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Effect of ECAP process on mechanical properties and microstructure of AA-6061 recycled chips

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HIGHLIGHTS

- Severe plastic deformation was utilized to improve mechanical properties, microstructure, and microhardness.
- Improvements in tensile strength and yield strength are reported for the ECAPed Al-6061 alloys.
- Ductility decreased from 27% to 13% after one pass.
- The micro-hardness increased from 47.55 HV to 79.36 HV, an increase of about 67% after four pass.

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ABSTRACT

The oxidation of aluminum machined chips makes successful recycling via the traditional re-melting technique difficult. A viable solution to this problem is to utilize solid-state recycling to transform aluminum machined chips directly into semi-finished goods, eliminating the costs of the re-melting process and minimizing CO₂ emissions. In the present study, Al-6061 alloy chips have been recycled using hot extrusion followed by severe plastic deformation at ambient temperature to examine the material's mechanical properties and microstructures. The current study investigates the effects of equal channel angular pressing samples after (1, 2, and 4) cycles using a die with 90° and 20° angles, a strain of 1 was applied each pass for a maximum of four cycles at room temperature, through different routes (BC,C) on the mechanical properties of recycled specimens before and after the process. Observations showed a significant improvement in the characteristics of recycled chips exposed to hot extrusion followed by the ECAP technique. Maximum ultimate tensile strength (265, and 238 MPa) maximum yield strength (149, and 136 MPa), and elongation to failure (46 and 41%) for both routes were obtained. Moreover, each pass increases yield strength ultimate tensile strength, and micro-hardness while increasing elongation to failure. The mechanical characteristics of ECAPed specimens were higher than the extruded specimen. The mechanical properties and microstructures of solidstate recycled Al-6061 alloy chip samples were significantly influenced by the number of passes and route types.

1. Introduction

Based on its favored properties such as moderate toughness, strong corrosion resistance, medium to high strength, and outstanding workability, The 6061 aluminum alloy represents among the best commonly used 6000 series aluminum alloys. A significant amount of waste (chips and discards) is formed when aluminum goods are fabricated [1]. Since recycling materials are cheap, recycling waste has become a particularly economical means of manufacturing resources [2]. Scrap recycling is divided into two categories: traditional and solid-state recycling [3]. The traditional method involves melting the waste for recycling. It is defined by large operating costs enormous power utilization and an enormous variety of operations [4].

The recycling of wastes without re-melting is known as solid-state- recycling problems associated with traditional methods. Solid-state recycling of aluminum scraps may save 40% of the materials 26-31% of energy and 16-60% of the worker when compared to traditional recycling [5]. Chips formed during semi-finished aluminum products being machined are extremely challenging to recycle using traditional techniques due to their small sizes elongated spiral shapes machining oil, and surface contamination with oxides. [6]. Haase recycled aluminum chips utilizing Solid-state recycled [7]. Solid-state was used to make composite materials from Aluminum and AlCu4 alloy chipped and tungsten powders [8, 9]. This approach has a significant benefit in that it may utilize nearly 95% of the source material by reducing the loss of metal during the casting technique.

Furthermore, Gronostajski et al. [1] presented a listing of criteria influencing mechanical characteristics of patterns for chipbased extrusion, including extrusion rates, extrusion temperatures, extrusion ratios, cold pressing variables, and chip sizes. The essential difficulties in the process of aluminum chip consolidating are strong bonding between chips and the avoidance of porosities, Both have a considerable influence on the mechanical features of extruding profiles. A particular level of strain is essential from a metallurgical viewpoint in order to breakdown oxides layers on the chip's surfaces and obtain strong bonding between chips. It is essential to improve the features of recycled materials to fulfill the growing demand for aluminum alloys with superior mechanical properties. Many earlier investigations on the solid-state recycling of machining chips using extreme plastic deformations (SPD) had been conducted.SPD techniques induce extremely substantial plastic strains into bulk materials, as a result of which super textured microstructures with grain size smaller than one micrometer and grain boundaries with high misorientation angles [10]. Equal-channel angular pressing (ECAP) is a unique technology capable of introducing significant shear strains without changing the cross-section of the workpiece throughout several cycles of deformations via a specific die [11].

Haase combined extrusion with equal channel angular pressing[12]. Al-Alimi et al. [13] evaluated SPD by ECAP of Al-6061 alloy utilizing solid-state technique (direct recycling), which includes chip preprocessing cold compaction, and hot extrusion followed by the ECAP process. Extrusion specimens with four passes of hot ECAP and heat treatment exhibited a maximum UTS of 291 MPa, compared to 324 MPa for the as-received Al-6061 specimen.

M.Harnicárová et al. [14] investigated the SPD of an Al-6000 alloy. The grain size was reduced from $28.90\mu m$ to $4.63\mu m$. A remarkable enhancement in the material's strength by 45% and a big decrease in ductility to 60% were verified instantly after the initial extrusion.

