

# Investigation of the Effect of Mechanical Alloying Time on Microstructure and Mechanical Properties in Hybrid Composites Produced with Fe<sub>3</sub>O<sub>4</sub>/nanographene Reinforcements in Al7020 Alloy

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## Anahtar Kelimeler

Mekanik Alaşım  
Hibrit Kompozit  
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Mikroyapı  
Karakterizasyonu

## Graphical/Tabular Abstract (Grafik Özet)

In this study, Fe<sub>3</sub>O<sub>4</sub> and nanographene were added as reinforcements at 10% and 0.25%, respectively, to the Al7020 alloy, and the effect of mechanical alloying (MA) time (1, 2, 4, and 8 hours) was investigated. / Bu çalışmada Al7020 alaşımına Fe<sub>3</sub>O<sub>4</sub> ve nanografen sırasıyla %10 ve 0.25 oranlarında takviye edilerek, mekanik alaşımlama (MA) süresinin (1, 2, 4 ve 8 saat) etkisi araştırılmıştır.

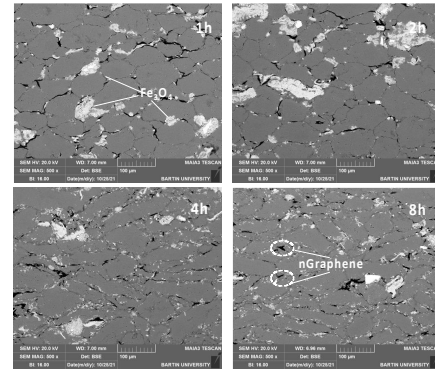
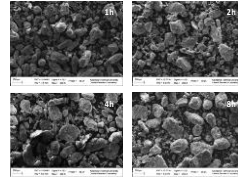
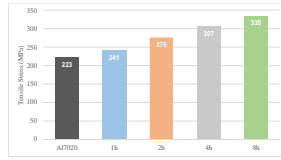


Figure A: Composites produced by reinforced Al7020 with Fe<sub>3</sub>O<sub>4</sub> and nanographene /Şekil A: Al7020 Fe<sub>3</sub>O<sub>4</sub> ve nanografen takviyesi ile üretilen kompozitler

## Highlights (Önemli noktalar)

- The composites were produced by incorporating Fe<sub>3</sub>O<sub>4</sub> and nanographene as reinforcement materials into the Al7020 matrix. / Al7020 matris içerisine Fe<sub>3</sub>O<sub>4</sub> ve nanografen takviye malzemesi olarak kompozitler üretilmiştir.
- The microstructure and mechanical properties of the composites were examined at different MA durations. / Kompozitlerin farklı MA süreleri ile mikroyapı ve mekanik özellikleri incelenmiştir.
- The highest strength was determined in the metal matrix composite sample processed for 8 hours of MA. / En yüksek dayanımın 8 saat MA işlemi yapılan metal matrisli kompozit numunede olduğu belirlenmiştir.

**Aim (Amaç):** The study aims to achieve a homogeneous distribution of reinforcements (Fe<sub>3</sub>O<sub>4</sub> and nanographene) within the matrix through MA processing at different durations and to enhance the mechanical properties of the Al7020 matrix alloy. / Çalışma, farklı sürelerde MA işlemine tabi tutulan takviyelerin (Fe<sub>3</sub>O<sub>4</sub> ve nanografen) matris içerisindeki dağılımı homojen olarak gerçekleştirmek ve Al7020 matris alaşımının mekanik özelliklerini geliştirmeyi amaçlamaktadır.

**Originality (Özgünlük):** The originality of the study is the production of hybrid metal matrix composites using different reinforcements and the improvement of their mechanical properties. / Çalışmanın özgünlüğünü, farklı takviyeler kullanılarak, hibrit metal matrisli kompozitlerin üretiminin gerçekleştirilmesi ve mekanik özelliklerinin geliştirmesi oluşturmaktadır.

**Results (Bulgular):** In the produced hybrid composites, the highest hardness (126 HV) and tensile strength (335 MPa) were observed in the sample subjected to 8 hours of MA processing. / Üretilen hibrit kompozitlerde en yüksek sertlik (126 HV) ve çekme dayanımı (335 MPa) 8 saat MA işlemi yapılan numunede olduğu tespit edilmiştir.

**Conclusion (Sonuç):** To enhance the mechanical and microstructural properties of the Al7020 alloy, it was found that the Fe<sub>3</sub>O<sub>4</sub> and nanographene reinforcements improved the mechanical properties at various MA durations. / Al7020 alaşımının mekanik ve mikroyapı özelliklerinin geliştirilmesi için takviye edilen Fe<sub>3</sub>O<sub>4</sub> ve nanografen takviyelerin, farklı MA süreleri içerisinde mekanik özellikleri geliştirdiği tespit edilmiştir.



