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DEPARTMENT OF NANOSCIENCE AND NANOTECHNOLOGY



PRODUCTION AND CHARACTERIZATION OF AL-SI HYBRID
NANOCOMPOSITES

Ph.D. Thesis

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ACCEPTANCE AND APPROVAL OF THE THESIS

The study entitled “**PRODUCTION AND CHARACTERIZATION OF AL-SI HYBRID NANOCOMPOSITES**” prepared by **Mohammed Jabbar Fouad ALOBAIDI** and supervised by **Assist. Prof. Dr. İbrahim İNANÇ** was found successful and unanimously accepted by committee members as Ph.D. thesis, following the examination on the date 8.8.2023

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ÖZET

AL-Sİ HİBRİT NANOKOMPOZİTLERİN ÜRETİMİ VE KARAKTERİZASYONU

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Ondokuz Mayıs Üniversitesi
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Doktora, Tammuz/2023
Danışman: Dr. Öğr. Üyesi İbrahim İNANÇ

Bu tez Al-Si'yi temel matris kompoziti olarak kullanıyor ve MoS₂ ve BN hibrit nanopartiküllerin eklenmesiyle, kendi kendini yağlayan parçalar üretmeyi amaçlamaktadır ve üretim sürecinde, metal matrisli nanokompozitlerin (MMNC'ler) üretimi için tercih edilen bir strateji olan Toz metalurjisi teknolojisi kullanılmaktadır. Alüminyum alaşımları, analiz ve üretim tahminlerinden temel alaşımlar olarak MMNC'lerde yaygın olarak uygulanan kompozitlerden biridir. Aşındırıcı bir malzeme olan Bor Nitrür (BN), uzun bir servis ömrüne ve hissedilir aşınma direncine sahiptir. Molibden disülfür (MoS₂) iki boyutlu bir yarı iletken madde olabilir; yoğunluğu ve aşınma önleme özelliklerini sahiptir, yapışmayı güçlendirir ve aşınmayı azaltır. Bu sayede ağır ısı ve sıkıştırma şartlarında katı yağlayıcı olarak kullanılabilen bir yağlama özelliğine sahiptir. BN'nin termal stabilitesi elmasan çok daha fazladır ve demir bileşenlerden daha mükemmel kimyasal stabiliteye sahiptir. Bu nedenle BN, bileşenlerin korunması için kullanılmaya, aşınmaya ve korozyona karşı korumaya uygundur.

Alaşımlama prosedürü için en uygun koşulları elde etmek amacıyla çeşitli deneyler hazırlanmıştır. Numuneler XRD, SEM, EDS, fiziksel muayene, mikrosertlik, triboloji gibi farklı yöntemlerle karakterize edilmiştir. Bu çalışmada, Al-Si'nin baz toz alaşımına MoS₂ ve h-BN nanopartiküllerinin ağırlıkça % 2, 4 ve 6 oranında katılım ile kullanıldı. Tribolojik karakterizasyonun öncesinde, mekanik frezeleme, 7 tonluk presleme ve 550 °C'de sinterleme yoluyla yeni, olağanüstü nanokompozitler üretilmiştir. Üretilen parçaların mükemmel kendi kendine yağlanması, yüksek sertliği ve iyi yüzey kalitesi, hibrit ilavesi % 4'e çıktığında aşınma oranının azalmasına neden olduğu sonucuna varılmıştır.

Anahtar Sözcükler: Al-12 % Si, Nanokompozitler, Toz Metalurjisi, Kendinden Yağlama, Aşınma.

ABSTRACT

PRODUCTION AND CHARACTERIZATION OF AL-SI HYBRID NANOCOMPOSITES

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Ph.D., July/2023

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This thesis uses the Al-Si as a base matrix composite, adding the hybrid nanoparticles of MoS₂ & BN. It aims to fabricate self-lubrication parts, and the manufacturing process utilizes the technology of powder metallurgy is a favored strategy for manufacturing metal matrix nanocomposites (MMNCs). Aluminum alloys are one of the widely applied composites in MMNCs as base alloys from analysis and manufacturing estimation. As an abrasive material, Boron Nitride (BN) has a long service life and sensible wear resistance. The Molybdenum disulfide (MoS₂) could be a 2-dimensional semiconductor matter; it has anti-wear properties, reinforces cohesion, and reduces abrasion. Thus, it has a greasing realization that might utilized as a solid lubricator within severe heat and compression conditions. The thermal stability of BN is far more than that of diamond and has more excellent chemical stability than iron components. Therefore, BN is suitable to use for the protection of components, anti-wear, and anti-corrosion.

Several experiments have been prepared to achieve optimal conditions for the alloying procedure. The samples were characterized by different methods such as XRD, SEM, EDS, physical examination, microhardness, and tribology. This study used 2, 4, and 6 weight % addition of nanoparticles MoS₂ and h-BN to the base powder alloy of Al-Si. Before tribological characterization, novel, outstanding nanocomposites are produced by mechanical milling, 7-ton pressing, and sintering at 550 °C. It is concluded that the excellent self-lubrication, high hardness, and good surface finishing of produced parts caused the wear rate to decrease whenever the hybrid addition increased to 4 %.

Keywords: Al-12 % Si, Nanocomposites, Powder Metallurgy, Self-Lubrication, Wear.

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SYMBOLS AND ABBREVIATIONS

AFM	: Atomic Force Microscopy
Al	: Aluminum
AMCs	: Aluminum Matrix Composites
AMNC	: Aluminum Matrix Nano Composites
Ar	: Argon
ASME	: The American Society of Mechanical Engineers
ASTM	: American Society for Testing of Materials
BN	: Boron Nitride
BNNp	: Boron Nitride Nanoparticles
BNNT	: Boron Nitride Nano Tube
CNT	: Carbon Nano Tube
EDS	: Energy-Dispersive Spectroscopy
h-BN	: Hexagonal- Boron Nitride
HV	: Vickers Hardness
MMC	: Metal Matrix Composite
MMNCs	: Metal Matrix Nano Composites
MoS ₂	: Molybdenum disulfide
M-BN	: Modified Boron Nitride
MWCNT	: Multi Wall Carbon Nano Tube
NT	: Nano Tube
PM	: Powder metallurgy
SEM	: Scanning Electron Microscopy
Si	: Silicon
SMP	: Shape Memory Polymer
SPS	: Spark Plasma Sintering
TiO ₂	: Titanium Dioxide
XRD	: X-Ray Diffraction

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1. INTRODUCTION

Metal Matrix Nano Composites (MMNCs) of Aluminum are the foremost widely used matrix because of their denseness, high stiffness, high specific strength, high specific modulus, and low thermal expansion coefficient. In recent projects, the hybrid in nanocomposite refers to double or triple nano oxides, nitrides, or other addition to induce new properties. Powder metallurgy (PM) could be a methodology that permits the preparation of qualitatively new Al-matrix alloys. We selected Al-Si alloys attributable to their low denseness, high wear resistance, wise specific strength, and stiffness in conjunction with low thermal expansion. These are widely utilized in various engineering applications like automobile, electronic, and aerospace industries.

Comparing micron-sized and nano-sized particles, nano has several benefits. A smaller particle size would offer a far larger surface area for molecular collisions and increase the reaction speed creating an improved catalyst and chemical. An increase in wear behavior signals a greater risk of component damage. In modern research, all scientists tried to decrease the energy loss and consumption of materials resulting from friction of countered components. The service life of a lubricating substance in a system influences friction and wear behavior. Choosing the nanoparticles as an additive material to oil are let us pay more money for every change to the oil of the engine, but if we are thinking of a new alloying element of manufacturing parts, that will make it cheaper in the long scope. Now days, we have electrical vehicles with a moving components, which means the problem of friction will appear.

1.1 The aim of this research:

1) Manufacture advanced moving components without lubrication by the fabrication process of the hybrid nanocomposites (Al-12wt%Si) matrix reinforced with nanoparticles of (MoS₂ & BN) by using the mechanical milling and powder metallurgy techniques,

2) Study the wear behavior of the produced nanocomposites under dry sliding conditions.

3) Study the topography of worn surfaces of nanocomposites by using scanning electron microscopes.

4) Using Boron nitride with nanocomposites instead of graphite, the effect of white lubricant. That will produce the engine parts, less expensive materials, and be more effective in producing a new engine with clean energy.

1.2 Project Plan

The work plan starts with nanocomposite as powder preparation and the tests in all stages to reach the characterization of wear and self-lubrication of Al-Si matrix hybrid nanocomposite.

The flow chart in figure 1.1 represents the start base alloy of Al-12Si that is achieved from preparing samples for the tests. The mixing by ball mill to produce the nanocomposites of 2, 4, and 6% of hybrid addition of MoS₂ and BN, it comes after preparing the alloy of Al-12% Si. The manufacturing process and investigations are in the diagram below for the research steps.

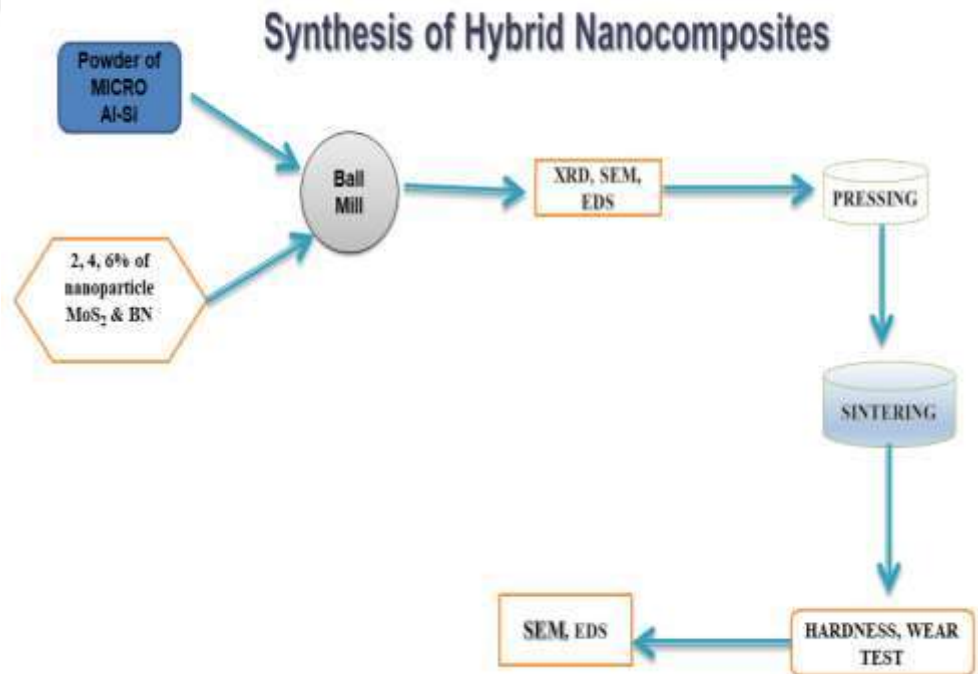


Figure 1.1. The manufacturing process and investigations of Al-Si matrix hybrid nanocomposite

2. THEORETICAL PART

The micro and nanomaterials have different properties, Aluminum and Silicon in the micro-size have many applications. Aluminum-Silicon alloys, also known as Silumin, are a class of lightweight, high-strength aluminum alloys based on the AlSi system, with silicon as the quantitatively most significant alloying element, as well as for nanoscale addition for MoS₂ and BN. In this chapter, we have reviewed the manufacturing process of Metal Matrix Nanocomposites (MMNs) and tribology science with its divisions, especially the wear and lubrication.

2.1. Aluminum-Silicon Alloy

Al-Si alloys show high efficiency in mechanical applications, engine parts manufacturing, and aerospace equipment, according to their tribological properties, elevated strength-to-weight ratio, low thermal expansion, and low corrosion. 3% to 25% silicon is frequently present in Al-Si alloys. Al-Si alloys are primarily used in casting, although they can also be used in powder metallurgy and quick solidification techniques. Even more silicon may be present in alloys used in powder metallurgy instead of casting—up to 50%. Silumin is effective in humid situations due to its excellent corrosion resistance. (Key to Metals, 2012).

In their study, M. J. Fouad and M. K. Abbass showed that the Al- alloy would resist wear by increasing the Si content to the eutectic limit of 12 wt.%. From (S.P. Nikanorov et al. 2005), the hyper-eutectic alloys which goal for wear-resistant applications, the phase diagram of Al-Si is shown in Figure 2.1.

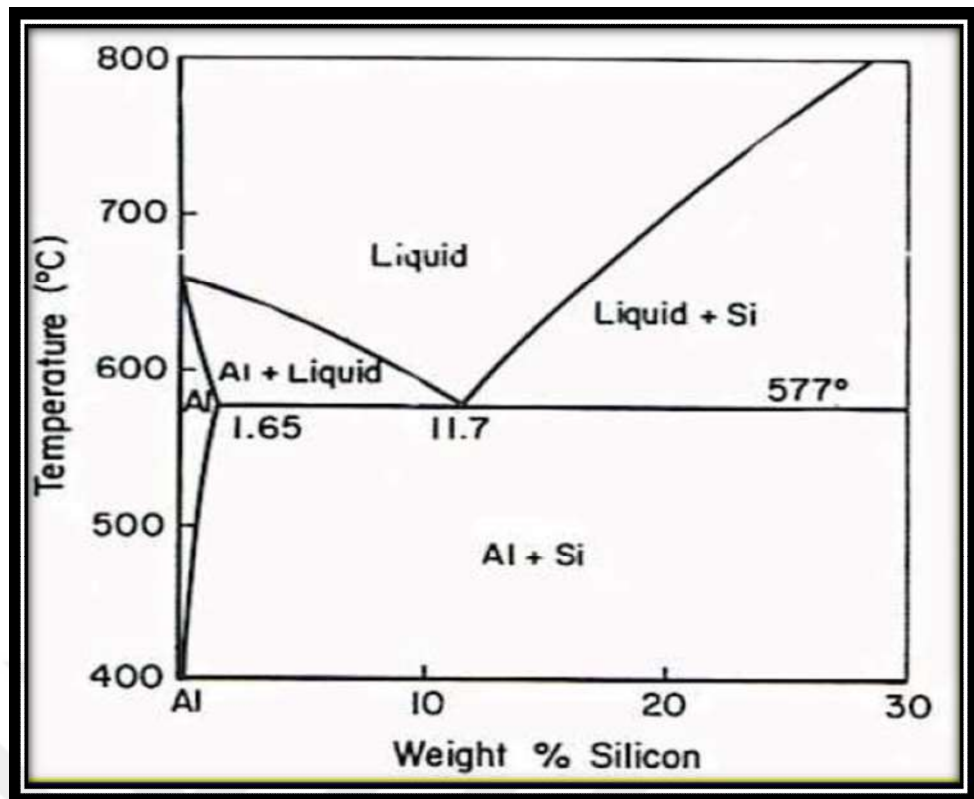


Figure 2.1. Aluminum-Silicon phase diagram (S.P. Nikanorov et al. 2005).

Increasing the Si in the alloy leads to an increase in the strain hardening of the powder, and thus the grain size is reduced. This effect can be described by hardening the solution as a result of the effect of increasing the proportion of Si. From, the Figure 2.2, we can observe the percentage of micro strain, crystal size, the effect of Si content, and the most important factor, the milling time. Of course, increasing the milling time with balls reduces the crystal size of the powder as a whole, but the presence of Si has a significant effect on this small crystal size. (A.H. Bahrami et al. 2013).

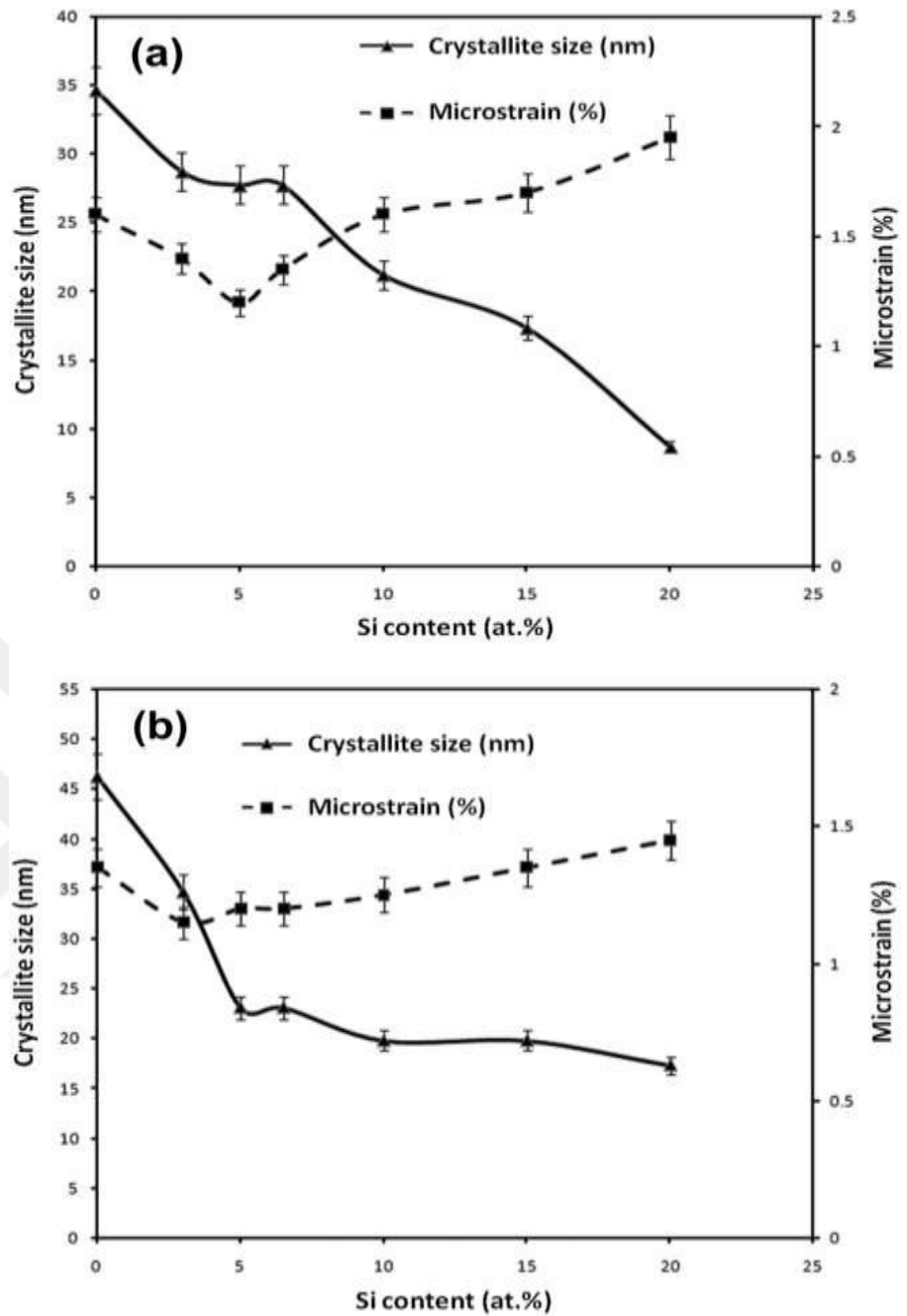


Figure 2.2. Effect of Si content on microstructure. (A.H. Bahrami et al. 2013)

2.2. Ceramics

Ceramics is a composite material; the best and most straight forward definition is the participation of two elements, one metal and the other non-metal. For this reason, ceramic materials consist of metals and their oxides, carbides, or metals with nitrides or sulfates, and others. It is a chemically stable substance with hybrid bonds between atoms, where they are ionic and

covalent bonds. As a result of these bonds, ceramic materials are characterized by properties such as high melting temperature, chemical stability, few dislocations, and difficulty in the movement. The last-mentioned property produces high hardness and great resistance to creep but gives the characteristic of brittleness. Ceramic compounds may contain several phases of two or more materials or two ceramic materials with different crystalline or microstructure sizes. (R. Warren, 1990).

2.2.1. Molybdenum Disulfide (MoS_2)

Nanostructures are receiving considerable attention because of their potential applications. Chhowalla et al. and Rapoport et al. 2005 have shown MoS_2 nanoparticles as solid lubricants at particular process parameters, showing extrextremecion and wear characteristics.

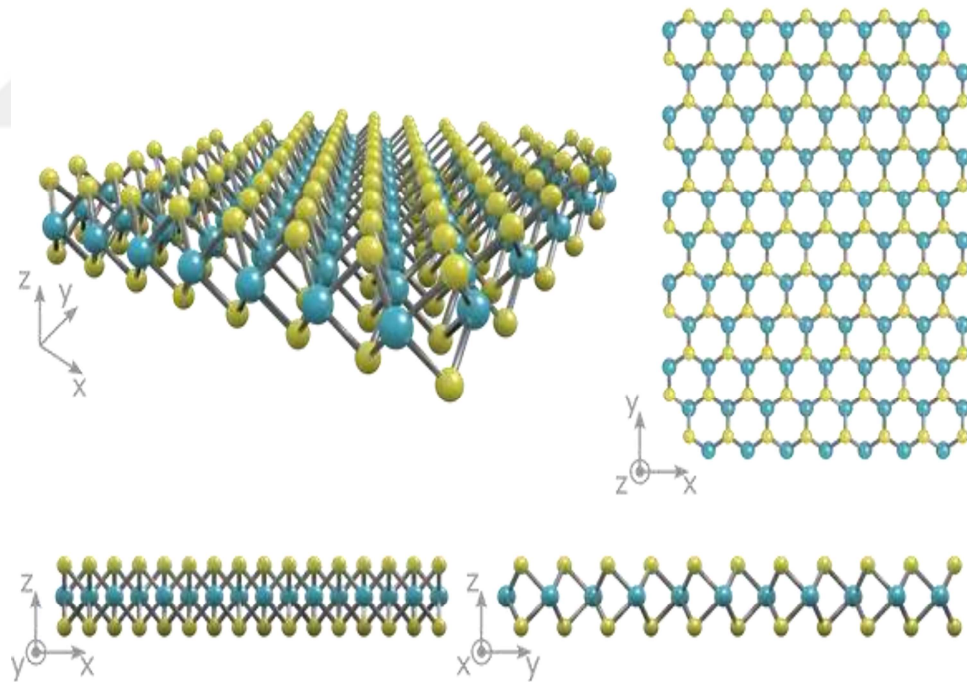


Fig. 2.3 The crystal structure of monolayer MoS_2 showing a layer of molybdenum atoms (blue) sandwiched between two layers of sulfur atoms (yellow). (Kin Fai Mak et al. 2010).

2.2.2. Boron Nitride (BN)

Hexagonal-Boron Nitride (h-BN) has several properties as a solid lubricant, and has a relatively low density, good thermal conductivity, high-temperature stability, and is also chemically stable.

BN is known as white graphite because it has a hexagonal structure similar to Graphite, and a crystalline structure that has high lubricating properties, and making it superior to Graphite, and it has the following specifications:

Because it has a coefficient of friction from 0.15 to 0.70, it is considered low and has good lubrication, Chemically stable, Electrical insulation, Does not retain heat stable at high temperatures above 1000°C and at higher temperatures up to 1800 °C with inert gas protection, low volumetric expansion, dimensional preservation.

BN comes in different nano shapes and sizes, including nano-sheets and nano-granules. (h-BN) is one of the best types of additives that improve the wear properties and reduce the friction of metal compounds and polymers because it has a multi-layer structure. We used Boron Nitride Nanoparticles (BNNP) in this research because of their exceptional hardness and abrasion resistance. It also has the characteristics of high toughness and chemical stability. Also, the production process has less energy consumption and less environmental pollution than other super hard materials. BN nanostructures are super reinforcing for lightweight MMNCs.

So far, the production of BN in nano-tube form and BN in graphene-like structure (BNNTs) in large quantities and with outstanding quality are considered to be a significant challenge. But the latter BNNTs have the advantage of high tensile strength and are ideal for strengthening nanostructures.

As we noted earlier, BN in nanostructure is manufactured in different forms and types, including the zero dimensions, nano-tubes are one-dimensional, similar to graphene in two dimensions, with a three-dimensional

diversity.

Also, a second classification of BN is in the form of nanowires, or nano fibers, nano assemblies, nano sheets, and polyhedral nanoparticles, and these are some of the shapes shown in Figure 2.4.

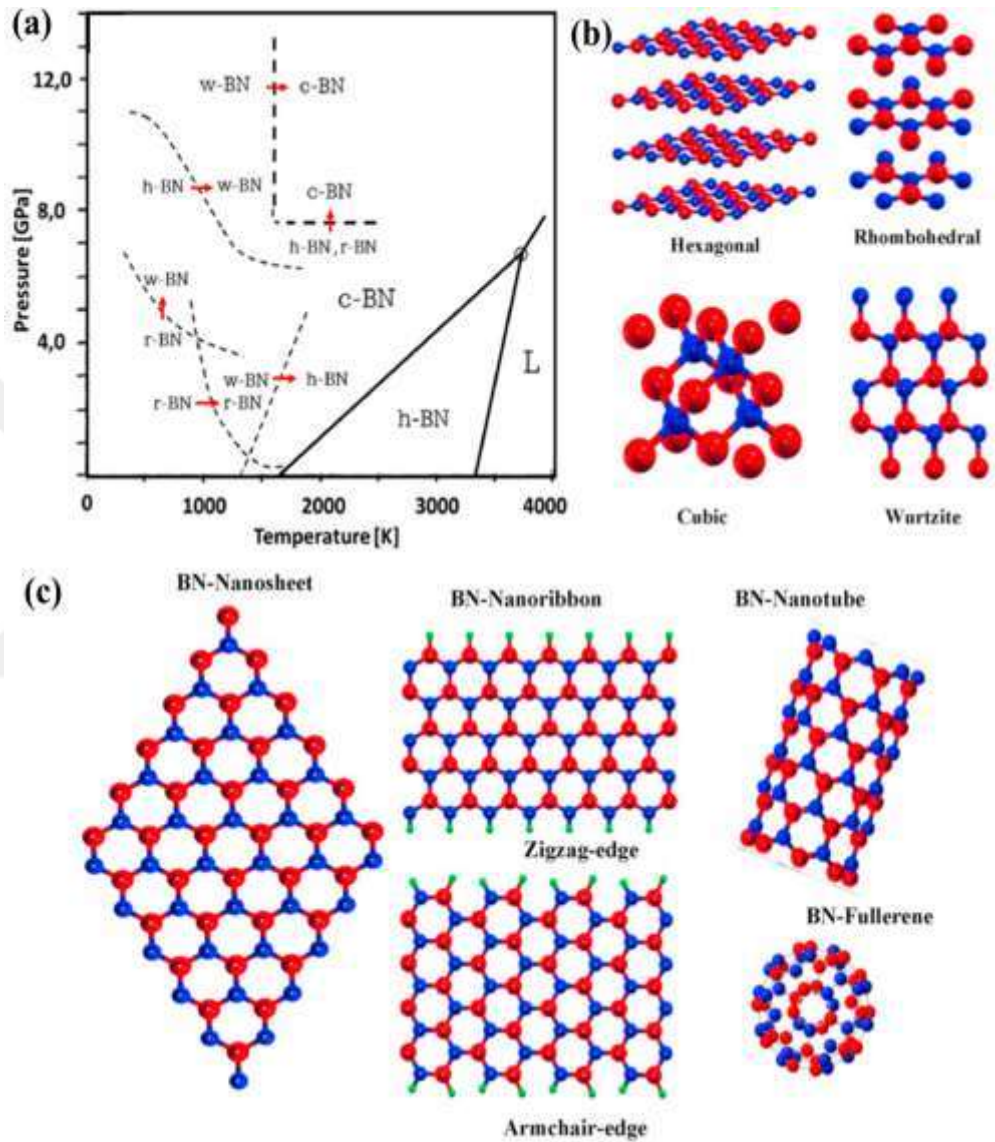


Figure 2.4. The phase diagram of BN and its various allotropes, a) phase diagram of BN, replicated with (Shayan, A. et al. 2022) b) various crystal structure of bulk BN, c) BN based nanostructures, (Arenal, R.& Lopez-Bezanilla, 2015).

2.3. Nanocomposite

A suitable definition of a nanocomposite is a material consisting of at least two immiscible phases isolated by an interface region. The matrix is the most widely used component of the nanocomposite as the filler is dispersed, (Nguyen et al. 2018).

The dominant part of the composite, tissue habit with properties to be strengthened, can define the matrix. The matrix is usually a metal, ceramic, polymer, or mixture with dimensions more significant than the nanoscale.

2.4. Manufacturing of MMNCs

Manufacturing methods used to produce metal matrix composite (MMC) can be casting or powder metallurgy (PM). However, PM has a high potential to avoid defects arising from casting processes and dimensional limitations. In the manufacture of composites of nanostructures as metal matrix nanocomposites (MMNCs), we avoid using casting because it may not give good mixing potential, isolation problems, or aggregates of agglomerates of nanoparticles, and therefore heterogeneity in the crystal structure, which reduces the mechanical properties that is desired to be achieved by improving specifications.