Gupta et al. [15] performed an experimental study examination of the Al-6063 alloy by ECAP. The observations revealed that after 6 passes at ambient temperature, the hardness improved to 85 HV. Only one pass at ambient temperature enhances hardness by up to 83%. Furthermore, the average grain size of the specimen after one pass at higher temperatures is ~20 μm.

Selmy et al. [16] studied severe plastic deformation in aluminum alloy (AA-6061) chips recycled at ambient temperature employing ECAP. The findings demonstrated that the ECAP technology influenced the mechanical characteristics of recycled specimens. After 6 passes at 500 °C, the maximal UTS (403 MPa), highest micro-hardness (110 HV), and elongation to failure (14.6%) were achieved.

Kadiyan et al. [17] used ECAP to conduct an experimental investigation on the Al-6063 alloy using several types of routes A, BC, and C. Processes were done with four, eight, and ten runs. Maximum tensile strength, yield strength, and elongation to failure (176MPa, 159MPa, 10.95%) were obtained with route C, respectively.

Incremnetal-ECAP was used by M. Ciemiorek et al. [18] to study plates of the Al-3030 alloy process done with one, two, three, and four runs. The findings indicated that maximum tensile strength and microhardness 240MPa and 70 HV were achieved, correspondingly.

Furthermore, Al-8176 alloy rods were examined by G. Shuai [19] using ECAP at two different temperatures: room temperature, and 150 °C. The process was done with one, two, four, six, and, eight passes. The results demonstrated that, at 150 °C, maximum UTS and elongation 186MPa, 10.5% were obtained, respectively.

Extrusion or ECAP processing of SPD materials is now becoming investigated for the production of ultrafine-grained structures for various engineering materials. High-strength, semi-finished goods made from ECAP-treated aluminum powders or wastes can be employed in the power, automobile, and aerospace industries. Fasteners including such screw rivets and screws utilized in the construction of aluminum aircraft structures, in addition to other engineering structures or aspects for aviation parts (skins, stringers, plates, and so on) are examples of these items [20].

Al-6061 is a common aluminum alloy in the metals industry. Many aluminum-producing firms are manufacturing the alloy at a low cost for various uses such as pipes and fittings, agricultural and irrigation purposes, furniture manufacturing, and automobiles [21]. Al-6061 is a medium-strength alloy with great formability, heat treatability, machinability, weldability, and corrosion resistance.

2. Experimental

2.1 Materials and Equipment

The aluminum alloy AA6061 (as-received) was employed in this investigation, and it is chemical composition, mechanical properties, and physical features are mentioned in Tables 1,2, and 3. Figure 1 also illustrates the setup for the experimental study. The used chips were produced by dry turning of the as-received bar at the specified cutting variables: 88 m/min cutting speed; 1 mm depth of cut; and 1 mm/rev feed.[22]. Chips were compressed at 300 °C with a pressure of 200 bars at this level, followed by extrusion at 370°C for 20 minutes through the conical-face die at a speed of 10 mm/sec with an extrusion ratio of 7.11 and plastic strain level \approx 2 and pressure was 100 bars at this stage. The (15) mm diameter extruded specimens were then processed by ECAP (1, 2 & 4) passes with plastic strains at different levels ($\varepsilon = 1.05$, $\varepsilon = 2.1$, and $\varepsilon = 4$) at ambient temperature via route BC, and C utilizing an ECAP die channel angle $\Phi = 90^\circ$ and arc of curvature angle $\Psi = 20^\circ$ targeting an improvement of their mechanical properties.

Table 1: Chemical Compositions of AA6061 aluminum alloy [23, 24]

| | | | | | | | | | | Base |
|-----------------------|------|------|------|------|------|------|------|-------|--------|--------|
| Elements | Mg | Si | Fe | Cu | Mn | Cr | Zn | Ti | Others | Al |
| %Min | 0.8 | 0.4 | - | 0.15 | - | 0.04 | - | - | 0.15 | 95.8 |
| %Max | 1.2 | 0.8 | 0.7 | 0.4 | 0.15 | 0.35 | 0.25 | 0.15 | | 98.6 |
| Al-6061 Current study | 0.92 | 0.60 | 0.26 | 0.23 | 0.15 | 0.12 | 0.03 | 0.013 | 0.115 | 97.562 |

Table 2: Mechanical Properties of AA6061 aluminum alloy [23, 24]

| Property | 6061-T4 | 6061-T6 |
|-----------------------|----------------------|----------------------|
| Tensile Strength | 241 MPa 35000 psi | 310 MPa 45000 psi |
| Yield Strength | 145 MPa 21000 psi | 276 MPa 40000 psi |
| Modulus of Elasticity | 68.9 GPa 10000 ksi | 68.9 GPa 10000 ksi |

