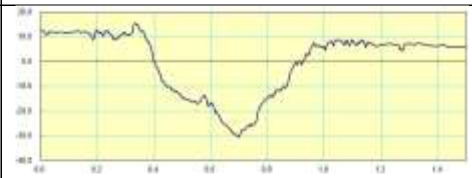
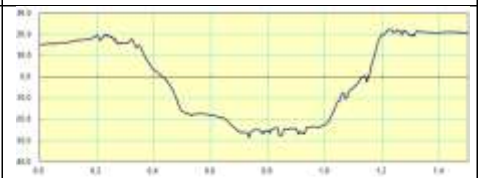
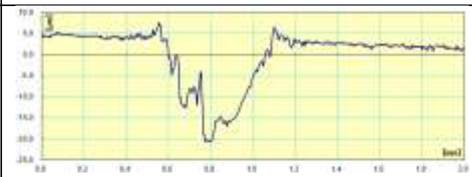
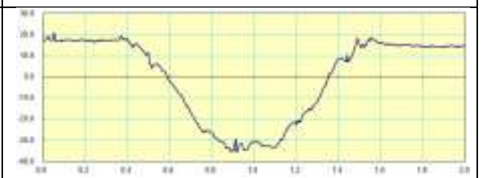
	30 N	60 N
Fe+0,45C +0 AlC		
Fe+0,45C +0,2 AlC		
Fe+0,45C +0,5 AlC		
Fe+0,45C +1 AlC		
Fe+0,45C +2 AlC		

Figure 5.2. Two-dimensional wear surface profile images measured with the surface profilometer device after the wear test under 30 and 60 N loads.

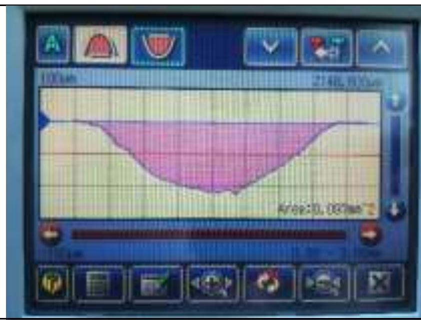
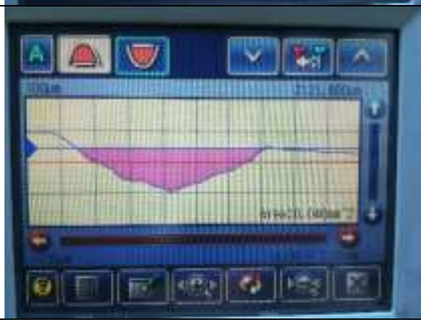
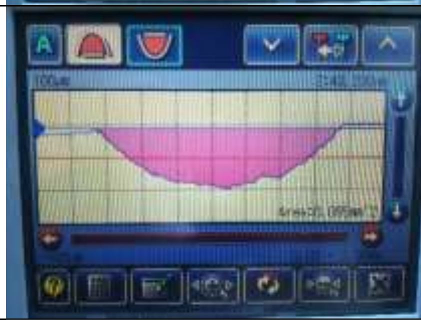
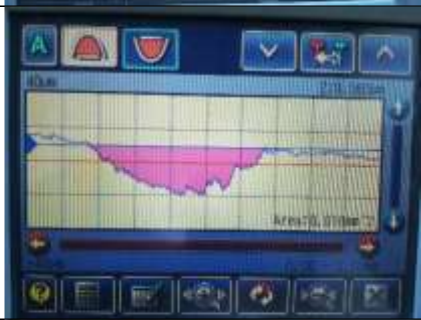
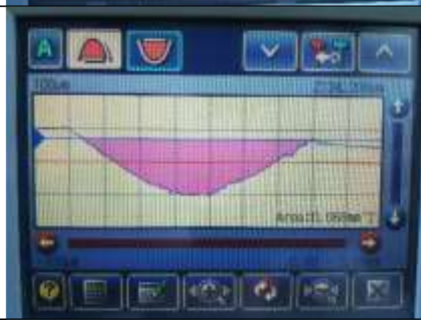
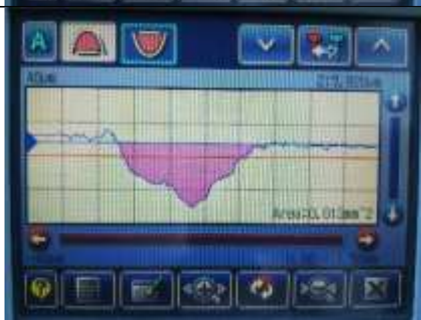
	30 N	60 N
Fe+0,45C +0 AlC		
Fe+0,45C +0,2 AlC		
Fe+0,45C +0,5 AlC		
Fe+0,45C +1 AlC		
Fe+0,45C +2 AlC		

Figure 5.3. Area loss images measured with the surface profilometer device from the wear marks at 30N and 60N loads.