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## Abstract

The objective of this work is to enhance the microstructural and mechanical characteristics of Aluminium (Al) 7020 alloys, which are receiving growing application in the aerospace sector. The mechanical properties of Al 7XXX series alloys are at high levels compared to other Al alloys. However, the need for Metal Matrix Composites (MMC) is increasing day by day to increase the mechanical properties to the desired levels. Powder metallurgy (PM) is one of the advanced production techniques for MMCs. It is known that microstructural and mechanical properties are improved by mechanically alloying (MA) and homogeneous mixing of powders in the structure. Therefore, the effects of different mechanical alloying times (1, 2, 4 and 8 hours) on the mechanical and microstructure properties of the composites produced by reinforcing 10% Fe<sub>3</sub>O<sub>4</sub> and 0.25% nanographene into Al7020 alloy were investigated. Density, XRD and SEM analyses were performed on all composite samples for characterization purposes. After that, the mechanical characteristics of the hybrid composite samples were determined by accurate hardness and tensile testing methods. The sample who received mechanical alloying for 8 hours achieved the highest tensile strength of 335 MPa.

## Al7020 Alaşımı İçerisine Fe<sub>3</sub>O<sub>4</sub>/nanografen Takviyeleri ile Üretilen Hibrit Kompozitlerde Mekanik Alaşımlama Süresinin Mikroyapı ve Mekanik Özelliklere Etkisinin İncelenmesi

## Makale Bilgisi

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## Öz

Bu çalışmada özellikle havacılık ve uzay endüstrisinde kullanımı artarak devam eden Alüminyum (Al) 7020 alaşımlarının mikroyapısal ve mekanik özellikleri özelliklerinin geliştirilmesi amaçlanmıştır. Al 7XXX serisi alaşımların mekanik özellikleri, diğer Al alaşımlarına kıyasla yüksek seviyelerdedir. Ancak mekanik özelliklerin istenilen seviyelere çıkarılabilmesi için Metal Matrisli Kompozitlere (MMK) olan ihtiyaç gün geçtikçe artmaktadır. MMK'ler için toz metalurjisi (TM) yöntemi ileri düzey üretim teknikleri arasındadır. Tozların mekanik olarak alaşımlandırılması (MA) ve homojen olarak yapı içerisinde karıştırılabilmesi ile mikroyapısal ve mekanik özelliklerin arttığı bilinmektedir. Bu nedenle Al7020 alaşımı içerisine %10 Fe<sub>3</sub>O<sub>4</sub> ve %0.25 nanografen takviye edilerek üretilen kompozitlerin farklı mekanik alaşımlama sürelerinin (1, 2, 4 ve 8 saat), mekanik ve mikroyapı özelliklerine olan etkileri araştırılmıştır. Üretilen tüm kompozit numunelere karakterizasyon amaçlı yoğunluk, XRD ve SEM analizleri yapılmıştır. Ardından mekanik özelliklerin belirlenmesi için sertlik ve çekme testleri hibrit kompozit numunelere uygulanmıştır. Elde edilen sonuçlar neticesinde en yüksek çekme dayanımına 335 MPa'lık değer ile 8 saat mekanik alaşımlama işlemi görmüş numunede elde edilmiştir.

## 1. INTRODUCTION (GİRİŞ)

Aluminum alloys have widely found application in the engineering sphere owing to a range of superior properties: lightness, high strength, corrosion resistance, and machinability. The main advantage

it offers is in sectors such as aviation, automobiles, and defense; it is critical because it has both low density and very high strength [1]. Al-Zn-Mg or 7XXX aluminum alloys are strengthened primarily by Zn and Mg elements to form high-strength structures. Alloys belonging to the 7XXX class find

extensive application in several industrial sectors, notably on aircraft, due to their exceptional strength-to-weight characteristics. One of the most outstanding members among these series, the Al7020 alloy, is notable for its fine mechanical characteristics, especially high strength, hardness [3]. The high strength properties made this Al7020 light alloy applicable to the aircraft industry. The material has also been increasingly used in automobile and other industrial applications [4]. Development of metal matrix hybrid composites (MMHC) in years has opened new opportunities for improvement in the endurance and microstructural characteristics of these alloys. These composites are developed by incorporating various ceramic, metal oxides, and carbon-based nanomaterials in the aluminum matrix. Such hybrid composites result in major improvements in properties like strength, hardness, wear resistance, and thermal stability [5]. The application of MMHCs with aluminum alloys provides great advantages in high-performance areas such as aerospace, automotive, electronics, and biomedical [6]. The aluminum matrix hybrid composites increase their load-carrying capacity to meet the critical engineering requirements, i.e., weight reduction and energy efficiency [7]. In the production of such materials, the mechanical alloying (MA) method, which is an important production technique especially for powder metallurgy, is important.