Table 3: Physical Properties of AA6061 aluminum alloy [23, 24]

| Property | 6061-T4 | 6061-T6 |
|----------|---------------------------------------|---------------------------------------|
| Density | 2.70 g/cc 0.0975 lb/in ³ | 2.70 g/cc 0.0975 lb/in ³ |

2.2 Experimental Work Preparations



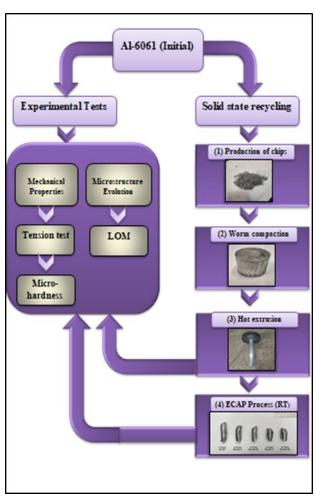


Figure 1: Represented experimental work layout (Flow chart)

In the present work, Al-6061 alloy chips were machined through the dry turning of the as-received bar according to the following cutting parameters cutting speed 88 m/min depth of cut 1 mm and feed 1 mm/rev [22] as shown in Figure 2.

The chips were compacted at 300°C using heat during the compaction stage to increase the weldability and achieve a more compacted sample. The compacted sample's dimensions were = Φ 45 x 80 mm, and a pressure of 200 bars was used at this stage, as shown in Figure 3.





Figure 2: Al-6061 alloy prepared of chips

Figure 3: A compacted sample of 6061 aluminum alloy chips

Both compacted sample and extrusion die were heated until they reached 370°C and held for 20 minutes (to make sure all the compacted sample and die were heated enough).

The semi-final products with a 15 mm diameter were produced by extruding at 370° C using a conical-face die and an extruded ratio of (R = 9), which is a level of the plastic strain of \approx 2 using a horizontal hydraulic press of 200 tons at a pressing speed of 10 mm/s under an extrusion pressure of 100 bar, as shown in Figure 4



Figure 4: (15) mm diameter extruded samples of Al-6061 aluminum alloy chips

To ensure sound bonding between chips, two conditions must be met [25]. The first condition is for sufficient plastic strains to be present in order to breakdowns the oxide layers on the surfaces of the chip and get a good material bond. The next stage is to generate perfect conditions of temperature and high pressure in order to complete the bonding of the chips altogether. If the oxide layer is partially disrupted, the chips will not fuse perfectly [25]. Hot extrusion, which involves intense shear pressures and compressive was used to achieve complete consolidation of AA6061 compaction recycled chips [16].

Circular specimens of (15) mm in diameter and 60 mm in length Figure 4 in form of billets were prepared from extruded specimens. The ECAP procedure was carried out using a two-piece split die, as shown graphically in Figures 5, 6 and 7(a,b and c). The die material was H13 tool steel. This type of die is suitable for reducing the time of the process because it doesn't have

bolts. The channel intersecting angle is $\Phi = 90^{\circ}$ and the outer corner angle is $\Psi = 20^{\circ}$. In order to achieve better grain refinements and mechanical properties both rout BC and C were selected for conducting the ECAP process based on a recommendation in [26].

The ECAP process applies to billets up to four passes at room temperature. was attained, i.e., specimens were pressed at a speed of 10 mm during the process. Using a 100-ton vertical hydraulic press with a 10 mm/s pressing speed, The corresponding strain in each pass is roughly 1.05 according to the ECAP technology [27]. The specimen was subjected to large, simple shear forces [28].

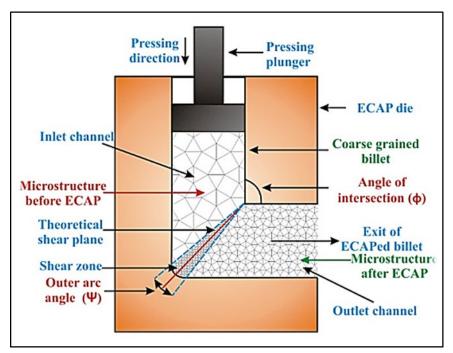


Figure 5: Principle of ECAP [29]

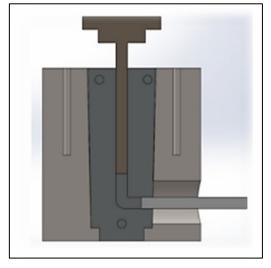


Figure 6: Schematic illustration of ECAP Die [28]



Figure 7: (a) Appearances of ECAP Die (b) two pieces split die parts (c) ECAPed sample during the process