To obtain high mechanical specifications and wear resistance for the production of Aluminum alloys used in various applications, including self-lubricating bearings in engines or engine parts and valve seats in the automotive or aircraft industries, we used the powder technology as a manufacturing method. Different types of ceramics in nano sizes, or nanoparticles are added to strengthen the composite structure of Aluminum alloys. Among the materials used in previous research and practical applications in Aluminum matrix composites (AMCs) and Aluminum matrix nanocomposites (AMNCs) are the nanoparticles including Molybdenum Disulfide (MoS_2), Aluminum Nitride (AlN), Boron Carbide (B_4C), Alumina (Al_2O_3), Titania (TiO_2), Silicon Carbide (SiC), Boron Nitride (BN), Graphene, in addition to using Carbon nanotubes (CNTs).

The mechanical properties of using dispersed nanoparticles and

fabricating metal matrix nanocomposite (MMNCs) are better and higher than the mechanical properties resulting from using dispersed micrometer scale particles in the metal matrix composites MMCs. Only 1 % of SiC at a scale of 10 nm was added to the Al alloy, and 15 % of the same ceramic, but at a scale of 3.5 μm , was added. The output of the first is called MMNC, and the second is called MMC, and it was found in mechanical tests that the yield stress and tensile stress of MMNC are much higher than MMC.

2.5. Sintering

Sintering uses heat without air to prepare a product after pressing various metallic or ceramic powders. During sintering, agglutination and consistency of the granules that make up the powder occur in its wet phase, the process of raising the temperature to a level below the melting point of the main component of the alloy.

The diffusion process between the particles and the adjacent grains occurs during the solid state of the materials, and the final sintered product is of high density and almost free of gaps.

The sintering process gives the sintered parts a coherent structure, but the factors affecting this process must be carefully designed. Because sintering is a process of strengthening the microstructure by applying controlled heat energy while protecting it from external weather conditions such as moisture and oxygen sources. The affecting factors are most often controlled using a furnace system with a vacuum and a gaseous gas to protect the surface from interaction with oxygen.

2.6. Tribology

Tribology is the study of the engineering of surfaces subjected to relative motion, and this science has different classifications on which mechanical aspects depend, as the types of friction, lubricants, and mechanical wear that we call wear are applied, and it is what we seek to reduce in our study.

The geometry of surfaces subjected to relative movement in relation to tribology has important correlations that affect the product's expected life. One of these influences is surface topography, which is a very influential factor in bearings. The other influencing factor is the material from which the bearing is made, the value of the loads to which the bearing is exposed, and, as we mentioned above, the relative movement and the type of lubricant.

Friction is a mechanism that appears between opposite surfaces and is a resistance to the resulting movement between them. In some definitions, it is mentioned that friction appears between solid bodies where the force or load is perpendicular to the opposite surface.

But friction exists between different materials, whether they are solid, liquid, or gaseous, wherever they provide the load, such as the pressure applied, where the atoms rub against each other, and each friction generates thermal energy, and this energy may have adverse effects in most cases by causing it to consume and wear out the opposite surfaces.

The force of friction consists of a resistive force parallel to the direction of motion; there are two forces to generate friction, which are static friction force and kinetic force of friction. The static friction force is a tangential force to start sliding, while the kinetic friction force is a tangential force that we need to stay in a slipping state.

Wear is a mechanical corrosion that leads to the consumption of materials, caused mainly by sliding two objects on each other. Wear is an engineering problem that studies seek to solve because it leads to the failure of mechanical facilities and equipment subjected to movement. One of the contemporary solutions used is lubrication to reduce the contact of the contacting surfaces, which are subjected to relative loads and movement, or to make a spacer between the parts so that the bodies are not exposed to direct contact.

Friction and wear reduction are well-known issues affecting various materials, from engine and motor parts to medical instruments and micro-and sensor mechanisms. The importance of solid lubricants, as they are used

in mechanical parts to reduce friction and extend the life of the equipment.

The use of nanoparticles as a solid lubricant and a liquid in our time has given great economic importance. There are comprehensive tribological studies to diagnose the addition of particles or nanomaterials as lubricants and their effect on the final product by reducing friction losses. (M. Gulzar et al. 2016).

2.6.1. Wear Mechanisms

Adhesive actions, abrasion, fatigue methods, and oxidation processes are the four main mechanisms a material might wear under the mentioned scenarios. More complicated wear events involve the interaction of wear mechanisms, according to ASM Metals Handbook, Friction, Lubrication, and Wear Technology 1992.

Several properties and mechanisms frequently appear when surfaces subject to wear are examined. However, in most tribological systems, one type of mechanism is dominant or dominant over the other. One of the types of wear is adhesive or abrasion; the mechanism of the emergence of these types has causes or influences, including surface roughness and the type of material. Adhesive wear processes are most commonly associated with sliding. However, because of the slip that is commonly present in certain settings.

The adhesive wear mechanism occurs under rolling and shock conditions, depending on the surrounding conditions. The wear rates of these mechanisms are variable for several values of influences.

2.6.2. Wear Design

Determining the factors in designing a tribological system to achieve an acceptable wear mechanism is essential. The procedure to obtain this is called wear design. The goal is to achieve an acceptable low wear rate. Tribological design factors are material type, opposite surfaces, lubrication, and roughness. However, these are not all factors to consider when

designing. For example, loading, relative motion, and weather variables can sometimes be used as variables. (ASM Metals Handbook, Materials Selection and Design 1997).

Lubricants are used to separate two opposite surfaces to reduce friction and wear and also reduce the heat generated by friction. There are different types of lubricants, but the most widely used are oils in liquid form with chemical additives, and these additives improve the type of oil by reducing friction, extending the life of the oil, and bearing heat, and also from these additions are nanoparticles. Among the lubricants, nanoparticles are used with metal alloys, and there are some uses for gas lubricants.

Solid lubricants are materials that have the ability to reduce friction due to the ability of the material's grains to reduce shear strength within the material composition itself. Among these mineral materials, the most famous are graphite, boron nitride, and molybdenum disulfide, so these materials have atomic layers that absorb the shear stresses inside them. Also, noble metals are used as hard lubricants, as well as ductile metals that have resistance to low shear stresses.

3. LITERATURE REVIEW

M. J. Fouad 2013 used Al-12wt % Si as the base alloy and added a hybrid of Al_2O_3 and TiO_2 nanoparticles to strengthen the structure of the alloy compounds and increase the hardness and wear resistance of the nanocomposites.

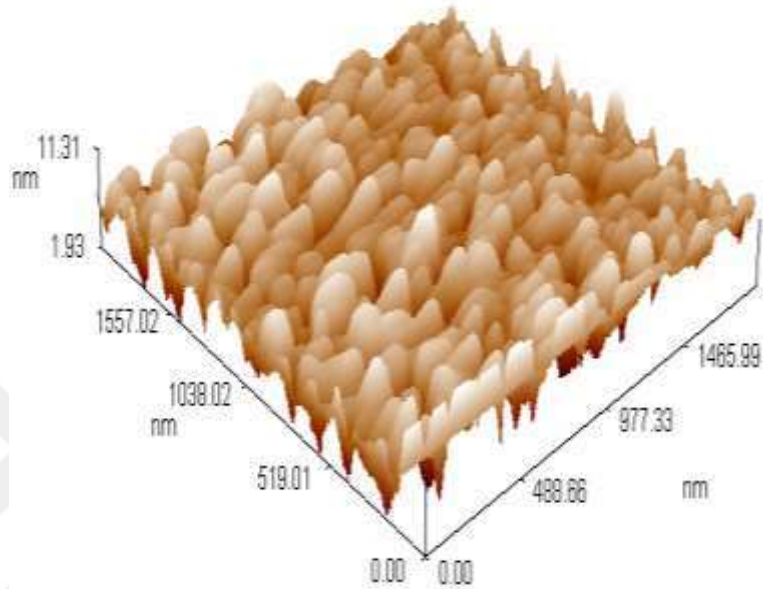


Figure 3.1 AFM results for mixed powders of hybrid nanocomposite of base alloy (Al-12%Si) +4 wt % (Al_2O_3 + TiO_2), (M. J. Fouad, 2013).

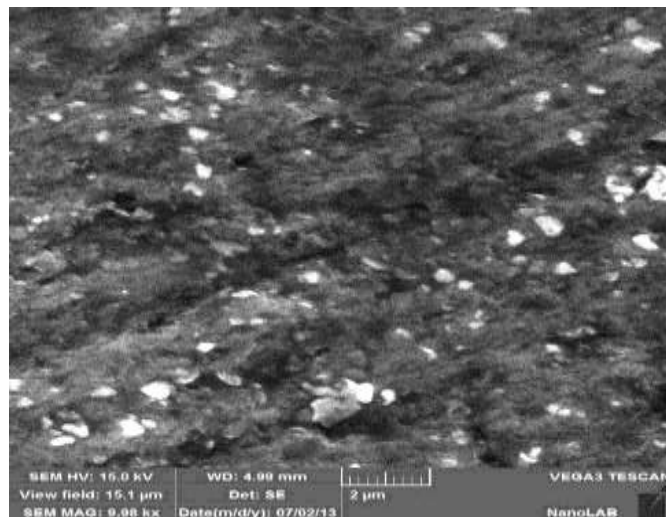


Figure 3.2 SEM micrographs for sintered MMNC sample of Al-12 % Si +4 wt % (Al_2O_3 + TiO_2) hybrid nanoparticles; at 10 kX. (M. J. Fouad, 2013)

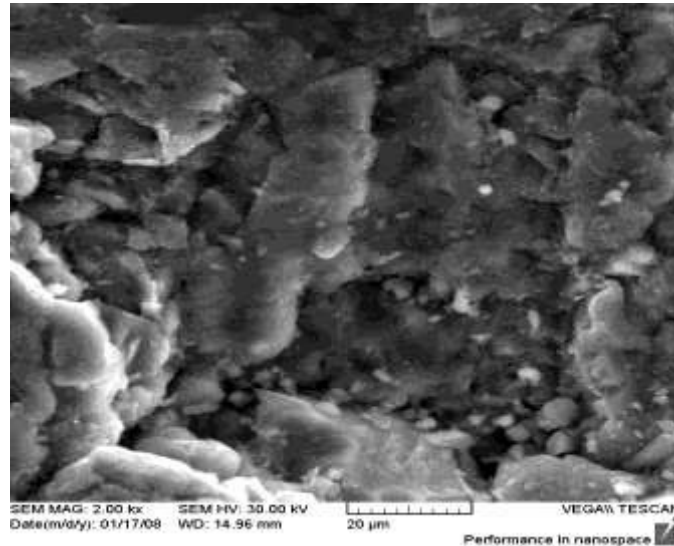


Figure 3.3 SEM micrographs of worn surface of hybrid nano composite with 6 wt % ($\text{Al}_2\text{O}_3+\text{TiO}_2$) nanoparticles under a normal load (12.5 N); at 2 k x. (M. J. Fouad, 2013)

Use Alumina balls for the mixing process, producing an even distribution of nanoparticles in Al-12wt%Si alloy. Note an increase in the density to reach near the molded density with an increase in the percentage of addition of nanoparticles to the main alloy. The percentage of porosity in all samples was less than 1%, which indicates a close final specification for the shape of the cast without gaps.

Diagnosis by atomic force microscopy (AFM) of the mixed powder by ball-mixing method or mechanical mixing between micro- and hybrid nano-particles indicates homogeneity and good distribution. While examining the wear using the (pin-on-disc) method and increasing the applied weight and the friction time, we find an increase in weight loss for all test samples, but the wear rate is lower for the samples with hybrid nanocomposites added.

When examining the wear of different samples, which is the base alloy and additives 2 %, 4 %, and 6 %, an improvement in wear resistance was found after nano-hybrid additives consisting of Alumina and Titania.

George et al. 2005, used balls for mixing Al powder with multiwall Carbon Nano tube (MWCNT), the mixing time was only 5 min, and they

used a cylindrical mold to press the mixture with a pressure of 120 kN. The sintering process was carried out at 580°C for 45 min under atmospheric protection by N₂ gas. They concluded that the mixing process did not damage the CNT, nor did chemical reactions occur, so no carbide phases were found.

In one study of M. Khorshid 2016, a nanocomposite was made of Aluminum and Graphene, and the weight additives were in various proportions using powder technology. The effect of adding Graphene on the tribological properties of the nanocomposite product, including self-lubrication, was investigated. Under the dry wear test conditions, it was found that the addition ratio of 1 % was preferable for improving the self-lubricating specifications of the alloy manufactured by adding Graphene as a nano-strengthening element to the Aluminum.

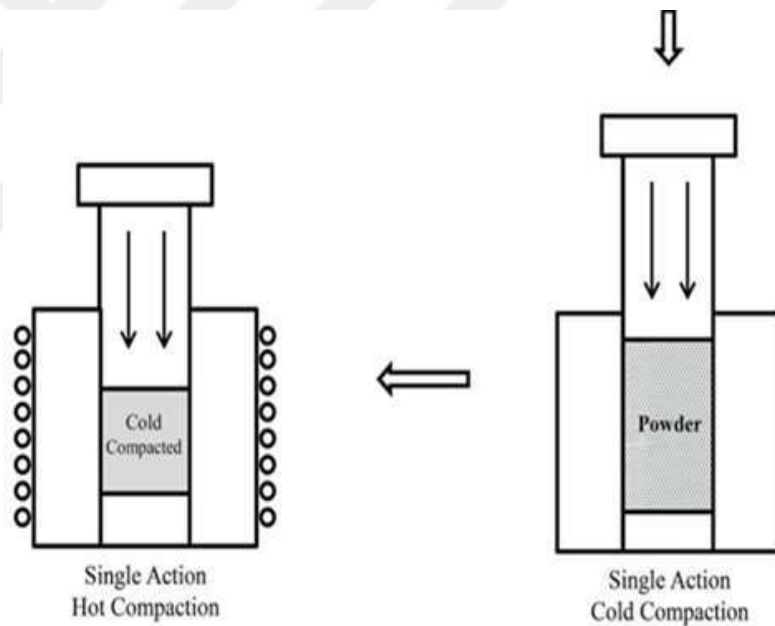


Figure 3.4 The two stages of molding process; cold & hot compaction. (M. Khorshid, 2016)

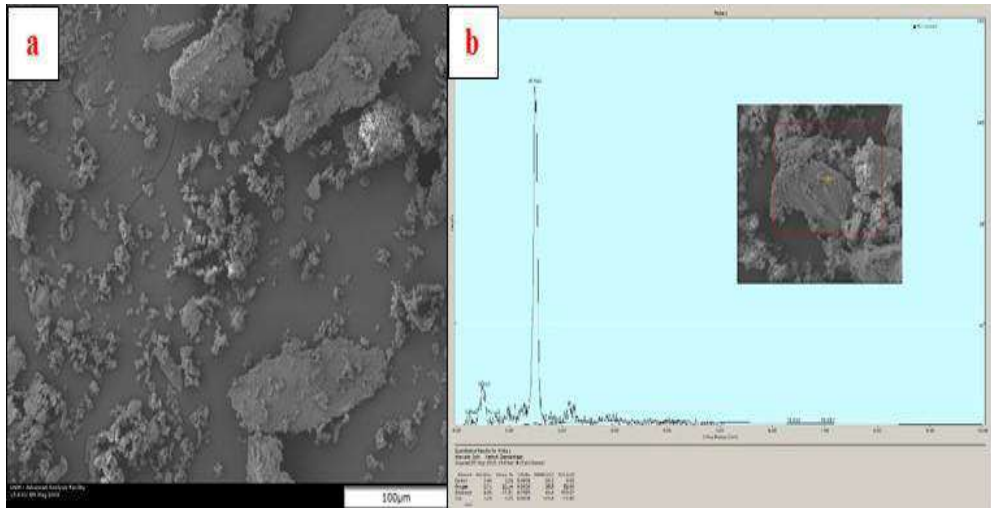


Figure 3.5 (a) Micrograph by scanning electron microscope (b) EDS of wear debris for Al with 1 % of Graphene nanoparticles. (M. Khorshid, 2016)

In research by W. Han et al. 2017, adding Boron Nitride nanoparticles improved the wear resistance of AA6082/TiB₂ composite Aluminum alloy with Titanium and BN as the addition as a solid lubricant and produced a three-layer composite with new properties.

K. L. Fierstein et al., in 2017, prepared a composite of Al and added to it Boron Nitride in the nano form and the micro type, and determined the addition of proportions to examine the mechanical properties with the highest specifications. MMCs are made from an Al base by Spark Plasma Sintering (SPS) method.

After the tests to diagnose the manufacturing products, they found that BN in the micro-type is more homogeneous by mixing with the powder using balls than the nano-addition of the same material. There is a 50 % increase in the strength of the resulting material by adding only 4.5 BNNPs compared to Al alone. A good yield stress value was shown up to 115 MPa at test conditions at 300°C.

M. Penchal Reddy et al. 2018 prepared samples of Al nanocomposite by powder technology by adding different amounts of BN in the form of nanoparticles, sintering using a microwave to heat the produced parts, and then they performed the extrusion process.

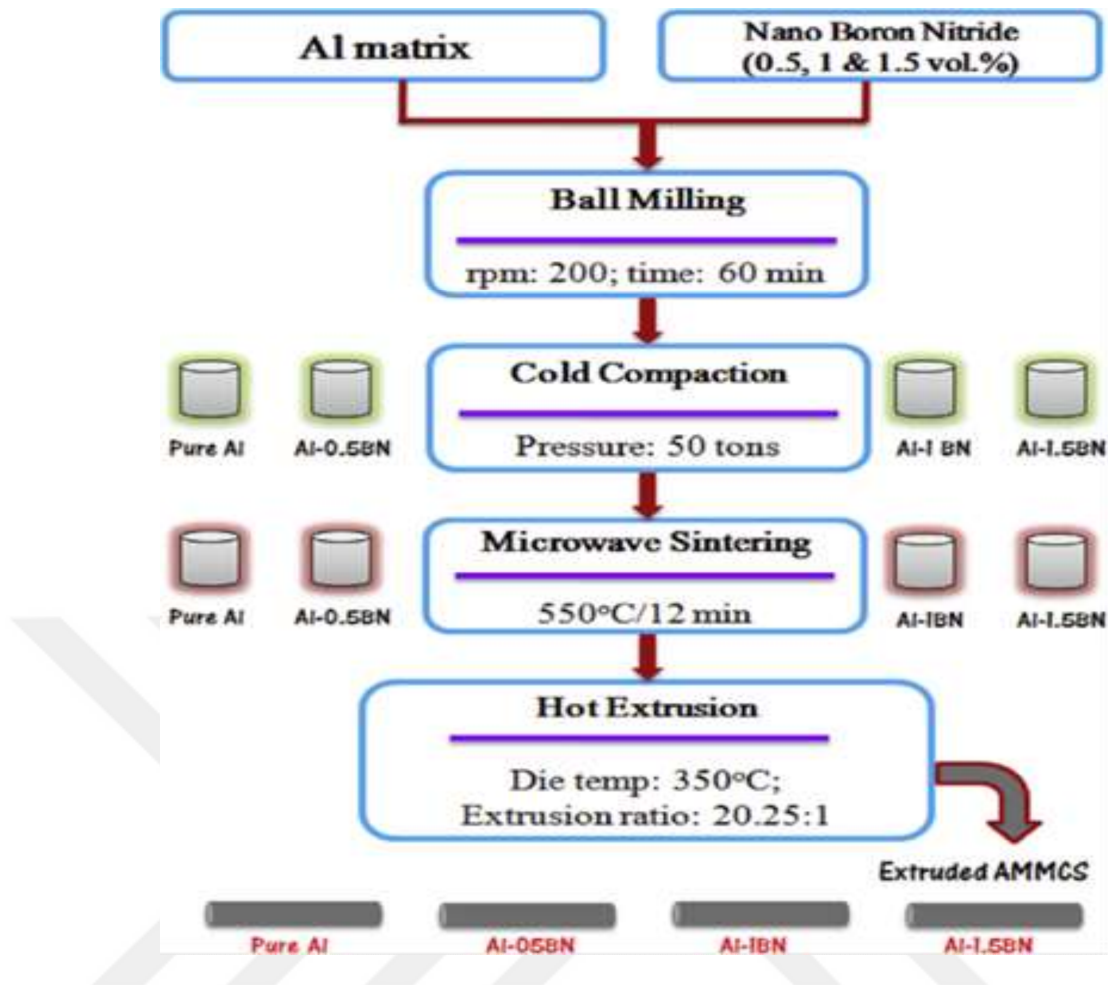


Figure 3.6 Preparation Al-nanocomposites contain different quantities of Boron Nitride (M. Penchal Reddy et al. 2018)

Upon mechanical testing by tensile, small gaps they have appeared on the broken surfaces. Aluminum's damping capacity and modulus of elasticity improved after increasing the amount of BN addition to 1.5 % nanocomposite compound and increasing the hardness value.

In a study by Alexander et al. 2018, they examined the effect of improving the mechanical properties in two ways, either a single addition or a hybrid, and the materials LiN_3 , BN, and B, as powders were added to Al and mechanical mixing, was done using balls, and spark plasma sintering (SPS) was used.

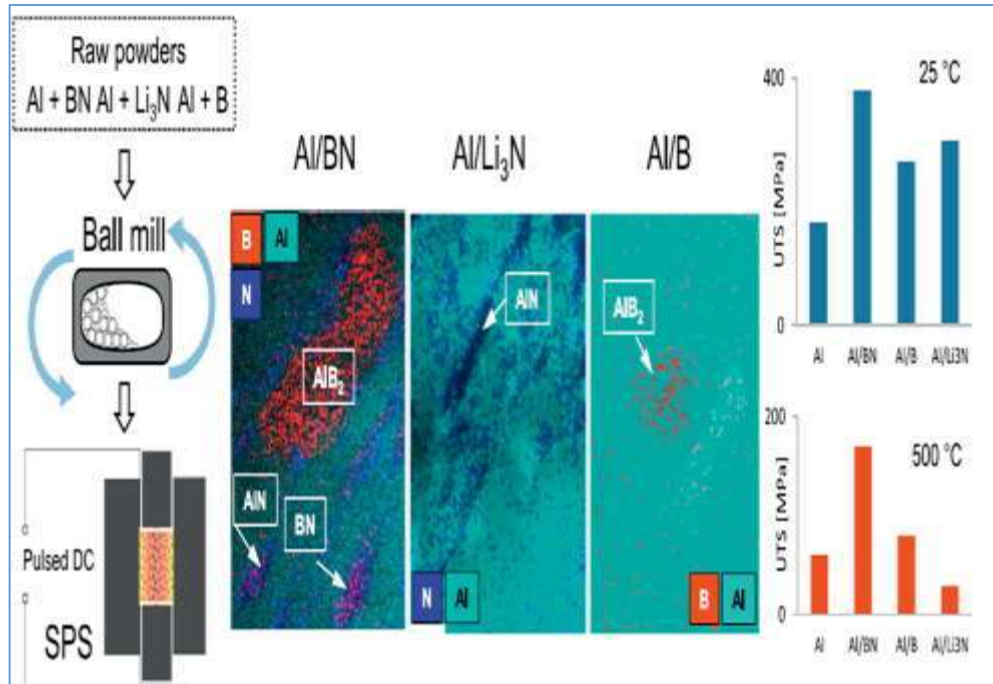


Figure 3.7. Al-composites are prepared by mechanical mixing of Al/BN, Al/B, and Al/Li₃N. (Alexander et al. 2018)

It was found that the composite consisting of adding BN to Al is better in terms of mechanical properties under the same manufacturing conditions compared to the rest of the additives. The tensile strength was 380 MPa at standard conditions, and the phases of Aluminum Boron and Aluminum Boron Carbide appeared thermally stable at 500 °C.

The hexagonal shape of the Boron Nitride (h-BN) gave preference to homogeneity within the structure during the mixing process because of its lubrication effect between the mixture and the balls, which affected the improvement of the high mechanical specifications of the final product.

L. Chen et al. 2018 improved the specifications of the Al-Si alloy to resist wear. They used the surface anodizing process with a coating of nano-MoS₂. The wear test was tested by Pin-on-disc, and the result was a 22 % decrease in the friction coefficient for samples coated with MoS₂, in addition to self-lubrication in the presence of nanoparticles, which increases the life of the produced alloy.

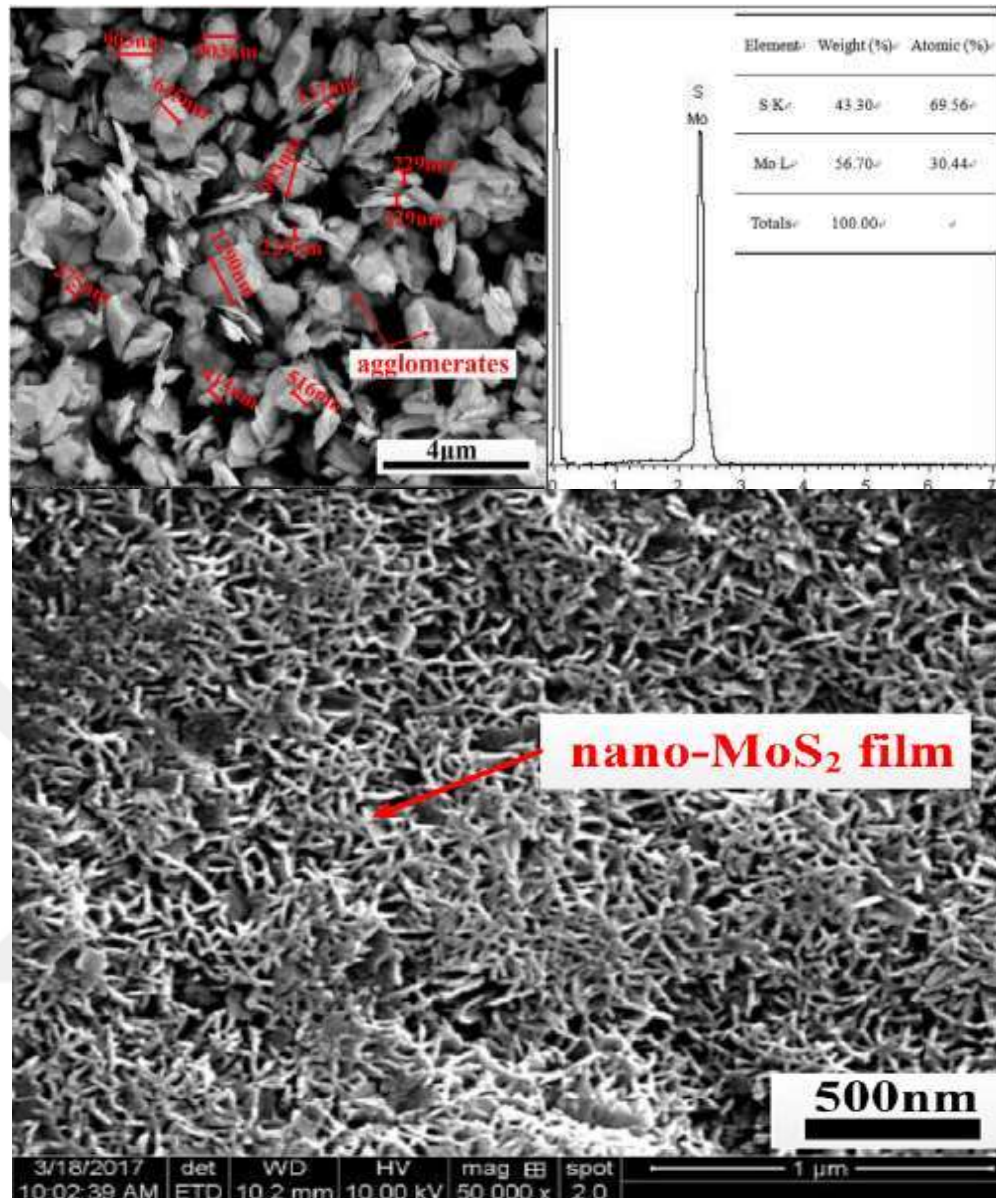


Figure 3.8. SEM and EDS for nano-MoS₂ coating of Al-base (L. Chen et al. 2018)

V.V. Monikandan et al. 2018, through their study of the Al 6061 with the addition of only one addition of 10 % B₄C and comparing it with the addition of a hybrid of B₄C with MoS₂ of 7.5 % because the latter has the effect of self-lubrication, which reduces the coefficient of friction. And they made other experiments to compare the addition of MoS₂ and Graphite; they tested the wear test under dry conditions by increasing the temperature to 250 °C, and they found that the alloy containing MoS₂ had a lower wear rate than that contains Graphite.