The MA is the process of combining one or more basically solid materials in a high-energy grinding medium to produce new, stronger materials. It is widely used in composites, usually metals, ceramics and a combination of both [8]. The MA process, which is a critical production process in powder metallurgy methods, enables the alloying process to be realized together with the plastic deformation applied to the powders repeatedly by high-energy balls. Thus, it is possible to control the internal structure of the material with determined parameters [9].

Elevated efficiency of  $\text{Fe}_3\text{O}_4$  in improving the microstructural integrity and mechanical performance of aluminum matrix composites has been demonstrated. [10].  $\text{Fe}_3\text{O}_4$  is characterized by high hardness and possesses magnetic properties; the properties of this compound make a composite material much harder and more resistant to tensile strength [11]. By increasing the microhardness, the addition of  $\text{Fe}_3\text{O}_4$  into Al7020 alloy enhances the

mechanical strength of the alloy and guarantees the long-term durability of the composite construction. [12]. The reinforcement by  $\text{Fe}_3\text{O}_4$  increases the thermal stability of the alloy. There is an improvement in the performance of alloys with increasing temperatures [13].

In carbon-based nanomaterials, including nanographene, the material is highly strong, flexible, and electrically conductive [14]. The use of nanographene as reinforcement in aluminum alloys improves the microstructural integrity and mechanical properties by preventing dislocation motion and consolidating the grain boundaries, respectively [15]. Indeed, nanographene has the ability to enhance the tensile strength and hardness of composite materials, as well as promote the thermal and electrical conductivity characteristics of the alloy. [16]. For instance, the development of hybrid composites reinforced with nanographene and  $\text{Fe}_3\text{O}_4$  in Al 7020 alloy provides innovative solutions, especially for areas that require high performance such as aerospace [17].

The present work aimed to examine the effects of varying times of  $\text{Fe}_3\text{O}_4$  and nanographene reinforcement on the mechanical alloying time of Al7020 alloy. Although Al7020 alloy can show high strength properties, the production of hybrid composites is needed to develop the required mechanical characteristics. For this reason, it was aimed to determine the optimum parameters in composite materials after the characterization and mechanical testing processes performed on all produced samples.

## 2. MATERIALS AND METHODS (MATERIALS AND METHOD)

The effect of mechanical alloying times of 1, 2, 4 and 8 hours on hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforcement to Al7020 alloy was investigated. The Al7020 alloy used has a comprising the chemical components listed in Table 1.

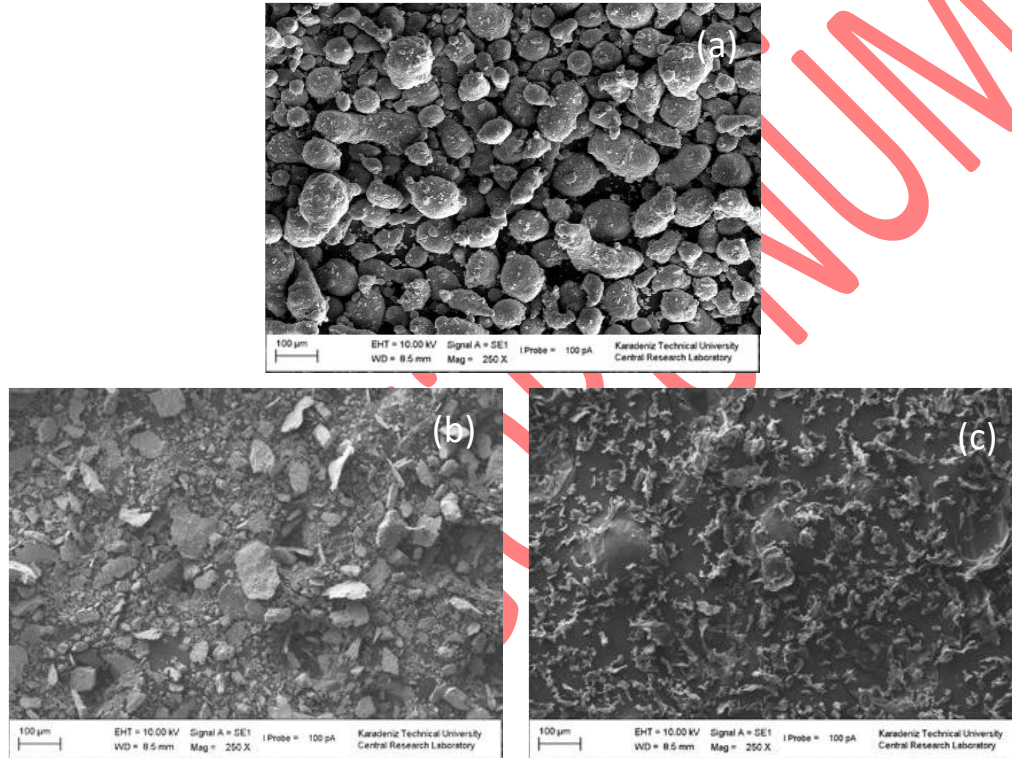
For the hybrid composite samples, the blended powders were subjected to mechanical alloying with 10 wt%  $\text{Fe}_3\text{O}_4$  and 0.25 wt% nanographene reinforcement in a 3D high energy milling machine for 1, 2, 4 and 8 hours. The MA process was determined as 200 Rpm in X direction and 500 Rpm in Y direction after preliminary studies. Figure 1 shows SEM images of the initial powder images. It