Figure 6The billets (Φ 15 x 50) mm process was carried out using a die through an angular die with two-channel intersection angles of 90° and 20° arc of curvature. The number of passes processed by 1, 2, and 4 at various plastic strain levels (E = 1.05, E = 2.1, and E = 4) via routes BC and C at room temperature (RT-ECAP) was investigated. The comparable strain for each specimen is around 1.05 per run. The most effective route for producing UFG materials with a homogeneous microstructure is Route BC, which employs a 100-ton vertical hydraulic press with a pressing speed of 10 mm/s. Molybdenum disulfide (MoS₂) was used as a lubricant for each pressing [30, 31] as demonstrated in Figure 8

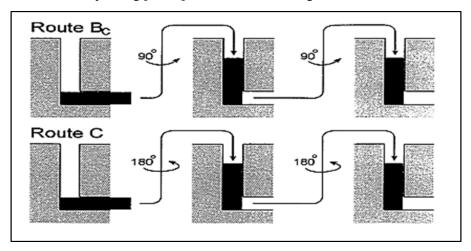


Figure 8: Schematic demonstration of route BC and C [30, 31]

The billets were pressed repetitively for 1, 2, and 4 passes, giving a maximum total strain of \sim 4. The pressings were performed using a pressing speed of 10 mm/sec and processing route BC, in which the billet is rotated about the longitudinal axis by 90° in the same direction between each pass [32]. This processing route was selected because using a die with $\Phi = 90^{\circ}$ leads most expeditiously to an array of equiaxed ultrafine grains separated by boundaries having high angles of misorientation [30]. As shown in Figure 9 (a,b,c,d,e)

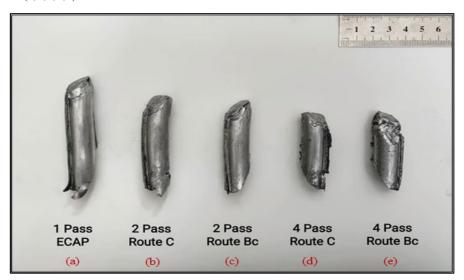


Figure 9: (a) 1 Pass (b) 2 Pass route C (c) 2 Pass route BC (d) 4 Pass route C ,and (e) 4 Pass route BC samples

2.3 Testing Characterizations

Tensile testing on the UTM (Model Name: SANTAM STM-50) made of ECAP processed material was carried out in the laboratory. Electro-discharge machining (EDM) wire-cutting machine was used to cut all specimens. The dog-bone-shaped tensile specimens were cut in the plane perpendicular along the extrusion direction, with a gauge length of 5 mm and a thickness of 3 mm.

A micro-hardness analyzer was used to determine the micro-hardness of the specimens during ECAP processing. A 200-gram load was applied for 10 seconds throughout this process.

Microstructure to appropriately polish the specimen's various grades of emery paper were utilized. Additionally, the samples were polished with abrasives such as alumina. The images were obtained from an optical microscope with a (125x, 250x, 500x, and 1000x) magnification zoom.

3. Results and Discussion

The investigations were carried out on commercially AA6061 aluminum chips recycled by hot extrusion, followed by SPD processed through routes BC and C ECAP die, and their results are discussed in the parts that follow.

3.1 ECAP Passes Have an Influence on Stress-Strain Behavior

The tensile properties of both as-built and ECAPed Al-6061 samples were tested to mechanically characterize and evaluate the effect of SPD on the initial sample. All samples were cut via electro-discharge machining (EDM) wire-cutting machine. The dog-bone-shaped tensile samples with 5 mm gauge length and 3 mm thickness were cut on the longitudinal plane along the extrusion direction as shown in Figure 10(a,b). ASTM E8/E8M-16 standards were followed for all samples sub-size tensile samples 5 mm gage length 3 mm width for the reduced section and approximately 3 mm thickness [33].

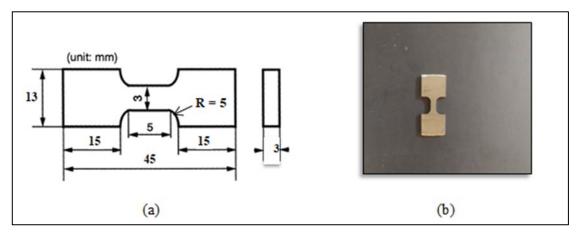


Figure 10: Appearance specimen; (a) tensile test sample dimensions in millimeters (mm); and (b) actual tensile test sample [33]

According to Figure 11, the beginning section of the curve for the as-received material presents an essentially direct correlation and achieves the highest value of stress of approximately 250 MPa and strain of around 0.3. The equivalent values of stress and strain after one pass were 209 MPa and 0.15, correspondingly. They were 232 MPa and 0.22 after the second pass. The readings climbed to 265 MPa and 0.46 after the fourth pass. The interplay of the shear and slip bands promotes grain growth at sub-micron levels, which is the last mechanism for grain refinement.