Table 5.2. The amount of volume loss after the wear test under 30N and 60N loads calculated from the data in Figure 5.3.

Composition	30N	60N
Fe+0,45C +0 AlC	0,18 mm^3	0,291 mm^3
Fe+0,45C+0,2 AlC	0,15 mm^3	0,255 mm^3
Fe+0,45C+0,5 AlC	0,66 mm^3	0,234 mm^3
Fe+0,45C+1 AlC	0,039 mm^3	0,096 mm^3
Fe+0,45C+2 AlC	0,021 mm^3	0,081 mm^3

Figures 5.2 and 5.3 show the values obtained as a result of the wear test applied to the samples produced by powder metallurgy. In Table 5.2, the wear volume values given in Figure 5.3 are the volume losses obtained by multiplying the stroke value of 3 mm. As it can be seen in Table 5.2, when the wear volume loss values are examined, it is seen that it is compatible with the hardness result and the wear volume loss decreases with the increasing AlC addition. It is seen that the wear volume loss decreased with the increasing AlC addition in the 30N and 60N load values applied in the wear test.

When we examine Figure 5.1, it cannot always be mentioned that there is a correct relationship between the depth of wear and the loss of volume. However, the alloy with the highest wear depth is the Fe+0,45C+0AlC alloy with the lowest hardness and the highest wear volume loss. While the wear scar of this alloy under 30 N load has a depth of 80 microns, the wear scar under 60 N load has an average depth of 100 microns.