is seen that the dimensions of Al7020 gas atomized powders are approximately 45µm in size and spherical shape, the size of Fe<sub>3</sub>O<sub>4</sub> ceramic particles used as 10% reinforcement is approximately 30µm

in size and in a flat form, and finally the size of the nanographene reinforcement used is 100 nm and in a flaky structure.

**Table 1.** Chemical composition of Al7020 alloy (Al7020 alařının kimyasal kompozisyonu)

Elements	Al	Zn	Mg	Ti	Cr	Mn	Fe	Si	Cu
%wt.	Balanced	4,67	1,34	0,3	0,29	0,48	0,09	0,02	0,01



**Figure 1.** Initial powder images a) Al7020, b) Fe<sub>3</sub>O<sub>4</sub>, c) nanographene (Bařlangıç toz görüntüleri a) Al7020, b) Fe<sub>3</sub>O<sub>4</sub>, c) nanografen)

The produced hybrid powder composites were pressed with a mold prepared from H13 tool steel at 550 °C temperature for 1 hour in a hot pressing device to prepare them as bulk samples. Then, metallographic processes for microstructural characterization of the composites were carried out in ATM Sapphire 330 device. In the microstructural characterization processes, after sanding at 400, 600, 800 and 1200 grit, the processes were completed with 9, 6 and 3 micron felts prepared with diamond suspension for polishing, respectively. For etching, Keller solution (1mL HF + 200mL H<sub>2</sub>O) was used. Density measurements were performed using the Archimed principle (Equation 1) with a Sartorius CP224S device.

$$ps = \frac{(ma)}{(ma)-(mw)} \times (pw) \quad (1)$$

Here ps symbolize the density of the material in grammes per cubic centimeter, pw denotes the volume of water at room temperature, ma represent the mass of the material, and mw denote the weight of the material included in the water.

SEM analysis (ZEISS EVO LS 10) for microstructure images and XRD analysis (Bruker D8 Advance) for crystal structure and phase determination were applied to all composite samples. Determination of mechanical properties microhardness Vickers tests (HV10) were carried out on Emcotest Duravision 200 Universal hardness

tester in accordance with ASTM E92 standards by taking the average values of five measurements horizontally in the center of each sample. Continuous tensile testing was conducted using the Instron 3369 test machine until the specimens fractured.

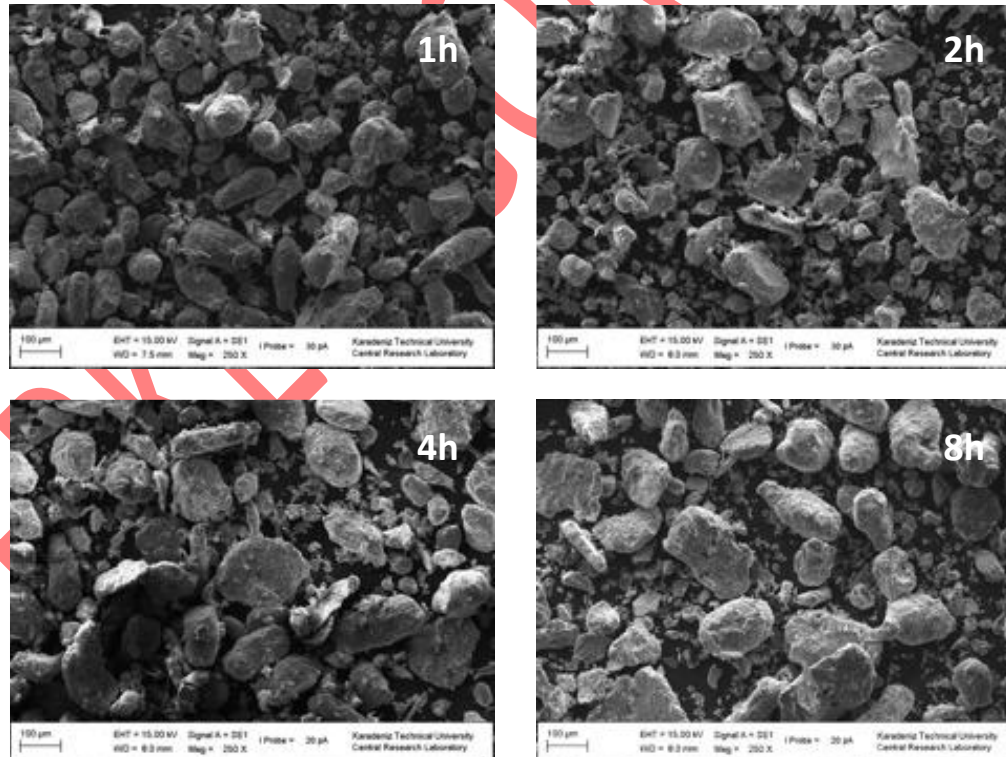
### 3. RESULTS AND DISCUSSION (BULGULAR VE TARTIŞMA)

