

# Enhancement of Microstructure Evolution and Tribological Properties of Al6063 Alloy Through Grain Refinement via ECAP Technique

Taha Alper YILMAZ<sup>1\*</sup> 

<sup>1</sup>Gazi University, Vocational School of Technical Sciences, Department of Machinery and Metal Technologies, Ankara, Turkey

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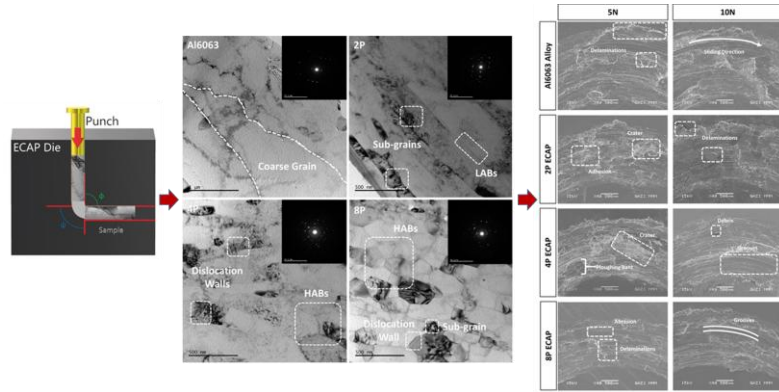
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## Anahtar Kelimeler

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## Graphical/Tabular Abstract (Grafik Özet)

The ECAP technique effectively refines the grain structure of Al6063 alloy, improving its hardness and tribological performance. TEM and SEM analyses revealed microstructural evolution from coarse grains to equiaxed sub-micron grains. The 8 pass sample showed the lowest wear and friction values, indicating optimum structural enhancement for industrial use. / ECAP tekniği, Al6063 alaşımının tane yapısını etkili şekilde incelterek sertlik ve tribolojik performansını artırmaktadır. TEM ve SEM analizleri, iri tanelerden eş eksenli sub-mikron tanelere yapısal evrimi göstermiştir. 8 pasoluk numune en düşük aşınma ve sürtünme değerlerini sunarak endüstriyel kullanım için en uygun yapısal gelişimi sağlamıştır.



**Figure A:** ECAP prosesinde mikroyapısal ve worn surface süreci / **Şekil A:** ECAP prosesinde mikroyapısal ve worn surface süreci

## Highlights (Önemli noktalar)

- Severe plastic deformation via ECAP was applied to Al6063 alloy at room temperature using route BC. / Al6063 alaşımına, Bc rotası kullanılarak oda sıcaklığında ECAP yöntemiyle şiddetli plastik şekil değiştirme uygulanmıştır.
- Microstructural evolution from coarse grains to equiaxed sub-micron/nano grains was observed with increasing ECAP passes. / Artan ECAP geçiş sayısı ile iri tanelerden eş eksenli sub-mikron/nano tanelere doğru mikro yapısal dönüşüm gözlemlenmiştir.
- After 8 passes, the sample showed the highest hardness (119 HV) and lowest coefficient of friction (0.398). / 8 geçiş sonrasında numune en yüksek sertlik (119 HV) ve en düşük sürtünme katsayısını (0.398) göstermiştir.
- ECAP significantly improved the tribological performance and wear resistance of Al6063 alloy. / ECAP işlemi, Al6063 alaşımının tribolojik performansını ve aşınma direncini önemli ölçüde artırmıştır.

**Aim (Amaç):** This study aims to improve the microstructure and tribological properties of Al6063 alloy via ECAP at room temperature for advanced industrial applications. / Bu çalışma, Al6063 alaşımının mikro yapı ve tribolojik özelliklerini, oda sıcaklığında uygulanan ECAP ile geliştirmeyi amaçlamaktadır.

**Originality (Özgünlük):** ECAP was applied with BC route at room temperature in 2, 4, and 8 passes. Advanced techniques such as TEM and 3D profilometry were used. / ECAP işlemi BC rotasında ve 2, 4, 8 geçişle uygulanmış; TEM ve 3D profilometre gibi ileri teknikler kullanılmıştır.

**Results (Bulgular):** With increasing passes, grain size decreased and hardness improved. After 8 passes, hardness reached 119 HV, and CoF dropped to 0.398. / Geçiş sayısı arttıkça tane boyutu küçüldü ve sertlik arttı. 8 geçişte 119 HV sertlik ve 0.398 CoF değeri elde edildi.

**Conclusion (Sonuç):** ECAP significantly enhances the wear resistance and hardness of Al6063, offering potential for demanding applications. / ECAP işlemi, Al6063'ün aşınma direncini ve sertliğini artırarak zorlu uygulamalar için uygunluk sağlamaktadır..



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### Abstract

Present study aimed to increase the applicability of the material in the aviation, aerospace, and automotive industries by improving the microstructural and tribological properties of Al6063 alloy using the ECAP technique at room temperature with different passes (2, 4 and 8) and by selecting the BC route. XRD, SEM, EDS/Mapping, 3D Profilometer, TEM and TEM/MapViewer methods were performed on all samples in the microstructural characterization processes. Microhardness and dry sliding wear tests were conducted to evaluate the mechanical and tribological properties. It was determined that with the number of passes being 8, the hardness of the equiaxed sub-micron/nano grains increased to 119HV and had the best tribological properties (CoF: 0.398 and Ra: 0.620).

## ECAP Tekniği ile Tane İnceltme Yoluyla Al6063 Alaşımının Mikro Yapı Evrimi ve Tribolojik Özelliklerinin Geliştirilmesi

### Makale Bilgisi

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### Anahtar Kelimeler

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TEM

Tribolojik Özellikler

### Öz

Bu çalışma, oda sıcaklığında farklı geçişlerle (2, 4 ve 8) ve Bc rotası seçilerek ECAP tekniği kullanılarak Al6063 alaşımının mikro yapısal ve tribolojik özelliklerini iyileştirerek malzemenin havacılık, uzay ve otomotiv endüstrilerinde uygulanabilirliğini artırmayı amaçlamaktadır. Mikro yapısal karakterizasyon süreçlerinde tüm numunelerde XRD, SEM, EDS/Mapping, 3D Profilometer, TEM ve TEM/MapViewer yöntemleri gerçekleştirildi. Mekanik ve tribolojik özellikleri değerlendirmek için mikro sertlik ve kuru kayma aşınma testleri yapıldı. Geçiş sayısının 8 olmasıyla, eş eksenli alt mikron/nano taneciklerin sertliğinin 119HV'ye çıktığı ve en iyi tribolojik özelliklere sahip olduğu (CoF: 0,398 ve Ra: 0,620) belirlendi.