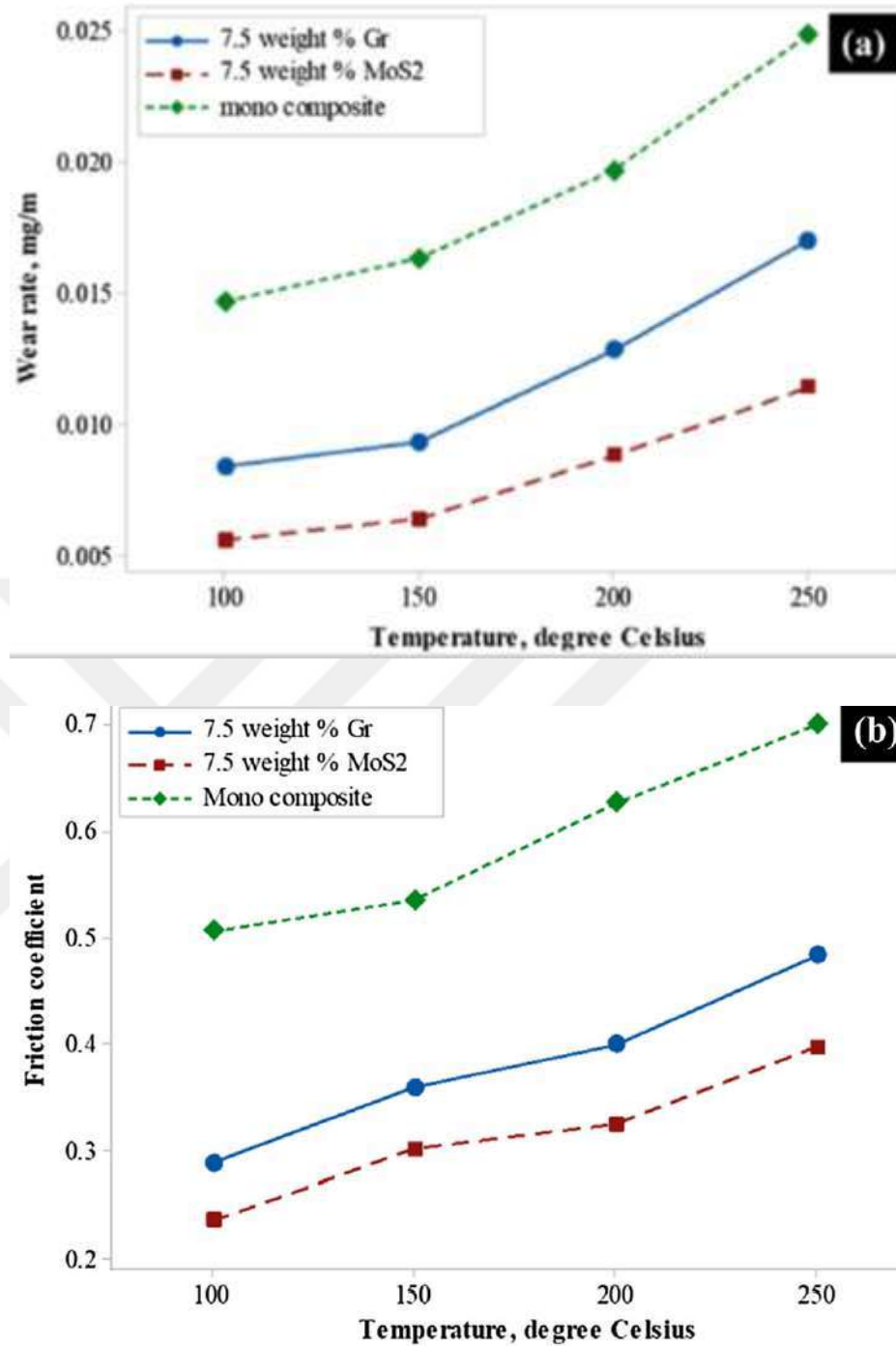


Figure 3.9. Comparison of Al-alloys with added Graphite or Molybdenum Disulfide as hybrid additives or without co-additives on a) wear rate b) coefficient of friction.

(V.V. Monikandan et al. 2018)

In a study by H. Ribeiro et al. 2019, MoS₂ and h-BN were used as two co-additives to the epoxy, that is, a nano-hybrid addition. Hybrid addition was done by adding different ratios to study their effect on the composition, mechanical specifications, and temperature sensitivity.

The resulting additive hybrid mixture had high results for the epoxy composition, where there was an increase in tensile strength by up to 95 percent and 60 % in yield stress, as for the thermal conductivity, an increase of 203 % compared to the compound without additives. Through this research, a nano-epoxy composite with various mechanical and thermal applications was manufactured with high specifications.

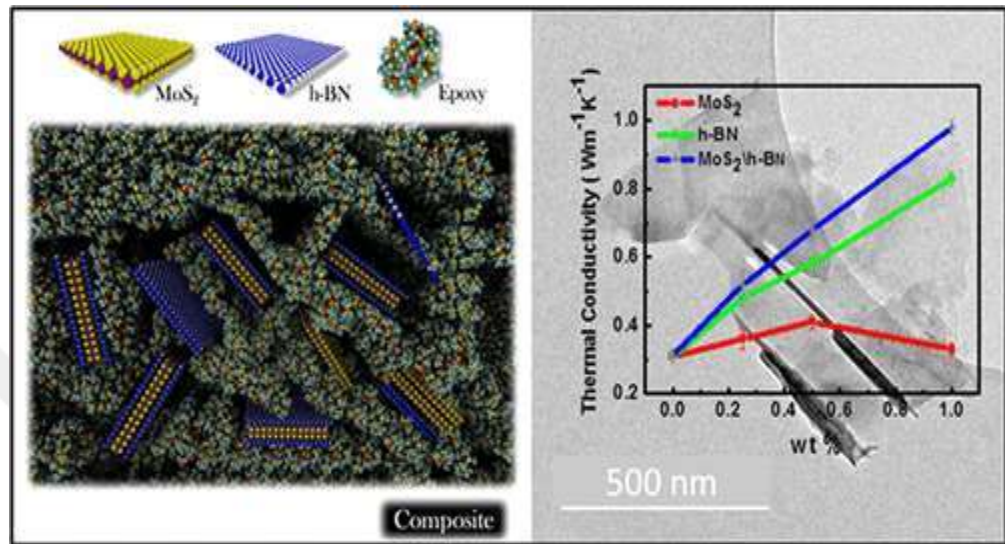


Figure 3.10. The nano-hybrid addition of MoS₂ - BN. (H. Ribeiro et al., 2019)

X. Xiao et al. 2020, worked to improve the wear resistance, hardness, self-lubricating property, and thermal conductivity of BN, as the material used was Shape Memory Polymer (SMP), the modified Boron Nitride (M-BN) was used in this study. The hardness, wear, and thermal conductivity tests were carried out to know the effect of the addition of 10 % M-BN nanoparticles, and they found an improvement in the wear resistance of the adhesive type, and an increase in the hardness of the resulting improved compound, as shown in figure 3.11.

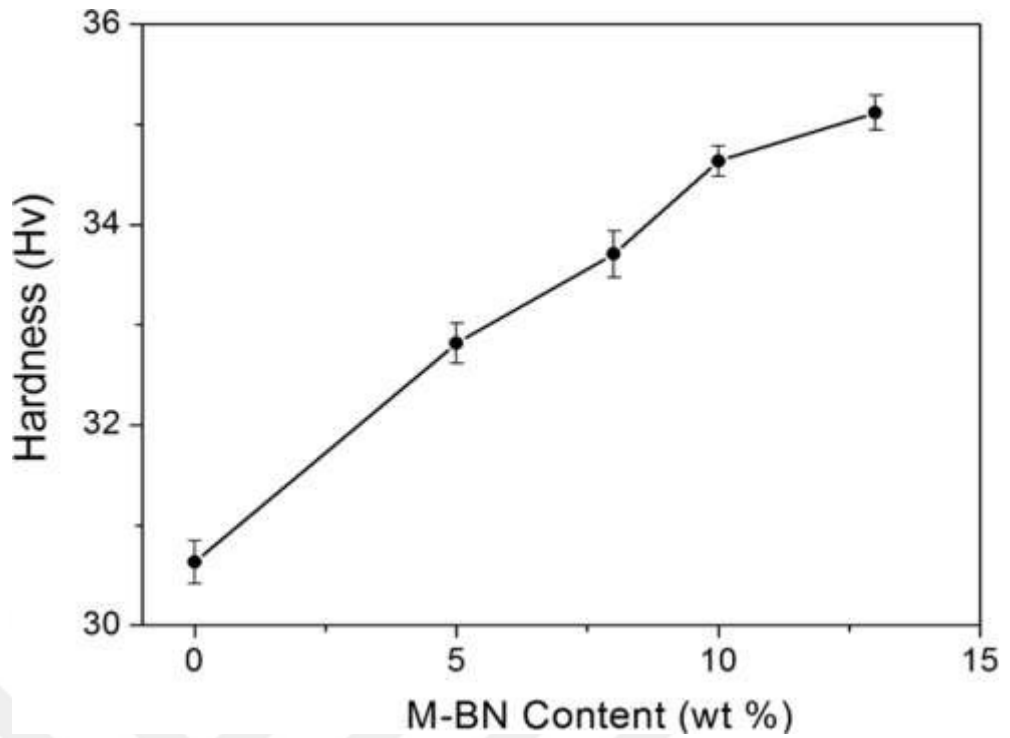


Figure 3.11. The effect of M-BN on a) the hardness & addition of M-BN. (X. Xiao et al. 2020)

U. Aybarç and M. Ö. Seydibe 2021 used A356 as the primary alloy of Al and varied from one addition of nano- Al_2O_3 and Graphene to strengthen the structure. The hybrid casting process of mechanical and ultrasonic was used to produce a homogeneous distribution of nanoparticles, and all parts of the product were subjected to heat treatment. A mechanical test check was performed to characterize the specification. The obtained results showed an increase in yield strength and ultimate tensile strength with the addition of 0.5 % Al_2O_3 and with the addition of 0.25 % Graphene compared to the base alloy.

4. MATERIALS AND METHODS

4.1. Introduction:

The nanopowder and the micro-scale powders were prepared to fabricate the basic Aluminum alloy, and then nanocomposite was prepared by the powder technology method. Mechanical mixing was used in the presence of Zirconia balls (2.5 mm), then pressing within a 10 mm mold, and the final process was sintered with Ar gas protection. Al-12 wt.%Si was used as a matrix reinforced with 2, 4, and 6 % of both MoS₂ and BN and 4 % of single addition MoS₂ or BN.

Hybrid nanocomposites were prepared in three steps: mixing (blending) the Al and Si powder with nanopowder via the mechanical alloying method, compaction (pressing), and sintering.

On the practical side, after preparing the nanocomposite powders, we conducted a SEM examination to find the homogeneity by mixing. We took the mixture of powders and pressed them, and then sintered them with the aforementioned protection conditions. To determine the resulting properties of the nano-alloys, we performed the Vickers hardness test and density and porosity tests by applying Archimedes' theory.

To find out the extent to which the wear-resisting properties improved, the friction properties were tested by the abrasive method in dry conditions, and the weight was fixed at a constant value with the change of time and thus, the distance changes, and at each time we calculate the lost weight for each alloy we made, and compare them.

The topography is studied with SEM examination of the wear to determine the type of wear from friction between surfaces. We collected the powder generated by friction after stopping the device and took out the test sample debris and analyzed the elements in it by EDS.

4.2. Material

As we mentioned above, the materials used are powders of different particle sizes, where the Al and Si with it are micro in size from Nanografi Technology, while the MoS₂ and BN are in nanoparticle sizes.

Table 4.1 shows the elements and compounds with approximate particle size and purity measurements.

Table 4.1 The elements and compounds powders.

Powder	Purity %	Approximate particle size	Symbol
Aluminum	99.99	44 (μm)	Al
Silicon	99.99	44 (μm)	Si
Molybdenum Disulfide	99.90	50 nm	MoS ₂
Boron Nitride	99.80	30 nm	BN

Table 4.2 Weight % of powders used in nanocomposites samples.

Sample	Al-12 wt% Si	wt (g)	MoS ₂ %	MoS ₂ wt (g)	BN %	BN wt (g)
1	100	10	0	0	0	0
2	98	9.8	1	0.1	1	0.1
3	96	9.6	2	0.2	2	0.2
4	94	9.4	3	0.3	3	0.3
5	96	9.6	4	0.4	0	0
6	96	9.6	0	0	4	0.4

The six different alloys by powder metallurgy techniques were prepared from the four types of material as micro and nanopowders. Table 4.2 shows the different weight percent and weights in grams to prepare the nanocomposite samples.

It is also known that material with a size of less than 100 nm is considered to have properties with high specifications that apply to the description of nanotechnology. When adding nanoparticles to MMCs, a clear improvement in mechanical and physical properties and fracture resistance strength was observed.

So, the specifications and characteristics of MMNCs are metallic alloys with nano-particles that can overcome difficult working conditions, longer life, wear, and fatigue resistance.

4.3. Experiments and Methods

First of all, were prepared the powders of the micro size of Al, Si, and nano size of BN and MoS₂. The mixing was according to Table 4.2.

4.3.1. Method of Preparation Aluminum Matrix Composite AMC

Twelve % was taken as the weight calculation when preparing the alloy by powder technology. The total weight of the alloy is 10 grams, which is 12 %, equal to 1 and 2 tenths of a gram, which is the weight of Si. As for Al, it weighs eight and eight-tenths of a gram. The powder material was supplied with a ball mill at 250 rpm in a plastic container for two hours. After mixing, we take a sample of Al-Si powder, provided that it does not exceed 1 g for XRD, SEM, and EDX tests.

4.3.2. Method of Preparation Aluminum Matrix Nanocomposites

Alloy powders (Al-12 wt % Si) were mixed with nanoparticles (MoS₂ and BN) by dry blending. These powders are mixed using a ball mill to obtain a good dispersion. Powder mixing was performed by adding the alloy powder (Al-12 wt % Si) in a 20:1 ratio together with various wt % mixed

additives (nano-MoS₂ and nano-BN) into a ball mill (2.5 mm ball diameter with 20 number for every gram), and using only single addition of MoS₂ or BN. The mixing time was 2 hours at average 250 rpm in a dry vessel to achieve a good particle distribution. To prepare the first alloy of the nanocomposite, as shown in Table 4.2, Al-12 % Si hybrid addition of 2 % of MoS₂ and BN. To obtain a hybrid addition of 2 % of nano BN and nano MoS₂, we take 0.1 g of BN + 0.1 g of MoS₂ as nanopowders and mix them with a matrix alloy of Al-12 % Si with a weight of 9.8 g.

The addition of 4 % hybrid nanoparticles (MoS₂ + BN) was 0.2 + 0.2 g. It was added to the base alloy of powder Al-12% Si, weighing 9.6 g, and mixed by ball milling in a plastic jar at a speed of 300 rpm and for 2 h.

The addition of 6 % hybrid nanoparticles in the third alloy nano (MoS₂ + BN) was 0.3 + 0.3 g. It is added to the base alloy of the powder and mixed by ball milling in a plastic jar at 300 rpm for 2 h.

The mixing process was carried out using zirconia balls with diameters ranging from 2.5-5 mm and the jar or cylindrical plastic container, rotation speed of 300 rpm, for 2 h; the number of balls was 20:1.

The fourth alloy, 4 % nanoparticles with only one addition of MoS₂, 0.4 g is added to the base alloy of Al-12 % Si powder, and mixed by ball milling in a plastic jar for 300 rpm for 2 h.

For the fifth alloy, 4 % nanoparticles, only one addition of BN, 0.4 g is added to the Al-Si alloy and mixed by ball milling in a plastic jar of 300 rpm for 2 h.

4.3.3. Powder Compaction

The process of pressing the mixture powder is by using a piston and a mold with an inner diameter of 10 mm. The mixed powder is subjected to a uniaxial cold pressing process to obtain good pressing pressure and produce a blank with fewer defects. To select a suitable pressure, more than one experiment was carried out. A mold with a hole diameter of 10 mm and a

hydraulic press with a capacity of 12 tons are used. Powder compaction weights of 6, 7 and 8 tons. Experimental work and literature (Schey & John, 2007), (M.E. Fayed & L. Otten, 1984), (Cathleen H. 2007) showed that the optimal applied pressure was 7 tons for all samples used in this work.

The pressing was carried out under normal atmosphere conditions without increasing the temperature and at pressure rates of more than 7 tons. We settled with this pressure for two minutes, and these values are among the influential variables in producing pieces with high specifications and obtaining values with high conditions that came from experiments, and every mixture and proportions can change these values.

There are several factors on which the value of the pressure applied to the prepared powder depends; including the size of the powder, as the nanopowders differ from the micro powders by the value of the pressure applied, and also to obtain the pressure value at an optimum level based on experiments.

One of the important factors is the type of powder elements and their chemical composition, which have an important role in the convergence of atoms and their bonding with a certain pressure. Since the pressure will vary according on the area to which the force is applied, measure the mold by measuring its inner diameter. We used a three-piece mold, the first cylindrical piece is a punch that will be subjected to direct pressure by the piston in the form of a column, and its circular face will be against the powder. The second part of the mold is die, where we will fill the powder inside; the smaller part represents the base of the die and is at the bottom, the first thing lifted after the end of the pressing process of the powder.

During the work, this mold is subjected to damage due to constant pressure, which leads to the expansion of the diameter of the shaft and its corner inside the mold. Using another mold with the tool's specifications, stainless steel, with a finely smooth surface and precise dimensions, to produce the samples with the best measurements and free from defects in their consistency.

The following Figure 4.1 represents the three parts of the mold, represents the upper part, and we call it the punch with diameter 10 mm, the second part is die, and the last part is the base. The actual images of the mold used in this study, where the image on the left shows the mold parts in a cylindrical shape and the other in a rectangular shape. It is possible to make cylindrical and cubic shapes for manufactured samples, and this is clear from the picture on the right, but according to the standard specifications of the wear test, the samples produced must be cylindrical with a diameter of 10 mm.

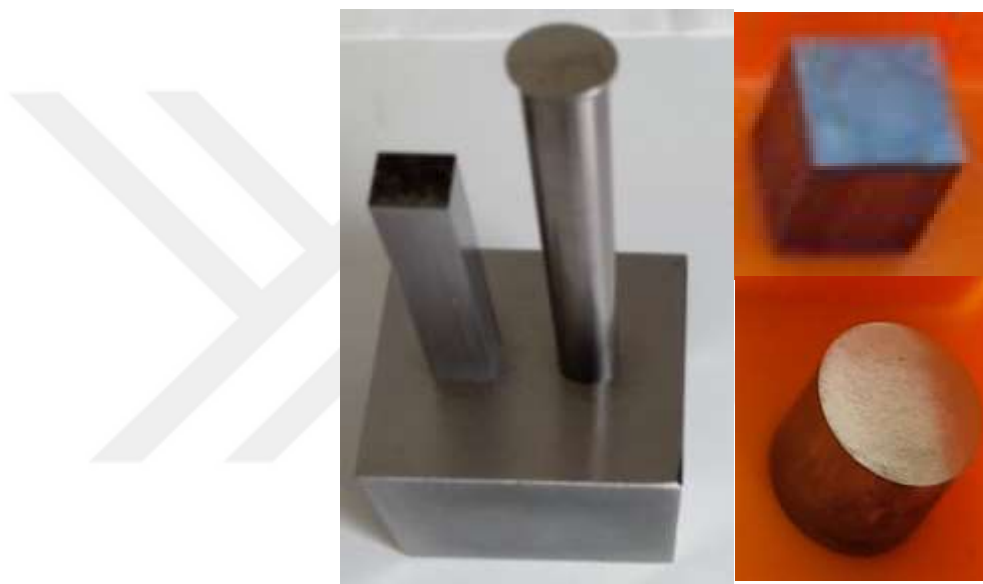


Figure 4.1 The different shapes of molds; cubic and cylindrical

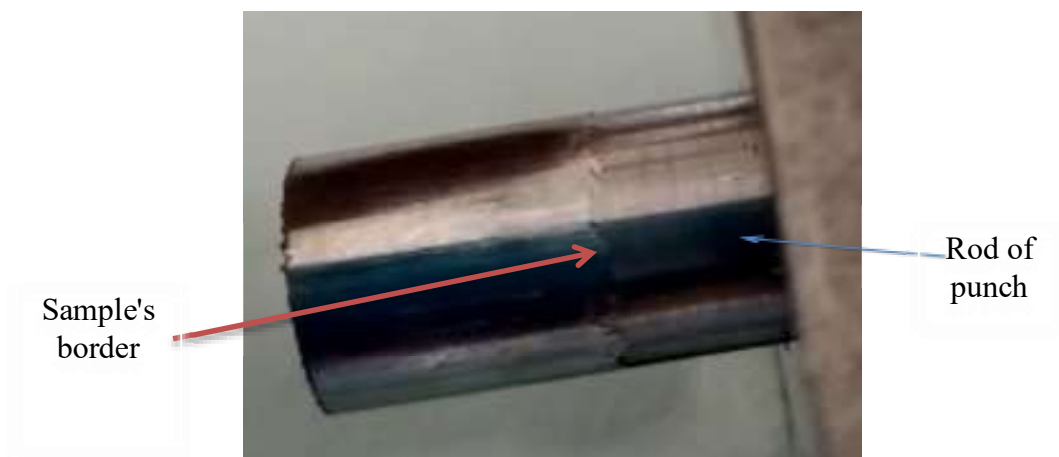


Figure 4.2. The sample after pressing

The pressing process and the pressure selection depend on the powder's type and size. Through the experiments, we used a pressure of 7 tons and fixed it for two minutes. After it is removed from the mold, the process of adhesion between two different metals occurs temporarily due to the pressure of the steel on the Al powder. The image in Figure 4.2 shows the boundary between a sample of the Al alloy and the steel rod used in the dies.

Figure 4.3 shows the sample of Al powder after separating it from the welding of the steel column and reaches a height of 15 to 20 mm and a diameter of 10 mm. Note that when Al powder is mentioned because it is the main element with which the rest of the elements are mixed in its micro or nano form of different powders ratios.



Figure 4.3. The sample, after being pulled from the rod

4.3.4 Sintering

The process of sintering in the presence of inert gas protection, which is Ar, protects the samples during high temperatures from the entry of Oxygen into the chemical compounds of which the nanocomposite is made.

The process can be compared to TIG welding, which is a process of

welding metals, especially Al, using a Tungsten electrode and with the protection of an inert gas such as Ar, but the difference in sintering is at a temperature lower than the melting point and equal to approximately 0.8 of it and according to the type of metal. The melting of Al is more than 660 °C and varies according to the alloying elements added, and we used an average sintering temperature of 550 °C.

Within the tube furnace shown in Figure 4.4, 6 samples were placed inside 6 ceramic flasks, where first was the base alloy, and the rest were nanocomposite samples. The sintering conditions were the same for all samples; the sequence of heat exposure inside the furnace was in three stages, the first stage was raised from room temperature to 350 °C, and we kept it for 30 min. In the second stage, the temperature was raised to 550 °C and stabilized for 90 min; the last stage cooled to room temperature for over 90 min. All the stages of sintering are shown in the diagram in Figure 4.5, which represents the relationship between time and temperature; we conducted it with the presence of Ar gas pumping at a rate of 2 L/min. As shown below, the electric tube furnace connected with Ar gas to shield the entire green samples through the sintering procedure.



Figure 4.4. The electric tube furnace used for sintering

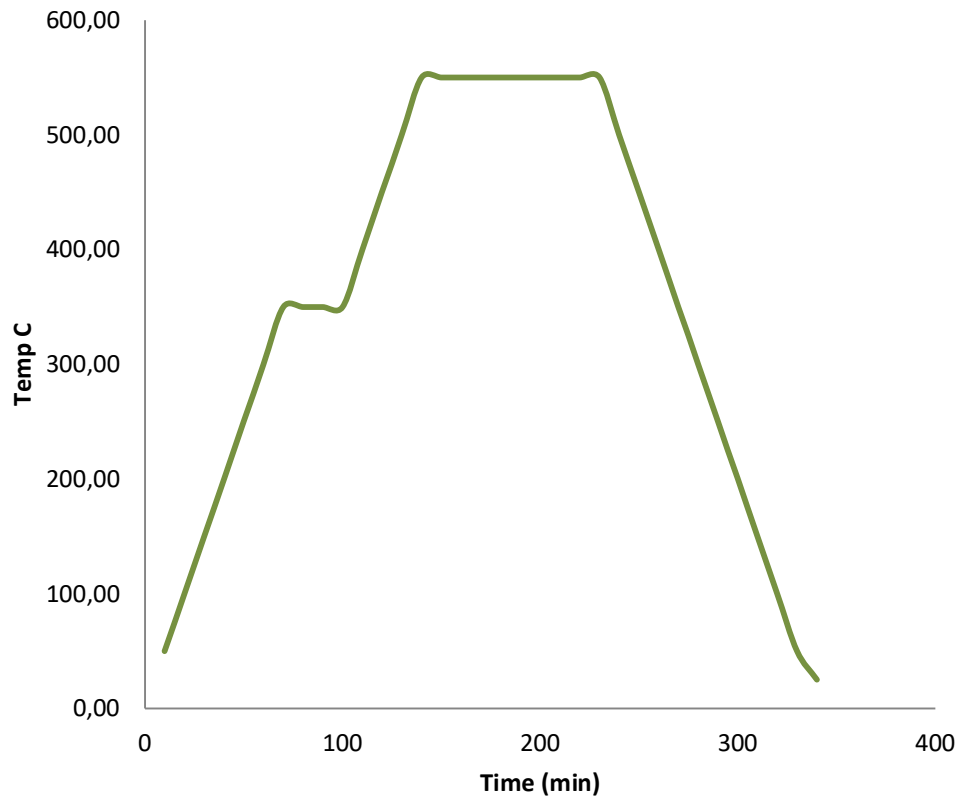


Figure 4.5. Temperature time cycle of sintering process.

From the experimental work and literature review of (W. W. Leong Eugene & M. Gupta, 2010), it seems that the same procedure as above was performed on the sintered samples.

4.3.5. Electron Microscopy and Energy Dispersive Spectroscopy

Using the scanning electron microscope (SEM) is important in preparing nanomaterials so that different properties of the manufactured material are determined.

One of the properties that can be determined is the surface topography of the prepared compound, which shows the roughness or smoothness of the surfaces, and this is related to the elements and compounds from which the alloy was prepared and its granular size.

Since we mentioned the particle size, we can know the measurements and dimensions of the particle size after the mechanical mixing process of the

powders, the crystalline limits of the alloy and the different phases after the manufacture of the compound, and the different ratios of the elements in a particular compound from a part of an examined surface. The device used in our research is located in the Black Sea Research Center at the Ondokuz Mayıs University of Samsun, model Jeol JSM-7001F. This SEM is equipped with an energy-dispersive spectrometer (EDS), secondary electron detector (SE), and electron backscatter diffraction (EBSD).

EDS and BSD are important in determining the map of components through electronic image processing by defining the element's property and extracting the composition automatically.

We benefited from SEM to determine the homogeneous distribution between micro and nanopowders for Al-Si- MoS₂-BN, analysis for each element of the powders, especially the percentage of nanoparticles before and after the wear test, and surface imaging to determine the wear type of nanocomposite after carrying out the wear test.

The analysis principle by EDS depends on the characteristic X-ray resulting from the collision of electrons on the sample's surface and the emission of these rays, which indicate the type of atoms according to the emitted spectrum. From the sensor that captures the emitted energy in a spectral form, it turns into a mapping of the elements and shows their proportions on the surface that was scanned.

The resulting characteristic X-ray depends on the intensity of the accelerating voltage; the value of 10 kV does not give the same penetration under the metal surface as the effect of 15 kV.

There is integration in the work of SEM and EDS where at the same time, those electrons collide on the sample's surface, leading to the formation of SE, BE, and X-rays.

From the results above, some detectors receive BE, SE, and EDS, thus giving an integrated description of the surface of the sample examined as an image of a map on which the proportions of the elements are indicated.

Rather than and this is shown in Figure 4.6.

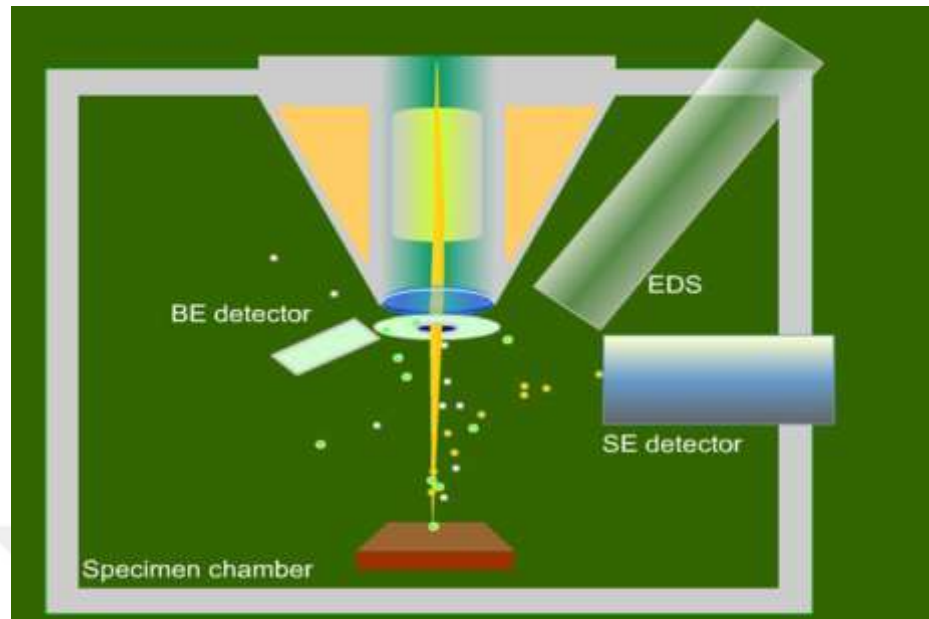


Figure 4.6 Schematic of Electron Microscopy.