#### 3.1. Effect of Mechanical Alloying Time on Hybrid Composite Powders (Hibrit Kompozit Tozlarına Mekanik Alaşım Süresinin Etkisi)

The powder morphologies of 10% Fe<sub>3</sub>O<sub>4</sub> and 0.25% nanographene powders reinforced in Al7020 alloy after 1, 2, 4 and 8 hours MA process are shown in Figure 2.

When the figure is examined, it is seen that the spherical shaped Al7020 matrix alloy gradually takes a flaky form with the MA alloying time.

Especially in the hybrid composite with 1 hour MA treatment, while the spherical form is generally preserved, the transition to the flaky form started with 2 hours of MA treatment. It is seen that the deformation, which continues with MA alloying, passes into full flaky form at the 4th hour. In the 8-hour MA process, it can be said that the deformation has reached saturation, the powders are no longer flattened and the powders broken by cold welding are recombined. In the nature of MA, the effect of mechanical impact forces applied to the powder particles by the balls in the chamber increases the surface area of the particles and provides a homogeneous distribution [18,19]. At 8 hours of MA process, the cold weld formed by the formation of a sufficient amount of plastic deformation between the flattened powder particles enabled them to coalesce at the atomic level. Thus, a strong bond formation can be observed between the hybrid composite powders and the homogeneous internal structure can contribute to microstructurally more equiaxed grain formation [20,21].

