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Research Paper

Experimental Investigation of Heat Treatment Effects on Mechanical Properties and Microstructure of AA6063 Alloy in ECAP Process

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ABSTRACT

Severe Plastic Deformation (SPD) methods have gained significant attention in modern metal forming due to their ability to create desirable microstructures. One highly regarded SPD method is Equal Channel Angular Pressing (ECAP), which induces substantial shear strain to refine grains during the forming process and subsequent recrystallization. Numerous factors influencing ECAP's efficiency and material quality have been subjects of extensive research. This study investigates the effects of various heat treatment cycles on the mechanical properties and microstructure of 6063 AA alloy subjected to ECAP. The ECAP process was performed up to 4 passes at room temperature, following the BC route for aluminum samples of 6063 AA. Additionally, the combination of ECAP and in-passe aging heat treatment was examined. To evaluate the mechanical properties and microstructural changes, tensile and hardness tests along with Scanning Electron Microscopy (SEM) were utilized. Results indicated a significant increase in strength and hardness, coupled with a reduction in elongation, attributed to work hardening and grain refinement. SEM images confirmed ECAP's effectiveness in producing ultrafine grains. Overall, natural aging for 30 days post-ECAP showed no effect on mechanical properties compared to immediate solutionizing and subsequent ECAP. For applications requiring high strength and substantial elongation, artificial aging post-ECAP is recommended.

Keywords: Plastic deformation; Ultra finely grained structure; Equal Channel Angular Pressing (ECAP); Mechanical properties; Microstructure.

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1 INTRODUCTION

ALUMINIUM alloys have been widely used in various industrial applications due to their lightweight, high strength, and excellent corrosion resistance [1,2]. Among them, 6063 AA is particularly notable because of its mechanical properties and widespread application in structural components such as window frames, irrigation tubing, and automotive parts [3]. Equal Channel Angular Pressing (ECAP) is a prominent severe plastic deformation (SPD) process that enhances mechanical properties by producing ultrafine-grained materials [4]. Combining ECAP with various heat treatments can further modify the microstructure and mechanical properties of 6063 AA, paving the way for optimized performance in industrial applications.

Nanomaterials, despite appearing to be a modern innovation, trace their origins to ancient civilizations like Rome. The Lycurgus cup from the 4th century BCE illustrates the use of nanoscale particles of gold and silver to produce striking color changes under varying light conditions. These techniques resurfaced in the Middle Ages with stained glass windows in cathedrals [5]. Advancements in nanotechnology in the 1960s and 1970s included the development of nanoscale metal powders for data storage applications [6]. Richard Feynman's 1959 lecture "There’s Plenty of Room at the Bottom" marked a significant milestone by discussing the manipulation of materials at the atomic level [7].

Among the various SPD techniques, ECAP stands out for its ability to induce significant plastic deformation without changing the workpiece's overall dimensions [8]. ECAP involves pressing a metal sample through a die with two intersecting channels, resulting in ultrafine-grained microstructures with enhanced mechanical properties [9]. Other SPD techniques include Accumulative Roll Bonding (ARB) and Dual Equal Channel Lateral Extrusion (DECLE), each contributing to material property enhancement [10].

Cold working introduces substantial changes in a material's internal structure, including alterations in the crystalline lattice and increased defect density, leading to improved mechanical properties such as hardness and strength [11]. ECAP helps refine grain structures, thus enhancing material properties while maintaining ductility [12].

Heat treatment processes, such as age hardening, play a crucial role in further improving the mechanical properties of aluminum alloys [13]. Age hardening involves heating the alloy to a specific temperature, followed by rapid cooling and reheating at a lower temperature to promote the formation of fine precipitates [14]. This process helps achieve a uniform distribution of phases, increasing the yield strength and hardness of the alloy. Combining age hardening with ECAP can lead to even superior mechanical properties [15].

The 6063 aluminum alloy, part of the 6000 series, is renowned for its excellent mechanical properties and versatility (ASM Handbook, 1993). Comprising magnesium and silicon, the 6063 alloy forms the Mg2Si phase, contributing to its strength and hardness [16]. This alloy is widely used in various structural applications, including door and window frames, architectural structures, and irrigation systems [17]. Incorporating ECAP and heat treatment processes can significantly enhance the alloy's mechanical properties [18].

Recent studies have investigated the impact of different heat treatment processes on the microstructure and mechanical properties of ECAP-processed 6063 aluminum [19-22]. Findings indicate that combining ECAP with age hardening results in a more refined microstructure and better mechanical properties compared to either process alone [23]. For instance, after undergoing four passes through ECAP, followed by natural and artificial aging, the 6063 alloy exhibited a significant increase in yield strength, hardness, and resistance to fatigue [24].

Combining solution heat treatment with ECAP has also been explored to optimize the mechanical properties of 6063 AA. Solution treatment involves heating the alloy to a high temperature to dissolve solute elements, followed by rapid quenching to retain a supersaturated solid solution [25-27]. Integrating solution treatment with ECAP refines the microstructure, resulting in ultrafine grains and improved hardness [28].

Optimizing ECAP parameters such as die geometry, number of passes, and lubrication is crucial for achieving uniform grain distribution and superior mechanical properties [29,30]. Studies have shown that varying the ECAP process route, such as rotating the sample between passes, can lead to more homogeneous grain structures and enhanced mechanical properties [31,32].

In conclusion, the combination of ECAP and various heat treatment processes significantly improves the mechanical properties and microstructure of the 6063 aluminium alloy. The integration of age hardening, solution treatment, and optimized ECAP parameters contributes to the development of high-performance materials with tailored properties for a wide array of applications. As research continues to advance, the potential for further improvements in ECAP-processed aluminium alloys remains promising, offering new opportunities for industrial applications.

2 METHODOLOGY

The overall sample fabrication flowchart for conducting the experiments in this research, as outlined in Figure 1, is thoroughly explained in subsequent parts of this section. After completing the sample fabrication, all specimens underwent tensile tests, hardness measurements, and Scanning Electron Microscopy (SEM) analysis. In this study, the 6063 aluminum alloy, a widely used variant of aluminum, was selected as the test material. The impact of Equal Channel Angular Pressing (ECAP) on the mechanical properties and microstructure of this alloy was investigated. The preparation process of the specimens for testing and the execution of the ECAP process are elaborated in detail.

Initially, extruded aluminum samples of 6063 alloy were prepared for the experiments. The chemical composition analysis of the samples, conducted using quantitative methods, is presented in Table 1. As seen in the sampling flowchart, there are two general categories for sampling. At first, the first category (samples that have only been subjected to various thermal processes) is as follows.