The improvement in strength properties after each ECAP process due to increasing accumulated strains of the specimen might be attributed to the specimen material's reduced grain size, or producing UFGs. Furthermore, The decrease in grain size following each ECAP procedure might be the source of the decline in elongation. If grain reduction is the source of the higher tensile strengths and reduction in elongation [27]. Following that, the Hall-Patch formula is used, followed by deformation material. A few Al ECAP-processed investigations [34]. Tensile strength and elongation decreases showed a similar trend.

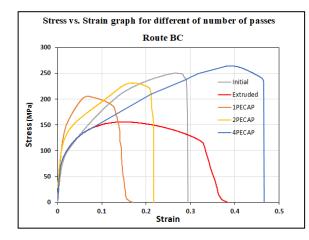


Figure 11: Comparative stress vs. strain curve as received extruded (Initial), (1, 2, and 4) pass, ECAP route BC of the experimental aluminum alloy Al 6061 chips recycling

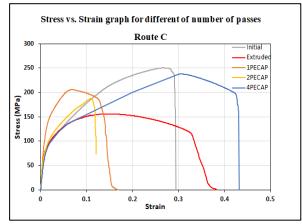


Figure 12: Comparative stress vs. strain curve as received (Initial), extruded, (1, 2, and 4) pass, ECAP route C of the experimental aluminum alloy Al 6061 chips recycling

According to Figure 12, the beginning section of the curve for the as-received material exhibited an essentially direct correlation and achieved a high value of stress of approximately 250 MPa and strain of around 0.3. The comparable values of stress and strain after one pass were 209 MPa and 0.15, respectively, and 182 MPa and 0.11 after the second pass. The findings after the fourth run were 238MPa and 0.42, respectively. Route BC provides the most appropriate method for generating UFG materials with homogeneous microstructures [30, 31], as shown in Figure 8.

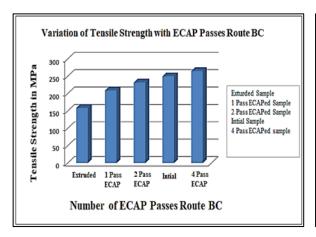
3.2 ECAP Passes Have an Effect on Tensile Strength

Tensile strength is an essential material's major characteristic and was determined on the UTM of the model (SANTAM) STM-50 using the IS 1608-2005 (RA 2017)/IS 1586-1:2018 protocol. Seven different kinds of specimens were organized: one as receiving material (Initial), one without a pass (extruded specimen) one with one pass two passes four passes route BC, and two passes and four passes route C. Figure 13(a), and (b) represented tension samples before and after tensile test. Figure 14 illustrate graphs comparing the difference in tensile strength after each ECAP pass using route BC, and route C. Moreover, Tables 4 and 5 are examples of Mechanical properties of Al-6061 alloy before and after ECAP using route BC, and route C. Owing to the large imposed shear strain and grains consolidating, the tensile strength increased dramatically after the first pass from 159 to 209 MPa, then marginally enhanced after the second pass to 232 MPa, and ultimately increased to 265 MPa after the fourth pass due to additional grain refining. The obtained findings followed a similar pattern to those of an earlier investigation. The findings were experimentally validated, and it has been observed that there is a considerable increase in the AA6063 tensile strength after four passes using 90-degree channels [35, 36].



Figure 13: Actual specimens' appearance; (a) Various samples following ECAP preparation and (b)

Tensile test tension samples; and (c) After a tensile test, the specimens were fractured



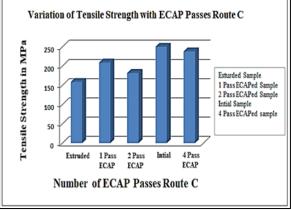


Figure 14: Comparative graphs demonstrating tensile strength variations after each ECAP cycle using route BC, and route C

The tensile characteristics of Al6061 alloy before and after ECAP are tabulated in Tables 4 and 5 The ultimate tensile strength (UTS) and yield strength (YS) of the no-pass sample (extruded sample), were 159 MPa and 105 MPa, respectively, with a 33% elongation. The YS and UTS values climb as the number of runs increases. Furthermore, following the fourth pass, the UTS value of this sample improves to 265MPa and the YS value improves to 149MPa with a 46% elongation. Though the alloy's strength rises as the number of passes increases, the percentage of elongation improves from 33% to 46%. This might be related to the processing of ECAP and the resulting strain-hardening of materials. It is estimated that the increases in the strength of the alloy preparation procedure utilizing ECAP are mostly due to grain refinements [37]. After a single run of ECAP, the grain size is dramatically reduced and purified. Furthermore, partition manifests itself in certain grains and grain borders and is enhanced as a result. Furthermore, twins play an important role in modifying the YS, which may serve as a barrier to disengagement slide and contribute to superior features. As an outcome, the growth of YS is driven by grain refinement and the combination of twinning and separation or dynamic recycrestalization mechanism. It is apparent that following the critical pass, prolongation is reduced. The malleability of materials generally diminishes after they have been twisted. The decrease in development can be attributed to metals' exceptional plasticity, and partition advancement cannot anticipate a role in refining microstructures [38].