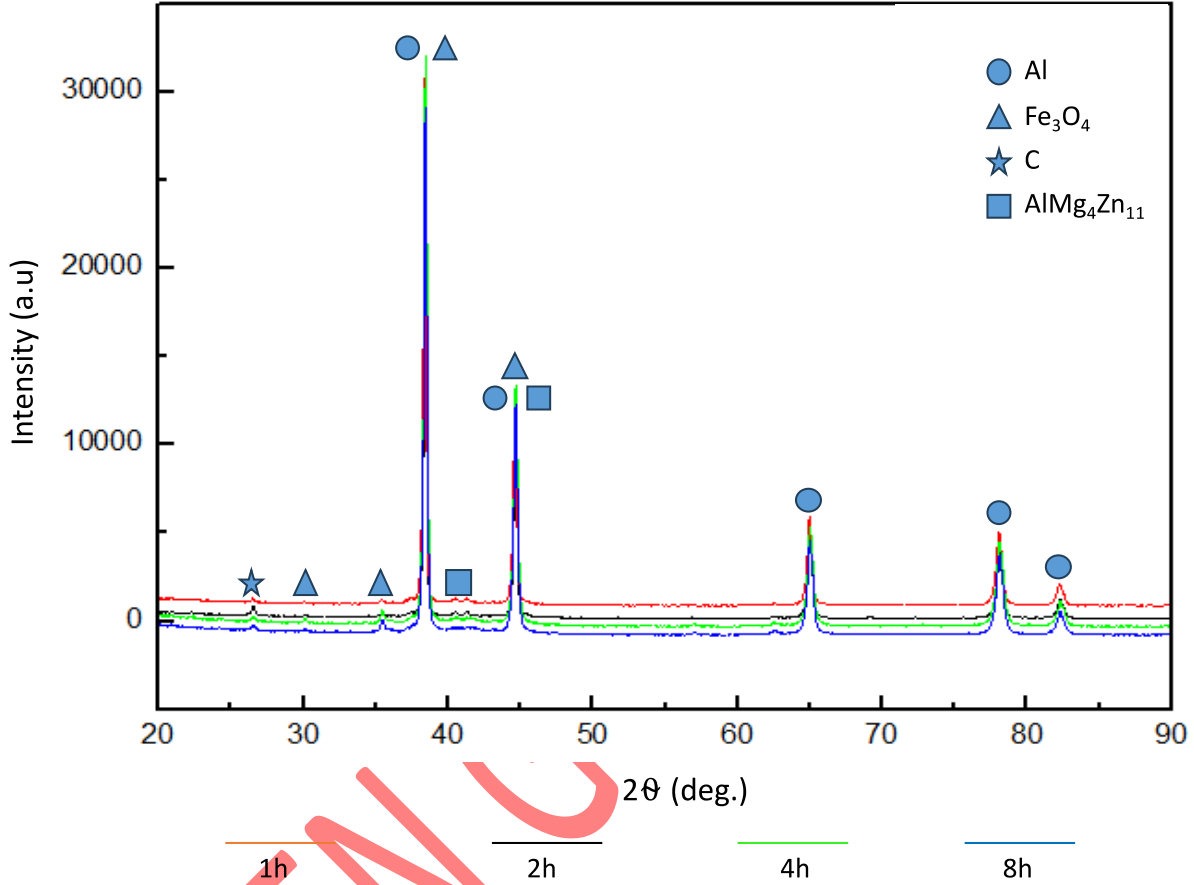


**Figure 2.** Powder images of hybrid composites with 10% Fe<sub>3</sub>O<sub>4</sub> and 0.25% nanographene reinforcement to Al7020 alloy (1, 2, 4 and 8 hours) (%10 Fe<sub>3</sub>O<sub>4</sub> ve %0,25 nanografen takviyesi ile Al7020 alaşımına sahip hibrit kompozitlerin toz görüntüleri (1, 2, 4 ve 8 saat))

### 3.2. Microstructure Analysis (Mikroyapı Analizi)

Figure 3 shows the XRD patterns of hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforced in Al7020 alloy. In XRD analysis, the matrix phase Al, the reinforced

ceramic particle  $\text{Fe}_3\text{O}_4$ , the reinforced carbon-based nanographene and finally the intermetallic phase  $\text{AlMg}_4\text{Zn}_{11}$  in the structure was observed.



**Figure 3.** XRD patterns of hybrid composites (Hibrit kompozitlerin XRD paternleri)

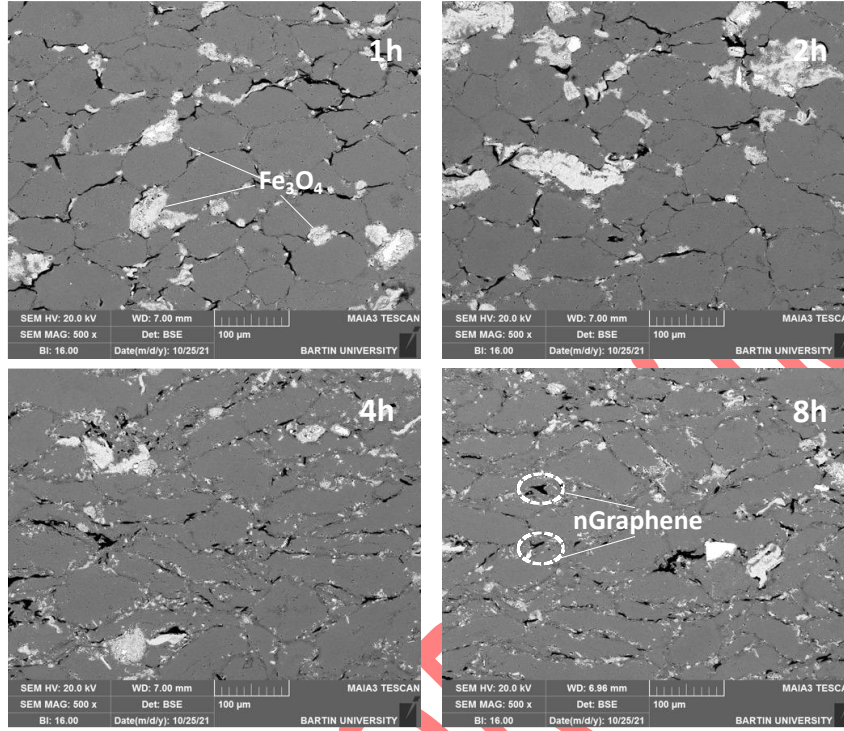
When the figure is examined, it is seen that XRD peaks generally appear at the same intensity and degree. Slight shifts in the peaks were observed with the increase in the MA time. These phenomena arise as a result of plastic deformation. As the deformation time increases, the internal stresses occurring in the crystal lattice structure change the distances between the planes, causing the peaks to shift [22,23]. Figure 4 displays the scanning electron microscope (SEM) pictures of hybrid composites following 1, 2, 4, and 8 hours of MA alloying.

SEM images of hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforcement to Al7020 alloy with 1, 2, 4 and 8 hours MA treatment are

shown. No major changes in grain size and spherical grain boundaries were observed in the sample with 1 hour MA time. In addition, it was determined that the white  $\text{Fe}_3\text{O}_4$  ceramic reinforcement was homogeneously distributed in the structure. Upon examination of the sample with an MA time of 2 hours, it was seen that the grains began to elongate at a rather modest rate compared to the initial stage. This can be explained by the fact that the MA process flattens the powder particles during mixing. Similar to the previous image, the light colored regions located at the grain boundaries show  $\text{Fe}_3\text{O}_4$  ceramic particles and the dark black regions show nanographene reinforcement. With the MA time of 4 hours, it was determined that the deformation effects were further intensified on the powder

particles and the grains were flattened. When the last hybrid composite sample, the sample with 8 hours MA treatment, was examined, it was found

that the grains became much flatter and the grain boundaries and grain boundary angles decreased.