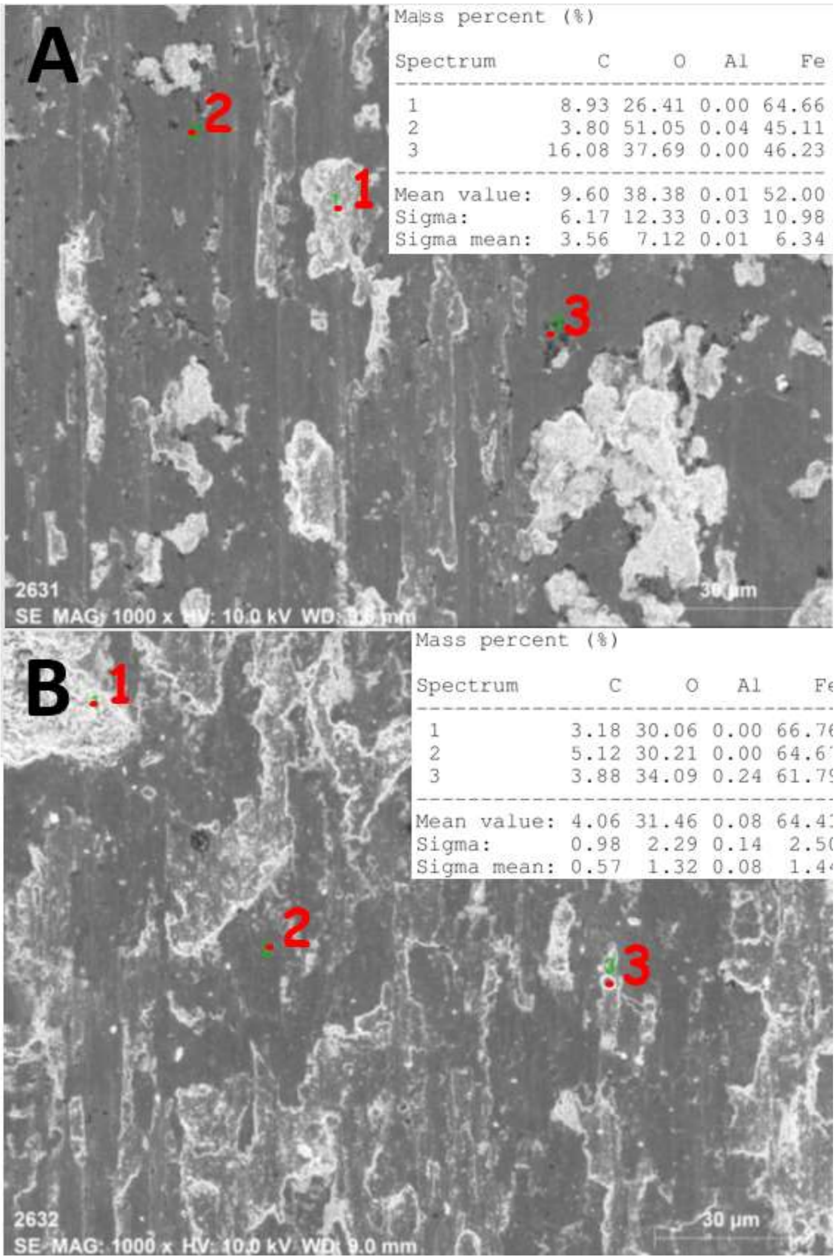
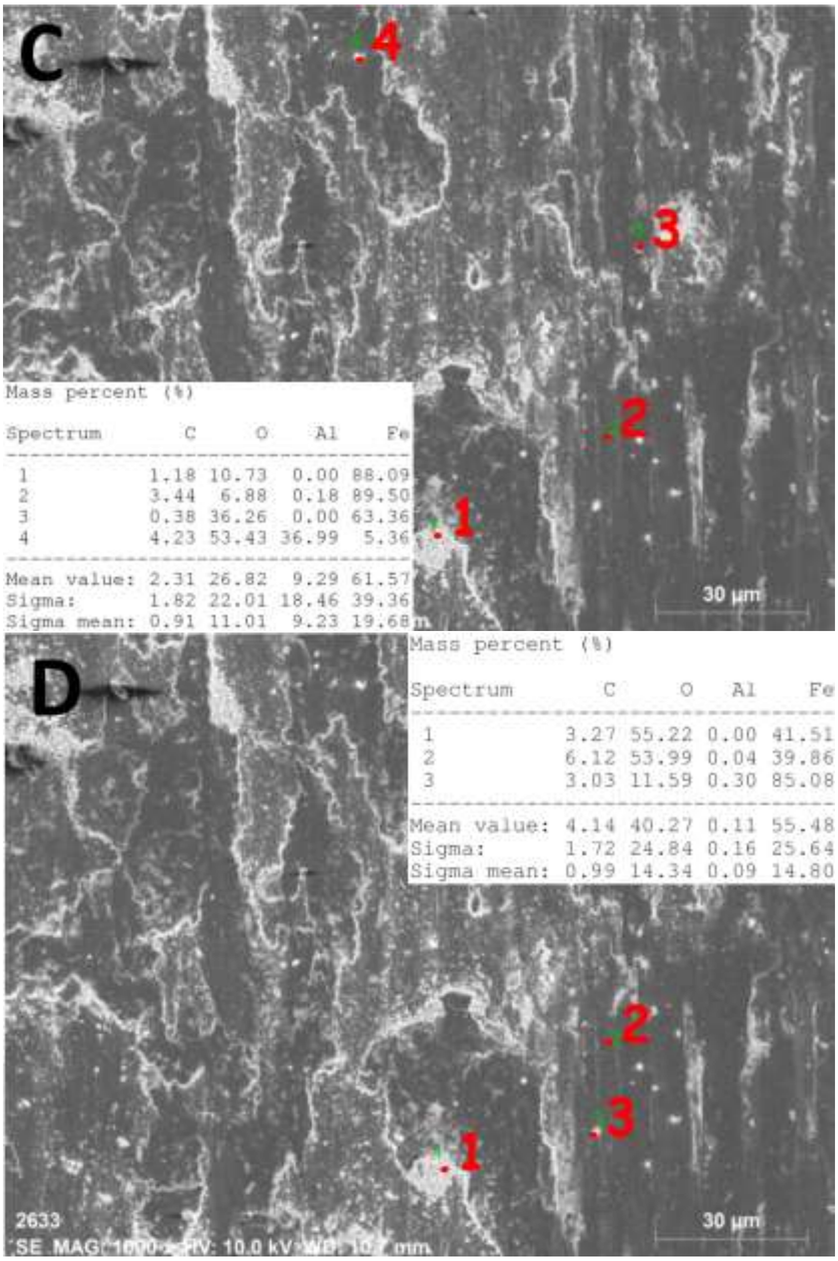