1. Raw material (O)
2. Solution heat treatment (S)
3. Natural age hardening heat treatment (30 Days) (T4)
4. Artificial age hardening heat treatment (T6)

In the following, the second category (various heat treatments), ECAP was also performed on the samples, dividing them into the following categories:

1. ECAP Process (O + ECAP)
2. Solution Heat Treatment + ECAP (S + ECAP)
3. Solution Heat Treatment + ECAP + Natural Aging (30 days) (S + ECAP + T4)
4. Solution Heat Treatment + ECAP + Artificial Aging (S + ECAP + T6)
5. Solution Heat Treatment + One Pass ECAP + Natural Aging + One Pass ECAP (S + 1P ECAP + T6 + 1P ECAP)

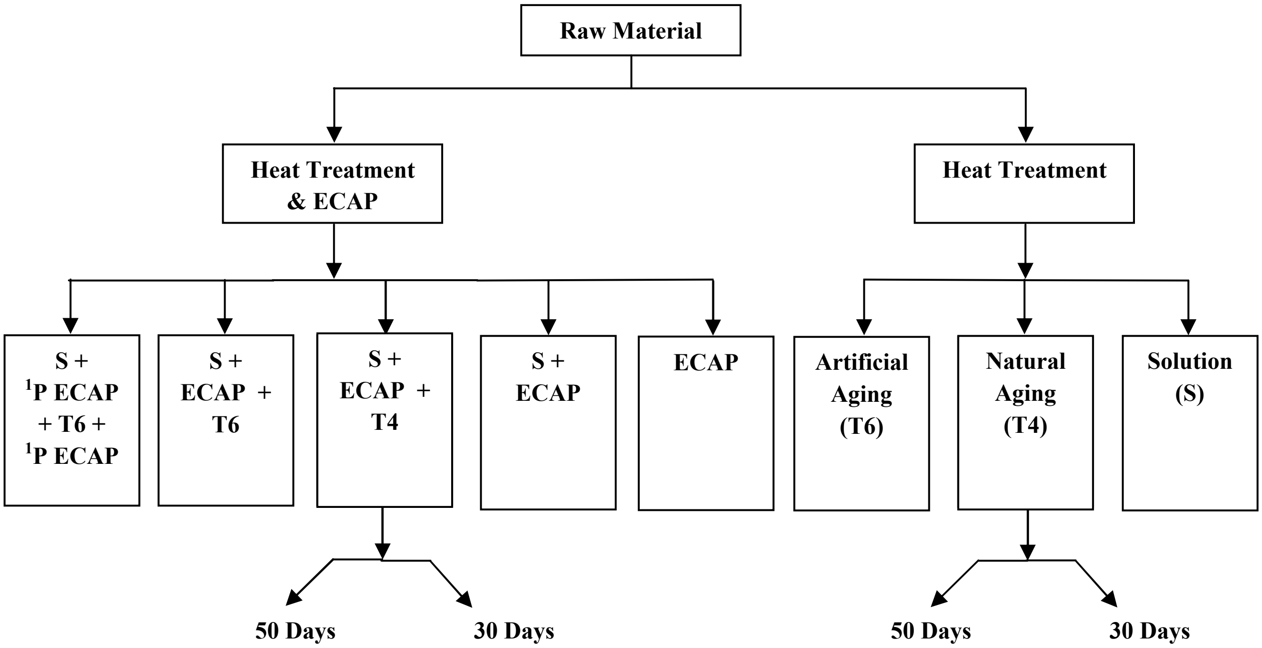


Fig. 1
Overall sample fabrication chart.

Table 1
Sample analysis

Element	Al	Si	Fe	Cu	Mg	Zn	Ti
Percent	98.93	0.28	0.16	0.02	0.51	0.04	0.02

2.1 Solution heat treatment (S)

To conduct this experiment and prepare the desired sample, it was essential to determine the solution temperature and duration. Using available resources, the temperature and time for the solution treatment for the investigated alloy were selected [33]. The chosen temperature for this operation was 520 °C, and the duration was one to two hours. Once these two crucial factors were identified, a thermal operation was conducted using a furnace named "Esaghoolian Furnace". The furnace was initially heated to 520 °C, and the sample was maintained at this temperature for one hour. After this process, the sample was promptly and without delay quenched in water. This immediate quenching is critical as any delay between the heat treatment and quenching can cause the part to undergo unwanted precipitation hardening.

2.2 Natural aging heat treatment (T4)

The preparation of this sample followed a similar approach to the previous method (Solution Heat Treatment). However, after the raw material was subjected to the solution heat treatment at 520 °C for one hour and promptly quenched in water, the sample was maintained at room temperature in open air for 30 and 50 days. As a result, the sample underwent natural aging (T4).

2.3 Artificial aging heat treatment (T6)

The preparation of this sample involved an artificial aging heat treatment, similar to the previous phase. However, instead of natural aging in open air, the sample was aged in a furnace. Here's how the process was conducted:

As we know, in the artificial aging process, temperature and time are crucial factors. Therefore, two specific temperatures, 180 °C and 200 °C, along with various durations (5-10-20-30 minutes, and 1-2-4-6-8-10-12 hours) were selected. After conducting the heat treatments as specified, hardness tests were performed on the samples to determine the maximum hardness. As indicated in the tables and graphs, the maximum hardness at 200 °C was achieved after 4 hours, while at 180 °C, the maximum hardness was obtained after 8 hours. This is expected since temperature and time in artificial aging are inversely related—higher aging temperatures require shorter durations [34].

However, the data revealed that a higher hardness was achieved at 180 °C for 8 hours compared to 200 °C for 4 hours. Therefore, based on the hardness test results, the highest hardness for the 6063 AA alloy was found at 180 °C for 8 hours. These findings are consistent with existing literature. Similar parameters can also be found in the ASM Heat Treating Handbook. Following the initial solution heat treatment at 520 °C for one hour and subsequent quenching in water, the sample was then artificially aged in a furnace at 180 °C for 8 hours.

2.4 Heat treatments and ECAP Process

Before delving into the sample fabrication process, it's essential to discuss the die, press, routes used, and other specifications employed in conducting the Equal Channel Angular Pressing (ECAP) experiments:

- Die Type: The samples were fabricated using a die with a channel angle (φ) of 90 degrees and a corner angle (ψ) of 20 degrees with a rounded channel cavity, as illustrated in Figures 2.
- Type of Press: A 40-ton hydraulic press with a speed of 5 millimeters per minute was used to move the sample into the die, as shown in Figure 3.
- Sample Route: For the ECAP process, samples with dimensions of approximately 19 mm diameter and a maximum length of 140 mm were prepared. As mentioned before, the BC route exhibits the highest hardness and strength among different ECAP sample routes, as concluded by Sun et al. [35]. In the BC route, the sample is continuously rotated 90 degrees in the positive direction. Hence, the ECAP process was conducted using the BC route.
- Lubricant Type: Molybdenum disulfide (MOS_2) was used as the lubricant for the ECAP operation.
- Number of Passes: The sample underwent 4 passes through the die.
- Operating Temperature: The process was conducted at room temperature.