**Figure 4.** Microstructures of hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforced Al7020 alloy (1, 2, 4 and 8 hours) (%10  $\text{Fe}_3\text{O}_4$  ve %0,25 nanografen takviyeli Al7020 alařımlı hibrit kompozitlerin mikro yapıları (1, 2, 4 ve 8 saat)

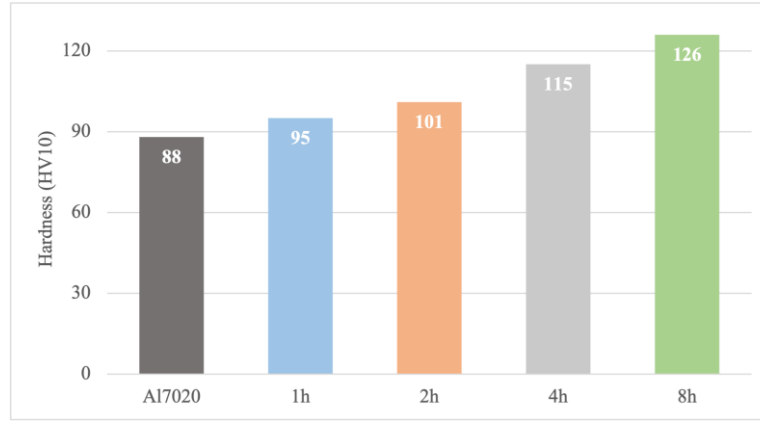
### 3.3. Physical and Mechanical Properties

(Fiziksel ve Mekanik Özellikler)

Physical and mechanical properties of samples reinforced with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene in Al7020 alloy were determined by hardness and tensile testing after to 1, 2, 4, and 8 hours of MA treatment. The hardness findings of hybrid composites generated by MA treatment with varying durations are shown in Figure 5.

When the figure is examined, it is determined that the average hardness value of the unreinforced Al7020 alloy is 88 HV. With the increase in MA time, it was measured as 95 HV for 1 hour, 101 HV for 2 hours, 115 HV for 4 hours and finally 126 HV

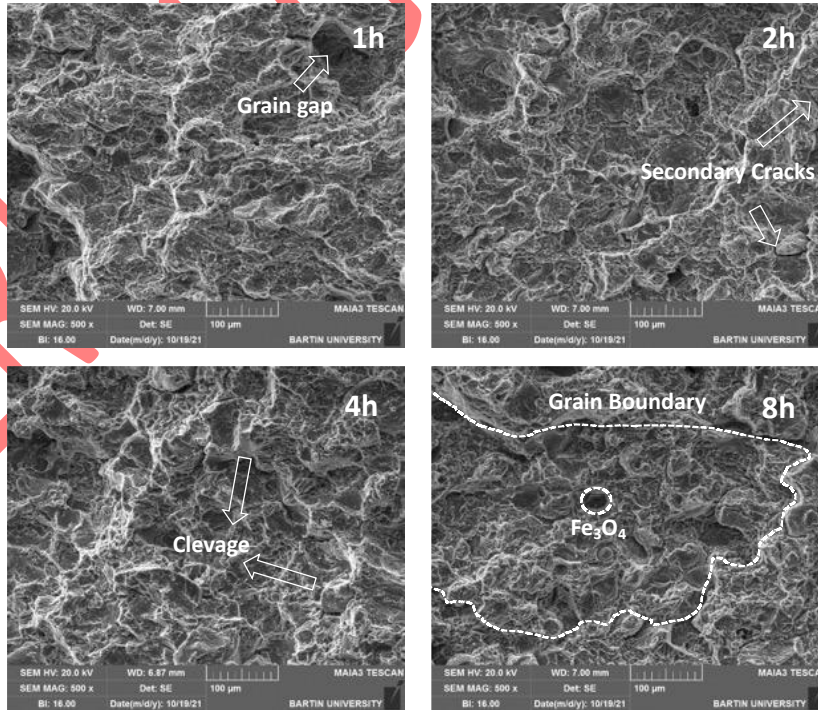
for 8 hours. The highest hardness value was reached by the sample with 8 hours MA treatment. This is a natural result of deformation flattening the grain structure and the  $\text{Fe}_3\text{O}_4$  and nanographene reinforcements used in the production of hybrid composite samples. On the other hand, Karabulut et al. similarly produced composite samples by cold pressing method by adding 10%  $\text{Al}_2\text{O}_3$  to pure aluminum by mechanical alloying and stated that the hardness value increased with the increase of mechanical alloying time [24]. These results support the results of increasing hardness with increasing mechanical alloying time.