Figure 4.7. Scanning electron microscope (Jeol JSM7001F model)

4.3.6. XRD Characterization

XRD X-ray diffraction was used to determine the phases of the elements included in the powder mixture in the first stage only at the micro level of Al and Si, and after the second stage of preparation by adding nanoparticles by mechanical mixing with the initial powder of the micro powder. And the measurement conditions were as follows; X-Ray 40 kV, 30 mA, and scan speed / duration time of 2 deg/min. The Rigaku Smart Lab located in the KiTAM Central Research Laboratory contains a water-cooled system for X-ray generation tube.

We used Bragg-Brentano method using "parallel focal beam geometry" for phase definitions for rough surfaces, poorly crystallized samples, and especially thin films.



Figure 4.8. X-ray diffraction device (Rigaku Smart Lab model)

XRD is based on the International Center for Diffraction Data (ICDD) database. They organized the Powder Diffraction File (PDF), which is a diffraction database for inorganic materials such as mineral and organic

powders used for phase determination and characterization of materials by powder diffraction. This PDF has been around for 75 years, and new examinations are added yearly as a database to determine the X-ray diffraction of the powders being examined.

PDF was developed into a database in the sixties and can nowadays be accessed via the Internet after it was transformed from paper to data recorded in the computer because it has become uneconomical to be in books and records that are difficult to access. The primary purpose of the PDF is to serve as a quality reference tool for those working on X-ray powder diffraction characterization. Its use has become imperative to obtain the structural and crystalline properties of the material, allowing for phase determination.

The main input for PDF is; the diffraction type (X-ray, electron, and neutron), wavelength apertures, intensity variables, and Inter planar spacing (d) and intensities (I); Miller indices are listed when available. (Gates-Rector and Blanton, 2019)

PDF is used for it provides structural details, such as determinants of the crystal lattice, atomic coordinates and thermal factors, composition formation, phase determination, and quantification. (Belsky et al., 2002).

The American Society for Testing and Materials (ASTM) published the first set of PDFs in 1941. At present, these data have been increased for different materials, including crystalline, amorphous, and modified nanomaterials. And as we explained in the data input, it is not only limited to X-ray diffraction only but also electron and neutron diffraction. (Bruno et al., 2017).

4.3.7 Microstructure and Hardness Tests

Sintered samples were prepared for microstructural studies through the sequencing process. The grinding process was carried out using SiC paper of 320, 500 and 1000 grit with water as coolant. The polishing process is carried out with a special cloth and diamond paste (1 μm), the etching is

carried out with an etchant consisting of 1 % hydrofluoric acid and 99 % distilled water. The samples were then washed with water and alcohol and dried in hot air.

To examine the microstructure, the surfaces of the prepared samples were examined at different magnifications using an optical microscope. A polarizing microscope was used to observe the topography of the sintered surface of various samples.

The hardness of all samples was measured using a micro Vickers hardness tester. The applied load is 1.96 N and the loading time is 15 s. Make 3 to 5 indentations on the surface of each sample and take the average value to determine the Vickers hardness.

4.3.8 Density and Porosity Measurements

The final density and porosity (after sintering) of the samples were measured according to ASTM D 792, which is based on Archimedes' principle. The specific gravity of the material is given by equation (4.1) and the porosity by equation (4.2)

First weigh the sample in air, then immerse it in a liquid of known density and weigh again, and finally calculate the density of the sample according to formula (4.1) and porosity formula (4.2).

$$\rho_{experimental} = \frac{m_{air}}{m_{air} - m_{water}} \times \rho_{water} \quad (4.1)$$

where m_a and m_w represent a mass of the sample in air and water, respectively, and ρ_w represents the density of the distilled water (0.998 g/cm³ at 20 °C).

$$\rho_{theoretical} = \rho_{Al-alloy} * W_{Al-alloy} + \rho_{Si} * W_{Si} \quad (4.2)$$

where W and ρ represent the weight fraction and density(g/cm³) of the element. (J. Kuma, et al. 2020)

$$\%Porosity = 1 - \frac{\rho_{experimental}}{\rho_{theoretical}} * 100 \quad (4.3)$$

4.3.9 Wear Rate Measurement

The wear rate of the samples was determined gravimetrically. The samples were weighed before and after the abrasion test using a sensitive balance with an accuracy of 0.0001 g. Weight loss (ΔW) is divided by the sliding distance and the wear rate is determined using the formula in (U.N.I.D.O., 1990).

$$\text{Wear rate } (W.R) = \Delta W / \pi D.N.t \quad (4.4)$$

where: D is the sliding circle diameter (cm), t is the sliding time (min), N is the drum or disc speed (rpm)

The material's actual wear behavior is affected by various external parameters (abrasive particle size, velocity, and angularity) as well as intrinsic wear material qualities (hardness, toughness, Young modulus, and so on). Wear-resistant materials hardness and toughness features are heavily influenced by the reinforcing phase's quantity, size, and mechanical properties (Priit Kulu et al. 2008).

The nano-tribological behavior can aid in developing new solid lubricants, such as those used in hard disk storage technology, and the repeatable and reliable manipulation of micro/nano-objects on surfaces (Metin Sitti, 2003).

5. RESULTS AND DISCUSSION

5.1. Introduction

We started the tests after preparing the micro and nanopowders, where the first results were the X-ray diffraction of the basic alloy, Al-12wt%Si, once, and the other after adding nanoparticles in a hybrid way.

We also conducted SEM for the prepared powders, showed the results, and discussed the distribution of the prepared powders by mechanical mixing. Also, in addition to detailing the characterization of the components included in the powders, we present the analysis results by EDS for different powders before and after mixing.

After the pressing and sintering procedures, there are results of different tests, which are the hardness, density, and porosities of the nanocomposites prepared by the powder technology method. The results of the wear tests, their discussion with the images of the electron microscope of the various samples, the improvement or failure that occurred in the experiments, and their reasons with a comparison with some previous studies will be presented.

5.2. X-Ray Diffraction (XRD)

The intensity and Bragg's angle θ is related to crystal structure and are according to the X-ray diffraction response for each powder. By comparing with the standard data defined for the previous PDF models of the materials, we can determine the peaks of the elements in the atomic levels.

Measurement conditions of X ray diffraction are given in the following table:

Table 5.1 XRD parameters

Scan-speed/ Duration time	2.0000 deg/min 40 kV, 30 mA
Voltage	
Step width	0.0100 deg
Scan axis	Theta/2-Theta
Scan range	1.0000-90.0000
Incident slit	1/2deg
Length limiting slit	10.0mm
Receiving slit #1	1/2deg
Receiving slit #2	0.300mm

We will start with the peaks that appeared after the examination of the device as it is, and then we will draw peaks showing the planes of the crystalline structure, and then we will draw comparison diagrams between the basic alloy and alloys with the nano-additions.

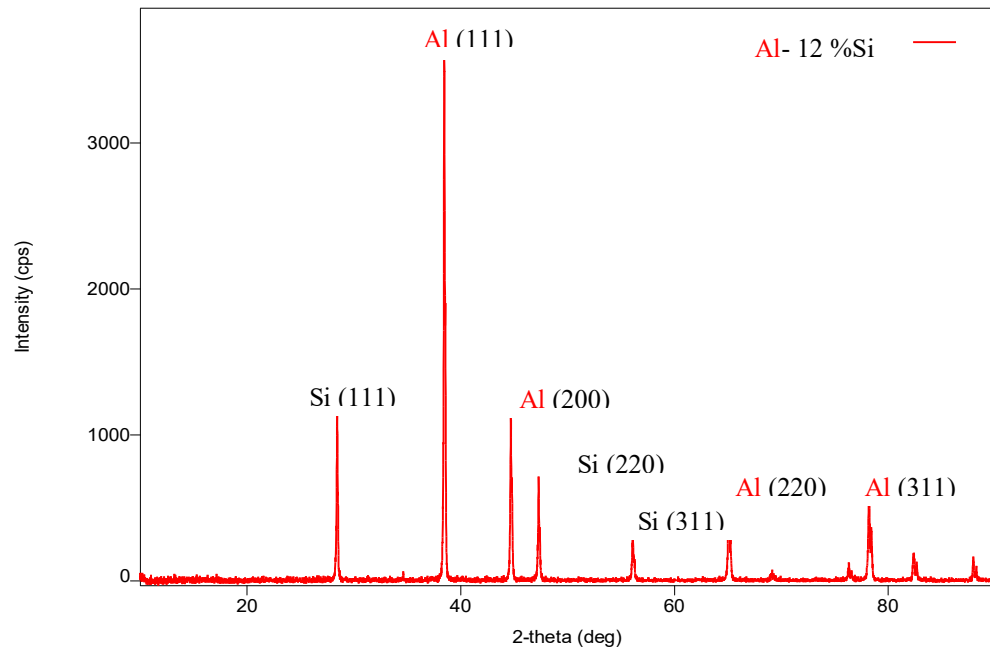


Figure 5.1 XRD of base alloy Al-12 % Si

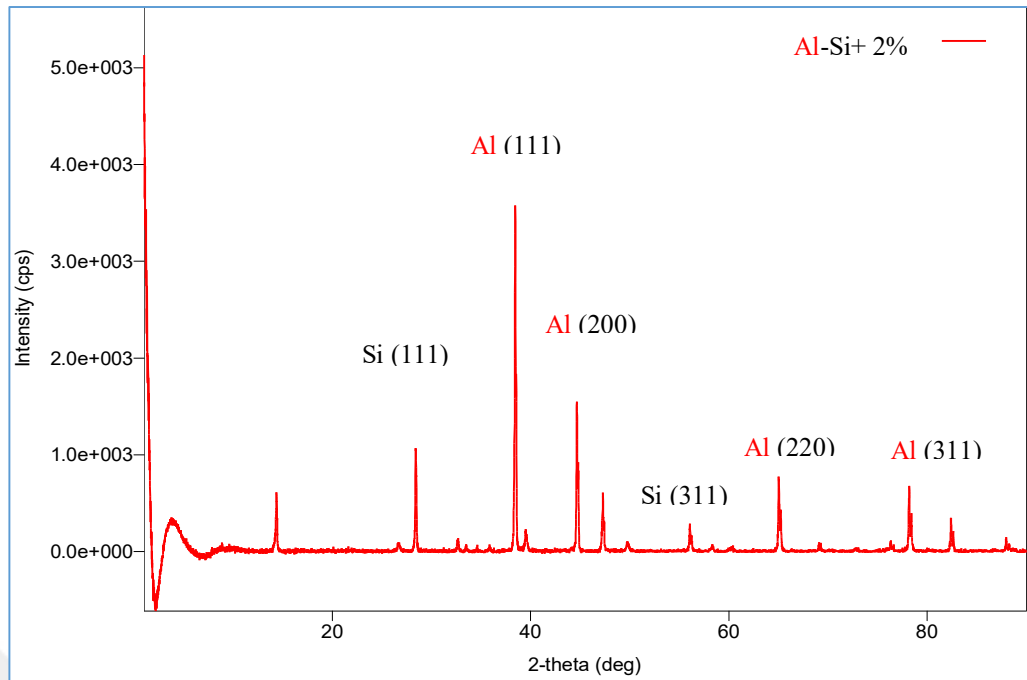


Figure 5.2 XRD of Al-Si+ 1% MoS₂+ 1% BN

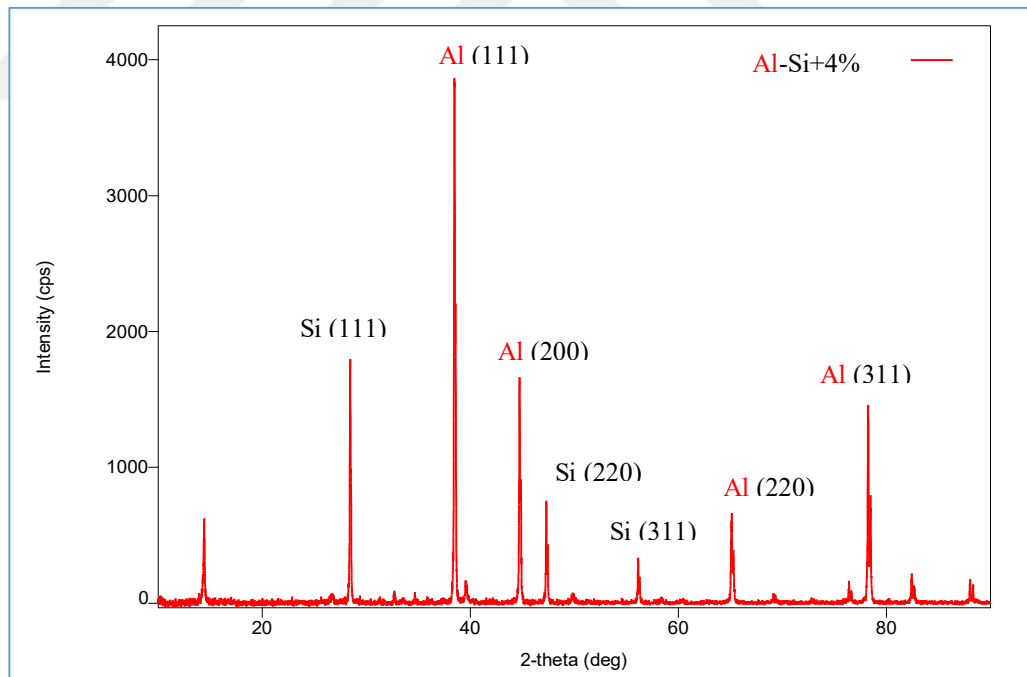


Figure 5.3 XRD of Al-Si + 2 % MoS₂ + 2 % BN

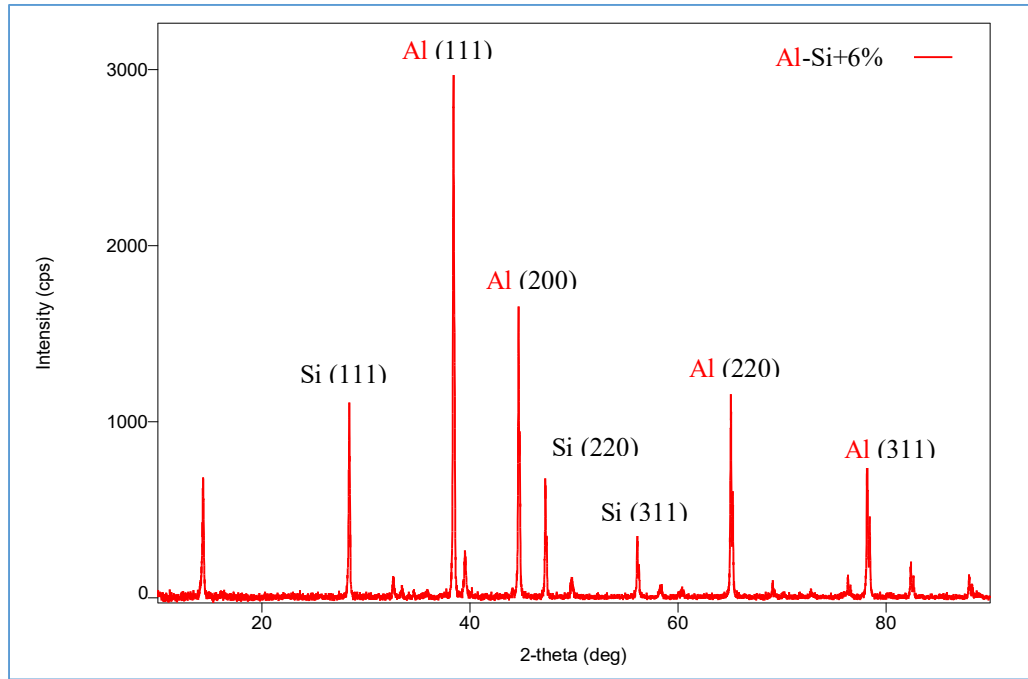


Figure 5.4 XRD of Al-Si + 3 % MoS₂ + 3 % BN

An X-ray examination was carried out using the X-ray diffraction method, beginning with the powder of Al-Si alloy for the first sample, after that the second sample of the mixture of Al-Si and the addition of 1 % of MoS₂ + 1 % of BN, as well as for the rest of the other common additives. We will notice all the diagrams for all the samples, that there is a very slight difference in the peaks and intensities at the Θ angles, the reason is that the amount of nano-elements is added in small proportions and the micrometer measurement is the most influential in the results of X-ray diffraction to determine the phases. Also, after these diagrams, we extracted only two diagrams showing the atomic levels compared to the results of previous PDF examinations

The first scheme represented the results of the basic alloy consisting of Al powders by 88 % and Si by 12 %. The peaks are clear for the levels because they are powders of a micrometer scale. Figure 5.1 shows the (111) plane.

Examination by X-ray diffraction depends on inputs, including the type of material, its quantity, and particle size, where the last effect has priority by the appearance of the phase clearly according to its quantity and

size to compare it with the data stored for examinations of elements previously.

The second stage of the diffraction test, as mentioned above, is after adding the nanoparticles of MoS₂ and BN to the parent alloy with the highest percentage of Al and Si. Compared to the ratios, the highest percentage added is 6 % hybrid addition, representing only 3 % for each nano-mixture. This may make us not notice a type or phase in a part of a sample of the powder that has been examined, and this is shown in Figure 5.2, where we did not show us the peaks explicitly or completely for the phases of the elements at the nano size. We still have clarity with peaks from the intensity relationship and two-theta for the two largest elements, which are Al-Si, of (111) and (200) planes.

Upon analyzing the figures, it becomes evident that distinct phases of pure Al are present at specific 2θ angles. These angles are measured at 38.50°, 44.74°, and 65.13° degrees. These findings align with the previous research conducted by (Muhammed R. et al., 2014), further validating our results. The presence of these phases indicates the crystalline structure of pure Al within the sample. The specific angles at which these phases appear correspond to the characteristic diffraction patterns associated with Al crystal lattice planes.

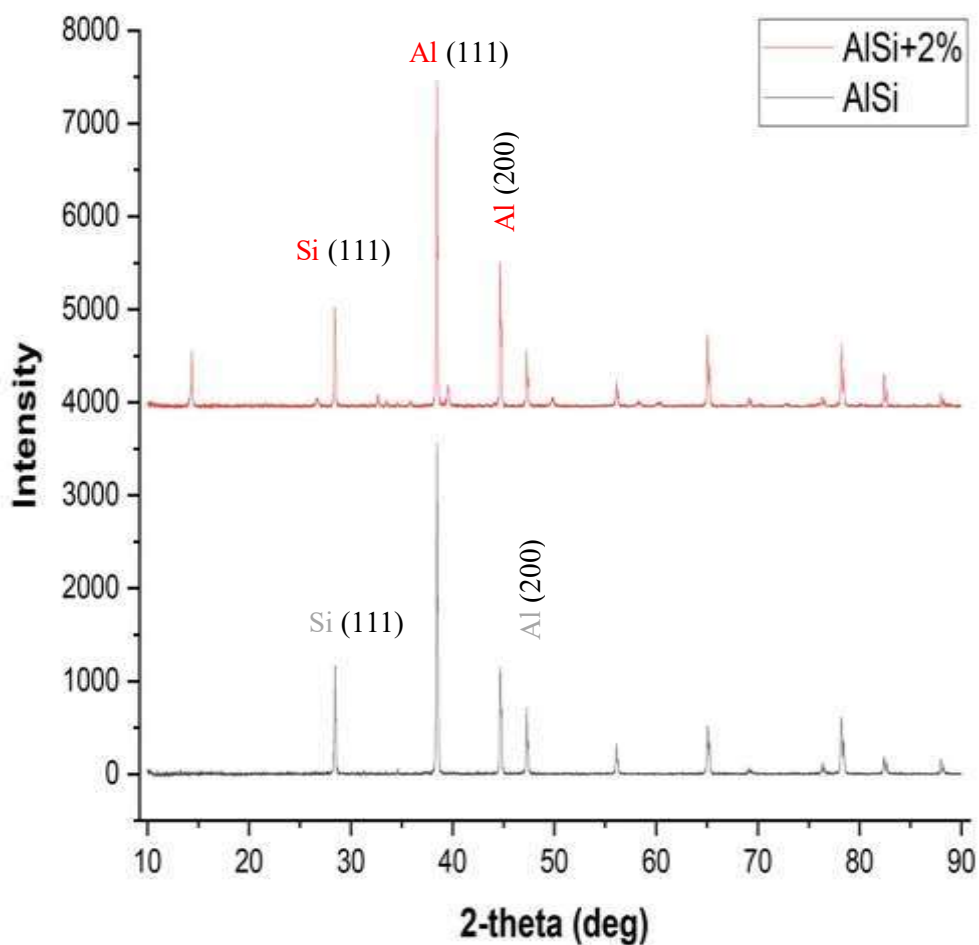


Figure 5.5 Comparisons XRD of Al-Si with 2% hybrid MoS_2 +BN

After observing the above figure, we notice an apparent deviation in the values of angles and intensity. As for the following two figures, the additions had no effect, so we note that the peaks are identical, with some difference in intensity. The reason may be in the different proportions of the elements affected during the passage of X-rays and their effect on the atoms. We considered the basis for the calibration test to be the primary powder consisting of Al with 12% Si, and the results for each sample we compared with it.

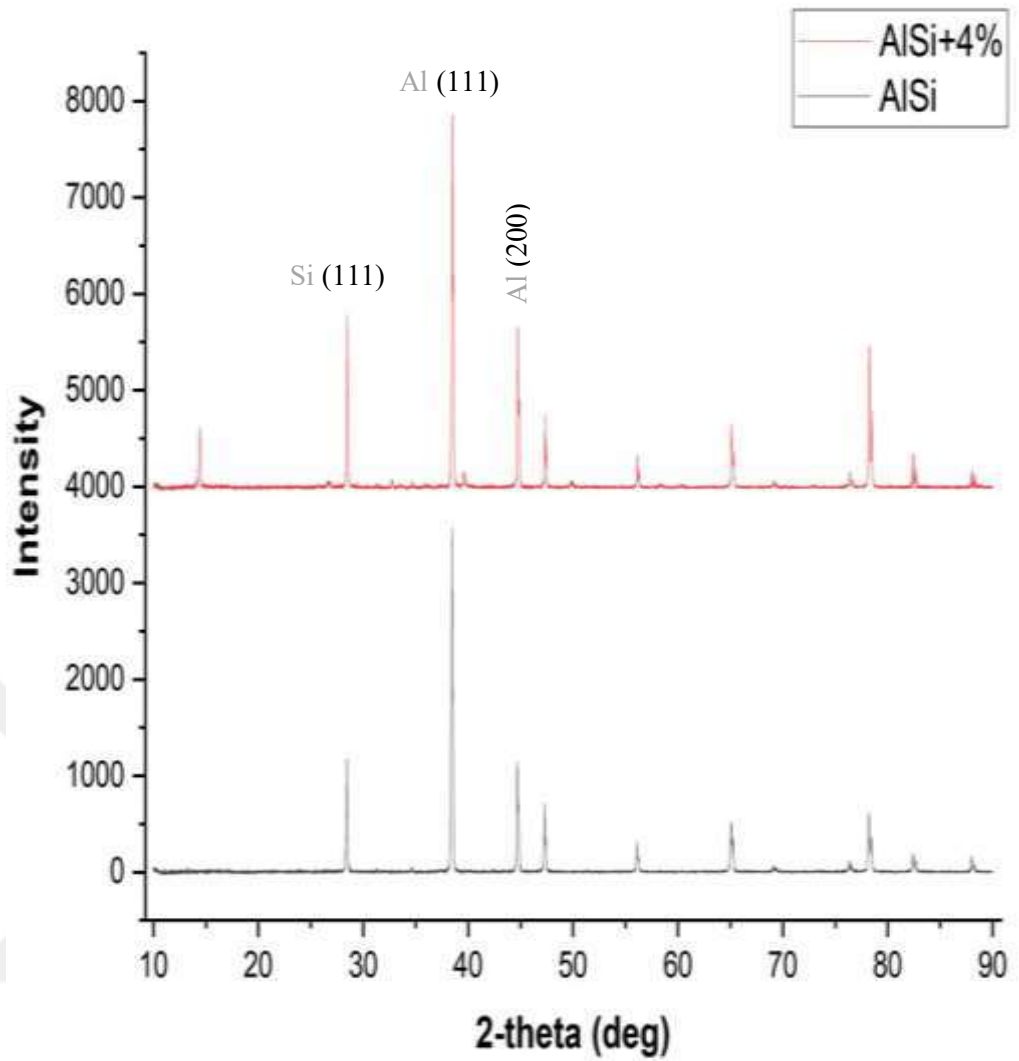


Figure 5.6 Comparisons XRD of Al-Si with 4% hybrid MoS₂+BN

By illustrating the difference in a red color for distinction, its non-appearance indicates a complete match between the base alloy and the nano-additives, and there is no slight deviation that distinguishes the angles of the two measurements.

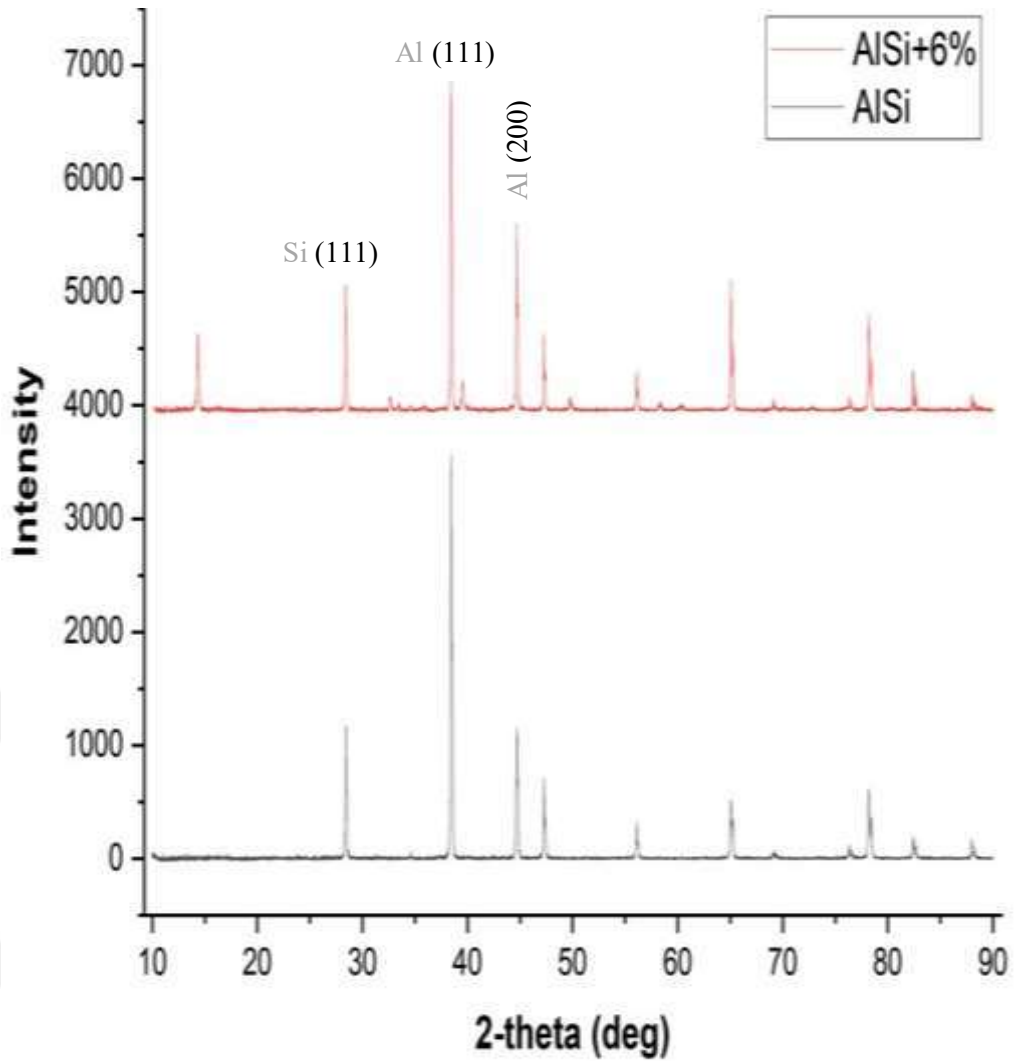


Figure 5.7 Comparisons XRD of Al-Si with 6% hybrid MoS_2 +BN

By comparing the results of the X-ray test with the combined diagrams of the base alloy and the nano-additive above, we notice the great convergence between the peaks of the large effect of Al as a host metal, followed by Si. The other reason for the clarity of the peaks for the Al and Si phases is: the micro-granular size, and the presence of nano-additives in small proportions. The appearance of simple peaks due to the effect of the added nano-elements forming crystalline phases is difficult to diagnose compared to the stock data for the crystalline phases of previously examined elements.