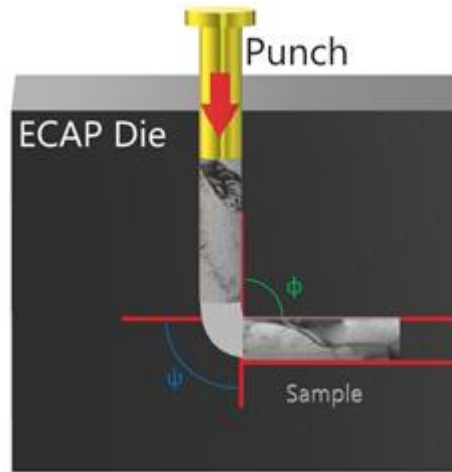
## 1. INTRODUCTION (GİRİŞ)

Powder metallurgy (PM) processes are widely used to produce ultrafine-grained (UFG) materials using nanoscale powders. This technology offers significant advantages because it efficiently produces nanoscale powders through pressing and sintering techniques. However, PM frequently encounters issues during the sintering phase, resulting in the formation of a porous microstructure that negatively affects the quality of the materials. Severe plastic deformation (SPD) techniques can be used as an alternative method to create microstructures with sub-micron or nanograined sizes [1–3]. This is achieved by extruding cast materials through a die with high strength. Equal

Channel Angular Pressing (ECAP) is a very efficient and useful technology for severe plastic deformation (SPD) that enables the creation of refined materials at a low cost. This technique entails exerting pressure on a large quantity of material by passing it through a die with a predetermined angle  $\Phi$  and a consistent cross-section [4,5]. The bulk sample goes through much shear stress during the pressing process. This causes it to deform while keeping its original cross-sectional area. Grains undergo homogeneous refinement due to dislocation motion [6]. Various methods are employed to rotate the sample around its axis for each pass. The process can be iterated until it reaches the minimum achievable grain size [7–9].

Multiple studies have shown that ECAP and other SPD methods can efficiently refine the grain structure [10–16]. Dislocations created during the plastic deformation process moved toward grain boundaries with high angles along their slip planes as distortion continued. This action promotes the sub-grains creation by transferring dislocations to sites with lower energy [17]. Once the initial grains have been sufficiently deformed, they come into contact with each other and change into a structure of fine grains whose edges are twisted at a high angle [18–20]. The ECAP process is affected by the angle at which the two channels intersect within the die and the radius of curvature of these channels

[21,22]. Figure 1 depicts the angles associated with the ECAP die. In most studies, the intersection angle ( $\phi$ ) is commonly fixed at  $90^\circ$ , whereas the curvature angle ( $\psi$ ) is generally set at  $0^\circ$ . The geometry and orientation of the die have a tremendous impact on the flow of material, greatly aiding the process. The angle formed by the intersection of two channels of equal size is denoted as ' $\phi$ '. If the angle  $\phi$  is less than  $90^\circ$ , a zone of undisturbed deformation can develop near the corner of the channel. Thus, setting  $\phi = 90^\circ$  is advisable to achieve optimal deformation characteristics and ensure effective deformation [7,23].



**Figure 1.** Angles in the ECAP pattern (EKAP kalbındaki açılar)

The ECAP process is an important method for improving the performance of aluminum (Al) alloys by significantly improving their mechanical properties, reducing their grain sizes equiaxially, and increasing their hardness values [24–26]. Grain refinement reduces the plastic deformation of the material and increases its resistance to surface damage that may occur during wear. Many early studies have shown that ECAP can significantly improve Al alloys' hardness and grain refinement. However, the tribological properties have not yet been fully optimized. Early studies have shown that the hardness and grain refinement can be significantly improved in Al alloys subjected to ECAP, while the tribological properties have not yet been fully optimized [27–29]. More studies are needed on how the properties such as wear rates, coefficient of friction and worn surface of the UFG grain structure can increase the overall strength of Al alloys. Ortiz-Ceuellar and his colleagues studied the tribological properties of the material. They used the ECAP process to look at the effect of severe plastic deformation on the microstructure of Al-Mg-Si alloys. They reported in their study that the hardness values increased with the increase in the

number of passes in the ECAP process, increasing the wear resistance [30]. In another study, Gao and his colleagues conducted a review study due to the contradictory results previously given by different SPD techniques in the literature. In their study, they stated that grain size, hardness, and flexibility are important factors affecting the wear resistance of materials. They also stated in their study that many parameters, such as surface quality of materials, lubrication conditions, wear load, speed and temperature, are determinants of wear properties and emphasized the need for increased studies on the subject [31].

This study aims to enhance the microstructural characterization and tribological properties of Al6063 alloy by applying the ECAP process with 2, 4, and 8 passes, facilitating the formation of an equiaxed and homogeneous microstructure. There is a significant gap in the literature on the tribological properties of Al6063 alloys after ECAP. Due to its low cost, Al6063 alloy presents a compelling case for further enhancement, particularly in its tribological performance. This study highlights the necessity of improving these

properties to expand their potential applications in various industries, including aviation, defense, and automotive. Therefore, investigating and optimizing the microstructural, mechanical, and tribological properties of ECAP-processed Al6063 alloy is crucial for advancing its industrial usability.

## 2. MATERIALS AND METHODS (MATERİYAL VE METOD)

### 2.1. Production of ECAP samples (ECAP Numunelerinin Üretimi)

The chemical composition of the Al6063 alloy on which the ECAP process was performed is given in Table 1. All samples were subjected to homogenization annealing at 400 °C for 2 hours to eliminate residual stresses originating from production and to obtain a homogeneous internal structure. Rods with a diameter of 19.8 mm were used as samples. All ECAP processes were performed at room temperature.

**Table 1.** Chemical composition of Al 6063 alloy (Al6063 alaşımlarının kimyasal kompozisyonu)

Elements	Mg	Si	Fe	Cu	Mn	Cr	Zn	Al
% wt.	1,01	0,67	0,28	0,2	0,04	0,05	0,06	Balance

ECAP processes applied to Al6063 alloys were carried out at room temperature to prevent grain size increase and to better see the effects of severe plastic deformation. A split ECAP die with a channel angle ( $\phi$ ) of 90° and a corner curvature angle ( $\psi$ ) of 20° was used for the severe plastic deformation. The BC route, which is accepted as the most effective route in the ECAP process, was preferred as the route. In order to examine the effects of the number of passes on grain refinement, 2, 4 and 8 pass ECAP processes were applied to the Al6063 alloy. The pressing speed in ECAP processes was determined as 0.025 mm/s. MoS<sub>2</sub> was used as the mold lubricant. After the ECAP processes, the effects of different pass numbers applied against the untreated material were investigated.

### 2.2. Microstructure characterization (Mikroyapı Karakterizasyonu)

All samples were put through characterization processes after the 2, 4 and 8 pass ECAP processes were used on Al6063 alloys. SEM and EDS (JEOL JSM-6060) analyses were applied to the materials to examine the microstructural properties of the samples. The results were characterized using X'Pert HighScore Plus software, and reference codes were determined. Since the grain sizes were expected to be in submicron scales, the samples on which the ECAP process was applied were first cut to approximately 5x10µm dimensions and 90nm thickness in the Focused Ion Beam (FIB) device (FEI Nova 650). Then, they were placed on TEM grids in the device. The TEM process (JEOL 2100 JEM HRTEM) was performed on all ECAP samples

to determine the microstructure and crystal structures. In addition, the atomic diffraction in the crystal structure was examined by applying the selected area electron diffraction (SAED) method. After the ECAP processes, the sample determined as the lowest grain size was subjected to EBSD analysis using the MapViewer software of the TEM device. The 3D Profilometer (Filmetrics Profilom 3D) device and software were used to determine the surface properties, wear rates, surface roughness, and wear marks of all the samples put through the wear process.