Figure 5.4. Wear surface images of the samples after the wear test under 60 N load, a) Fe+0,45C, b) Fe+0,45C+0,2AlC c) Fe+0,45C+0,5AlC, d) Fe+0,45C+2AlC.



Mass percent (%)

Spectrum	C	O	Al	Fe
1	1.18	10.73	0.00	88.09
2	3.44	6.88	0.18	89.50
3	0.38	36.26	0.00	63.36
4	4.23	53.43	36.99	5.36
Mean value:	2.31	26.82	9.29	61.57
Sigma:	1.82	22.01	18.46	39.36
Sigma mean:	0.91	11.01	9.23	19.68

Mass percent (%)

Spectrum	C	O	Al	Fe
1	3.27	55.22	0.00	41.51
2	6.12	53.99	0.04	39.86
3	3.03	11.59	0.30	85.08
Mean value:	4.14	40.27	0.11	55.48
Sigma:	1.72	24.84	0.16	25.64
Sigma mean:	0.99	14.34	0.09	14.80

Figure 5.4. Continues.

SEM image and EDS analysis were taken from the wear surfaces after the wear test performed at a sliding distance of 25 m under 30 N and 60 N loads, sintered at 1350 °C. SEM images were taken at 1000x magnification from the samples exposed to abrasion under 60 N load. EDS analysis was also taken from these samples and is shown in Figure 5.4. When the images are examined, it is seen that the abrasive ball generally forms lines parallel to the sliding direction. These lines show that the steel produced by the powder metallurgy method is exposed to the abrasive wear mechanism (Taşlıyan, 2020). It has been determined that there are wear channels in all materials in the SEM images taken from the wear surfaces. However, it is observed that the wear lines become deeper and wider depending on the sliding distance and the increase in the amount of applied load. In the SEM EDS analysis results, it was determined that the AlC particles were found in the form of particles on the wear surface and in the matrix. In addition, point EDS analysis results show that AlC precipitates are formed because these precipitates contain Al and C elements.

5.4. GENERAL CONCLUSIONS AND RECOMMENDATIONS

5.4.1. Overall Results

- Steel produced from Fe+0,45C+X AlC steel to which AlC was added by powder metallurgy method was supplied.
- In general, ferrite and pearlite were observed in the microstructure. An increase in the amount of perlite was observed with increasing AlC amount.
- In general, since the amount of AlC inhibited grain growth, finer grains were obtained with increasing AlC content, and this was reflected in the mechanical properties.
- The wear test values were directly proportional to the hardness results. The lowest wear volume and the lowest wear depth were obtained in the sample with the highest hardness.

5.4.2. Suggestions

- If large samples are produced, mechanical properties can be characterized in detail by performing tests such as tensile test and fatigue test.

REFERENCES

Askeland D. R., “The science and engineering of materials”, ***Chapman and Hall***, Third S. I. Edition, UK (1996).

Ateş, S., Uzun, İ., Çelik, V., Basınçlı İnfilitrasyon Yöntemi ile Üretilmiş SiC/Al 2014 Kompozitin Isıl İletkenliği Üzerine İnfilitrasyon Sıcaklığının Etkisi, ***6th International Advanced Technologies Symposium (IATS'11)***, 16-18, 2011, Elazığ- Türkiye.

Bakkali E. H. F, Chenaouia A, Dkiouaka R, Elbakkalib L. A. O. A., “Characterization of deformation stability of medium carbon microalloyed steel during hot forging using phenomenological and continoum criteria”, ***J Mater Proc Tech,*** 140–149 (2008).

Blank, J. R., and Gladman, T., “Quantitative metallograpy, tools and technique in physical metallurgy”, ***Marcel Dekker***, New York, 251 (1970).