5.3. SEM Images for Powders

The characterization of mixed powder by SEM shows the distribution and homogeneity of different components blended by ball milling. The first investigation for the first micro-alloy Al-12 % Si as mixed powder. The second and third characterizations for (h-BN) and MoS₂ as nanoparticles were added to base alloy.

As mentioned earlier, the nanometer level after pressing and sintering does not appear clearly, due to the aggregation and micrometric measurements of the main basic powders. This is confirmed by previous studies, as in the research of M. J. Fouad, 2014.

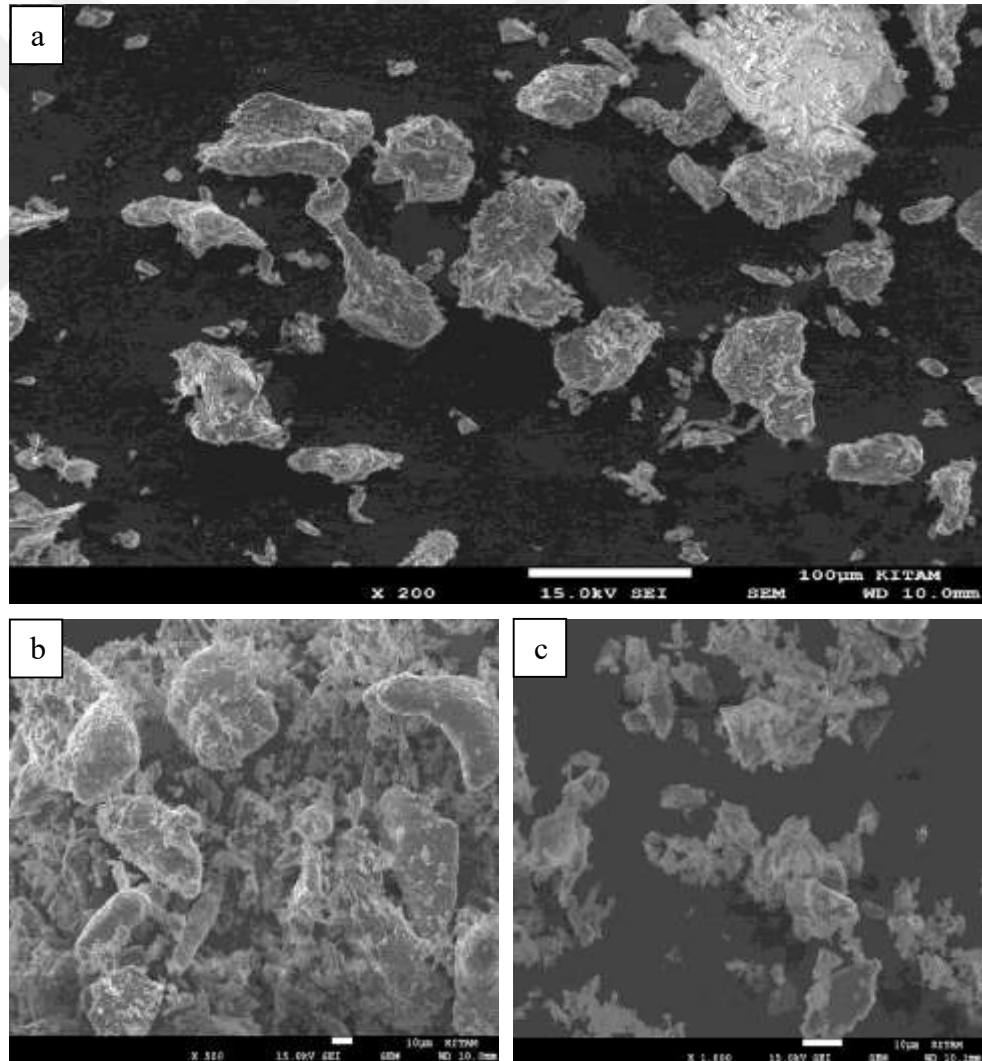


Figure 5.8. Al-12 % Si powder by SEM; a) 200 X, b) 500 X, and c) 1 kX

In Figure 5.8., SEM of the resulting mixture of Al and 12 % Si powders by 200 X magnification, at a level of 100 μm are seen. From 5.8. b, with a magnification of 500 X, and Figure 5.8. c with a magnification 1 kX a scale of 10 μm shows us the homogeneity between Al and Si after mixing by ball milling.

After mixing the granules of the powder 44 μm in relation to Al and Si, we notice the reduction of the granules as a result of the balls hitting during grinding. From Figure 5.8. c, we clearly notice size of the particles, are less than 10 μm . The magnification was up to 1 kX, and the scale was 10 μm .

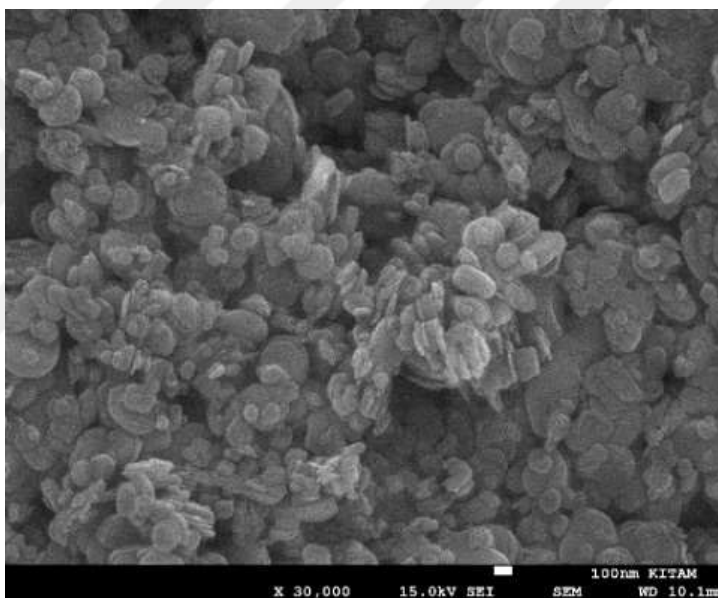


Figure 5.9. SEM image of h-BN nanopowder

From the Figure 5.9, the particle size of the powder is less than 100 nm, when was obtained by a SEM, with 30 kX magnification and a scale of 100 nm.

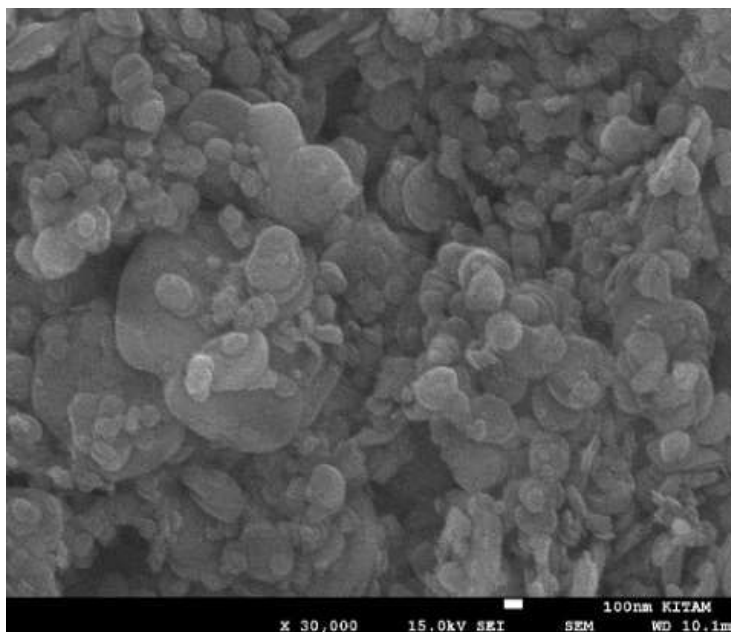


Figure 5.10. SEM image of MoS₂ nanopowder.

Figure 5.10 shows the MoS₂ nanopowder can see the distribution in the particle's size with a magnification of 30 kX, we notice a grain size of about 40 nm.

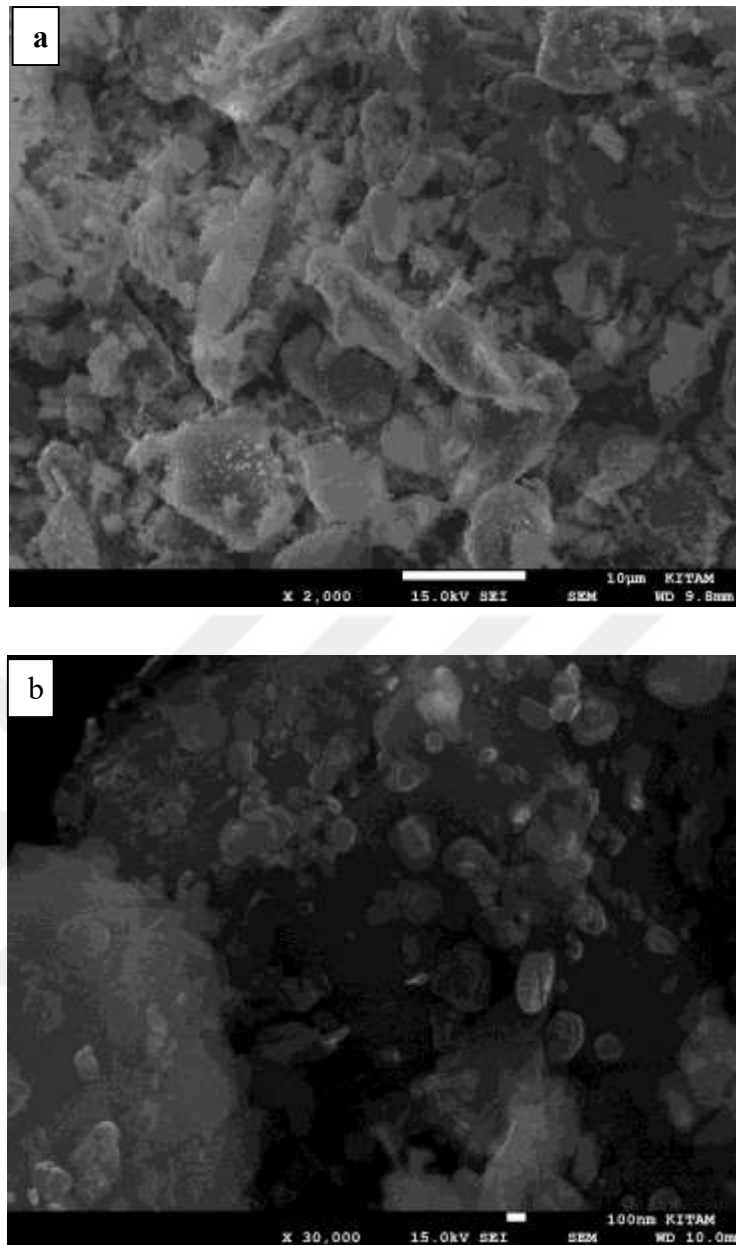


Figure 5.11. SEM for hybrid powder of Al-Si+ 2 % MoS₂-BN; a) 2 kX, b) 30 kX

After mixing the nanopowder and the micro powder, the characterization by SEM was achieved in different magnifications from (a) 2 kX with 10 μm scale and (b) 30 kX with 100 nm of scale in Figure 5.11. We notice the particles' homogeneity and presence at a scale of less than one hundred nano meter. This is evidence of the successful mixing process without additives, of dry mixing conditions, and without agglomeration.

We overcame the disadvantages of mixing with wet conditions or mixing during the melting process, so the process of producing

nanocomposites by powder technology method gave us satisfactory results for the final compound.

In the picture of Figure 5.11-b with a magnification of 30 kX and a scale of 100 nm for nanocomposite of Al and Si added by weight of MoS₂ and BN a hybrid additive with only 2 %. The nanoparticles are still clear for the nanopowder of MoS₂ and BN.

5.4. Energy Dispersive Spectrometry (EDS)

EDS showed us the results of the analysis of various elements of powders; most of them with peaks, as expected, are Al and then Si because it is an essential alloy with mixing ratios that almost obscure the rest of the elements.

Figure 5.12 showed quantities of other elements, including O, whose presence is explained by the speed of Al interaction with it, and small percentages of C and Fe for a small part of the sample shown by the map of the distribution of elements in the spectrum.

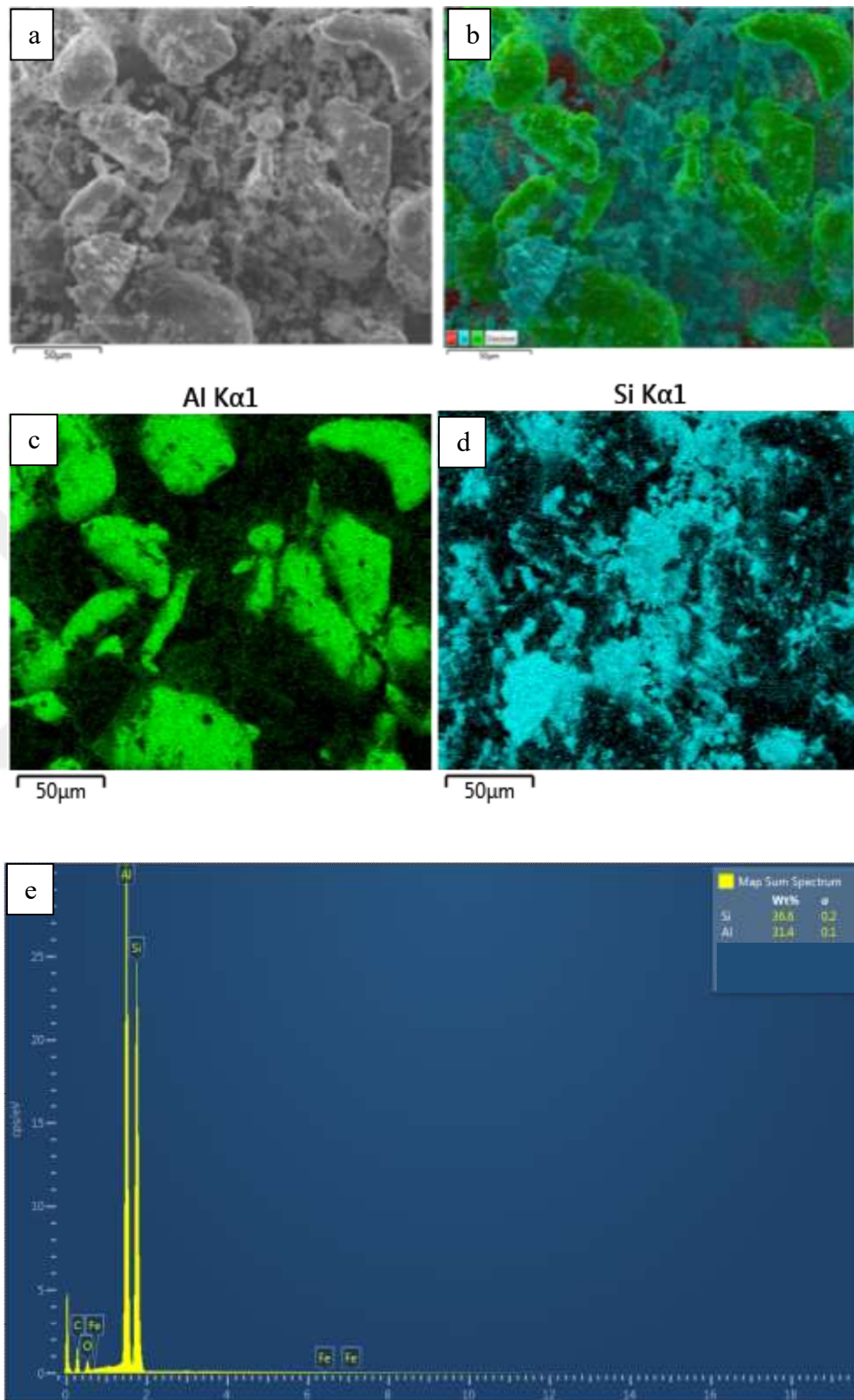


Figure 5.12. EDS of Al-Si powders; a) Al-Si image, b) Al-Si mapping, c) Al element, d) Si element, and e) spectrum of elements.

From the first image taken with an electron microscope at a scale of 50 μm , the grain size of Al appears clearly and homogeneously, as well as the following images of Si overlay, forming Al 12 % Si powder. Elemental analysis shows the clarity of the higher peaks of the Al and Si elements after preparing the basic alloy by ball mill.

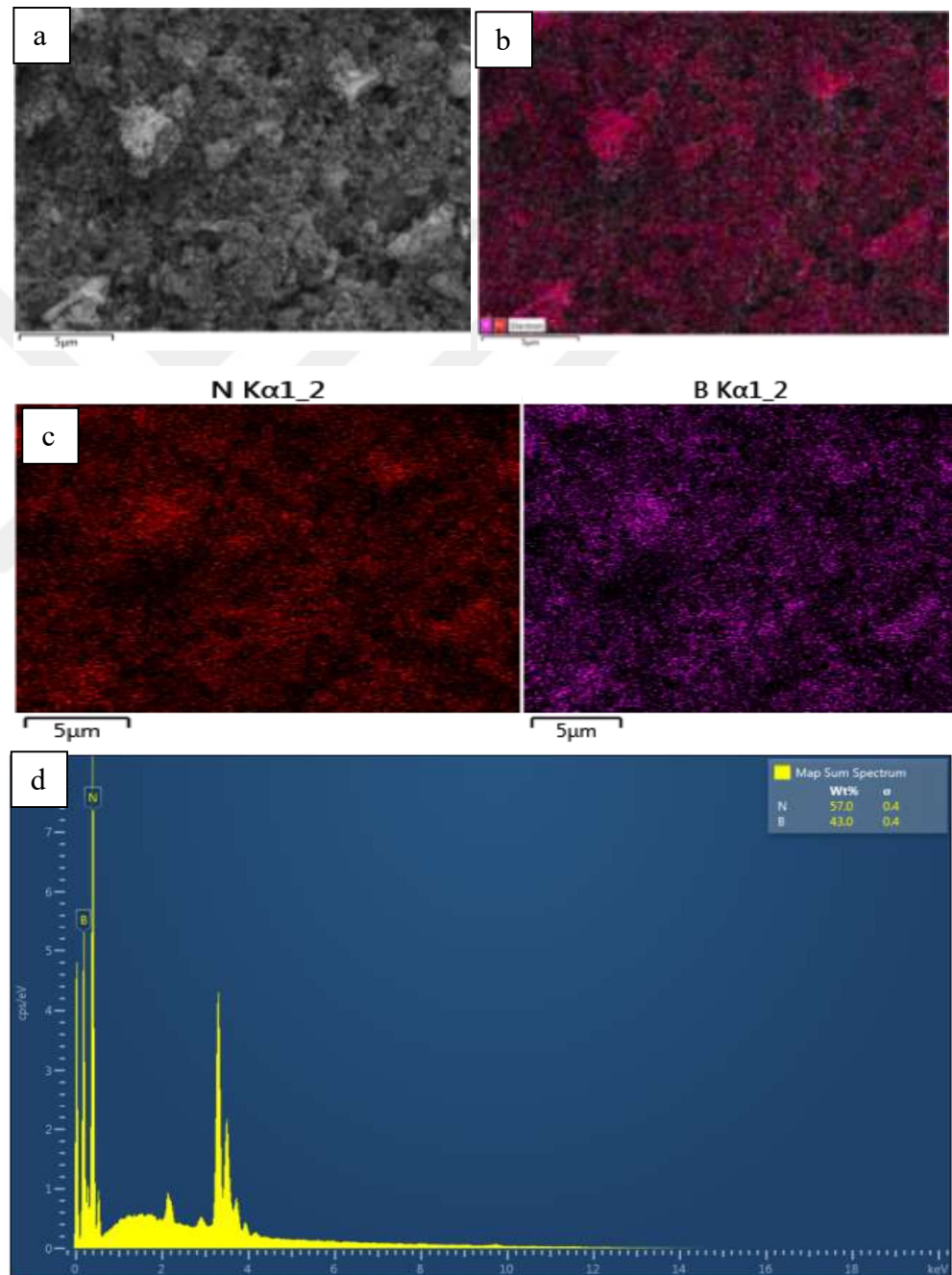


Figure 5.13. EDS of nanopowder h-BN a) BN image, b)BN mapping, c) element B and N, d) spectrum of elements.

Figure 5.13 shows the test by EDS, which can show the peaks of B and N. The analysis of the total spectrum gave the 57 and 43 wt. % respectively.

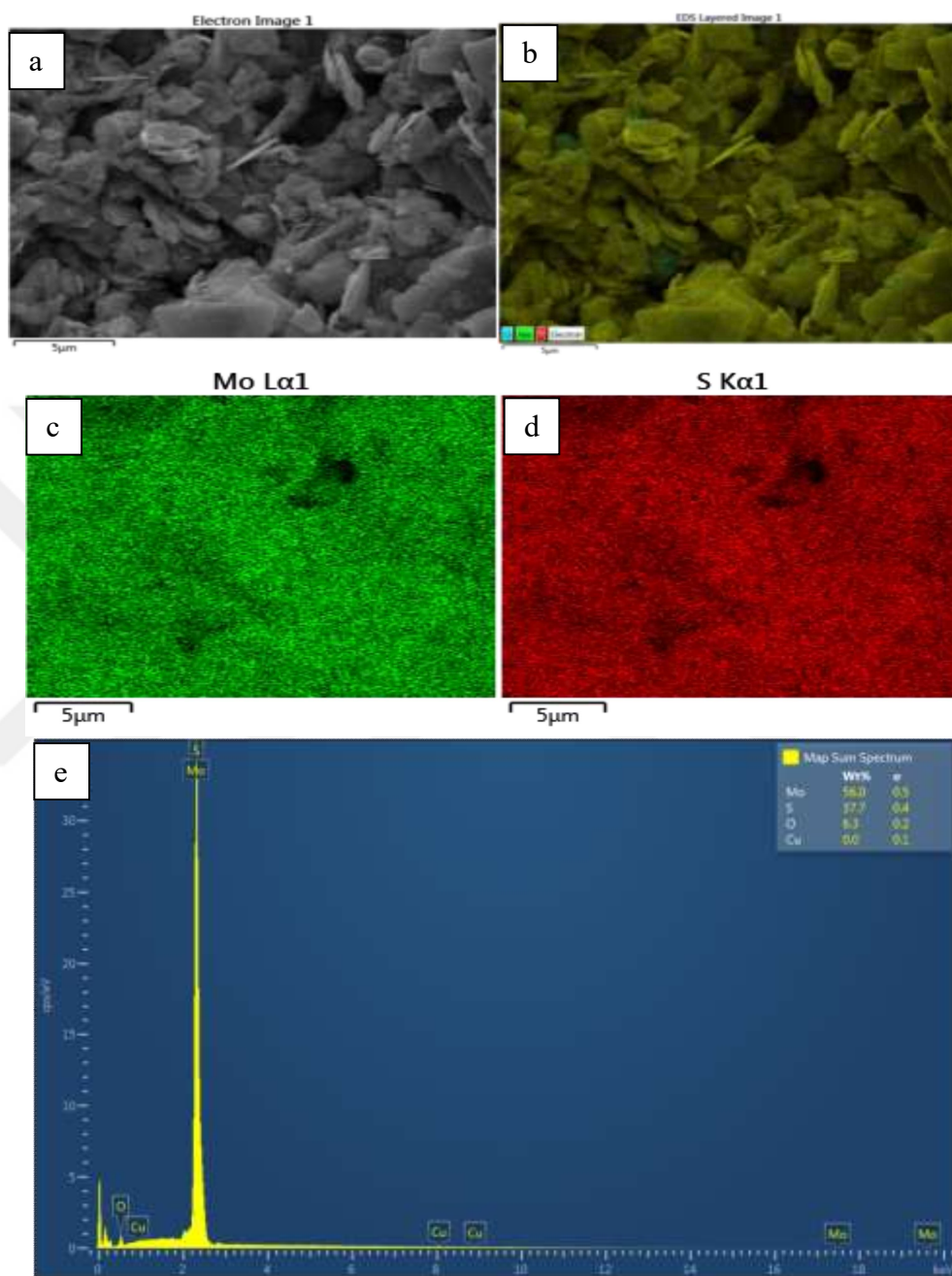


Figure 5.14. EDS of nanopowder MoS₂; a) MoS₂ image, b) MoS₂ mapping, c) Mo, d) S, e) spectrum of elements.

From Figure 5.14 it was seen the peaks of Mo and S from the MoS₂ nanopowder. Because the high energy of EDS enables it to show different elements and thus gives an accurate analysis for a specific part on the

spectrum map of the affected elements in the part of the prepared mixture. The total spectrum gave the highest weight percent of Mo reached 56 wt. %, and the 37 wt. % for S. The 6.3 wt. % O of oxygen appeared from surface oxidation of Al.

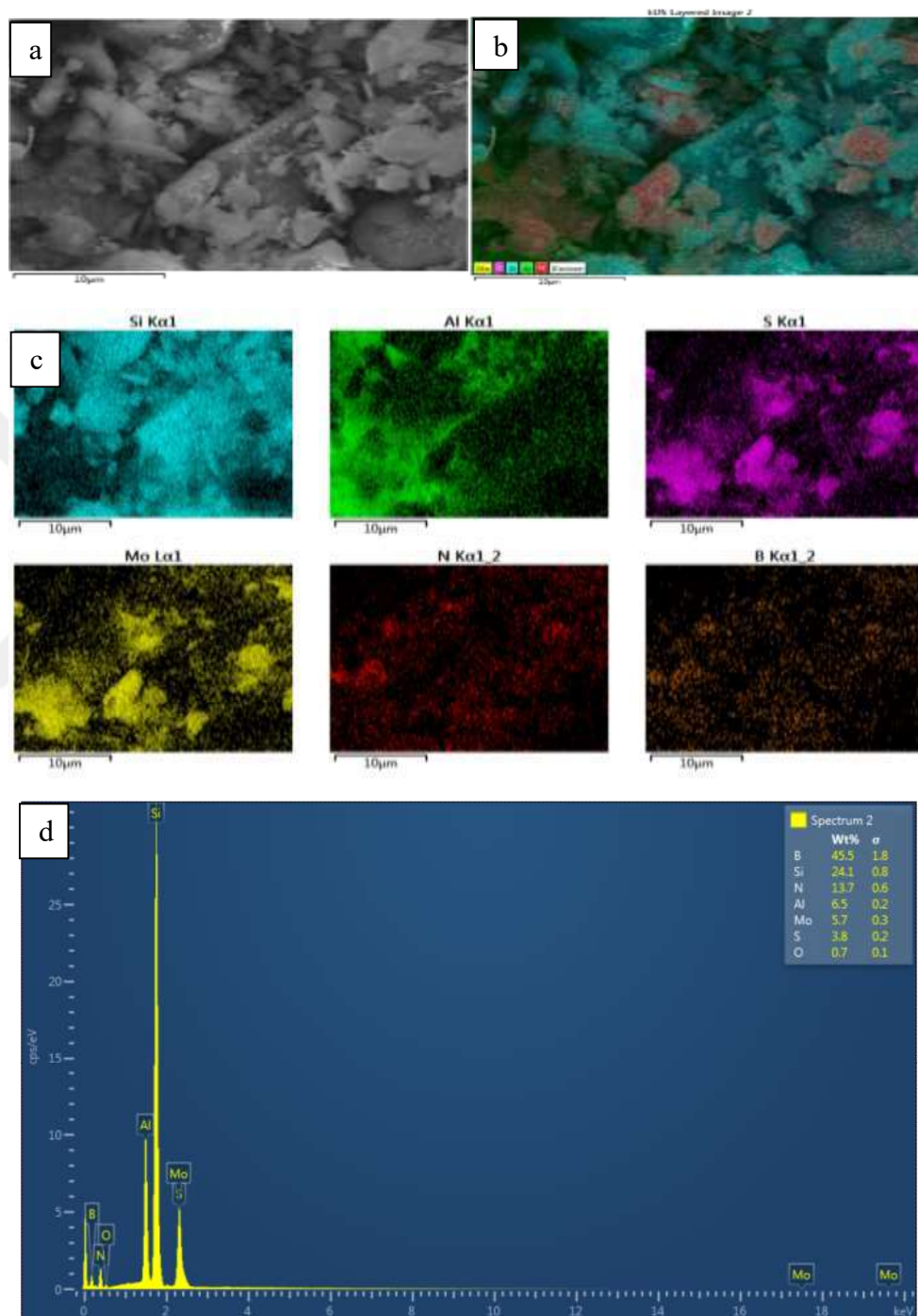


Figure 5.15. EDS of Al-Si-2 % MoS₂-BN; a) Al-Si-2% -MoS₂-BN image, b) mapping, c) all elements analysis, d) spectrum of elements.