### 2.3. Mechanical and Tribological Properties (Mekanik ve Tribolojik Özellikler)

Microhardness tests were performed on the materials to determine their mechanical properties. In the operations performed with the microhardness device (Shimadzu HMV-2), 5x5 measurements were taken under HV1 load, and an average of 25 measurements was taken. After the hardness and strength properties, wear tests (Anton Paar TRB3) were applied to untreated Al6063 alloy and ECAP-treated samples to examine the tribological properties. The materials and parameters used in the wear tests are given in Table 2. Wear tests were performed at two different loads, 5 and 10N, according to ASTM G99-05. After the wear test, the friction coefficient-distance curves were drawn by obtaining the wear data from the device software.

The wear rates were calculated using the data determined from the 3D Profilometer device's software and the formula in Equation 1.

$$\omega = \frac{V}{L \times N}$$

(1) Where  $\omega$  is the wear rate, V is the wear volume, L is the sliding distance, and N is the applied load.

**Table 2.** Wear test conditions of the samples subjected to ECAP process (EKAP işlemine tabi tutulan numunelerin aşınma test koşulları)

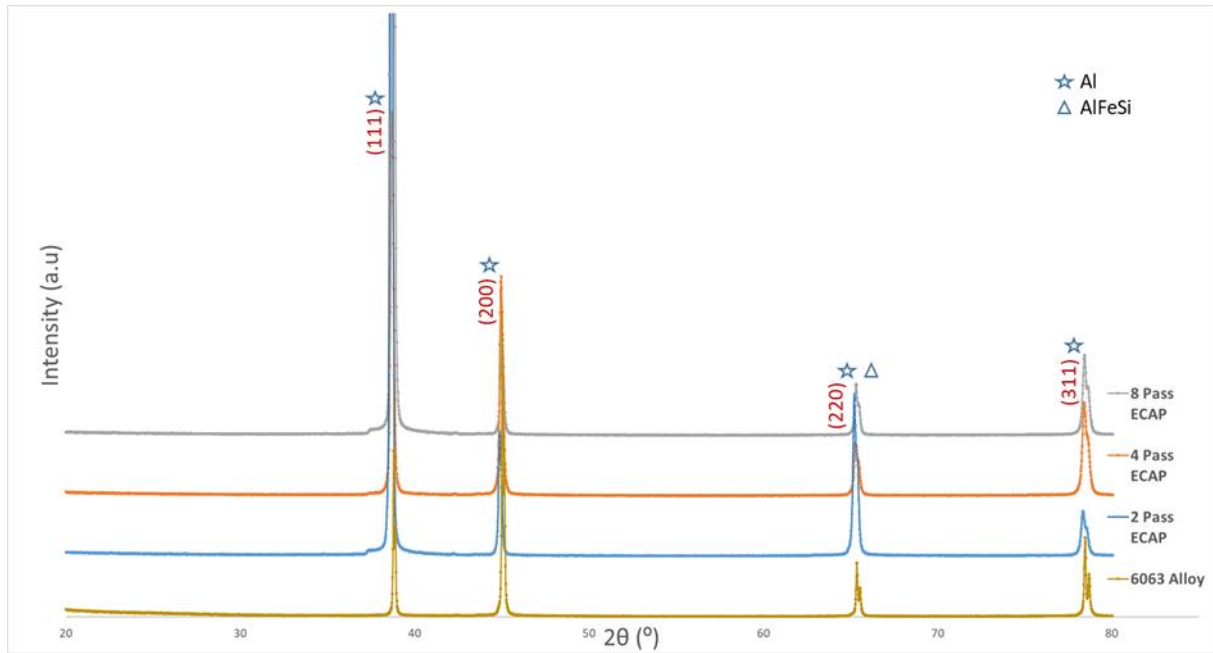
Parameters	Test Conditions
Applied Load (N)	5 and 10
Rotation Speed (rpm)	97
Trace Diameter (mm)	6
Ambient	Atmosphere
Temperature (°C)	22
Relative Humidity (%RH)	45-55
Distance (m)	100
Ball	ZrO <sub>2</sub>

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

#### 3.1. XRD Analysis (XRD Analizi)

Figure 2 shows the X-ray diffraction patterns of the Al6063 alloy and the samples subjected to 2, 4, and 8 passes of ECAP. All samples were subjected to a homogenization process to eliminate the possible presence of residual stresses and intermetallic compounds in the structure before the ECAP process, which determined the main phases in the

structure, Al and AlFeSi compounds. (Reference Cards: 00-001-1176 and 00-045-1206). The Mg<sub>2</sub>Si phase reported previously in the literature was not found in the structure [32]. In the samples subjected to ECAP, minimal shifts were detected in the peaks due to the effect of severe plastic deformation. It is thought that the shifts that occurred were due to the increase in the dislocation density in the material during deformation and micro stresses. In addition, it was determined that the peaks widened with the reduction of grain size with the ECAP process [33].



**Figure 2.** XRD patterns of Al6063 alloy, 2, 4 and 8 pass ECAP treated materials (Al6063 alaşımlarının, 2, 4 ve 8 geçişli EKAP ile işlenmiş malzemelerin XRD desenleri)

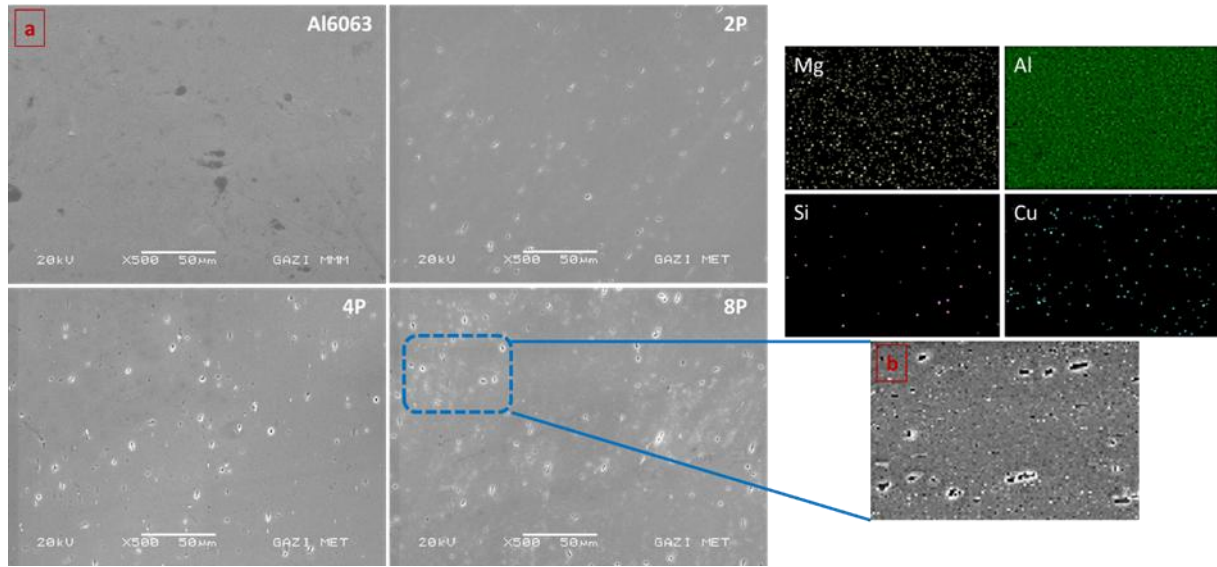


### 3.2. Microstructure Evolution (Mikroyapı Evrimi)

#### 3.2.1. SEM and EDS/Mapping (SEM ve EDS/Map)

Figure 3 (a) shows SEM images of untreated Al6063 alloy and samples subjected to 2, 4 and 8 pass ECAP processes. When the SEM analysis is examined, it is seen that there is a homogeneous internal structure only in the homogenized Al6063 alloy. It is seen that shear stresses occur due to severe plastic deformation; thus, the slope of the grains begins with the sample subjected to 2 pass ECAP [34]. This slope continues to increase with

the increase in the number of applied passes, and the orientation frequency in the extrusion direction continues to increase in 4 and 8 pass ECAP processes, respectively. The interaction of dislocations with each other becomes more complex with the increase in the number of passes. Dislocations gradually turn into nodes, then the dislocation walls dividing them into sub-grains become apparent in the microstructure. Since the increase in the number of passes will increase the number of sub-grains, grain orientation and the formation of equiaxed grains are expected [35].