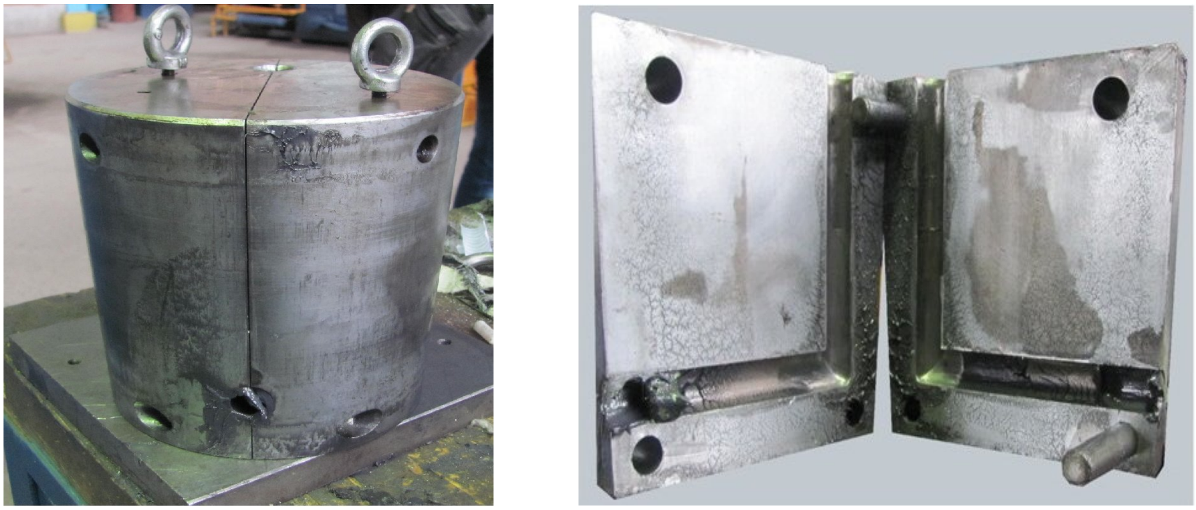

(a) (b)

Fig. 2
(a) ECAP close die and (b) ECAP open die used in experiments.

Fig. 3
40-ton hydraulic press used in experiments.

2.4.1 Equal Channel Angular Pressing (ECAP) process

To prepare a sample subjected to ECAP, the following steps were taken:

1. Sample Preparation: Initially, the samples were machined to the specified dimensions. The inner cavity of the die was then lubricated with MOS_2 lubricant.
2. Die Preparation: The die was closed and made ready for the ECAP process. The die was placed under the press, and the sample was inserted into the die channel.
3. Pressing Process: Using the punch and the previously mentioned press, the sample was pushed completely through the die channel. At this stage, the press force reached 30 bars.
4. Cleaning and Repositioning for Subsequent Passes: The die was removed from the press, and the sample was taken out. The die cavity was cleaned. For the next pass, the sample was rotated 90 degrees in the positive direction, reinserted into the die channel, and subjected to the same pressing process. The press force reached 35 bars during the second pass.
5. Repeating the Process for Third and Fourth Passes: All preceding steps were repeated for the third and fourth passes, with the press force reaching 40 bars and 42 bars, respectively.
6. Thus, a sample that underwent four passes of ECAP (O + ECAP) with the specified conditions was prepared.

2.4.2 Solution heat treatment + ECAP process

To prepare this sample, a raw material of the specified dimensions, as mentioned in previous methods, was first machined. After the heat treat furnace reached 520 °C, the sample was kept in the furnace for one hour and then immediately quenched in water. In the next stage, the ECAP process was conducted as explained in the previous methods, i.e., 1 to 4 passes using the BC route at room temperature and MOS_2 lubricant.

Thus, the sample subjected to both Solution Heat Treatment and ECAP (S + ECAP) was prepared.

2.4.3 Solution heat treatment + ECAP + Natural aging for 30 days process

The preparation of this sample was similar to the previous method. However, after the ECAP process, the samples were maintained in open air for 30 and 50 days. Thus, the sample subjected to both ECAP and Natural Aging (ECAP + S) was prepared.

2.4.4 Solution heat treatment + ECAP + Artificial aging process

The preparation of this sample followed the same steps as the previous methods, including raw material preparation, solution heat treatment, and the ECAP process.

However, in the final phase, artificial aging (T6) was applied. As previously mentioned, both temperature and aging time are crucial for this process. When a material undergoes solution heat treatment (Solid Solution) followed by cold working (like ECAP), the dislocation density increases, leading to quicker dislocation diffusion and precipitation (aging) [36]. Therefore, the same temperature and time used for raw material aging (180°C and 8 hours) can't be applied here. To determine the appropriate temperature and time for artificial aging, the experiment was conducted as follows:

The material subjected to solution heat treatment and ECAP (S + ECAP) was divided into several parts and placed in a furnace at 180 °C for varying durations (1, 2, 4, 6, 8, 10, and 12 hours). After artificial aging, hardness tests were performed.

Analysis of the hardness values and graphs revealed the maximum hardness was achieved at 180 °C for one hour. Notably, if the same maximum hardness time (8 hours) is to be reached as with the raw material's artificial aging (T6), a lower temperature must be considered. Therefore, for this sample's preparation, the raw material was first subjected to solution heat treatment at 520 °C for one hour, followed by the ECAP process with the specified conditions. In the final phase, the sample underwent artificial aging (T6) at 180 °C for one hour.

Thus, the S + ECAP + T6 sample was successfully prepared.

2.4.5 Solution heat treatment + Single Pass ECAP + Artificial aging + Single pass ECAP process

To prepare the sample with the specified characteristics, the following steps were taken:

1. Initial Preparation: A raw material with a diameter of 19 mm and a length of 140 mm was prepared. The sample was subjected to solution heat treatment at 520 °C for one hour, followed by quenching in water.
2. First ECAP Process: The ECAP process was conducted as in the previous methods, but in this cycle, the sample only underwent one pass (single pass) ECAP.
3. Artificial Aging: The sample was then subjected to artificial aging (T6) at 180 °C for 8 hours.
4. Second ECAP Process: In the final phase of this cycle, the sample underwent another single pass ECAP. During this ECAP operation, the sample was not rotated and the process was conducted using Route A.

The reasoning for selecting a single pass in this cycle is based on reports and references mentioned in previous section, which state that the most significant increase in strength and hardness occurs in the first ECAP stage. Additionally, if four passes of ECAP were selected, followed by artificial aging (T6) and then another ECAP process, the high hardness and strength developed in the sample could likely result in issues such as cracks or fractures in both the sample and the die.

2.5 Conducted tests on samples

Following the preparation of the samples, as detailed in the previous section, we now describe the various tests conducted on the samples:

2.5.1 Tensile test

After the Equal Channel Angular Pressing (ECAP) process, tensile test specimens were prepared from both deformed and non-deformed samples (only solution heat-treated). The gauge length was chosen in the longitudinal direction of the deformed sample, and the specimens were machined with a gauge length of 45 mm according to the standard ASTM B557M – 02a. All tensile tests were conducted at room temperature using a SANTAM STM-150 tensile testing machine, which stretched the samples at a speed of 1 mm per minute. The dimensions of the tensile test sample are given in Table 2.

2.5.2 Hardness test

To determine the hardness value of the samples, the hardness test was conducted using a KOOPA Universal Hardness Tester Model: UV1. With the suitable method for these samples being Vickers hardness testing, the samples were tested accordingly. The Vickers hardness test was performed at three different points on the sample surface, and the average hardness was recorded in the tables and charts.