Cuddy L. C., Raley J. C., “Austenite grain coarsening in microalloyed steels”, ***Metall. Trans.*** *A*, 14: 1989-1995 (1983).

Çengelli B., ***Sicak İzostatik presleme***, http://www.takimceligi.com (2014).

DeHoff, R.T., Rehines, F.N, “Quantitative microscopy”, ***McGraw-Hill***, Inc., USA (1968).

Du, B., Zou, Z., Wang, X. & Li, Q. “In situ synthesis of TiC-TiB_2 reinforced FeCrSiB composite coating by laser cladding” ***Surface Review and Letters***, 14:315-319 (2007).

Dutta B, Sellars C. M. “Strengthening of austenite by niobium during hot rolling of microalloyed steel”, ***Mater Sci Technol.***, 2, 146–153 (1986).

Doğan Ö. N., Hawk, J. A., Tylczak J.H, Wilson R.D., Govier R.D., “Wear of titanium carbide reinforced metal matrix composites”, ***Wear***, 225-229: 758-796 (1999).

Özdemirler, D., Gündüz, S., Erden, M.A., Karabulut, H., Türkmen, H., “Sinterleme sıcaklığının NbC ilave edilmiş TM çeliklerinin mikroyapı ve mekanik özelliklerine etkisi”, ***Afyon Kocatepe Üniversitesi Fen ve Mühendislik Bilimleri Dergisi,*** Özel Sayı 92-97, Afyon (2016).

Erden M. A., Gündüz S., Türkmen M., Karabulut H., “Microstructural characterization and mechanical properties of microalloyed powder metallurgy steels”, ***Materials Science and Engineering: A***, 616: 201-206 (2014).

German, R.M., “Powder metallurgy science”, 2nd edition, ***Metal Powder Industries Federation***, USA, 1-61 (1994).

Gladman, T. and Woodhead, J. H., “The accuracy of point counting in metallographic investigations” ***Journal of the Iron Steel***, 194, 189 (1960).

Gladman, T., “Physical Metallurgy of Microalloyed Medium Carbon Engineering Steels”, ***Ironmaking and Steelmaking***, 16, 241 (1989).

Galadman T., “Grain refinement in multible microalloyed steels. In: HSLA Steels Properties and Applications the Minerals”, ***Metals and Materials Society***, 1, 3-14 (1992).

Gilman, P.S. Benjamin, J.S. Ann. ***Rev. Mater. Sci.*** 13:279 (1983).

Gladman, T., “The physical metallurgy of microalloyed steels”, ***The Institue of Materials***, England, 1, 341 (1997).

George,F., Voort, V, “Practical applications quantitative metallography”, ASTM 839, Philadelphia, PA (1994).

Guang X., Xiaolong G., Guojun M., Feng L., Hang Z. “The development of Ti alloyed high strength microalloy steel”, ***Materials and Design***, 31, 2891–2896 (2010).

Gündüz S. and Cochrane R.C., “Clustering effect on high temperature tensile behaviour of vanadium microalloyed steel”, ***Journal of Materials Processing Technology***, 186, 246–252 (2007).

Jia Z., Misra R. D. K., Malley R. O., Jansto S. J., “Fine-scale precipitation and mechanical properties of thin slab processed titanium–niobium bearing high strength steels”, ***Mater. Sci. Eng. A***, 528: 7077-7083 (2011).

Kim, N. J., “The Physical Metallurgyof HSLA Line Pipe Steels”, ***A Reviev, J. Metals***, pp. 21-30 (1983).

Korczynsky, M., “Microalloying and thermo-mechanical treatment” In: DeArdo, A.J., Ed., ***Proceedings of International Symposium Processing***, “Microstructure and Properties of HSLA Steels”, Pittsburgh, 169-201 (1988).

Kostryzhev AG, Al Shahrani A, Zhu C, Cairney JM, Ringer SP, Killmore CR et al. “Effect of niobium clustering and precipitation on strength of an NbTi-microalloyed ferritic steel”, ***Mater Sci Eng A.***; 607: 226–235 (2014).

I.A. Petrusha, T.I. Smirnova, A.S. Osipov, V.F. Britun,Diamond Relat. ***Mater.*** 13 (2004).