**Figure 3.** a) SEM images and b) EDS/Mapping images of Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP process ((a) 2, 4 ve 8 geçişli ECAP işlemine tabi tutulan Al6063 alaşım numunelerinin SEM görüntüleri ve b) EDS/Haritalama görüntüleri)

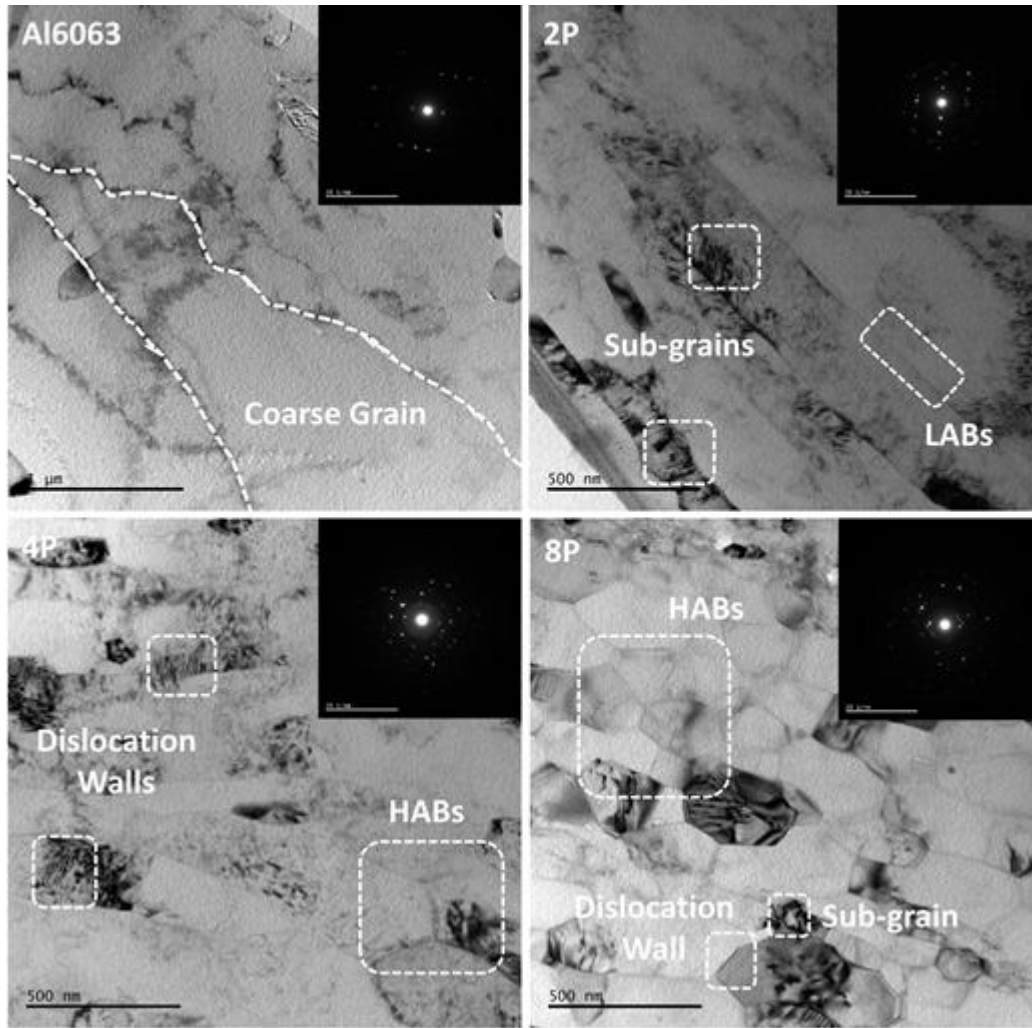
Figure 3 (b) shows the EDS element mapping analysis of the sample with 8 passes of ECAP. In the EDS/Mapping analysis made from the SEM image, in addition to Al, which forms the main phase, the elements with the highest density to the lowest density are Mg, Si and Cu. Especially in the sample with the highest number of passes and 8 passes of ECAP, the elements forming the alloy were detected to have a homogeneous presence in the structure in the deformation zones formed. In addition, it cannot be said that severe plastic deformation affects on the elemental distribution [36]. The EDS/Mapping analysis also determined that the elements in the structure did not form another phase except for the AlFeSi intermetallic compound determined in the XRD analysis.

#### 3.2.2. TEM Analysis and TEM/MapViewer (TEM Analizi ve TEM/MapViewer)

Figure 4 shows TEM and SAED images of Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP processes. It was determined that only the homogenization process Al6063 alloy had large grain sizes. As seen from the TEM images, it was determined that sub-grains started forming in the sample subjected to the 2 pass ECAP process and the grain sizes decreased. However, it was observed that long grains were formed. This situation shows that the inter-grain angles in the 2 pass ECAP process have LABs [37]. With the number of passes being 4, LABs started to give way to HABs. It was determined that dislocation walls were formed in the microstructure with a gradual increase in the dislocation density. This situation is critically important in increasing the mechanical strength [38]. As the number of passes increases to 8, it is seen that the ultra-fine-grained Al6063 alloy shows an equiaxed and homogeneous distribution.

In the applied 2 and 4 pass ECAP processes, the insufficient deformation caused the materials to have an anisotropic structure. However, it was concluded that the grains forming the microstructure with the 8 pass ECAP process had a sufficient isotropic structure [39]. In the analysis of SAED patterns, it is thought that the grain boundaries, together with the deformation effect, decreased the continuity of the diffraction rings with high misorientation and HABs increase [40]. At the beginning stage of the deformation process (2 pass), grains with long and irregular geometries were determined. With the increase in the number of passes, the accumulation and rearrangement of dislocations resulted in a decrease in grain sizes and contributed to the formation of new grains. Increasing the number of passes to 8 passes causes the dislocations in the material to accumulate along the LABs and the boundaries to turn into HABs gradually. The formation of the equiaxed structure with the highest pass, the similarity of grain sizes,