As we know, the Vickers method uses a diamond pyramid indenter with a square base, having an angle of 136 degrees between opposite faces. In this test, a force of 1 kgf was applied for 30 seconds. After removing the force, the diagonal lengths of the indentation were measured using an optical microscope, and the hardness values were calculated using standard tables or the Vickers hardness formula.

$$VHN = \frac{1.8544\ \mathrm{F}}{d^2} \tag{1}$$

2.5.3 Scanning Electron Microscope (SEM) test

To examine the microstructural changes in the aluminum samples, a Scanning Electron Microscope (SEM) was used, offering greater magnification than optical microscopes. Images were taken from the initial sample which underwent natural aging (T4) as well as from samples subjected to solution heat treatment, ECAP, and then both natural aging (T4) and artificial aging (T6). For this test, a SEM VEGA TESCAN was used, with electron acceleration voltages of 10 kV and 20 kV.

Table 2
The dimensions of the tensile test sample

Nominal Diameter	Dimensions, mm			
	Standard Specimen	Small-Size Specimens Proportional to Standard		
	12.5	9	6	4
G-Gage length	62.50±0.10	45.00±0.09	30.00±0.06	20.00±0.04
D-Diameter (Note 1)	12.50±0.25	9.00±0.10	6.00±0.10	4.00±0.05
R-Radius of fillet, mm	9	8	6	4
A-Length of reduced section, min (Note 2)	75	54	36	24

3 RESULTS AND DISCUSSIONS

3.1 Tensile test results

The results from the tensile tests (yield strength, ultimate tensile strength, and ductility) of the heat-treated samples and the samples that underwent ECAP and heat treatment are shown in Figures 4, 5, 6, and 7. As these charts indicate, the application of artificial aging and the severe plastic deformation process have led to significant improvements in mechanical properties.

The increases in yield strength, ultimate tensile strength, and decreased elongation percentage from the ECAP process can be attributed to the following reasons:

1. Cold Working: As a result of cold working the samples, dislocations are produced. With increasing applied strain, the density of these dislocations rises, leading to more interactions between them. Therefore, more pressing stages results in more cold work on the sample, which in turn increases its strength (work hardening).
2. Dislocation Boundaries and Grain Refinement: Severe strain introduces dislocation boundaries and divides the initial grains into smaller units called sub-grains. As the applied strain increases, the distance between these boundaries decreases, leading to a microstructure with very fine grains, thus increasing strength (grain refinement). The partial reduction in elongation percentage can also be attributed to work hardening.

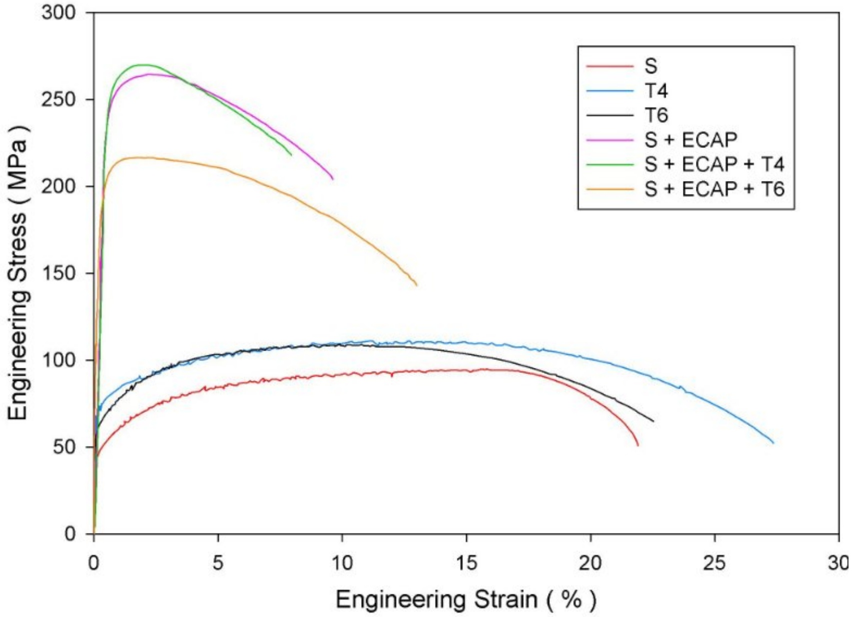


Fig. 4
Comparison of engineering stress-strain diagrams conducted from tensile tests.

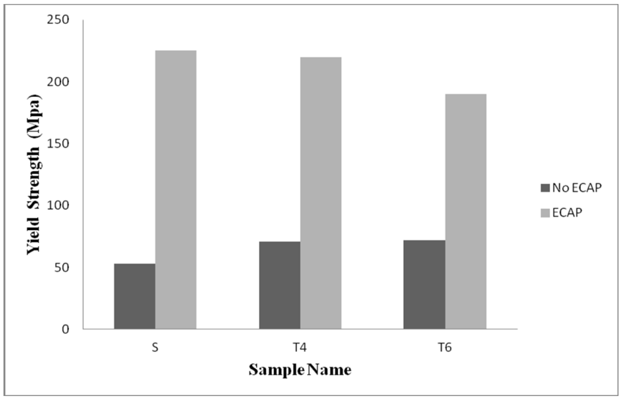


Fig. 5
Comparison of yield strength values, for samples in heat treatment mode and heat treatment with ECAP process.

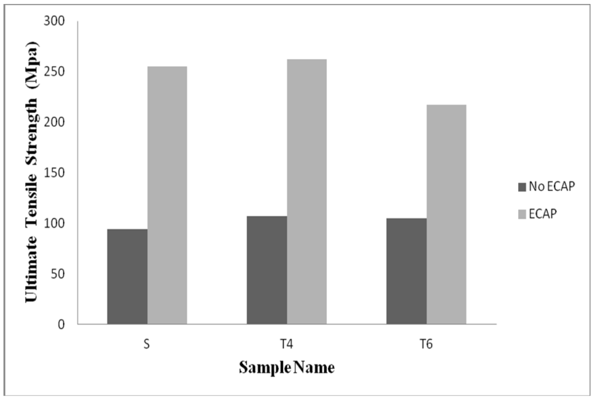


Fig. 6
Comparison of ultimate tensile strength, for samples in heat treatment mode and heat treatment with ECAP process.

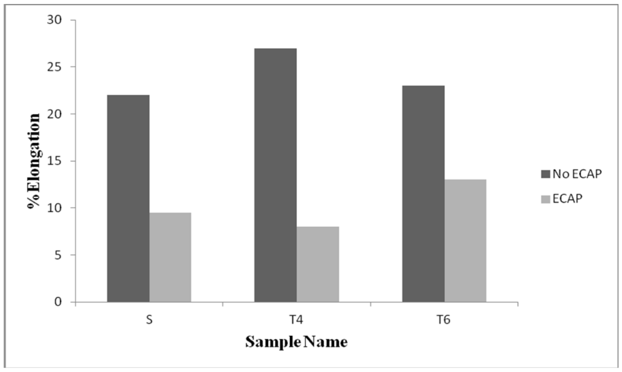


Fig. 7
Comparison of elongation percentage, for samples in heat treatment mode and heat treatment with ECAP process.