Tables 4 and 5 show the tensile characteristics obtained for up to four ECAP passes using varying routes. As illustrated in Figure 13(b), various tensile samples were constructed from received, extruded, and ECAPed billets with gauge lengths of 5 mm lying parallel to the longitudinal axis and cross-section areas of 3mm x 3mm.

| Condition, Route BC | Average Microhardness | Ultimate tensile Strength (MPa) | Yield Strength (MPa) | Elongation % |
|------------------------|--------------------------|---------------------------------------|-------------------------|--------------|
| As Received | 81.66 | 250 MPa | 110 MPa | 27 |
| Extruded | 47.55 | 159 MPa | 105 MPa | 33 |
| 1-pass | 61.16 | 209 MPa | 135 MPa | 13 |
| 2-pass | 62.36 | 232 MPa | 138 MPa | 22 |
| 4-pass | 79.36 | 265 MPa | 149 MPa | 46 |

Table 4: Mechanical properties of Al-6061 alloy before and after ECAP using Route BC

Table 5: Mechanical properties of Al-6061 alloy before and after ECAP using Route C

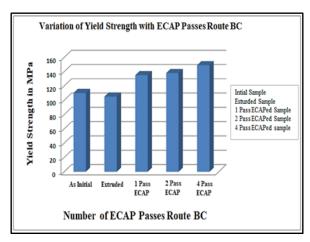
| Condition, Route C | Average Microhardness | Ultimate tensile Strength (MPa) | Yield Strength (MPa) | Elongation % |
|--------------------|--------------------------|---------------------------------------|-------------------------|--------------|
| As Received | 81.66 | 250 MPa | 110 MPa | 27 |
| Extruded | 47.55 | 159 MPa | 105 MPa | 33 |
| 1-pass | 61.16 | 209 MPa | 135 MPa | 13 |
| 2-pass | 68.34 | 182 MPa | 115 MPa | 12 |
| 4-pass | 86.06 | 238 MPa | 136 MPa | 41 |

Table 4 exhibits that route BC pressing increases ultimate tensile strength from 159 MPa to 265 MPa (an improvement of 67%) after four passes. This strength improvement is the result of grain refining caused by multi-pass ECAP. When compared to the extruded sample, the findings likewise show an increase in UTS one passes utilizing route BC. Moreover, Table 5 shows that route C pressing increases ultimate tensile strength from 159 MPa to 238 MPa (an improvement of 50%) after four passes. This strength enhancement is the result of grain refining caused by multi-pass ECAP. It was also shown that the UTS for fourth passes increased extraordinarily when compared to the extruded sample.

Work hardening by dislocation movement and grain refinement [39], caused by increased dislocation density strain-hardening, enormous stress applications, and cold working could cause changes in the strength of tensile properties of metals exposed to SPD. Significantly, UTS and yield strength increased near the end of the first pass of the cycle while the rate of elongation rapidly diminished. The increase in strength in consecutive runs of the activity was a fundamental outcome of the grain's developmental structures. (alteration of microstructure) rather than fine grain arrangements. As a result, the rates of growth in yield strength and reduction in elongation were virtually lower than in the first cycle.

3.3 ECAP Passes Have an Influence on Yield Strength (YS)

Table 5 reveals that route BC pressing increases yield strength from 105MPa to 149MPa (an improvement of 42%) after four passes. This strength improvement is the result of grain refining caused by multi-pass ECAP. When compared to the extruded sample, the findings likewise show an increase in YS one passes utilizing route BC. Moreover, Table 5 shows that route C pressing increases yield strength from 105MPa to 136MPa (an improvement of 30%) after four passes. This strength enhancement is the result of grain refining caused by multi-pass ECAP. It was also shown that the YS for fourth passes increased when compared to both of initial and extruded samples as shown in Figure 15



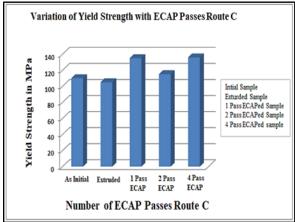
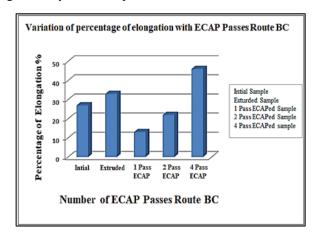


Figure 15: Comparative graphs demonstrating yield strength variations after each ECAP cycle using route BC, and route C

The relationship between YS and hardness was direct proportion, microhadness increased with increasing the yield strength. Yield strength was 135MPa and microhardness was 61.16 HV for one run. The value of hardness increased to 79.36 HV with increasing yield strength to 149 MPa after four runs for route BC. Meanwhile, the value of hardness increased to 86.06 HV with increasing YS to 136 MPa after four runs for route C, respectively.