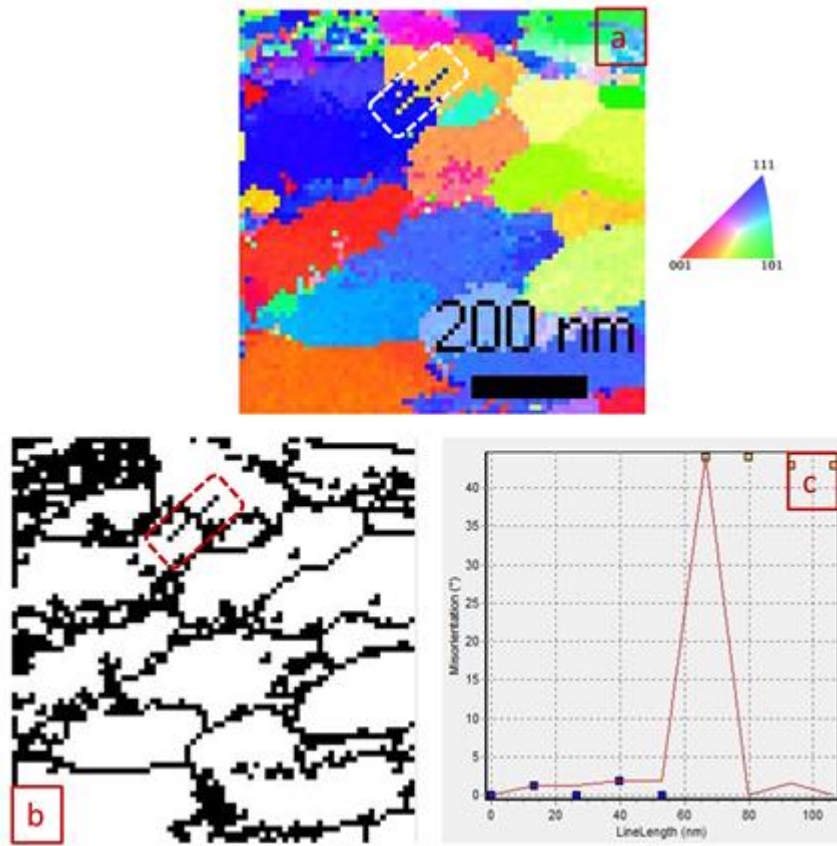
and the formation of submicron/nanograins. In a study conducted by Zhao et al. [41], they investigated the mechanical and microstructural properties of the material by applying a friction stir process to Al6063 alloy. They stated that the high density dislocations formed in the material with severe deformation transformed from LABs to HABs and that the grain refinement process was detected homogeneously in the microstructure. In another study, Rao et al. [42], investigated the mechanical and microstructural properties of the material by applying multidirectional forging to Al6061 alloy. The researchers reported in their study that the sub-grains formed an equiaxed structure with dense dislocation walls and formed with increasing cycle numbers. As can be understood from the studies, it was determined that the 6XXX series alloys, similar to this study, first support the formation of sub-grains by grain reduction with severe plastic deformation and then form an equiaxed and homogeneous microstructure.



**Figure 4.** TEM and SAED images of Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP process (2, 4 ve 8 geçişli ECAP işlemine tabi tutulan Al6063 alaşım numunelerinin TEM ve SAED görüntüleri)

Figure 5 shows the TEM analysis software MapViewer image of the sample with 8 pass ECAP process, together with this technique, similar to the EBSD method. It is seen that the grain sizes decrease to submicron/nano dimensions in the sample with the highest number of passes. In Figure 5 (a), it is also understood that the grains are in different orientations and different orientations. It is determined that the grains are generally oriented in the [111] plane, and then the orientation decreases in the [101] and [011] directions, respectively. A more distinct image of the grain boundaries is given in Figure 5 (b), independent of the orientation. Here, the grains are understood to consist of high-angle grain boundaries (HABs). This situation has made a significant contribution to the formation of an equiaxed and homogeneous internal structure. In

Figure 5 (c) is used to determine the misorientation angle between two randomly determined grains in (a) and (b). When the figure is examined, it is determined that the angle between both grains is higher than  $60^\circ$ . During severe plastic deformation, high density dislocations accumulate in the material's microstructure and contribute to the formation of HABs [43,44]. HABs act as a strong substantial to the movement of dislocations. Large shear strains occur in the deformation formed by the ECAP process. Thus, additional shear systems actively form within the structure, allowing the intensive formation of new deformation-induced boundaries [45,46]. The fact that the material subjected to the ECAP process at room temperature has HABs is due to the selection of the appropriate pass (8) and route (B<sub>C</sub>).



**Figure 5.** TEM/MapViewer analysis of 6063 alloy with 8 pass ECAP process (8 geçişli ECAP prosesi ile 6063 alaşımının TEM/MapViewer analizi)

### 3.3. Microhardness Test (Mikrosertlik Testi)

The microhardness test results of the sample with 2, 4 and 8 passes of ECAP process applied to Al6063 alloy and the untreated sample are given in Table 3. When the figure is examined, it is seen that the hardness values increase with the increase in the number of passes. As the deformation density increases with the number of ECAP passes, grain size decreases while strength increases. As it is

known, the Hall-Petch equation expresses the effect of the grain refinement process on the strength of metallic materials [47]. In the Hall-Petch equation, the decrease in grain size increases the hardness and therefore, the strength of the material. This study reported that the grain sizes decrease and the equiaxed grain structure gradually forms, as in Figure 4. Since the decrease in grain size will create more grain boundaries and the movement of dislocations tends to oppose the grain boundaries



that act as barriers, it is thought that the increase in hardness is appropriate according to the equation in question. As a result, a hardness increase of

approximately 86% was detected when the untreated Al6063 alloy was compared with the sample subjected to 8 pass ECAP process.

**Table 3.** Microhardness test results of Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP process (2, 4 ve 8 geçişli ECAP işlemine tabi tutulan Al6063 alaşım numunelerinin mikro sertlik test sonuçları)

Samples	Microhardness (HV1)
Al6063 Alloy	64
2 Pass ECAP	86
4 Pass ECAP	106
8 Pass ECAP	119