EDS in Figure 5.15 for the hybrid nanocomposites gave at this region

of the sample the faraway from expectations because the highest peak was for Si with 24 %, and the most percent for B was 45.5 wt. % instead of Al was 6.5 wt. %.

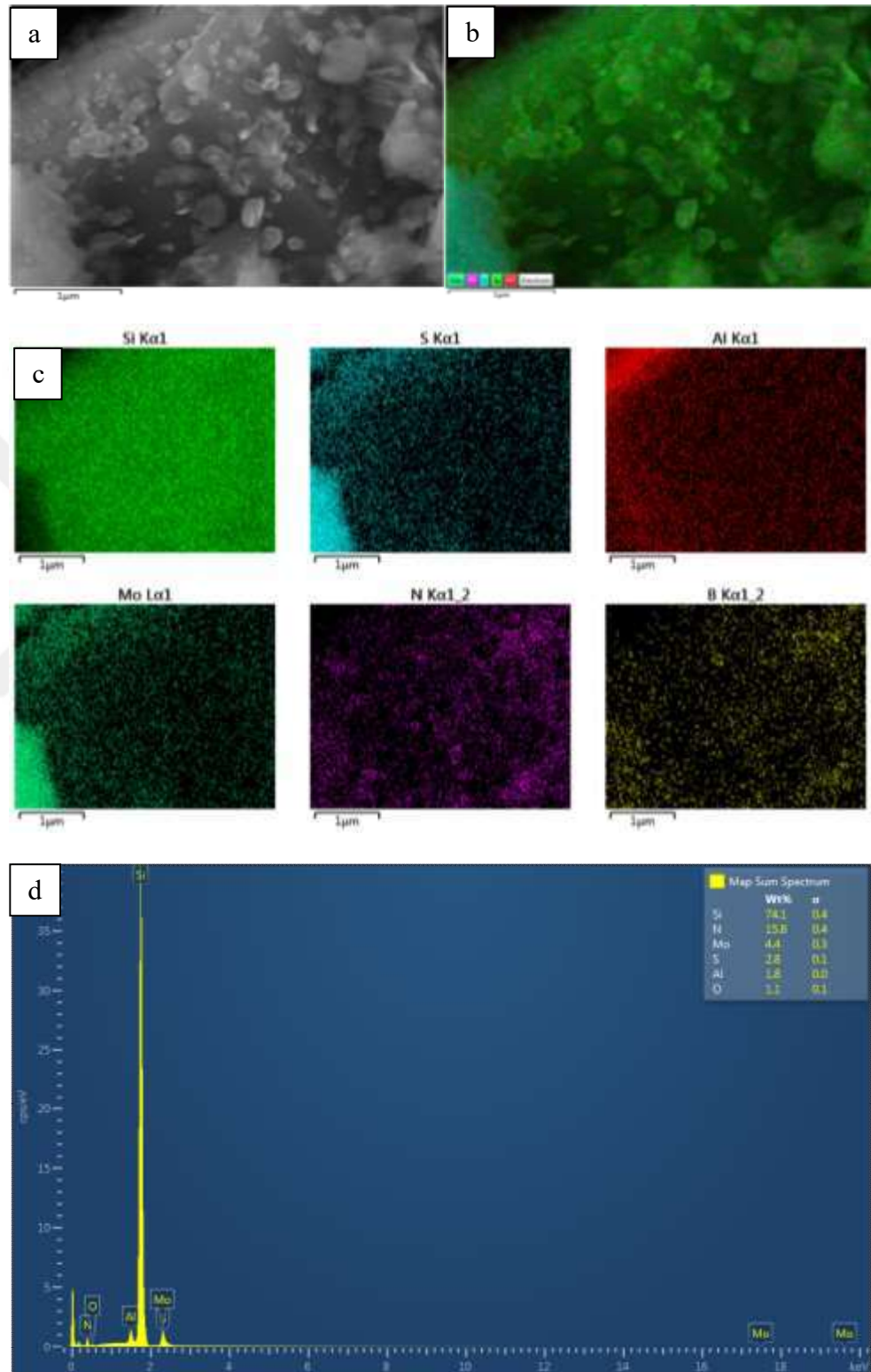


Figure 5.16. EDS of Al-Si- 4 % (MoS₂-BN); a) Al-Si-4 % -MoS₂-BN image b) mapping, c) all elements, d) spectrum of elements analysis

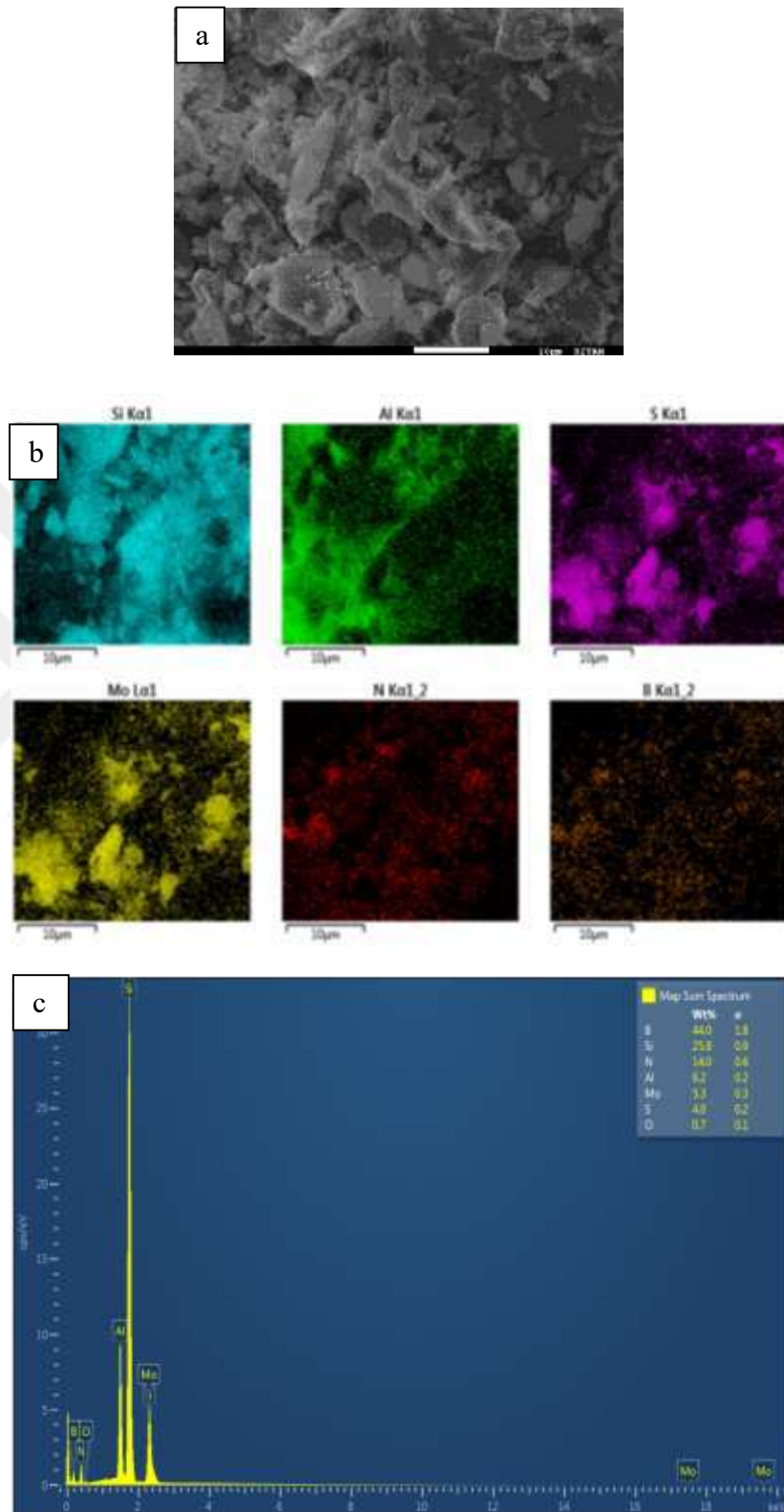


Figure 5.17. EDS of Al-Si 6 % ($\text{MoS}_2\text{-BN}$); a) Al-Si-6 % - $\text{MoS}_2\text{-BN}$ image, b) mapping all elements, and c) spectrum of element

5.5. Pressing powder

When it comes to Al-Si alloy properties, the compaction pressure plays a pivotal role. This pressure refers to the force applied during the manufacturing process of these materials, and it can greatly affect their characteristics and performance. High compaction pressures exerted on the Al-Si alloy result in a more dense microstructure, enhancing its mechanical strength and wear resistance. On the other hand, low compaction pressures lead to less density and increased porosity in the material, which can negatively impact its mechanical properties. Therefore, finding an optimal compaction pressure is crucial for obtaining desired features like improved hardness, thermal conductivity, and corrosion resistance in Al-Si alloys. It is through carefully controlling this variable that manufacturers are able to fine-tune their alloys for specific applications such as automotive engine components or electronic packaging materials. We decided to run a little experiment and put Al-Si powder under the mighty force of a hydraulic press cranked up to more than 7 tons. You see, when you apply such an extreme pressure on this fine powder mixture, it experiences some serious compression. The particles start bonding together, forming a solid mass that's tougher than nails. This process can be like magic, it takes loose powder and transforms it into a robust material ready to take on any challenge. We wanted to see just how far we could push the limits of this technology were we impressed. The resulting product displayed exceptional strength properties and left us feeling quite triumphant about our little experimentation with Al-Si powder and a powerful hydraulic press. (Q. Xu et al 2018, and Saboori, A. 2017).

5.6. Sintering of samples

By utilizing powder metallurgy, the solid-state mixing of the matrix and reinforcement materials eliminates the need for sintering to ensure intimate contact between the particles and promote diffusion bonding at the atomic level. As a result, the final product exhibits enhanced mechanical properties, including improved strength, hardness, and wear resistance. According to Kyung Ho Min et al. 2005, the sintering temperature is the

primary regulating parameter. A key factor is the choice of sintering temperature.

To investigate the impact of the Al size of the particles, sintering temperature, and sintering time on various aspects of the composite material, M. Rahimian et al., 2009 produced Al-Al₂O₃ composites using powder metallurgy. The temperature range of sintering is between 500- 600 °C. The sintering period was ranged from 30 to 90 min. The densification increased from 45 to 90 min. Additionally, it was shown that denser structures emerge at higher sintering temperatures due to faster diffusion rates. Accordingly, we selected 550 °C and 90 min. for our experiments.

5.7 Surface Microstructure and Hardness Tests

The samples that completed sintering were prepared for microscopy testing, where surface smoothing was carried out using SiC paper and three types starting from the coarse number 320. In the second stage, we replace it with 500; the last is 1000, being the smoothest on the surface. We performed the process with normal water flow to reduce friction and as a cooling medium.

The process continued by polishing the surface after it had been smoothed in the previous stage. A fine cloth and Alumina paste with a grain size of 1 µm were used. The exposing solution has been prepared for the metal surfaces and is considered a medium for corrosion, where we want to show the crystalline boundaries of the grains that make up the composites.

The solution prepared from 5 mL of Nitric acid and 3 mL of Hydrochloric acid is added to 190 mL of distilled water, placed on the sample's surface, and then washed with normal water; thus, it is ready for microscopic examination and hardness test.

An optical microscope was used to examine the surface structure of the sintered samples at different magnifications, as shown in the pictures in Figure 5.18.

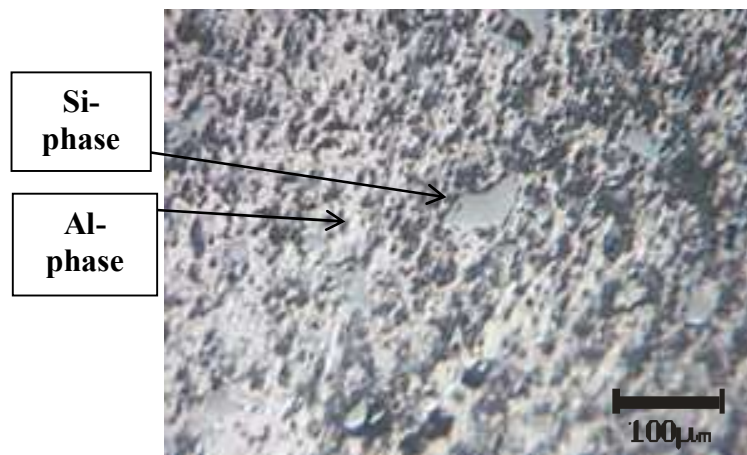


Figure 5.18 Microstructure of sintered Al-12 % Si alloy

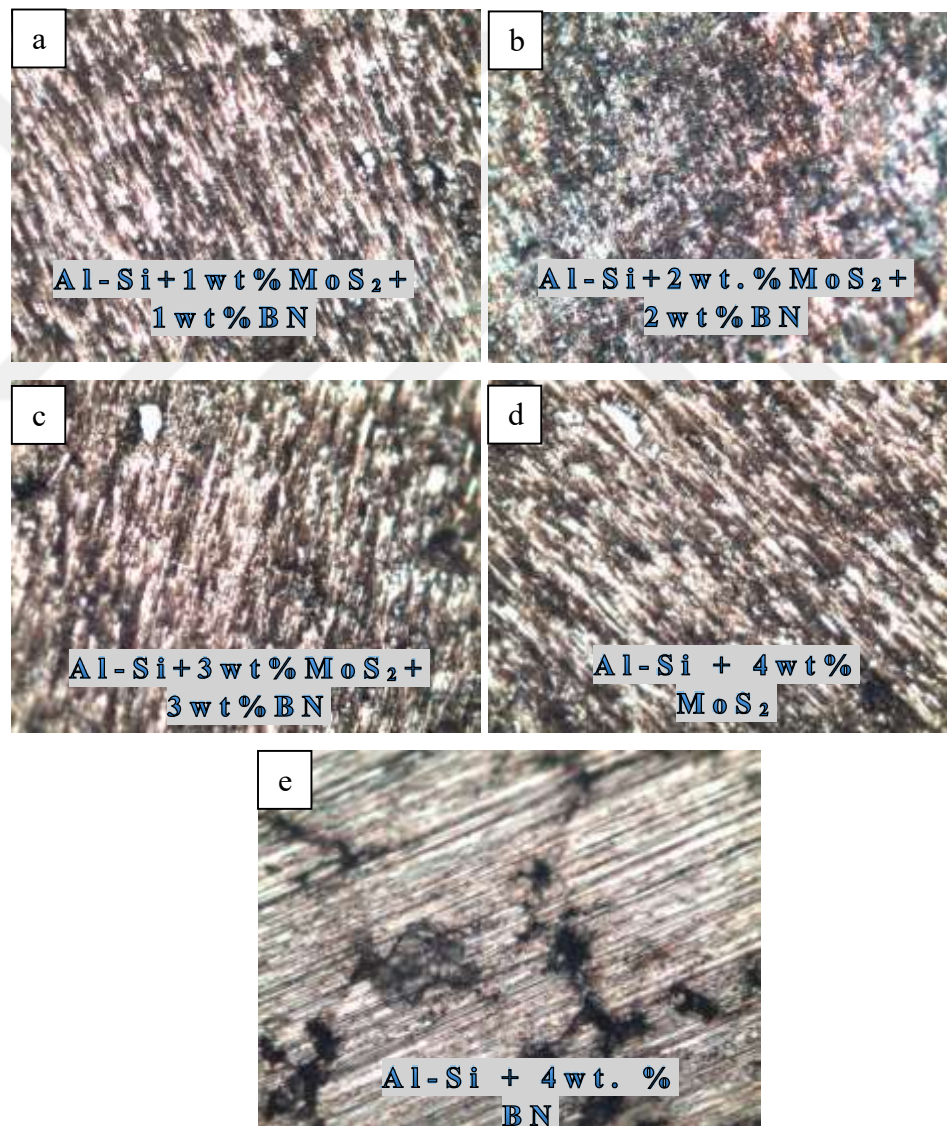


Figure 5.19. Images by optical microscopic for Al-Si hybrid nanocomposites after sintering

From the pictures shown above, which were taken from a polarized light microscope, the composition of Al with Si clearly showed us. The measurements of the granules are at the micrometer level. In other images, the nanometer cannot be seen by light microscope after the sintering process. Also, nano granules added to the base alloy do not appear, why? There was a process of agglutination of the micrometric granules with it. Therefore, we conducted an investigation before the pressing and sintering process, to ensure good mixing and the effect of combined or hybrid nano-addition. These tests were carried out at the stage of the mixed powders and the resulting samples after examining the wear by electron microscope, which we saw earlier, and we will see it later.

Hardness has significant results in the specifications of the product's composite, as for the type of examination using any device; it depends on the kind of material from which the product is prepared and the method of manufacture.

Concerning the manufacturing method and the materials in the nano form, which represent the case of our project, the method of powder technology and for the manufacture of acceptable samples had to check the micro-hardness, and the Vickers scale was chosen.

Multiple examinations were carried out for one sample, some of them 3 readings, and sometimes 5 repeated readings by taking different locations on the surface, and an average of measurements for the diameters observed was taken from the trace. One of the variables affecting the hardness test is the applied load, which was selected as 1.9 Newton and a loading time of 15 s. And in Table 5.2 shows us the readings obtained from the first alloy of Al-Si and, with it, nanocomposite alloys MMNCs.

Table 5.2 Hardness results for samples.

No.	Sample	HV (kg/mm ²)
1	Base alloy (Al-12 wt. %Si)	87
2	Base + 2 wt. % (MoS ₂ + BN)	112
3	Base + 4 wt. % (MoS ₂ + BN)	124
4	Base + 6 wt. % (MoS ₂ + BN)	134
5	Base +4 wt. % MoS ₂	132
6	Base +4 wt. % BN	145

Table 5.2 shows the different hardness, and we notice a significant increase from the base material after the additions of hybrid nanoparticles in proportions of 2, 4, and 6 %. And also the increase in hardness for one addition of nanoparticles compared to the base alloy.

All alloys with one or two types of nanoparticles added to them are called MMNCs. As for the base alloy of Al and Si, it can be called either metal matrix composite (MMC) or Al matrix composite (AMC).

We noticed the highest hardness achieved by adding one nanoparticle only, which is about 4 % of BN, which is higher than the hardness we got from two additions or even just by adding MoS₂. This result may be good in some aspects and bad in others, as we may show later, and its effect on the microstructure of MMNCs.

Emanuela et al. 2021 found that Al alloys' hardness ranged between 115-132 Vickers hardness measurements. The hardness is affected by several factors, including the density of the sample, the method of manufacture, and the temperature.

Al, as it is known, can be treated thermally, and it produces different hardness phases for us, according to the microstructure. Since we made the Al-Si alloy by powder technology, each stage of the work affects the resulting hardness of the final microstructure. The first stage is the mechanical mixing process of powders, and the influencing factors are; the ball mill's time, the number of balls used, materials, and ambient conditions. In the second stage, compression, the influencing factors are; temperature and ambient conditions, applied pressure value, and type of mold used. The third and final stage is sintering: it is also a governing stage, the most important of which is temperature control, isolating the sample from the outside environment and protecting it from the reaction of Oxygen.

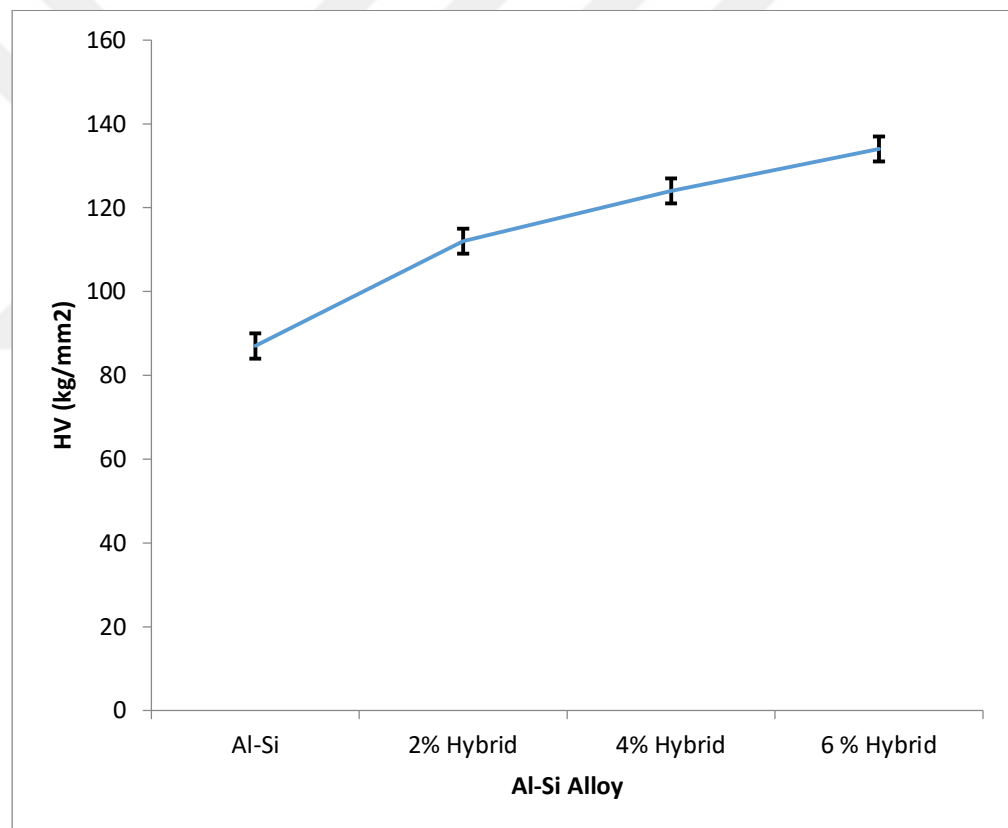


Figure 5.20. Effect of wt. % (MoS₂+ BN) nanopowders hybrid addition on hardness.

From Bhandari, P. (2023), we used standard deviation formula to estimate the variabilities for samples;

$$S = \frac{\sqrt{\sum (X - \bar{x})^2}}{n-1} \quad (5.1)$$

S: standard deviation, n: number of sample, $\sum$: sum of., X: each value, $\bar{x}$: sample mean.

Figure 5.20 represents the relationship between the hardness and its change with the percentage of the addition of the hybrid nanoparticles MoS₂+BN. From the diagram in Figure 5.20, we noticed an increase in the hardness with the increase of the hybrid nanoparticles, and the hardness was the largest at about 6 %. This effect was evident from the first nano-co-addition of 2 %, which is higher than that of the Al-Si alloy. And the average increase between high and low is at 4 % as a hybrid nanoparticle addition of MoS₂ + BN. The results of hardness and standard deviation as like for (J. Kuma, et al., 2020). The production of MMNCs by powder method and mechanical mixing significantly improved the hardness, which reflects the product's success and will significantly reflect the wear resistance tolerance. So, the hardness has a significant indication of the values of mechanical properties and specifications, as in previous research, it had given an inverse change in the relationship between hardness and wear resistance.

5.8. Density and Porosity Measurements

Our physical examinations were based on the Archimedes principle for floating bodies and liquid displacement. According to the American Society for Testing and Materials ASTM D792, the density and porosity of the samples produced by powder technology were calculated after the completion of sintering.

There are two equations previously used by (A. F. Hussien, 2010) and also (M. J. Fouad, 2014) in their researches after preparing nanocomposites, as we will explain below:

To find the density, the weight is found while the sample is in a suspended state in the air, and then we immerse it in a suspended state in the liquid. We also take the second reading of the weight. We applied the results to the equations as mentioned in chapter 4, noting adding the value of the density of the liquid used, which is ethanol; we first find the density of each sample that was weighed twice and then calculate the percentage of the gaps

within the composite structure. The tests were carried out at 20 °C, and the density of ethanol was consistently 0.7893 g/cm³.

Density and porosity measurement results of (Al-12Si) and (MMNCs) samples sintered at 550 °C for 180 min are presented in Table 5.3.

Table 5.3 Density and porosity calculations for Al-Si-nanocomposite alloy

Sample	Reinforcement content wt. %	Density g/cm ³	Porosity %
Al-12 wt. % Si (base alloy)	0	2.580	12.60
Base+2% (MoS ₂ +BN)	2 wt. % (MoS ₂ +BN)	2.601	9.88
Base+4 % (MoS ₂ +BN)	4 wt. % (MoS ₂ +BN)	2.648	9.75
Base+6% (MoS ₂ +BN)	6 wt. % (MoS ₂ +BN)	2.781	8.65
Base + 4 % MoS ₂	4 wt. % MoS ₂	2.712	9.50
Base + 4 % BN	4 wt. % BN	2.611	9.70

Table 5.3 shows that when MoS₂+BN hybrid nanoparticles are added to the basic Al-Si composite, the porosity ratio decreases, i.e., the gaps inside the new structure, and the density increases having a direct relationship with the increase of the added nanoparticles.

By looking at the calculations in the table for the porosity readings, it was found that all the resulting values are less than 1 %, and this is a good indication that the product is cohesive by the structural structure and that the pressing and sintering process is successful. Through experiments, if we reduced the pressure used in the pressing process, we would not have obtained samples with the required specifications of hardness and density, and thus affect the values of wear resistance rates. You can refer to other

researchers' results Francis et al., 2012 to compare their results.

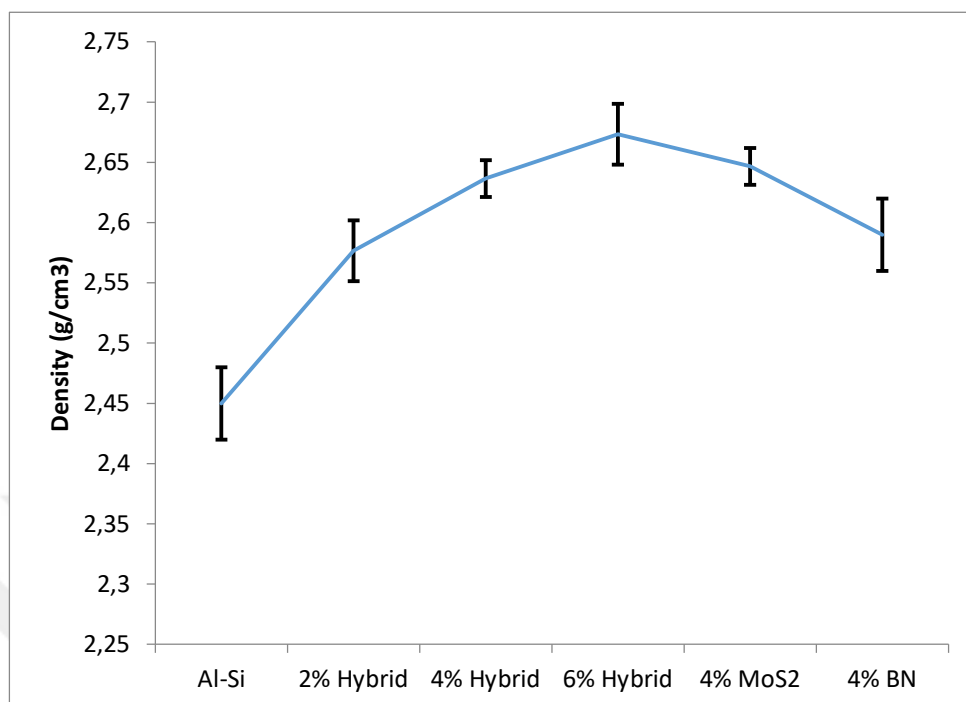


Figure 5.21. Density of Al-Si alloys after sintering.

It is noticed that the best results are from hybrid nanocomposite of (Al-12 % Si) + 6 wt. % (MoS₂+ BN) samples after sintering; the average percentage of porosity friction is 0.0865. There is an inverse relationship between density and porosity because the higher the porosity, the lower the density, and vice versa; the results are better for our product to be free of defects with the least percentage of gaps. Also, the increase in hardness is related to fewer gaps and an increase in density, so the relationship is direct between density and hardness and inverse with porosity. So, the possible effort on the practical side of manufacturing for any sample with low porosity is the closest to a higher of the wear resistance values. It is possible to observe the results after conducting a density test for the samples by applying Archimedes' theory, and a similar density appeared for Al alloys, which is 2.7 g/cm³.

A small deviation percentage appeared in the densities and their convergence in the cases of nano-additions. This is consistent with the lack of gaps and because of the pressure used during the manufacturing process by

cold pressing and the use of the hot sintering method at 550 °C. The temperature, pressing pressure, and adequate shielding against Oxygen entrance during sintering are the factors that affect density and porosity. We reached the highest-pressure values to ensure the success of the sample compression process after experiments with compressing mixed powders, and it was about 7 tons. As for the protection system, when sintering at high temperatures, we conducted a vacuum process by means of a vacuum pump, and then Argon gas was pumped before heating began, all to ensure that we obtained a product with the least structural defects. From previous studies, we can discuss their results in this way (E. A. Majeed 2006).