3.4 ECAP Passes Have an Influence on Elongation (ductility)

Figure 16 depicts the relationship between the number of ECAP passes via different routes BC and C and the percentage elongation or ductility. It was revealed that the ductility was substantially reduced from 27% to 13% just after a single run, and the cause for this was that the grains were processed in the die's principal deformed region. The material was amalgamated and empty spaces were eliminated. Following the second passing, the ductility was seen to progressively drop to 22% for route BC and 12% for route C) then suddenly increase to 46% for route BC and 41% for route C after the fourth pass. Previous studies indicate that ductility remains consistent or mildly diminishes after 4 or more cycles. Prior research also validated the % change in elongation [34]. which revealed a 33% decrease in elongation after 3 cycles of ECAP having a 90° die and specimen AA6063. The findings are very similar to prior studies.



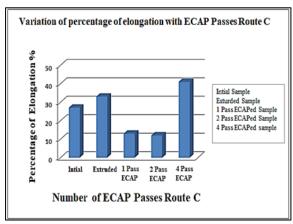


Figure 16: Graphs comparing the variance in elongation (ductility) after each run of ECAP using routes BC and C

3.5 ECAP Passes Have an Impact on Vickers Micro-Hardness

A Vickers micro-hardness analyzer was used to determine the micro-hardness of the specimens after the ECAP process. A 200-gram load was imposed for 10 seconds throughout this operation. All specimens (initial sample, extruded sample, one pass, two passes, and four pass ECAP samples) were measured at ambient temperature utilizing a Vickers hardness tester (OTTO WOLPERT-WERKE GMEH D-600) A. E92-17 [40]. The micro-hardness examination was carried out by pushing a square-based pyramidal diamond indenter with 136° face angles for around 10 seconds. Then, The measurements were repeated at several locations to determine their accuracy to at least three points.

The hardness of the examined ECAP-produced material, AA6061 aluminum alloy, was evaluated on hardness testing equipment at an ambient temperature of roughly 27°C. The findings of hardness in the processed specimen by both routes BC, and C are presented in Figures. 17and 18 It observed that the value of hardness has increased with each ECAP run. Hardness improves more after the first pass and then continues to improve, but at a slower rate. The hardness shift might be due to the difficulties of merging grains among every passing, and every passing grain consolidations increase. After 4 passes, the increased of hardness value from 47.55 HV to 86.06 HV. Comparable patterns have been found, leading to the conclusion that the hardness

of AA6061 has increased from 47.55 HV to 61.16 HV after the first pass. It simply demonstrates that there is the same tendency for the improvement of material hardness after many cycles of ECAP [41].

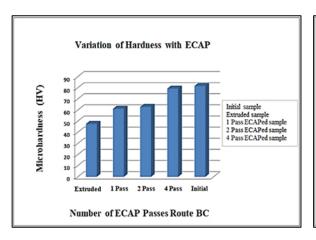
Micro-hardness (HV) is determined perpendicular to the deformation direction on all ECAPed specimens using 200 g of the test load. For better outcomes, HV is measured at three distinct sites along the axis. The average HV values of several specimens are reported in Table 6 for one, two, and four passes at ambient temperature for both Routes BC and C. It was observed that the hardness values begin to rise after the first pass. According to the HV values, the greatest hardness value is obtained from the fourth pass samples deformed at ambient temperature, i.e., 79.36 HV for route BC and 86.06 for route C respectively.

Furthermore, the hardness ratings begin to increase after the first run 61.16 HV. The maximum hardness value, i.e., 86.06 HV, is viewed from the fourth continuously passed sample deformed at ambient temperature, for route C, as shown by the HV values. Meanwhile, at ambient temperature, the specimen with four ECAPed passes showed the greatest rise in hardness value of 80 %. After one pass of ECAP, the rates of rising hardness values slow because practically all of the grains are equiaxed and elongated, leaving just a little gap between the grain boundaries.