Li, Y., Wilson, J. ACrowther, D. N., Mitchell, P. S., Craven, A. J. and Baker, T. N. “The Effects of Vanadium, Niobium, Titanium and Zirconium on the Microstructure and Mechanical Properties of Thin Slab Cast Steels”, ***ISIJ International***, 44(6): 1093–1102 (2004).

Llewellyn D. T., Hudd R. C., “Steels: metallurgy and applications”, 3th edn, ***Reed Educational and Professional Publishing Ltd***, Oxford (1998).

Matik U, “Akımsız nikel kaplamalarda ısıl işlemin sertlik ve aşınma özelliklerine etkisi”, Doktora Tezi, ***G.Ü. Fen Bilimleri Enstitüsü***, Ankara, 1-50 (2010).

Narita, K., “Physical Cemistry of The Groups Iva (Ti, Zr), Va (V, Nb, Ta) and The Rare Earth Elements in Steel”, ***Transaction ISIJ***, 15: 145 (1975).

Najafi H., Rassizadehghani J., Halvaaee A., “Mechanical properties of cast microalloyed steels containing V, Nb and Ti”, ***Mater. Sci. Tech.***, 23: 699–705 (2007).

Ollilainen, V., Kasprzak W., Hollapa L., **“**The effect of slicon, vanadium and nitrogen on the microstructure and hardness of air cooled medium carbon low alloy steel*”*, ***Journal of Metarials Processing Technology***, 134: 405-412 (2003).

Pickering F. B., Gladman T. “Metallurgical developments in carbon steels”, ***Iron and Steel Inst***., 81: 10-20 (1963).

Schade C, Murphy T, Lawley A, Doherty R., “Microstrucure and mechanical properties of microalloyed PM steels”, ***Int J of Powder Metall***. 48: 51-59 (2012a).

Schade, C., Murphy, T., Lawley A., Doherty R., “Microstructure and mechanical properties of PM steels alloyed with silicon and vanadium”, ***International Journal of Powder Metallurgy,*** 48 (6): 41-48 (2012).

Taşlıyan, M.F., “Toz metalürjisi ile üretilen mangan çeliğine TiC, TiN, TiCN ilavesinin mikroyapı ve mekanik özelliklere etkisinin araştırılması” ***Karabük Üniversitesi Fen Bilimleri Enstitüsü, Yüksek Lisans Tezi,*** Karabük (2020).

Türker, M., Özdemir, T., Ögel, B., ve Yavuz, A., “Al-SiC tozlarının mekanik alaşımlama değirmeninde öğütme zamanının kompozit toz yapısına etkisinin araştırılması*”*, ***Uluslararası Katılımlı 2. Ulusal Toz Metalurjisi Konferansı***, Ankara, 425-430 (1999).

Jung J. G, Park J. S, Kim J., Lee Y. K., “Carbide precipitation kinetics in austenite of a Nb–Ti–V microalloyed steel”, ***Mater Sci Eng A.***, 528: 5529-5535 (2011).

Qing-yun S., Gui-yan L., Li-feng Q., Ping-yuan Y., “Effect of cooling rate and coiling temperature on precipitate in ferrite of a Nb-V-Ti microalloyed strip steel”, ***Proceedings of Sino-Swedish Structural Materials Symposium***, 316-319 (2007).

W.A. Groen, M.J. Kraan, P.F. van Hal, A.E.M. De Veirman, ***J. Solid State Chem***. 120 (1995) 211.

Wang, Y. S., Zhang, X. Y., Fengchun L., Guangting Z., “Study on and Fe-TiC surface composite produced in situ”, ***Mater. Des***., 20, 233-236 (1999).

Wang, J., Wang, Y., Ding, Y., “Production of (Ti, V)C reinforced Fe matrix composites”, ***Materials Science and Engineering A***, 445-455, 75-79 (2007).

Yıldırım, M. M., Buytoz, S. and Ulutan, M., “Mikrostrukutrelle anderungen an SiC beschichteten und mit WIG schweistechnik erzeugten metalloberflacheneines 45Mn5 stahls”, ***Prakt. Metallogr***., 44, 59-69 (2007).

Xiang-done H., Xin-ping M, Sheng-xia L., “Effect of annealing temperature on recrystallization behavior of cold rolled Ti-microalloyed steel” ***J. of Iron and Steel Res Int***., 20 (9): 105-110 (2013).

RESUME

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