5.9 Wear Rate

The most important characterization in our research is the effect of adding nanoparticles to the Al-Si alloy on the wear resistance rate. Since the main alloy was made by powder technology and at a rate of 12 % Si addition, i.e., with a eutectic phase structure, then this composite is highly wear-resistant and therefore used to manufacture engines. In our current study, improvements and innovations were made to the idea of self-lubrication within the same alloy composition to extend life by resisting friction and reduce pollution by using conventional oils. There have been several examinations of alloys manufactured only from the grains of the Al-Si micro powder and nanocomposite alloys with nanoparticles added. The wear rate was calculated using the weight loss method after the friction procedure, the applied weight was fixed, and the change in the test time was adopted. The results for each sample were represented by a curve to compare them with each other and were discussed to conclude the best. The test applied by the abrasive instrument is shown in Figure 5.22 at a fixed load of 2.5 N and sliding distance equal to 20 m.



Figure 5.22. The abrasive test device

5.9.1 Wear For Different Periods

This test was applied for sintered samples with various hybrid nano addition from 2, 4, and 6 wt. %. The relationship between the test time on the X-axis and the wear rate on the Y-axis was represented.

The curve of the Al-Si alloy showed the highest wear rate, and a decrease in the rates appeared with the addition of nano-hybrid powders. (Al-12 % Si) and hybrid nanocomposites with 2, 4, and 6 wt % (MoS_2 +BN) under constant load (2.5 N) are shown in Figure 5.23 as well.

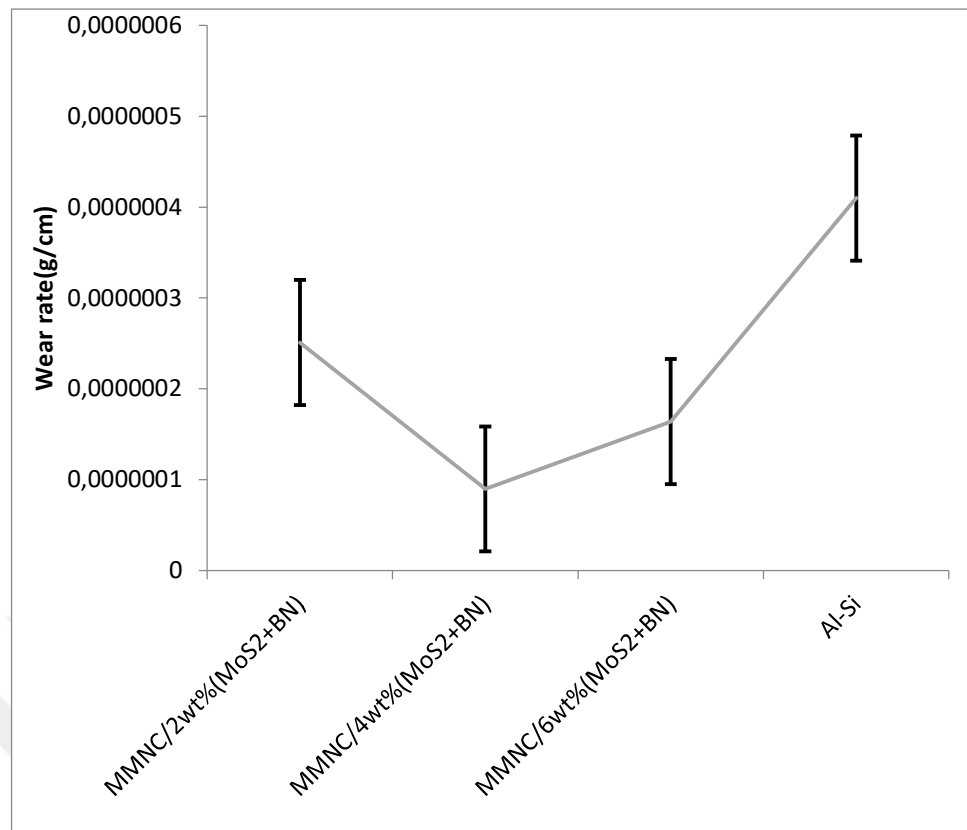


Figure 5.23. The relationship between the test time and wear rate.

Figure 5.23 illustrates the relationship between the dry friction time and wear of various alloys manufactured by powder technology. The constant load was more than 2.5 N because the weight of the arm in which the sample was installed was added to it. With the increase in the test time, there is an increase in the wear of all samples, knowing the load is constant, as mentioned above, but the weight loss is less for the nanocomposite hybrid, which means a higher resistance to wear.

It was found through the comparison of the resulting curves in the above figure that 4 % of the added nano-hybrid particles had a better effect than the other nanocomposite alloys. The reinforcement of the structure as a whole leads to an improvement in the wear resistance, which means that it gives self-lubrication to the compound by reducing the effect of friction, and thus reduces the resulting losses and changing the dimensions during working conditions against rough surfaces when dry friction and high temperatures.

5.9.2 Wear Different Test Weights of Hybrid Addition and Fixed Time

The effect of adding hybrid nanoparticles of ($\text{MoS}_2 + \text{BN}$) to the base alloy is shown in Figure 5.24 as the wear rate of MMNCs samples at a specific load (2.5 N) and a friction time of (15 min).

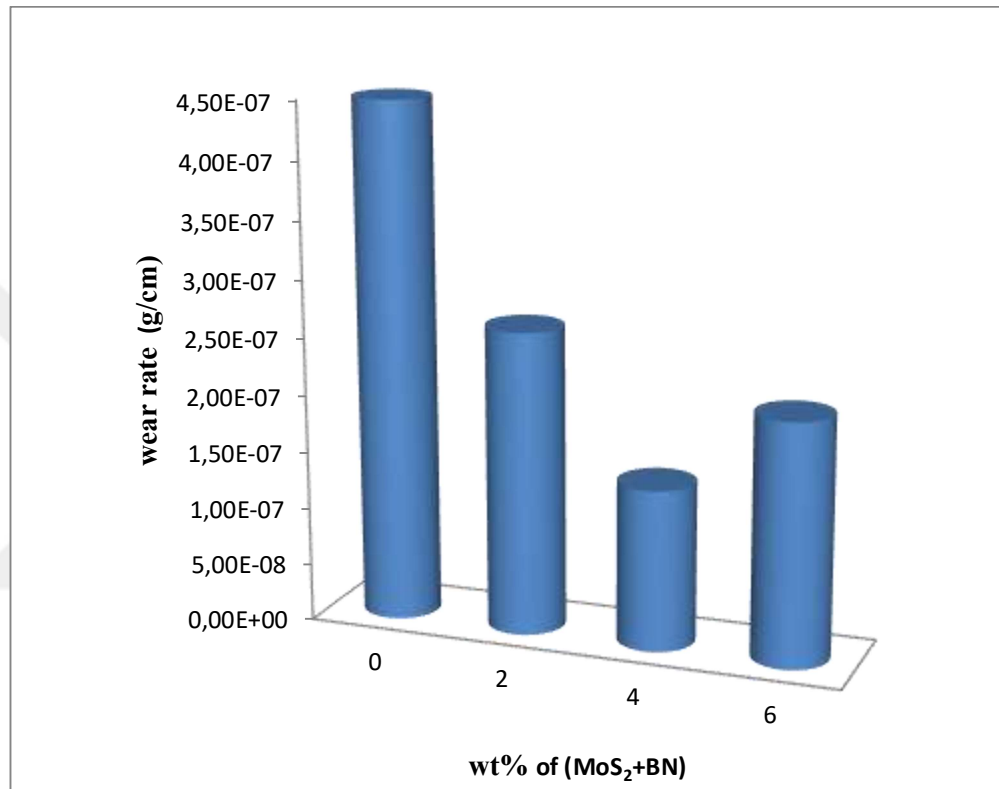


Figure 5.24. Effect of ($\text{MoS}_2 + \text{BN}$) hybrid nanopowder addition on wear rate for hybrid MMNCs samples under applied load (2.5 N) and constant sliding time (15 min).

The effect of the co-addition is evident by reducing the friction, which means that the nanoparticles act as self-lubricating and improve the microstructure of the manufactured nanocomposite. Where there was less lost weight for the samples to which nanoparticles were added, which led to a lower wear rate than the base alloy. ($\text{MoS}_2 + \text{BN}$) hybrid nanopowders added to the Al-Si matrix composite at applied loads of 2.5 N as shown in Figure 5.24, but some are increasing in weight loss in the alloy with 6 wt. % of hybrid addition of nanoparticles may be because of the increasing brittleness in the whole structure.

5.9.3 Effect of Nanoparticles as Single Addition on Wear Rate

From Figure 5.25, we called triple 4 %, such as the relationship between the addition ratios of only 4 % for one addition of BN or MoS₂ and a co-addition with the base composite.

As expected, it appeared in practice that the increase in the time of friction leads to a continuous decrease in weight for all materials. However, through the new nano-alloys, the effect of nanoparticles with self-lubricating properties demonstrated an improvement in frictional resistance.

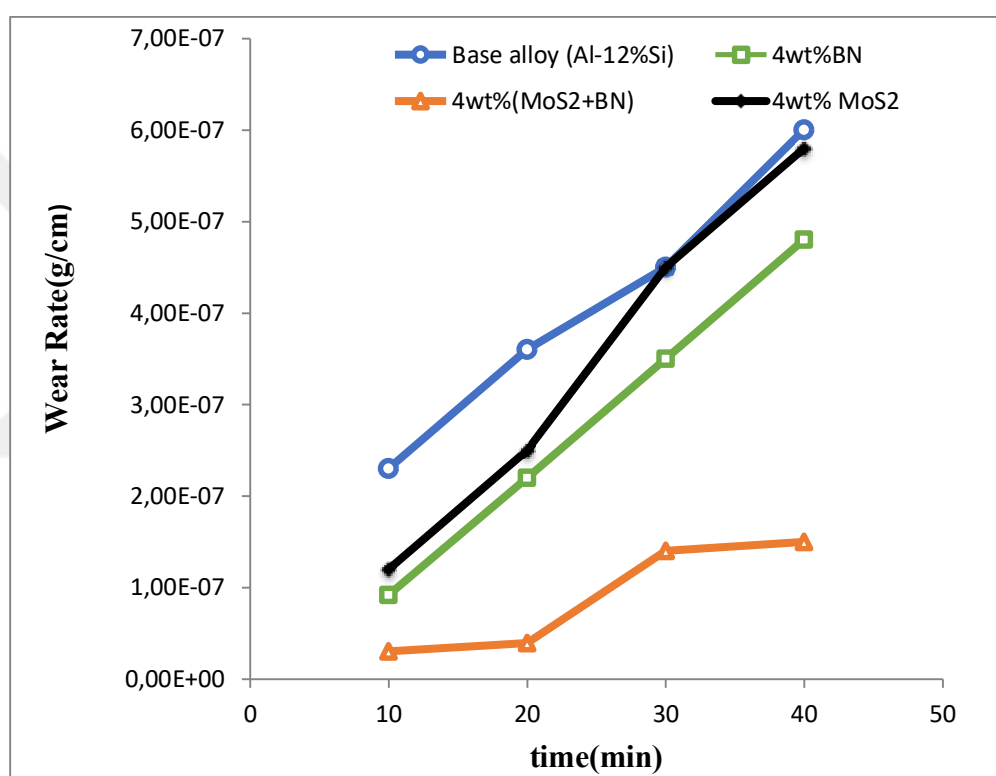


Figure 5.25. The relationship between wear rate with time for 4 % additions.

The combined addition of 4 % gave the best results for self-lubrication with higher resistance to friction and less weight loss. Let us compare the previous Figures (5.23 and 5.24) the percentage increase of 6 %, which deviates from what is required and fails to resist friction and perform the lubrication function. We will find that 4 % is balanced and moderate and gives a composition that performs the functional properties of self-lubrication and homogeneity of the distribution of grains. The BN gave the structure strengthening and obvious hardness, and the ratio of 2 % BN with 2 % of

MoS₂ had a homogeneous mixing without agglomeration that may lead to undesirable results.

The overlap between the base alloy and the addition of 4 % BN can be observed. An increase in the wear rate occurred as if there was no addition or no effect on the wear resistance as the base alloy.

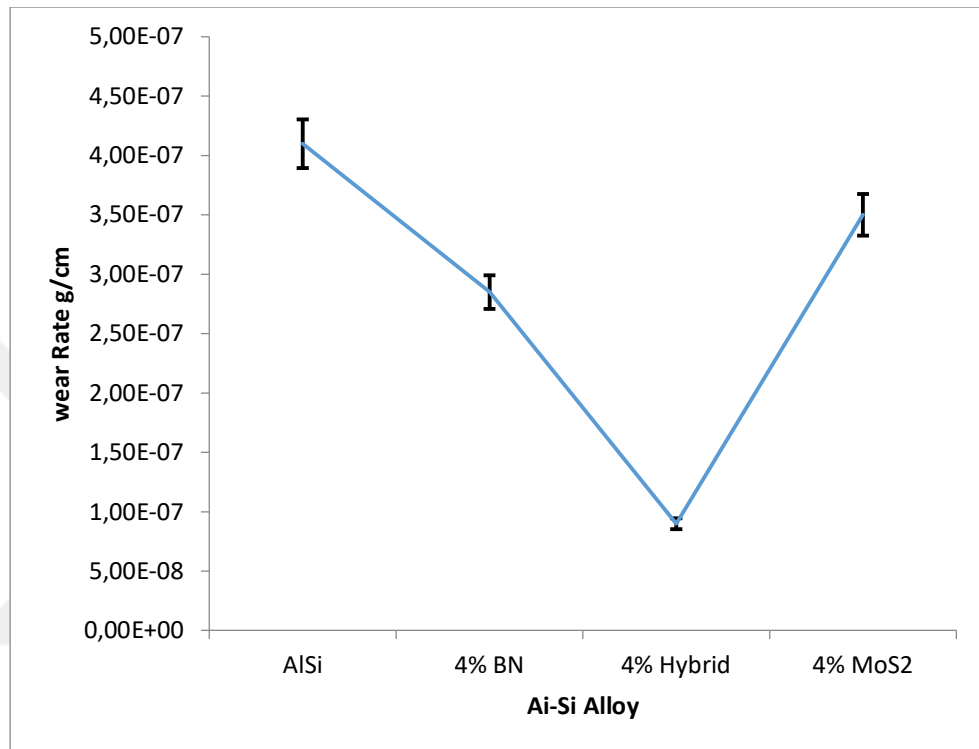


Figure 5.26. The average wear rate for Al-Si with 4 % addition.

The average wear rate is for adding one MoS₂ of only 4 %, which is somewhat acceptable but no better than the hybrid addition of nanocomposites.

5.10 Study of Worn Surfaces by SEM

The type of wear was studied by SEM, the imaging of surfaces after the friction procedure to identify the resulting topography and analyze the type of fracture that indicates the nature of the friction occurring and what is the effect of the additives or the manufacturing method to the method of examination. Different mechanisms of wear may be produced by one sample, including adhesive or abrasive type, which may differ according to the

composition if it is brittle or ductile. SEM images show the effects of the powder resulting from friction, as different sizes break them from one element or compound, and they are sometimes in the form of lumps or single fractions.

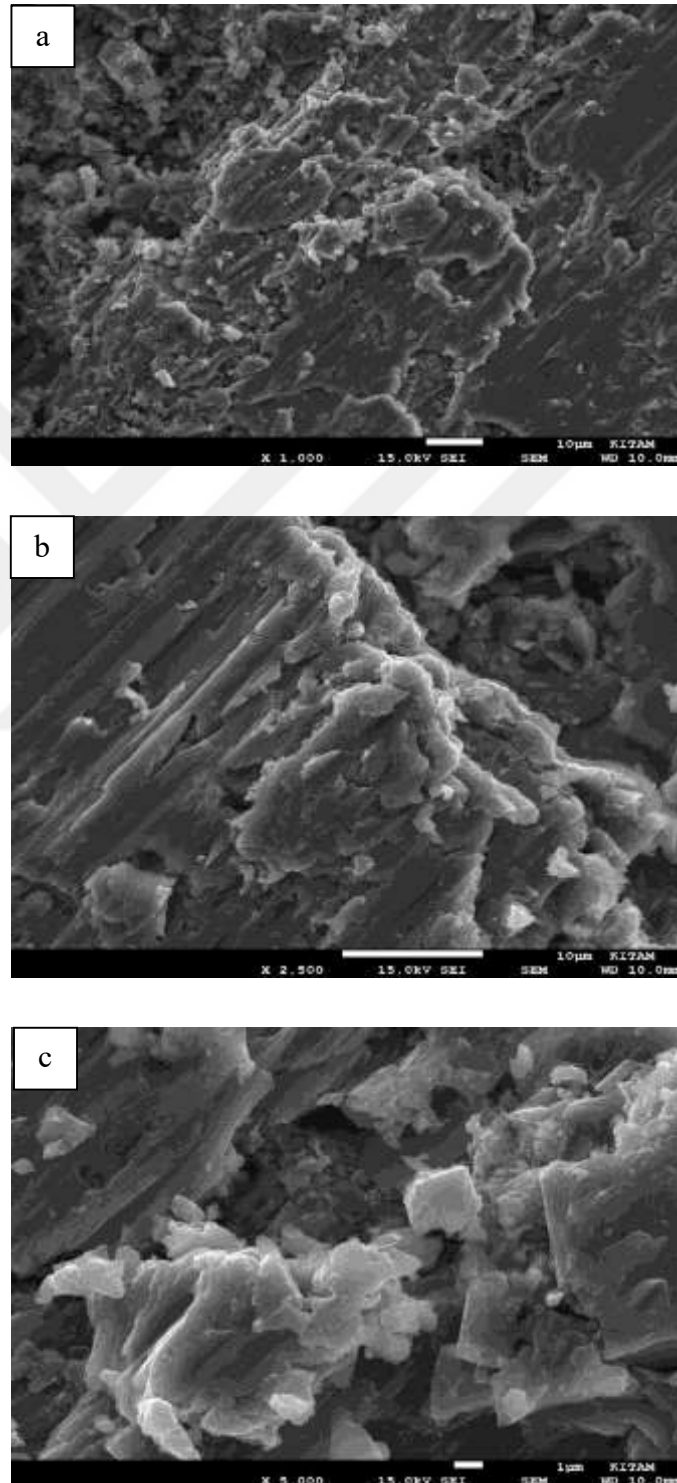


Figure 5.27. The surfaces of the basic alloy after the friction process; a) at 1 kX, b) at 2.5 kX, and c) at 5 kX magnification.

Figure 5.27 a, b, c shows the type of wear of the basic alloy of Al-Si without additives, where plastic deformation and adhesion between the particles result from friction with the opposite surface of the sample. The fragments break into an elongated shape and form of large lumps, which leads to a high weight loss of the sample during the friction test. Figure 5.27-c shows the SEM micrograph of the worn surface and debris, which reveals some features of the delamination mechanism and cracks.

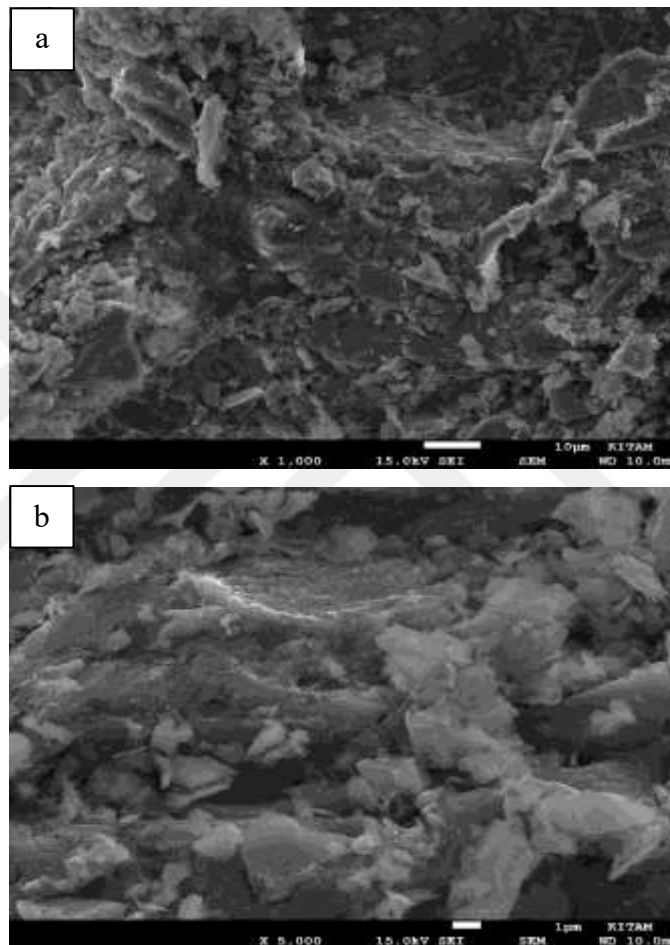


Figure 5.28. SEM images of the surface after wear of the 2 wt. % (MoS₂+ BN) hybrid nanocomposite; a) at 1 kX, b) at 5 kX magnification.

Figure 5.28 shows the effect of nanopowders 2 wt. % (MoS₂+ BN) that the alloy has strengthened and self-lubricating properties. It led to a tangible result from the images of the surfaces taken by the SEM of the parts that were subjected to friction. The nanoparticles acted as a lubricant, protecting the opposing surfaces when exposed to movement and reducing the effect of external load. Let us compare the previous images of the base material

without adding nanoparticles. We notice a significant and apparent change in the wear formed on the topography of the surface shape and exposure paths. Also, the resulting debris has become softer and turned into a fracture close to brittle without agglomeration. There is no significant weight loss as in the base alloy.

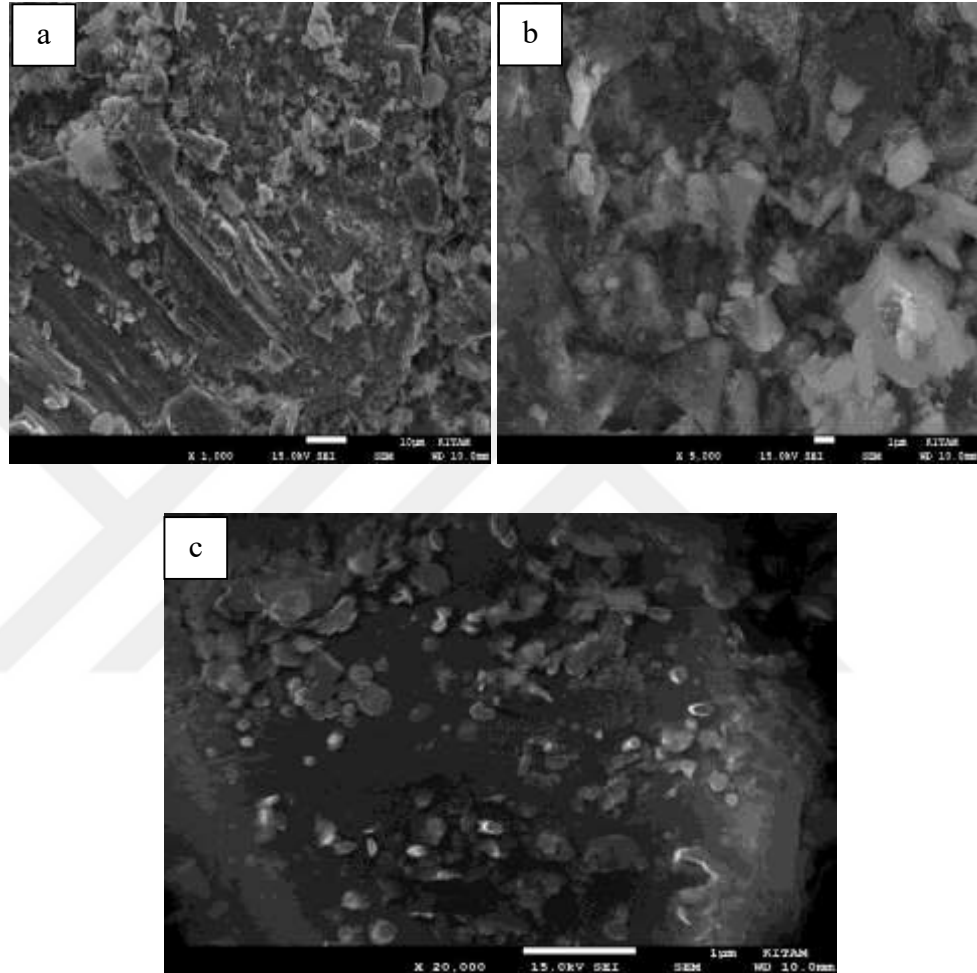


Figure 5.29. SEM image of the surface of nanocomposite with 4 % co-addition after friction; a) at 1 kX, b) at 5 kX, and c) at 20 kX magnifications.

In Figure 5.29 it is observed that fineness in the remaining grains on the surface of the sample that was subjected to the friction force, and this is a clear difference and an improvement in structure from the base alloy.

As we mentioned earlier, the mixing images of this alloy showed smoothness and homogeneity between the different powders, and the latter led to positive results in improving the wear results and the type of fracture with deformations resulting from friction. The small weight loss during

friction was evident from the microscopic structure that appeared from SEM examination, cohesion and bonding with the structure, no agglomeration, and if it cracked, it would fall into a fine powder. This leads to a small loss of dimensions if exposed to harsh working conditions such as heat or force.

From Figure 5.29-c, fine cracks may appear on the deformed surface, but some grains stop the crack from continuing or the failure of the specimen develops. Through the examination of surface, it was found that there is an overlap of two types of wear on the surface between some brittleness and ductile bonding, which stops the effect of friction and makes a protective layer on the surface. So the self-lubricating effect of a co-addition with the highest possible effect is the addition of a nano-hybrid by 4 %, and the strengthening function of the nanocomposite is added to it acceptably.

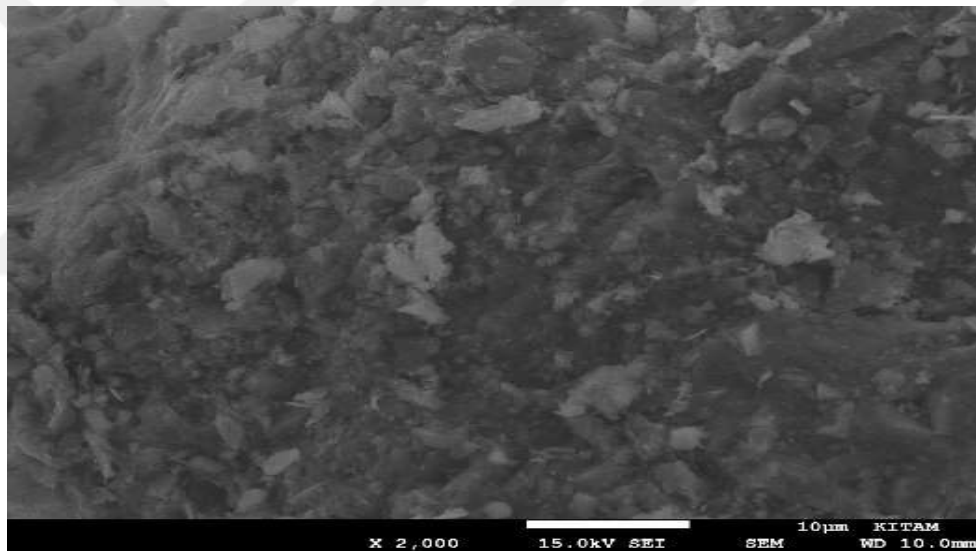


Figure 5.30. SEM image of surface of nanocomposite 6 % (MoS_2 + BN) hybrid addition of 2 kX magnification.

The image in Figure 5.30 shows the broken surface because of the structure's brittleness that contained 3 wt. % of MoS_2 and 3 wt. % of BN. 6 % MoS_2 + BN showed the wear mechanism of this sample in the form of powder disintegration and cracking brittle. We note that the surface is full of debris of scattered and unconnected grains; the amount of mixing may be more than the necessary limit to accommodate the overlap and homogeneity of the grains among them. Increasing the proportion of BN in the nano form gave a

negative effect on the structure; this sample only resisted friction for a short time. The failure occurred in a short period, and the obvious fracture was brittle; large pieces faded and other large pieces separated. In the worst nano-co-addition, the additional percentage should not reach this limit, which is 6 %, so we did not find the effect of reducing friction with the self-lubricating granules. The lumps that appeared in the mixing of the powder and the low wear rate value were confirmed by SEM images with the failure of this alloy.