### 3.4. Wear Test (Aşınma Testi)

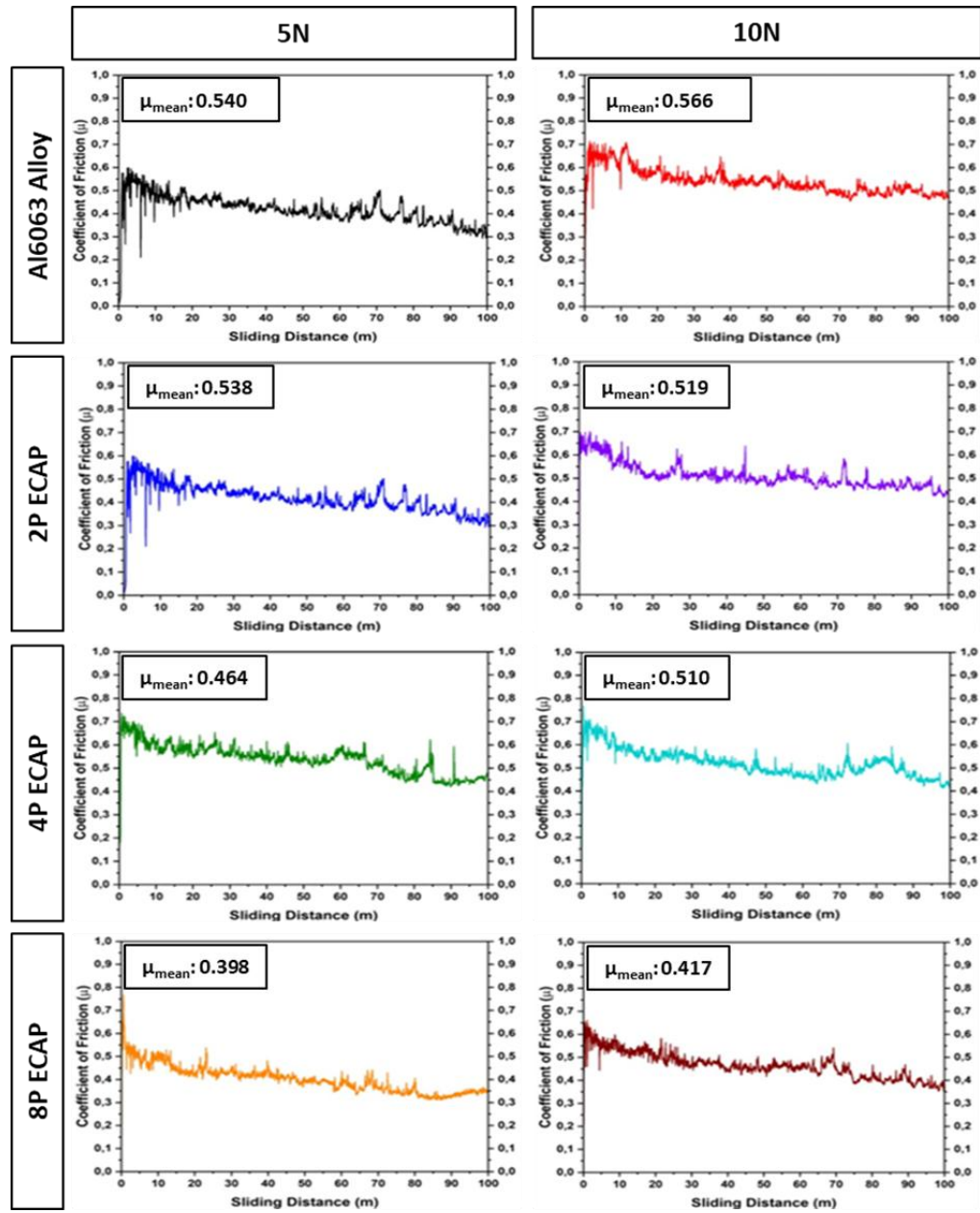
#### 3.4.1. Coefficient of Friction (Sürtünme Katsayısı)

Figure 6 shows the friction coefficient curves of Al6063 alloy and 2, 4 and 8 pass ECAP samples at 100 m distance under 5 and 10N loads. When the figure is examined, it is seen that the friction coefficient values decrease with the increase in the number of passes in the dry sliding wear test performed with a 5N load. Similarly, a decrease in the friction coefficient values was determined with the increase in the number of passes in the dry sliding wear test performed with 10N. An increase and fluctuations in friction coefficient values were observed in all samples. It is thought that the reason for this is that particles or residues such as debris increase the tension at the contact point during the wear test [30,48]. It was determined that the Al6063 alloy with coarse grains gave the highest friction coefficient values in both tests, and the sample with 8 pass ECAP had the lowest friction coefficient values in both tests. This situation can be explained by the fact that the dislocation density increases with the decrease in grain size, transitioning to equiaxed submicron/nanograins, and the hardness decreases the friction coefficient values [48]. When the load was increased from 5N to 10N in the dry sliding wear test and all samples were evaluated among themselves, it was determined that there was an increase in the friction coefficient values of all samples except the sample that had undergone 2 pass ECAP treatment. With the increase in the load, the area between the contact surfaces increases, causing the adhesion forces to increase. Thus, the

increased contact area and adhesion forces increase the friction coefficient. In addition, since the amount of plastic deformation occurring on the surface increases with the increase in the load, the formation of mechanical locking has been stated in previous studies [49]. It is thought that mechanical locking increases the friction coefficient by adhering the surfaces to each other more and creating an adhesive effect. When the friction coefficients performed in all wear tests were evaluated, it was determined that the highest wear rate at 5N load was in the Al6063 alloy with 0.540, and the lowest value was in the sample that underwent 8 passes of ECAP treatment with 0.398. Similarly, in the friction coefficient values of the wear tests performed with 10N, it was determined that the highest value was in the Al 6063 alloy with 0.566, and the lowest value was in the sample that underwent 8 passes of ECAP treatment with 0.417.

#### 3.4.2. Specific Wear Rate and Wear Mass Loss (Spesifik Aşınma Testi ve Aşınma Kütle Kaybı)

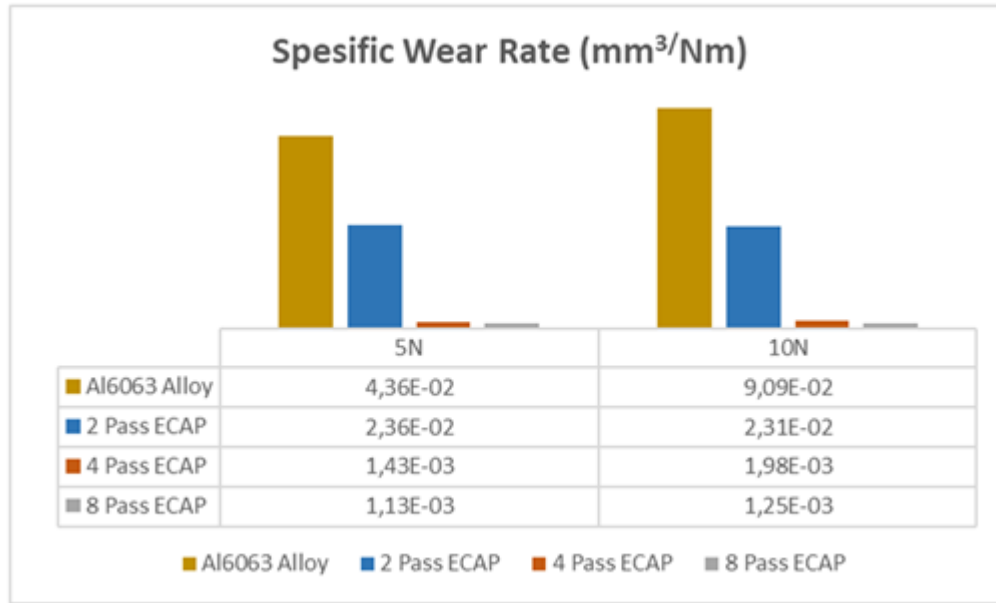
In Figure 7, the specific wear rates of the samples after the wear test under 5 and 10N loads for the Al6063 alloy and 2, 4 and 8 pass ECAP process were calculated from the 3D profilometer device software. When the figure is examined, the wear rates of the samples applied to both 5N and 10N wear tests decrease with the increase in the number of passes. However, when each sample is compared, the load increases the wear rate values. This situation created a wider wear area by increasing the force on the contact surface with the increase in the load. Similar to the friction coefficient curves, the wear rate also increased with the load.