**Figure 5.** Hardness results of hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforcement to Al7020 alloy (1, 2, 4 and 8 hours) (%10  $\text{Fe}_3\text{O}_4$  ve %0,25 nanografen takviyesi ile Al7020 alaşımına uygulanan hibrit kompozitlerin sertlik sonuçları (1, 2, 4 ve 8 saat))

Fig. 6 displays the tensile test outcomes of the Al7020 alloy, which was strengthened with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene, following 1, 2, 4, and 8 hours of MA heat treatment. Upon analysis of the results, it is evident that the unreinforced Al7020 alloy has the lowest tensile stress, while the sample subjected to 8 hours of MA treatment exhibits the highest tensile stress. The 8 hours MA treated specimen has 9% higher tensile stress than the 4 hours MA alloy, 21% higher than the 2 hours MA treated specimen, 39% higher than the 1 hour MA treated specimen and finally about 50% higher than

the unreinforced Al7020 alloy. Similar to the hardness results, this situation was similar in the tensile test. The growing trend in the test results can be ascribed to the amplified dislocation density resulting from the deformation effect as the MA time increases, together with the homogeneous dispersion of the reinforced ceramic particles in the matrix phase. [25]. Increasing the MA alloying time further will increase the dislocation density too much and will lead to interlocking. For this reason, 8 hours of MA time was deemed sufficient and the next step was not started.



**Figure 7.** Fracture surfaces of hybrid composites with 10%  $\text{Fe}_3\text{O}_4$  and 0.25% nanographene reinforced Al7020 alloy after tensile test (1, 2, 4 and 8 hours) (%10  $\text{Fe}_3\text{O}_4$  ve %0,25 nanografen takviyeli Al7020 alaşımılı hibrit kompozitlerin çekme deneyinden sonraki kırılma yüzeyleri (1, 2, 4 ve 8 saat))



Figure 7 depicts the fracture surfaces of hybrid composites consisting of 10% Fe<sub>3</sub>O<sub>4</sub> and 0.25% nanographene reinforcement after performing tensile testing on Al7020 alloy.

Figure 7 shows the fracture surface images of hybrid reinforced composite materials subjected to MA treatment for 1, 2, 4 and 8 hours. When the fracture surface images were examined, differences in fracture behavior were detected with increasing MA treatment time. In the fracture surface image of the sample subjected to 1 hour MA treatment, ruptures occurred in different layers. Especially the rupture behavior occurred in weak grains. With increasing MA time, secondary cracks were observed in the sample treated for 2 hours. It was determined that the increase in hardness value also reduced the layered rupture behavior. In the 4 and 8 hour MA treated specimens, breaks occurred from cleavage planes due to the increase in the hardness of the materials in the rupture behaviors, and it was determined that the grain boundary rupture behaviors were in large areas in the strength regions of the 8 hour treated specimen.

#### 4. CONCLUSION (SONUÇLAR)

In this study, the effect of MA time on hybrid composite samples was investigated by performing MA process for 1, 2, 4 and 8 hours with 10% Fe<sub>3</sub>O<sub>4</sub> and 0.25% nanographene reinforcement to Al7020 alloy. The results are listed below.

- With the increase of MA time, the hybrid composite powders changed from spherical to flattening due to the effect of deformation. The most prominent formation was observed in samples with 4 and 8 hours of MA.

- When XRD patterns were examined, it was determined that the peaks shifted due to the effect of deformation. In addition to Al, particle reinforcements Fe<sub>3</sub>O<sub>4</sub> and carbon forming the matrix phase, AlMg4Zn11 intermetallic phase was detected in the structure.

- In SEM analysis, it was determined that the low MA time did not change the grain size and form, and with increasing time, the grains and grain boundaries formed in the bulk material changed to flat form. In addition, reinforced Fe<sub>3</sub>O<sub>4</sub> and nanographene were detected in the surface images.

- When the hardness results of the mechanical properties of the hybrid composites were examined, it was determined that the sample with the highest value was the sample with 8 hours MA with 126 HV.

- When the tensile test results were examined, 335 MPa was obtained in the sample treated with MA for 8 hours.

- When the post-tensile fracture surface images were examined, while ruptures occurred in different layers in the 1 hour MA process, with the increase in the MA time, fractures occurred in the cleavage planes and rupture behavior was observed at the grain boundaries.

#### DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission.

Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

#### AUTHORS' CONTRIBUTIONS (YAZARLARIN KATKILARI)

**Taha Alper YILMAZ:** The author conducted the experiments, analyzed the results and performed the writing process.

Yazar, deneyleri yapmış, sonuçlarını analiz etmiş ve makalenin yazım işlemini gerçekleştirmiştir.

#### CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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