3.1.1 Tensile behavior of heat-treated samples

The yield stress and maximum tensile strength of samples subjected to natural aging (T4) and artificial aging (T6) are higher than those subjected to solution heat treatment alone. Based on Fig. 8, the following conclusions can be drawn:

a. Both natural and artificial aging heat treatments have been effective on this sample and improved its mechanical properties.
b. The mechanical properties (yield stress and maximum tensile strength) obtained by maintaining a sample at 180°C for 8 hours (artificial aging) are almost the same as those achieved by keeping it in open air for 30 days (natural aging). However, the elongation percentage differs.
c. As seen in Fig. 8, the elongation percentage of the naturally aged sample is higher than that of the solution heat-treated and artificially aged samples. Where elongation percentage is also important alongside mechanical properties, natural aging should be used.
d. The fluctuations observed in these graphs can be explained by the phenomenon of strain aging.

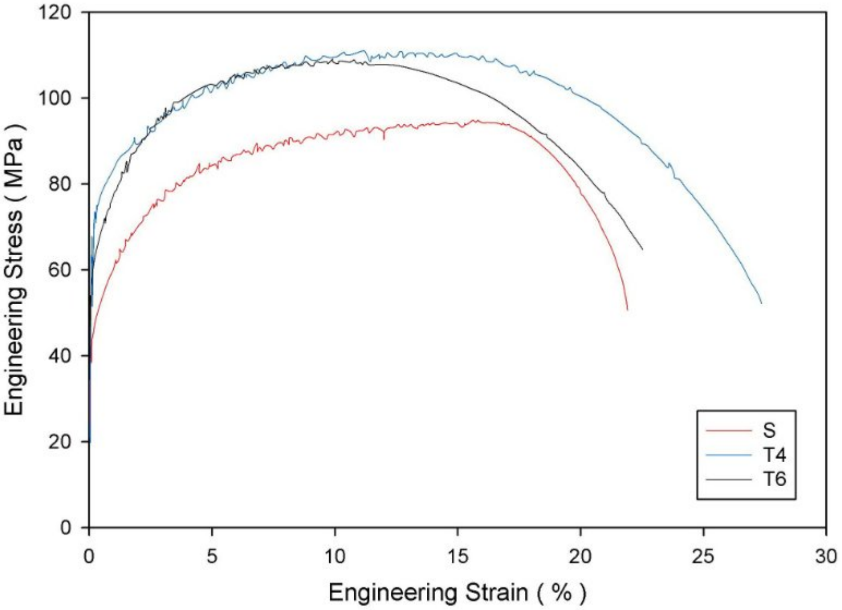


Fig. 8
Comparison of engineering stress-strain diagrams of samples subjected to different heat treatments.

3.1.2 Comparison of heat-treated samples with heat-treated and ECAP processed samples

As shown in Figures 9, 10, 11, and 12, the application of the ECAP process significantly increases the yield stress and maximum tensile strength of the samples (an average increase of about 150 MPa), while the elongation percentage decreases. Additionally, there's a larger elastic deformation region and a smaller plastic deformation region. The general reasons for these changes can be explained as follows:

a. Increased Work Hardening: The ECAP process results in significant work hardening.
b. Formation of Dislocation Forests: The severe plastic deformation creates dense dislocation networks, often referred to as dislocation forests.
c. Severe Plastic Deformation (SPD) and Grain Refinement: The SPD leads to the formation of ultra-fine grains, enhancing the material's strength.
d. Recovery and Recrystallization: These processes contribute to the overall structural changes.

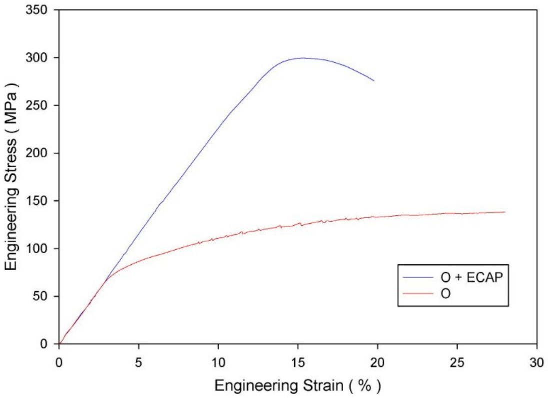


Fig. 9
Comparison of the engineering stress-strain diagrams of the raw sample with the ECAP raw sample.

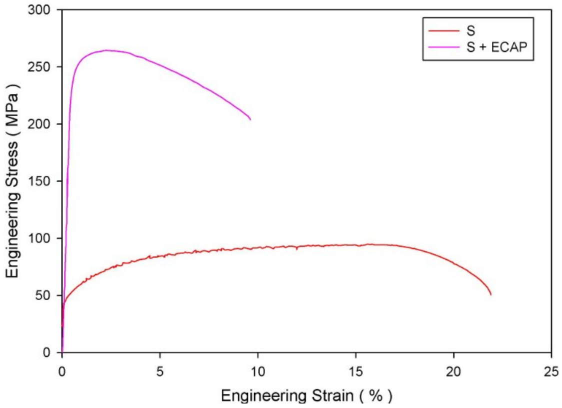


Fig. 10
Comparison of engineering stress-strain diagrams of sample S with sample S + ECAP.

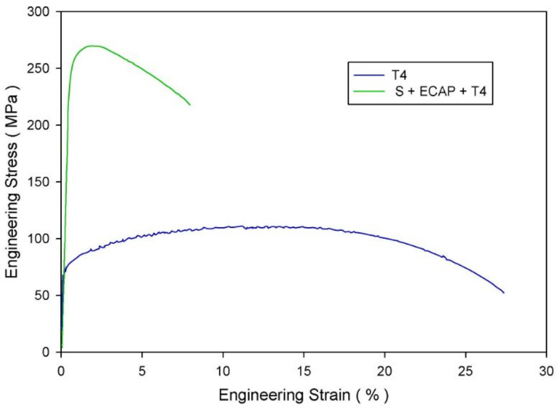


Fig. 11
Comparison of engineering stress-strain diagrams of T4 sample with S + ECAP + T4 sample in 30 days.

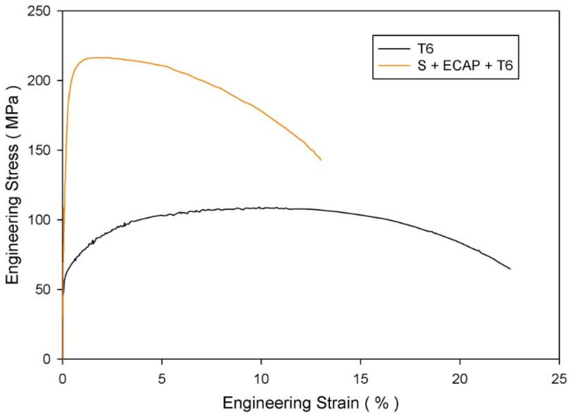


Fig. 12
Comparison of engineering stress-strain diagrams of sample T6 with sample S + ECAP + T6.