**Figure 6.** 5 and 10N friction coefficient – sliding distance curves of Al6063 alloy, 2, 4 and 8 pass ECAP treated samples (5 ve 10N sürtünme katsayısı - Al6063 alaşımının kayma mesafesi eğrileri, 2, 4 ve 8 geçişli ECAP ile işlenmiş numuneler)

In Figure 7, the specific wear rates of the samples after the wear test under 5 and 10N loads for the Al6063 alloy and 2, 4 and 8 pass ECAP process were calculated from the 3D profilometer device software. When the figure is examined, the wear rates of the samples applied to both 5N and 10N wear tests decrease with the increase in the number

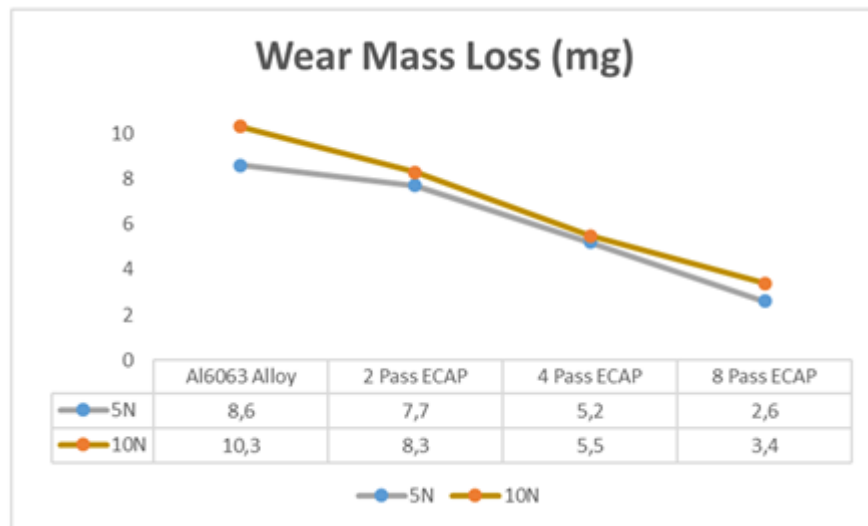
of passes. However, when each sample is compared, the load increases the wear rate values. This situation created a wider wear area by increasing the force on the contact surface with the increase in the load. Similar to the friction coefficient curves, the wear rate also increased with the load.



**Figure 7.** 5 and 10N specific wear rate curves and values of Al6063 alloy samples subjected to 2, 4 and 8 passes ECAP process

Figure 8 shows the mass loss of Al6063 alloy, which was subjected to wear tests at 5 and 10N loads, and samples subjected to 2, 4 and 8 passes of ECAP. In the figure, a decrease in mass loss values was determined with the increase in the number of passes. This situation can be explained by the decrease in grain size as the number of passes

increases and the resulting increase in hardness. Similar to the coefficient of friction and wear rate, when each pass was evaluated within itself, it was determined that mass losses increased with increasing wear load. The resulting material loss is more significant since the increased applied load widens the plastic deformation zone [12,50].



**Figure 8.** 5 and 10N wear mass loss curves and values of Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP process (2, 4 ve 8 geçişli ECAP işlemine tabi tutulan Al6063 alaşım numunelerinin 5 ve 10N aşınma kütle kaybı eğrileri ve değerleri)

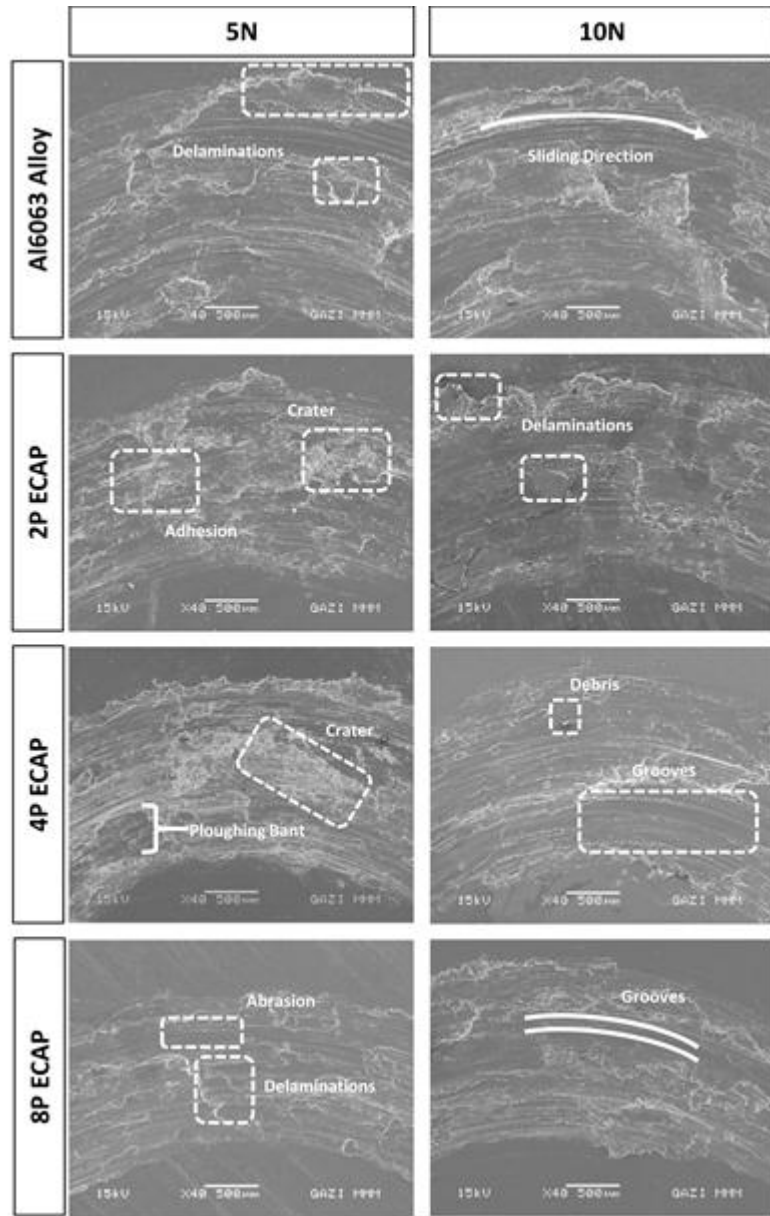
### 3.4.3. Worn Surface ve 3D Optical Profilometer

(Aşınmış Yüzey ve 3B Optik Profilometre)

The surface analyses of the Al6063 alloy samples subjected to 2, 4 and 8 pass ECAP process after wear under 5 and 10N loads are shown in Figure 9. When the worn surface images of the samples subjected to wear tests under 5N load are examined, it is seen that the wear track marks decrease with the increase in the number of passes. Similar results were obtained in the SEM images of the tests under 10N load. It was determined that the diameter of the trace formed in the wear tests conducted from 5N to 10N in all samples generally widened. Since a higher load increases the force applied to the contact surfaces, it results in higher pressure formation. Thus, the wear amount and rate of the samples cause deeper and wider worn surfaces to appear [51]. It is understood that the basic wear mechanism in all samples is adhesive wear. It is thought that this situation causes tension in the inner layers of the material due to the high pressure formed at the contact points as a result of microwelding [52]. The delamination amount is the highest in the Al6063 alloy sample without the ECAP process, with the lowest hardness value. With the increase in the number of passes, the amount of delamination in the samples subjected to the ECAP process tended to decrease. However, an utterly abrasive wear mechanism was not observed in all samples. Especially in the sample subjected to 8 passes of the ECAP process, a complex wear behavior was detected, and adhesive and abrasive wear mechanisms started to occur together. This situation can be explained by the increase in the hardness of the material and the decrease in grain size [53]. A study by Divya et al. investigated the effect of the route on wear properties by performing the ECAP process on AA2014 alloy. The study detected plastic deformation and cracks in wear tests performed at 10 and 30N. They stated that adhesive was the dominant wear mechanism observed with delamination under low load conditions. They