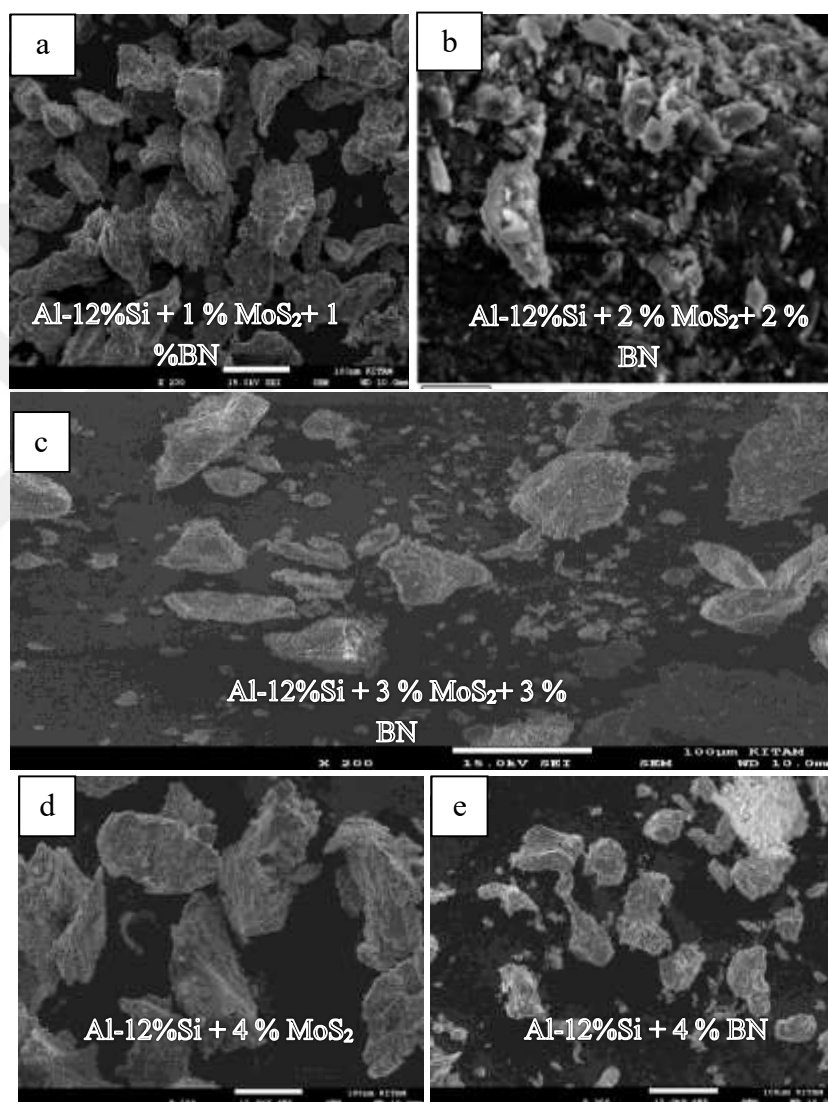


Figure 5.31. SEM of wear debris of hybrid nanocomposite of Al-Si and (MoS₂+ BN) nanoparticles

The wear debris of the nanocomposite is shown in Figure 5.31. As a result of the post-wear SEM images, it is shown that the resulting particles do not exceed 100 μm.

We can notice from the picture in Figure 5.31. a the roughness of the fracture residues are resulting from the wear process. 1 % of one nanoparticle with the addition of a second nanomaterial in the same proportion may have no effect on smoothing the microstructure of the base alloy.

From the second picture Figure 5.31.b we notice a somewhat acceptable homogeneity in the structure of the granules and lack of agglomeration. So far, and as a result of the various tests, this sample is the best in the tests, with longer life, due to its hardness, homogeneity in the crystal structure, and fineness of the microstructure affected by the addition of hybrid nanoparticles.

The third picture, Figure 5.31.c represents the result of examining the remaining sample that did not hold up much during the wear test. Co-addition or hybrid nanoparticles have a smoothing effect in some parts but agglomerate in many parts, and this is the reason for the brittle fracture of the sample surface during testing.

It turns out that increasing the ratios in addition to the hybrid nanoparticles is not in our favor. That is why we do not recommend a co-increase of more than four percent because of the failure of the sample containing 6 % of the nano-co-addition of BN and MoS₂. From the comparison with pictures Figure 5.31 d and e, we show the following: picture Figure 5.31.d shows one addition of MoS₂ and shows a kind of homogeneity and small grains compared to picture e) which shows large grains or clear agglomeration. Therefore, the increase in the percentage of adding a nanoparticle BN may be harmful to the microstructure, and this confirms that the previous increase of 3 % was harmful and agglomeration occurred when it was a hybrid addition to the third sample. We do not recommend adding more than 3 % for BN because the microstructure will suffer from fragility and agglomeration of the particles.

5.11. EDS of Hybrid Nanocomposites After Wear Test

For the importance of the characterization of the wear test, the friction test residue, which is a powder of different elements, these fragmented elements were collected from the tested sample and the friction test paper. The element analysis was examined by SEM, using the energy dispersive spectroscopy (EDS) feature. From the figures below, we can see the analysis of EDS for five samples of powders of debris after the wearing process achieved by the abrasive test.

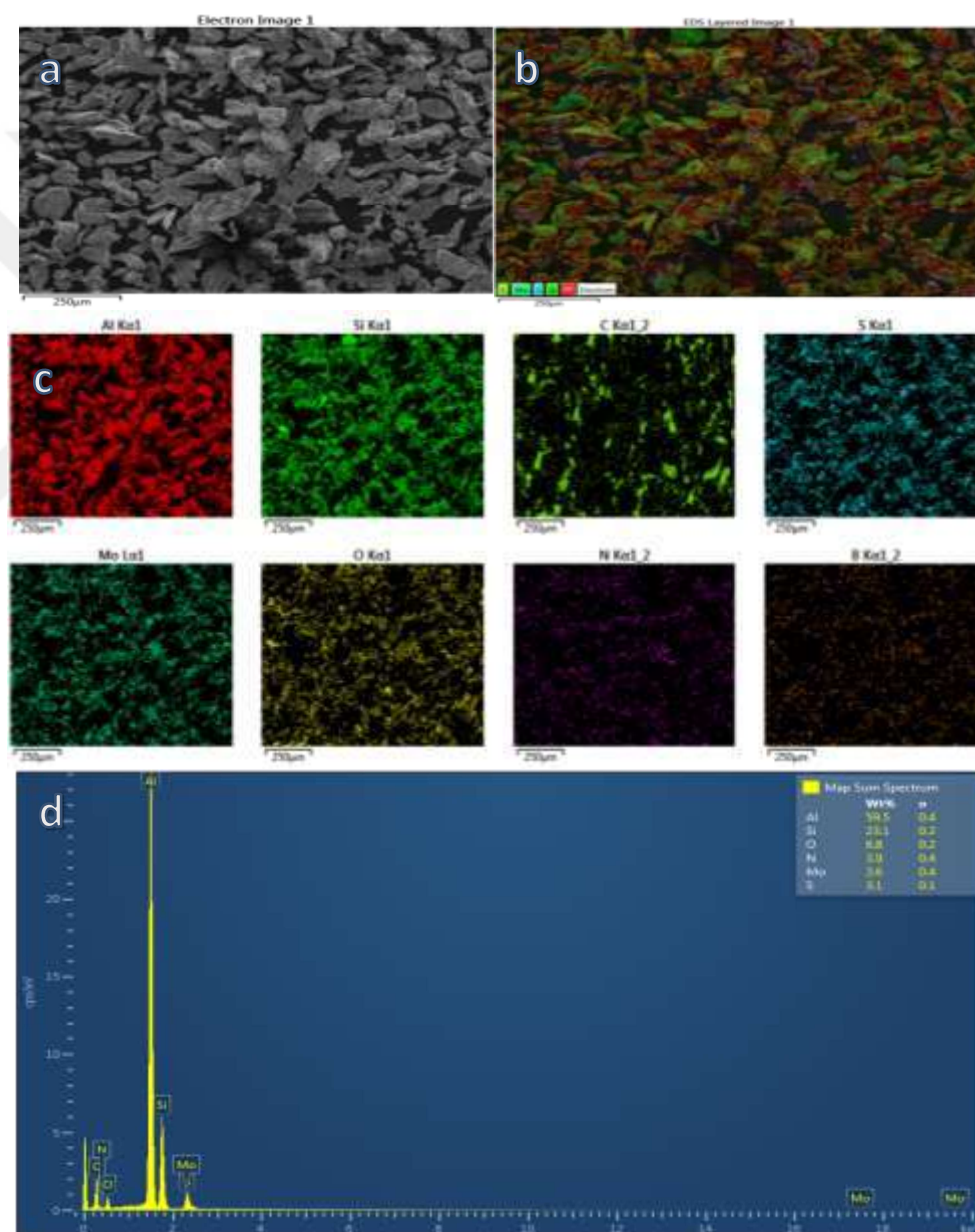


Figure 5.32. EDS of Al-Si base alloy + 1 % MoS₂ + 1 % BN. a) Al-Si-2 % MoS₂-BN image, b) mapping c) all elements, d) spectrum of elements

Figure 5.32 shows the wear debris analysis of Al-12 % Si and hybrid nano addition 2 % of MoS₂+ BN. The highest peak and weight % appeared to be the Al with 59.5 wt. % and the collection of variant percent of elements Si was 23.1 wt. %, other elements O, N, Mo, and S are also founded in different weights.

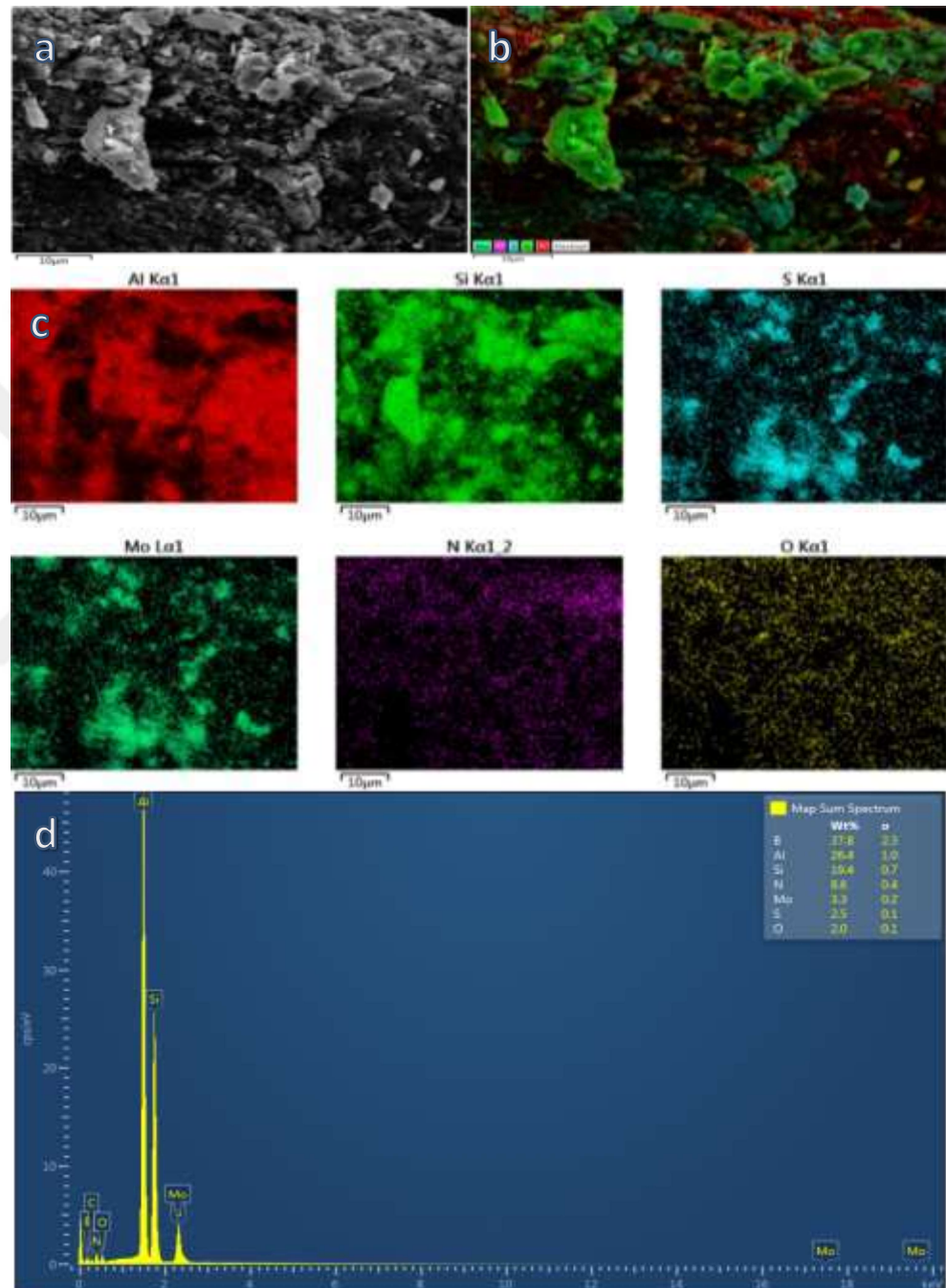


Figure 5.33. EDS of Al-Si base alloy + 2 % MoS₂+ 2 % BN. a) Al-Si-4 % MoS₂-BN image, b) mapping c) all elements, d) spectrum of elements.

Figure 5.33 shows the alloy with hybrid nano addition 4 wt. % MoS₂+

BN EDS test for wear debris. It shows the highest peak for Al, but the highest weight percent for B reaches 37.8 wt. %, and for Al, equal 26.4 wt. %. Si was 19.4 wt. %, N= 8.6, Mo= 3.3, S= 2.5, and some of the oxide up to 2 %.

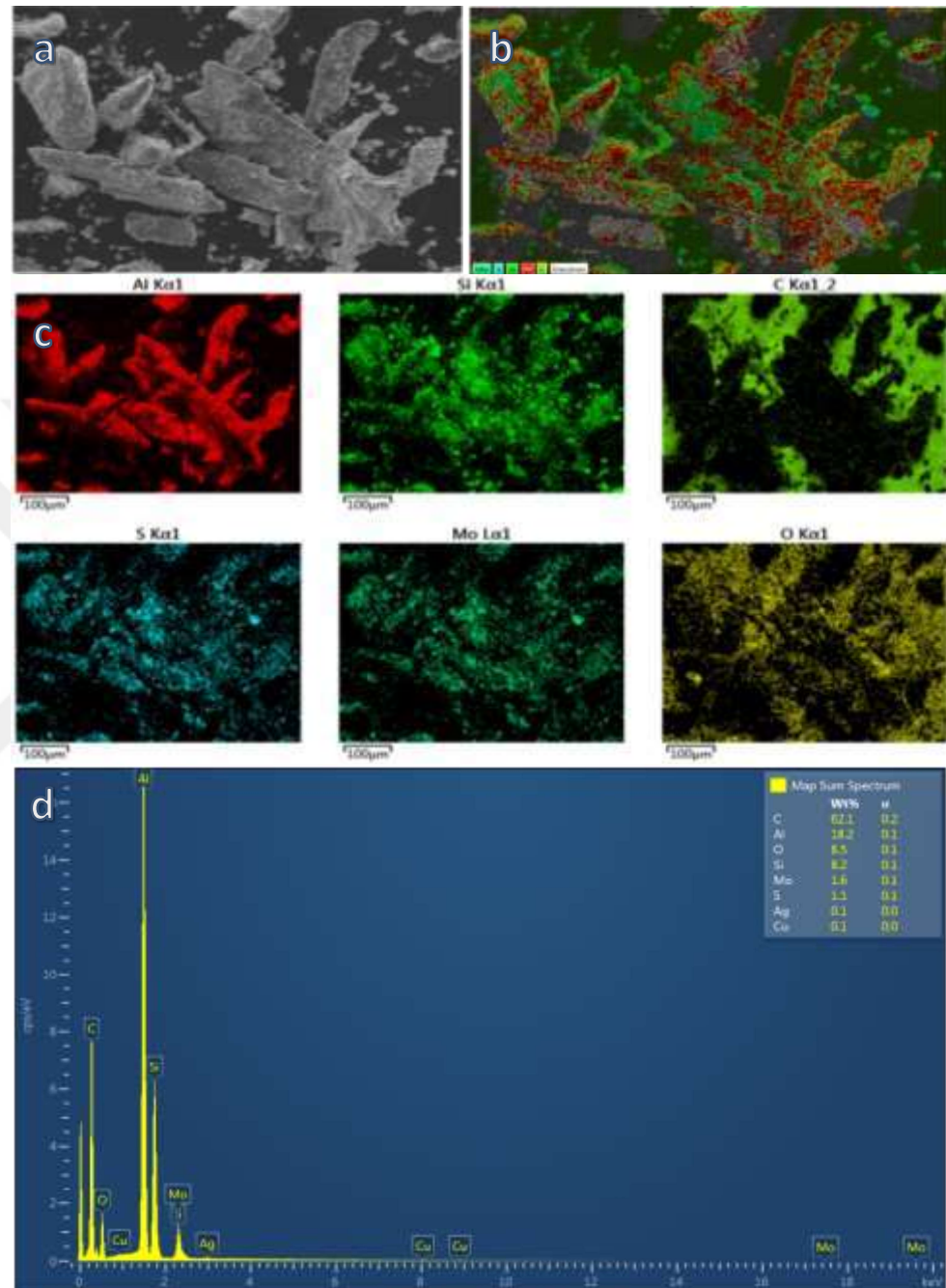


Figure 5.34. EDS of Al-Si base alloy + 3 % MoS₂+ 3 % BN. a) Al-Si-6 % MoS₂-BN image, b)mapping c) all elements,d) spectrum of elements

Figure 5.34 characterizations by EDS shows us the Al-Si alloy with the addition of MoS₂ and BN in nano form. The added weight percentage

was the highest, which is 6 %, as a hybrid addition of nanoparticles. During this inspection, the powder obtained from the wear process was collected after the mechanical wear process.

The result observed from it is that the highest peak, as expected, is for Al and then Si, but the weight percentages showed the presence of C with a high percentage of 62 %. Due to the excessive addition of nanoparticles, the crystal structure suffered from brittleness and could not withstand the assay conditions. As shown in the previous pictures, the surface suffered from brittle fractures, as evident from the SEM images. It was also found that there was a second reason, which is the impurities that were represented by an examination EDS such as C and some O.

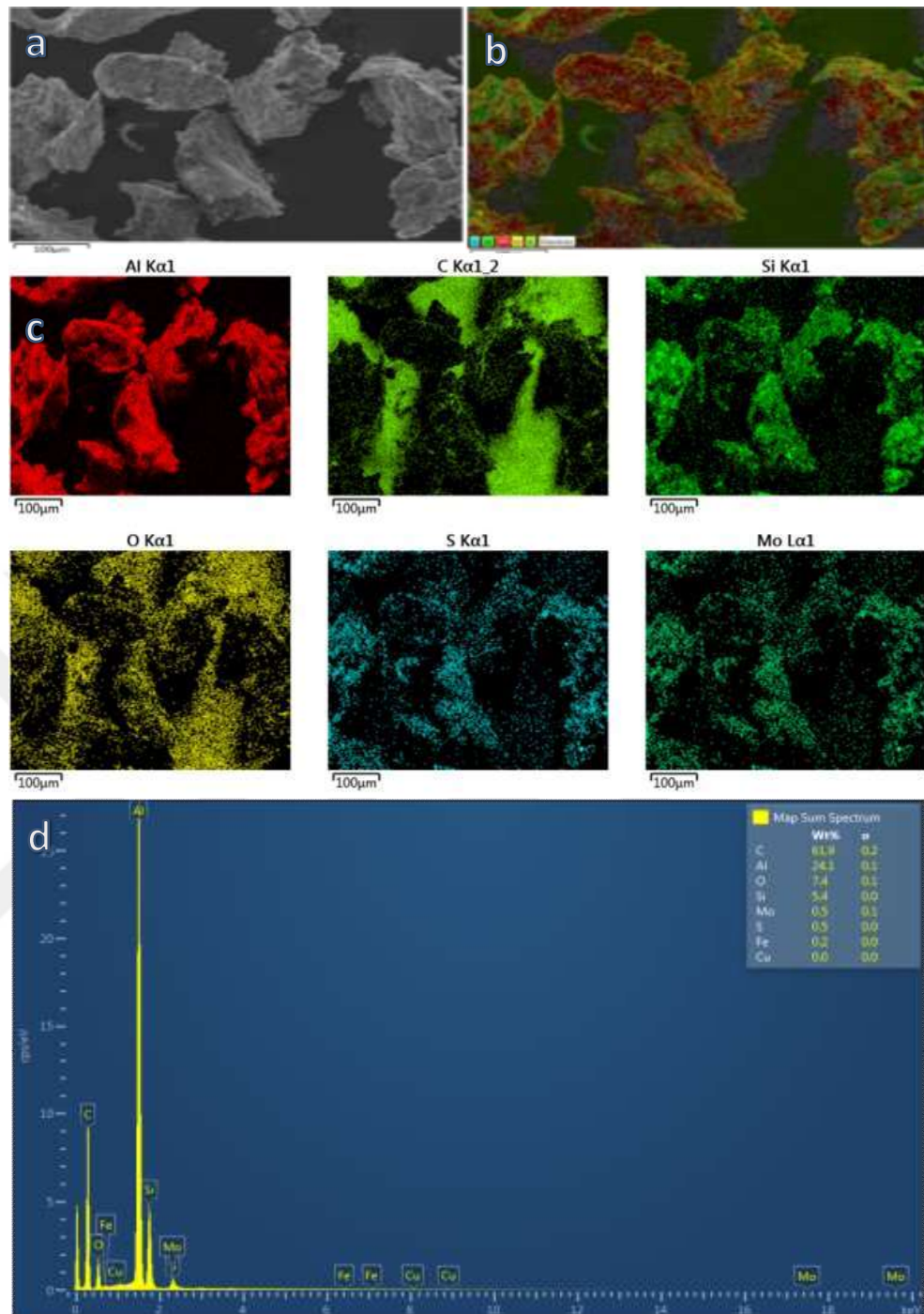


Figure 5.35. EDS of Al-Si base alloy + 4 % MoS₂ a) Al-Si-4 % MoS₂ image, b) mapping c) all elements, d) spectrum of elements

From Figure 5.35 for Al-Si powder with the addition of only 4 % of MoS₂. We called it powder because it was reverted to powders according to the component that made up the composites of nanoparticles and micro granules. Due to the different weight ratios, the result may need to be clarified in part of the examination sample. The highest peak was Al,

followed by C, and the latter appeared with increased rates of up to 62 %, followed by Si. High C weight ratios from different samples may be present from the friction of the surface opposite the test sample.

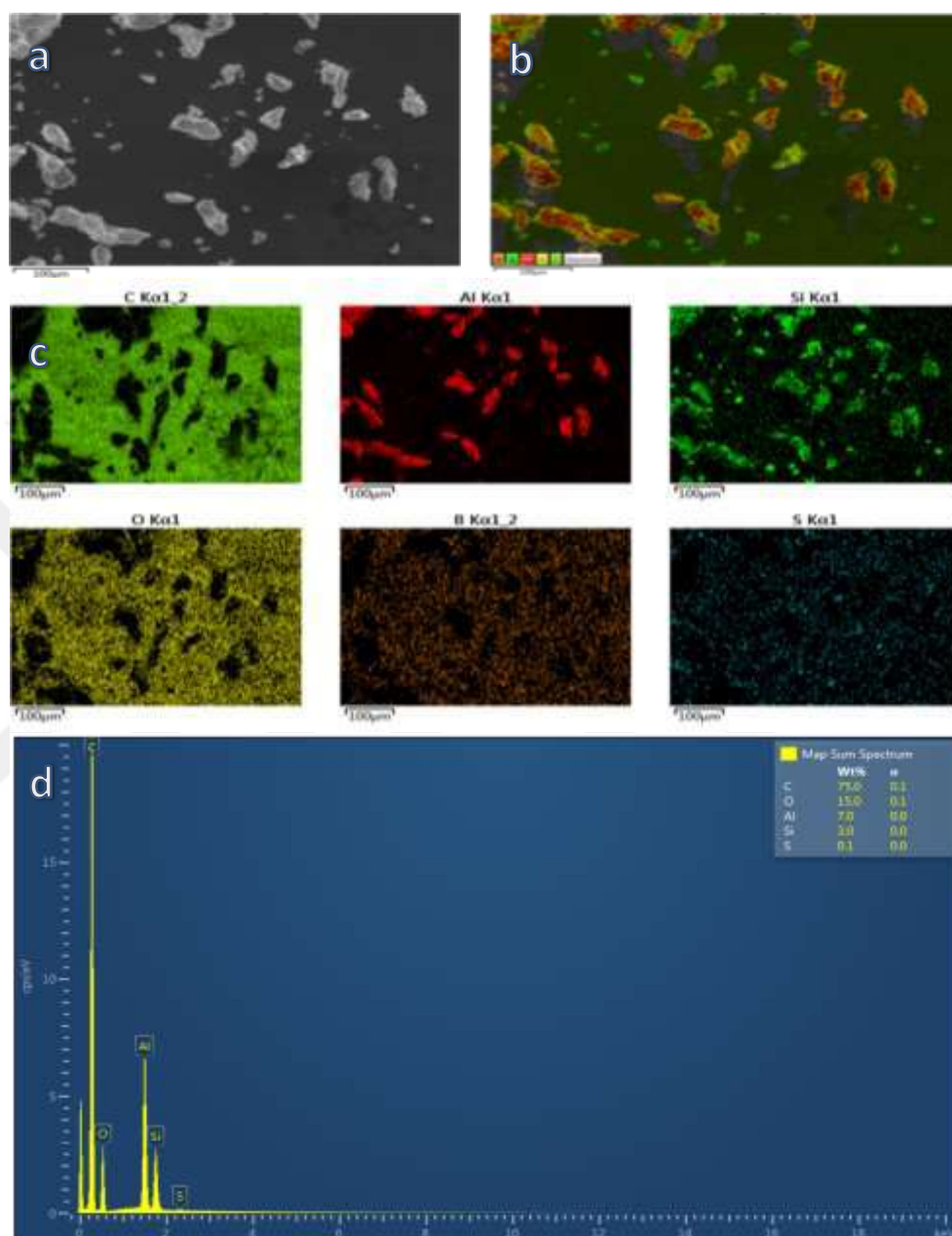


Figure 5.36. EDS of Al-Si base alloy + 4 % BN a) Al-Si-4 % BN image, b) mapping c) all elements, d) spectrum of elements

From Figure 5.36 for Al-Si powder with the addition of only 4 % of BN after the wear test. The result may be unexpected in part of the test sample to increase the weight percentages of the Carbon (C) element. The highest peak was C, and then Al and Si. The weight percentages of the

elements are as follows: 75 for C, 15 for O, 7 for Al, and 3 for Si, and the percentage of B and N did not appear in this part of the sample. The high percentages of O in the samples may be due to the oxidation process of the examined surfaces. During the reviews of previous studies, some works agree with the results of our research, and some of them differently because of the conditions of experiments or the manufacturing methods used.

Gnanavelbabu and his research team in 2020 worked on AA6061/B₄C/h-BN hybrid composites to study the effect of temperature on wear and found that B₄C and h-BN particles were evenly distributed in the AA6061 (B₄C + h-BN) phase, but there was a slight clumping of h-BN granules when 15 %, was used with casting method. The effect of B₄C is important by increasing the hardness and tensile strength, but BN increase it is counterproductive. The latter is considered useful as a solid lubricant and has a high value in reducing friction and wear rate in hybrid addition, and this is consistent with our work in the current research.

Through a research review by (Firestein et al. 2017), they prepared 12 samples of Al alloys with BN added in the nano form and the micro type for comparison between them. The powders were mixed by a mechanical method using ball mill, and then sintered by spark plasma sintering SPS. And they found that mixing micro-powders is better in distribution and homogeneity in composition to BN nanoparticles. The hardness of nano and micro-composites are close and from the EDS by SEM, they found high percentages of Al, according to what they expected, but the presence of some O atoms in the mixture as well.

CONCLUSION

From EDS and SEM morphology used, analysis of elements in the Al-Si base alloy indicated the good mixing in whole powder by the mechanical alloying process. Preparing the Al-12%Si by powder metallurgy process gives us a good result and prevents the drawbacks of casting manufacturing procedures. The characterization of Al-12%Si powder and with a hybrid of nanopowders of MoS₂+ BN by XRD, we can see the accurate peaks for base alloy because Al and Si were in micro-scale and the other addition in nanoscales.

The samples lost weight during friction, and the weight loss increases with increasing time, even for nanocomposite, but nanocomposite from the hybrid addition has an effect on improving the structure, which reduces weight loss due to the friction process. The self-lubricating Al-Si nanocomposite reinforced by 4 wt. % MoS₂+ BN gives the highest wear resistance. Good lubrication properties of 4 % h-BN and 4 % of MoS₂ were also observed.

The 6 % hybrid nano addition of MoS₂+ BN has the highest hardness but lower friction resistance because of the brittleness of the whole structure. We were observing that from the fracture of samples during pressing with cold and through the abrasive test.

Incorporating nanoparticles MoS₂+h-BN as hybrid addition in Al-Si resulted better performance for multifunctional application. The presence of MoS₂+BN reduces the wear rate. By the effect of self-lubrication, 2 % and 4 % additions of nanoparticles appeared to have a hybrid mechanism of wear and became brittle at 6 % hybrid addition.

For future work we suggest to use different types of nano additions to the base alloy of Al-Si, like BNNT, TiO₂, as nanotubes. Spark Plasma Sintering (SPS) process can be used. A patent will be filled and new alloys will be produced to produce moving or carrier parts.

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