3.1.3 Analysis of (S + ECAP) and (S + ECAP + T4) samples

The governing mechanism in S + ECAP and S + ECAP + T4 samples, besides reasons like cold working, work hardening, and dislocation forests, is due to dynamic recovery and recrystallization caused by the press pressure during the ECAP process. This leads to increases in yield stress, maximum tensile strength, hardness, grain refinement, and decreases in elongation percentage compared to samples before the ECAP process. As seen in Fig. 13, natural aging (T4) for 30 days after the ECAP process does not affect the yield stress and maximum tensile strength. It only causes a slight reduction in the elongation percentage. However, it can be generally stated that T4 heat treatment for 30 days has no significant impact on the mechanical properties of the S + ECAP samples.

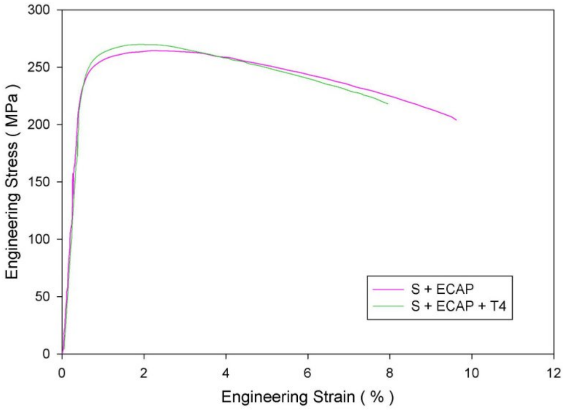


Fig. 13
Comparison of engineering stress-strain diagrams of S + ECAP samples with S + ECAP + T4 samples in 30 days.

3.1.4 Analysis of (S + ECAP + T6) sample

In all discussed cases, when the mechanical properties, such as yield stress and ultimate tensile strength, increase, the elongation percentage (% Elongation) decreases compared to samples before the ECAP process. This is not desirable when elongation is important. However, using T6 heat treatment after the ECAP process can improve these properties to some extent, as illustrated in Fig. 14.

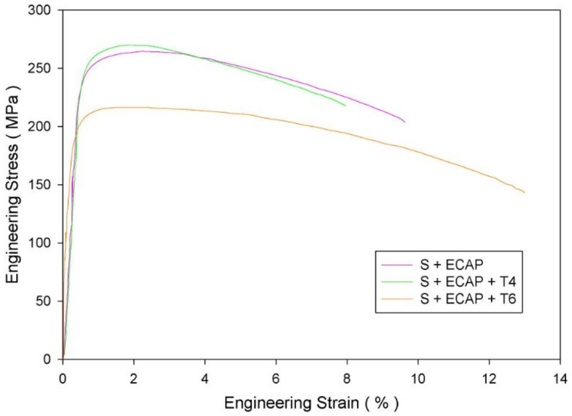


Fig. 14
Comparison of engineering stress-strain diagrams of samples subjected to different heat treatments along with pressing operations in ECAP process.

The reasons can be explained as follows:

a. Significant Work Hardening: The formation of dislocation forests due to severe plastic deformation.
b. Dynamic Recovery: Due to the heat generated by the pressing on the sample during ECAP.
c. Static Recovery: Due to the heat after the ECAP process for the T6 heat treatment.

As mentioned in previous section, both temperature and time are required for recovery and recrystallization.

During dynamic recovery, both factors (temperature and time) are minimal, especially since the use of lubricant reduces the temperature to its lowest level, and the entire process of one pass takes a maximum of one minute. However, in static recovery, both temperature and time are sufficiently present (180°C for one hour). The decrease in strength and increase in elongation percentage in this sample can be explained as follows:

Static recovery and recrystallization due to artificial aging after the ECAP process reduces some of the work hardening developed in the sample. These results in a slight decrease in yield stress and ultimate tensile strength and an increase in elongation percentage compared to the previous samples, as shown in Fig. 14.

3.1.5 Effect of ECAP after Artificial Aging (T6) on ECAP processed sample

As shown in Fig. 15, performing ECAP after artificial aging (T6) on a sample that had undergone ECAP increases the yield stress and ultimate tensile strength compared to a sample that underwent artificial aging after ECAP. However, the elongation percentage significantly decreases. The reasons for these differences can be explained as follows:

After the sample undergoes a single pass of ECAP and then artificial aging (T6), it becomes more prepared for the next pass due to recovery and recrystallization. This results in a significant increase in strength after the second pass, but the elongation percentage dramatically drops.

The reason the obtained strength in Fig. 15 doesn’t reach the same levels as other samples is due to the fact that this sample underwent 2 fewer passes of ECAP. Hence, less work hardening and severe plastic deformation were induced.

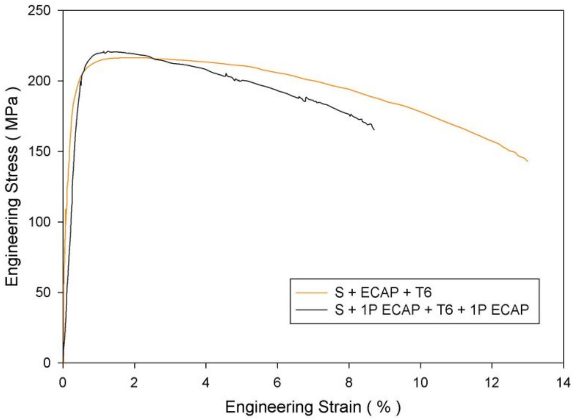


Fig. 15
Comparison of engineering stress-strain diagrams of S + ECAP + T6 sample with S + 1P ECAP + T6 + 1P ECAP.

3.2 Hardness test results

The hardness test results of the heat-treated samples and the samples that underwent both ECAP and heat treatment are shown in Fig. 16. The reasons mentioned for increasing strength can also explain the hardness increase in the process. As observed, the hardness of the solution heat-treated sample is approximately 56 Vickers, which increases slightly to 62 Vickers with natural aging (30 and 50 days). However, in artificially aged samples, the hardness rises to 75 Vickers, an increase of about 19 Vickers.

In samples that also underwent ECAP, the hardness of the sample subjected to solution heat treatment followed by ECAP reaches 75 Vickers. For the sample that underwent natural aging for 30 and 50 days after ECAP, the hardness increases to 82 Vickers, and for the sample that underwent artificial aging after ECAP, the hardness reaches 90 Vickers. So it can be concluded that by using the ECAP process and heat treatment of artificial aging, we have increased 35 Vickers.

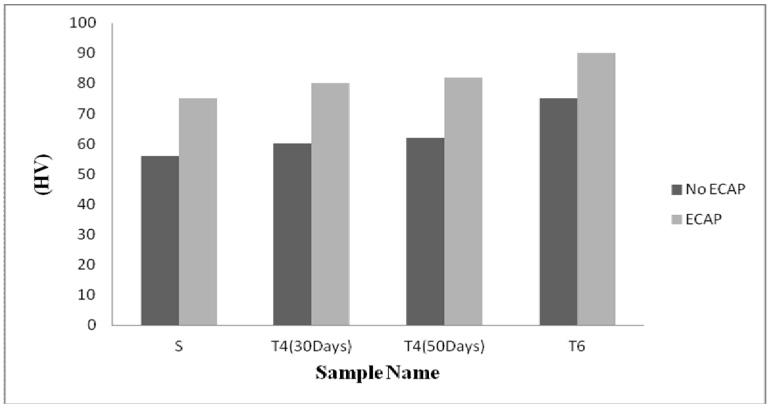


Fig. 16
Comparison chart of hardness values, for samples in heat treatment mode and heat treatment with ECAP process.