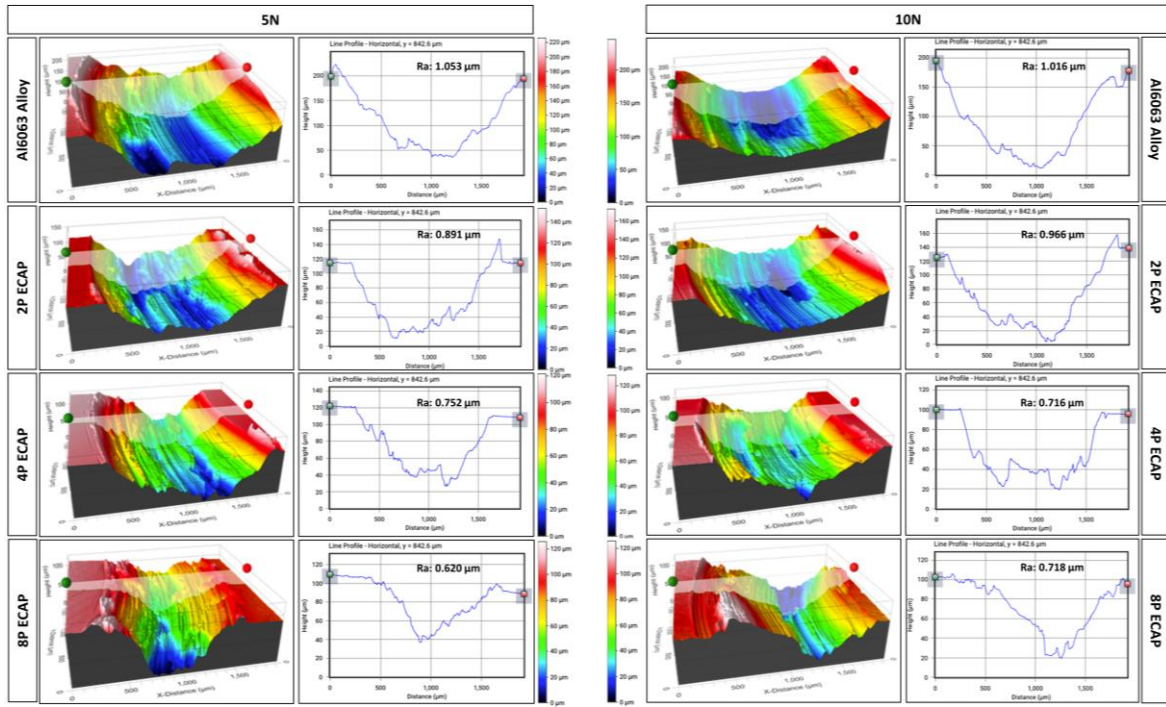
mentioned in their study that a transition from adhesive wear to abrasive wear occurred with the increase in load conditions [54]. Similarly, in this study, as the material hardens and plasticity decreases, the formation of ploughing bands (4P ECAP, 5N) and grooves (8P ECAP, 10N) by the scraping effect of hard particles such as debris formed on the surface is due to a transition from adhesive wear to abrasive wear. This sample with the highest strength is thought to be due to the increased resistance of the material surface to deformation.

Figure 10 shows the 3D profilometer analysis of Al6063 alloy samples subjected to wear tests under 5 and 10N loads and 2, 4, and 8 passes of the ECAP process. When the figure is examined, it is determined that the sample with the highest wear surface volume in the tests conducted under 5N load is Al6063 alloy. Wear volume decreased as the number of passes increased. In addition, the surface roughness (Ra) values also decrease with the increasing number of passes. Similarly, after the wear test was conducted under a 10N load, it was determined that the sample with the highest wear surface volume was Al6063 alloy, and the lowest wear volume was in the sample subjected to 8 passes of the ECAP process. Ra values also show a decreasing trend with the increasing number of passes as 1.016, 0.966, 0.716 and 0.718 $\mu$ m, respectively. Each sample showed increases in Ra values with the increase in the wear load. The formation of a regular microstructure by the fine-grained structure with the increase in the number of passes contributes to the reduction of surface roughness and irregularities [55]. Since the fine-grained structure also increases hardness, the resistance of the sample surface to deformation increases. [56]. Both situations are valid for this study. Due to the decrease in grain size and the increase in hardness with the number of passes, wear volumes and surface roughness values were observed [57].



**Figure 9.** SEM analysis of 5 and 10N worn surfaces of Al6063 alloy, 2, 4 and 8 pass ECAP treated samples (Al6063 alaşımının 5 ve 10N aşınmış yüzeylerinin SEM analizi, 2, 4 ve 8 geçişli ECAP ile işlenmiş numuneler)





**Figure 10.** 3D profilometer analyses of Al6063 alloy samples with 2, 4 and 8 pass ECAP treatment under 5 and 10N loads (5 ve 10N yük altında 2, 4 ve 8 geçişli ECAP işleme tabi tutulan Al6063 alaşım numunelerinin 3B profilometre analizleri)

#### 4. CONCLUSIONS (SONUÇLAR)

The findings of this study, which investigated the microstructural and tribological properties of Al6063 alloy subjected to 2, 4, and 8 passes of ECAP, can be summarized as follows:

- **Microstructural Evolution:** XRD analysis identified Al and AlFeSi phases in the alloy, with peak shifts due to severe plastic deformation. SEM and TEM analyses revealed progressive grain refinement with increasing ECAP passes, leading to the formation of equiaxed sub-micron/nano grains at 8 passes. High-angle grain boundaries (HABs) became dominant in 4 and 8-pass samples, significantly contributing to mechanical strengthening.
- **Mechanical Properties:** The microhardness of the samples increased with the number of ECAP passes. The sample processed with 8 passes exhibited an 86% increase in hardness compared to the untreated Al6063 alloy.
- **Tribological Performance:** Wear test results indicated that an increasing number of ECAP passes resulted in lower friction coefficient values. The highest wear resistance was observed in the 8-pass sample, which had the lowest CoF and wear mass loss. 3D optical

profilometry confirmed a decrease in worn surface area with increasing hardness.

- **Wear Mechanisms:** Adhesive wear was the dominant wear mechanism across all samples, while a transition to an abrasive wear mechanism was observed in the 8-pass ECAP sample due to increased hardness and grain refinement.

Overall, this study confirms that ECAP is an effective technique for significantly enhancing the microstructural, mechanical, and tribological properties of Al6063 alloy. The improvements observed in wear resistance and hardness suggest that ECAP-processed Al6063 alloy can be a promising material for applications in aerospace, automotive, and defense industries, where high performance and durability are critical.

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## 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:** He conducted the experiments, analyzed the results and performed the writing process.

Deneylemleri yapmış, sonuçlarını analiz etmiş ve maklenin 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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