3.2.1 Heat-Treated Samples

The hardness test results of the samples that only underwent heat treatment are shown in Fig. 17. From this figure, we can conclude the following:

a. The hardness of the sample subjected to artificial aging is higher than that of the samples subjected to solution heat treatment and natural aging. Thus, for this sample and alloy, we can deduce that natural aging up to 50 days results in lower hardness than artificial aging at 180°C for 8 hours.

b. Another conclusion from the hardness results is that the hardness of the naturally aged sample remains unchanged for 30 and 50 days.

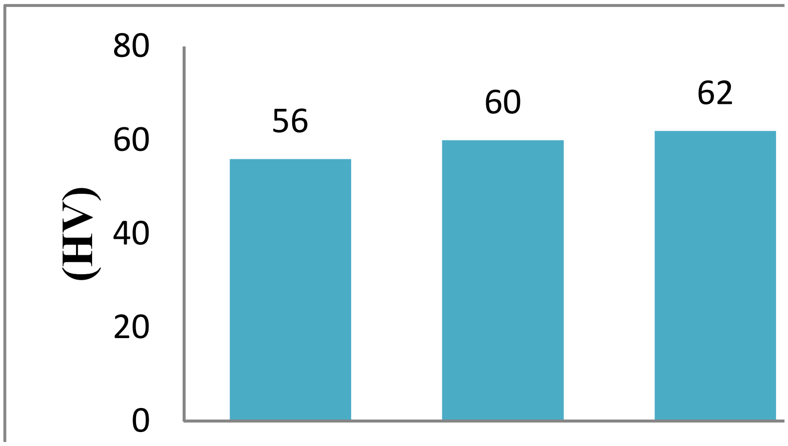


Fig. 17
Hardness chart obtained from the hardness measurement of samples that have only been heat treated.

3.2.2 Heat-Treated and ECAP samples

From Fig. 18, we can conclude the following:

a. The hardness achieved with artificial aging after ECAP is significantly higher than the hardness achieved with natural aging for 50 days after ECAP.

b. As natural aging after ECAP did not significantly impact the mechanical properties, it also does not substantially affect the hardness of the sample, to the extent that it can be almost disregarded.

c. Another reason for the increased hardness can be attributed to the uniform distribution of precipitates in the matrix (precipitation hardening phenomenon), which can be evidenced by the SEM images obtained from the samples (Figures 19, 20, and 21).

As observed in the images, Mg_2Si precipitates (hard intermetallic compounds) are distributed in the soft aluminum matrix, leading to increased hardness. Through the ECAP and T6 processes, the precipitates are broken down and distributed in the matrix, which results in increased yield stress, maximum tensile strength, and elongation percentage. This phenomenon is known as precipitation hardening. Another observation from the SEM images is that the material's constituent particles become very fine after the ECAP process.

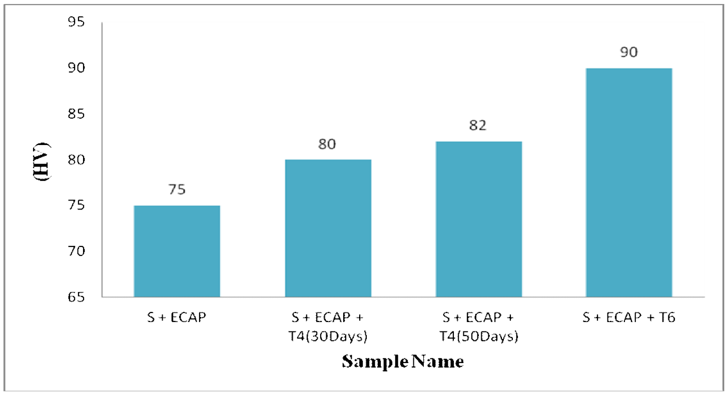
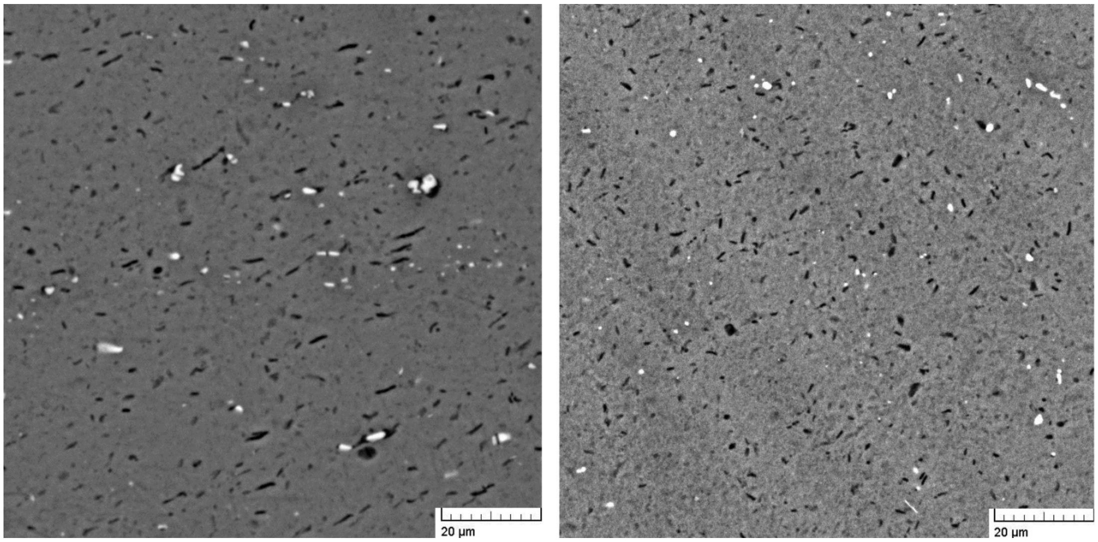
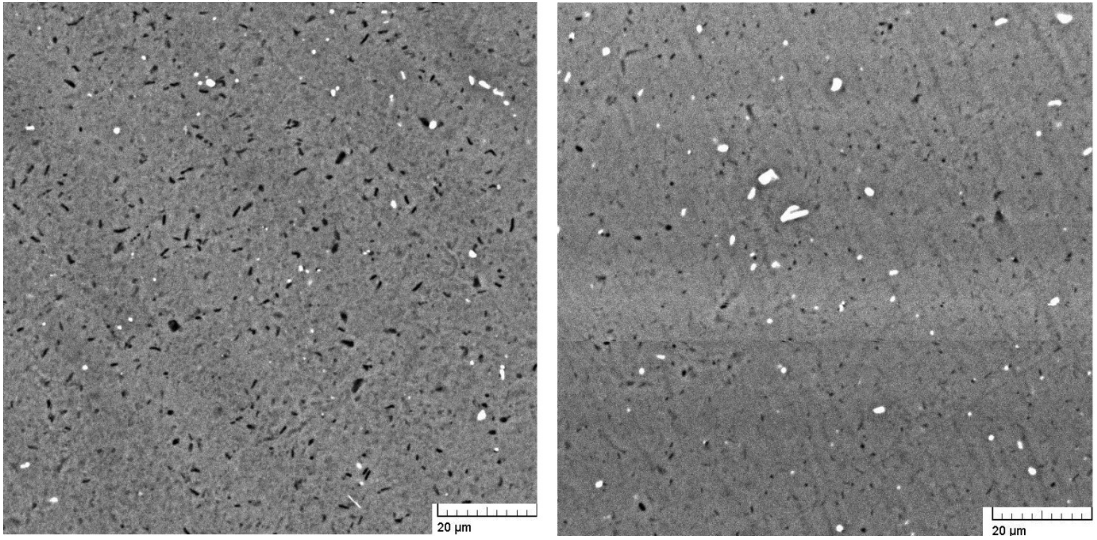


Fig. 18
The graph obtained from the hardness measurement of the samples that have undergone heat treatment and ECAP treatment.



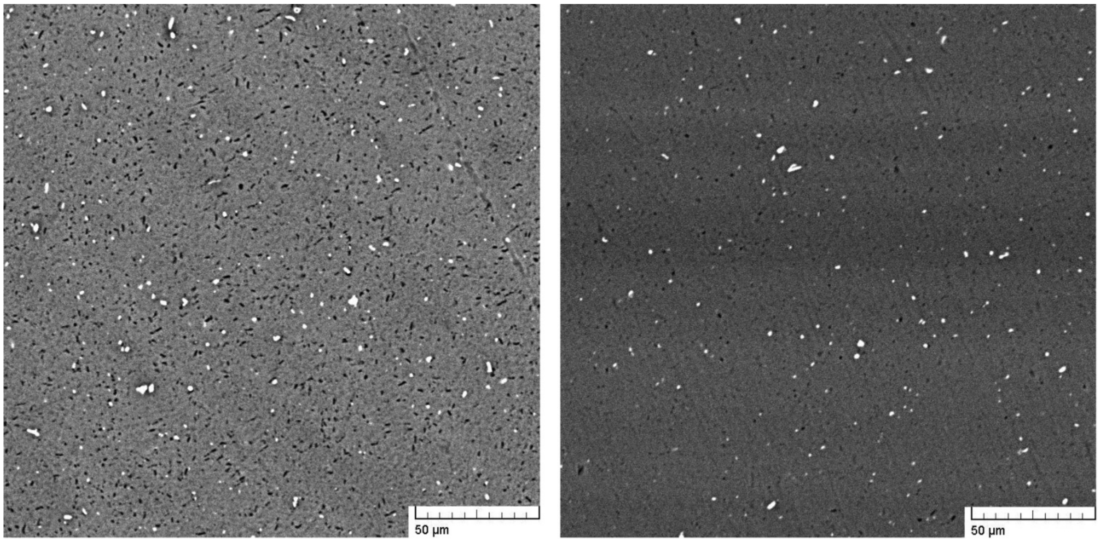
(a) (b)

Fig. 19
Images of a) S + ECAP + T4, b) T4.



(a) (b)

Fig. 20
Images of a) S + ECAP + T6, b) S + ECAP + T4.



(a) (b)

Fig. 21
Images of a) S + ECAP + T6, b) S + ECAP + T4.

3.2.3 Hardness comparison of S + ECAP + T6 with S + 1P ECAP + T6 + 1P ECAP samples

After examining Fig. 22, it was observed that the hardness of the S + ECAP + T6 sample is higher than that of the S + 1P ECAP + T6 + 1P ECAP sample. The primary reason for this difference is the number of ECAP passes. As referenced, the hardness increases with the number of passes until it reaches its maximum value, due to the increased dislocation density after each ECAP pass.

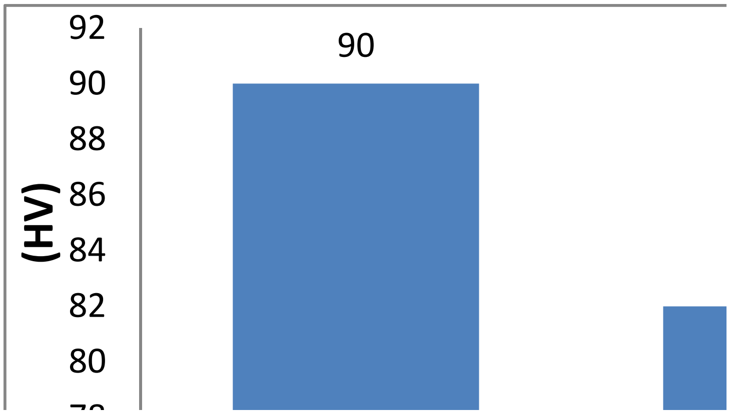


Fig. 22
The graph obtained from the hardness measurement of samples S + ECAP + T6 and S + 1P ECAP + T6 + 1P ECAP.

Table 3
The changes in the mechanical properties of the ECAP samples

Heat Treatments on Aluminum 6063		Yield Strength MPa	Tensile Strength MPa	%Elongation	HV
S	Before Deformation	53	94	22	55
	After Deformation	225	255	9.5	75
	Percentage of Changes	+325	+170	-43	+36
T4	Before Deformation	71	107	27	60
	After Deformation	220	262	8	80
	Percentage of Changes	+210	+145	-30	+33
T6	Before Deformation	72	105	23	75
	After Deformation	190	217	13	90
	Percentage of Changes	+163	+106	-56	+20

4 CONCLUSIONS

From the examination of the mechanical properties and microstructure of AA6063 alloy in various heat treatment conditions combined with the ECAP process, the following main conclusions were drawn, as summarized in Table 3:

- Significant Increase in Strength and Hardness: The ECAP process significantly enhances the strength and hardness of the samples. This increase is attributed to work hardening and grain refinement mechanisms.
- Minimal Impact of Natural Aging: Natural aging for 30 days after ECAP has a very minimal impact on the mechanical properties, to the extent that it can be disregarded.
- Effect of Artificial Aging: Artificial aging after the ECAP process slightly reduces the yield stress and maximum tensile strength, but increases the hardness and elongation percentage. This can be explained by static recovery and recrystallization due to heat. The sample from this process cycle can be considered the optimal one where both high mechanical properties and elongation are important.
- Precipitation Hardening Phenomenon: SEM images obtained from the samples indicate that the ECAP process combined with T6 heat treatment results in precipitation hardening, ultimately leading to increased hardness.
- No Impact from Natural Aging in T4 Condition: The hardness test results demonstrate no significant impact from natural aging for 30 and 50 days on the S + ECAP + T4 samples.
- Consistency with Literature: The obtained results are consistent with the findings of Yuntian et al. [39].

Overall, these results indicate that the ECAP process is highly effective for producing fine-grained samples with excellent and uniform mechanical properties.

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