The findings revealed that hardness acquired at normal temperature is significantly superior to hardness achieved at elevated temperatures, which is due to recrystallization beginning as the temperature rises. The greatest hardness growth in processed specimens is 86.06 HV at the fourth pass. However, the difficulty value for route BC increases after the fourth pass to 79.36 HV. The maximal hardness increase in processed samples is 79.36 HV on the fourth pass. Conversely, the difficulty value for route C is higher after the fourth pass to 86.06 HV.

| Micro-ha | rdness | As- received Sample | Hot Extruded Sample | 1 Pass ECAP Sample | ECA Route | | ECA Rout | |
|----------|--------|---------------------------|---------------------------|--------------------------|--------------|--------|-------------|--------|
| Condit | ions | Initial | Extruded | 1Pass | 2Pass | 4Pass | 2Pass | 4Pass |
| Micro- | HV1 | 83 | 45.787 | 60 | 62.553 | 80 | 67.728 | 87.782 |
| hardness | HV2 | 81 | 48.441 | 61.749 | 62.553 | 77.899 | 68.652 | 87.782 |
| (HV) | HV3 | 81 | 48.441 | 61.749 | 63.373 | 80.207 | 68.652 | 82.619 |
| Mea | n | 81.66 | 47.55 | 61.16 | 62.82 | 79.36 | 68.34 | 86.06 |

Table 6: Results of the hardness test



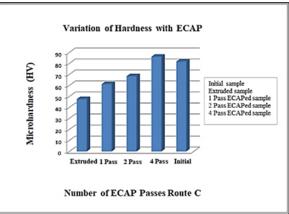


Figure 17: Graphs comparing the variation in microhardness after each run of ECAP employing Route BC and C

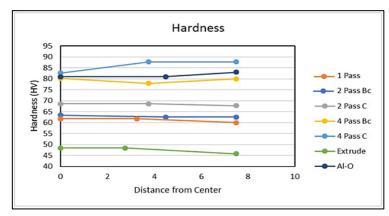


Figure 18: Graphs comparing the variation in microhardness after each run of ECAP employing Route BC and C

3.6 Effect of ECAP on Microstructure (Optical Microscopy)

Optical Microstructure to polish the specimens adequately, several grades of emery paper were utilized. Furthermore, alumina abrasives were used to polish the samples. Keller's reagent (190 mL $H_2O + 5$ mL HNO_3 (65%) + 3 mL HCl (32%) + 2 mL HF (40%)) was used as an etchant [42]. These specimens' microstructures were investigated using an (Olympus BH2-UMA) optical microscope.

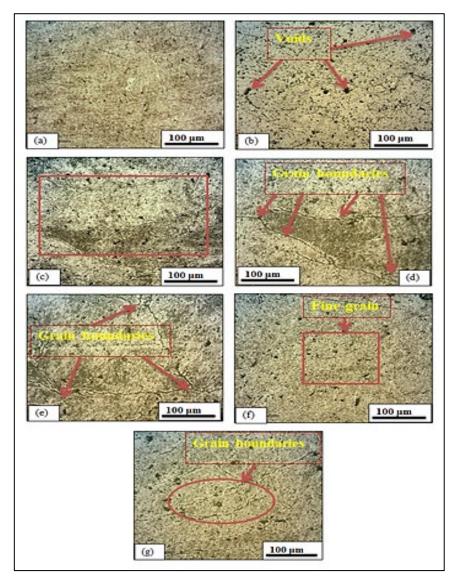


Figure 19: Optical microstructure at various magnifications (125x, 250x, 500x, 1000x), (c-g) show the microstructure of the ECAPed sample with the first, second, and fourth passes at room temperature, (a) show the microstructure of the initial sample, (b) show the microstructure of the extrusion sample at 370°C, respectively

Figure 19depicts pictures produced from an optical microscope with a magnifying zoom of (125x, 250x, 500x, and 1000x). The microstructure of specimens treated at ambient temperature with the first, second, and fourth ECAP passes is shown in Figure 19 (c–g). Depicts the microstructure of the initial specimen Figure 19 (a), and Figure19 (b) reveals the microstructure of the extruded sample. The as-received Al-6061 mostly features a coarse grain-like structure and an inaccurate orientation. With the commencement of the deformation process and an increase in the number of passes, grain elongation in the extruded direction may be detected, which eventually changes to UFGs. Sub-grain formation increases as the number of passes increases owing to the production of significant deformations and dislocations. Grain reductions, equiaxed grain formations, and grain orientations, on the other hand, are the fundamental factors of sub-grain developments. Shear bands have been seen in all samples. The density of shear bands inside the specimens is continuously increasing as a result of the ECAP procedure (Route BC). With each transit along this line, the samples rotated 90°. Shear bands are typical findings of localized strain in samples that signal the beginning of grain refinement. The interplay of the shear and slip bands promotes grain growth at sub-micron levels, which is the last mechanism for grain refinement [43].

The characteristics are largely related to the microstructure handled by the ECAP technology. As a result of low dislocation density, dislocation development during the first pass of ECAP is quicker than dislocation obliteration during multi-pass ECAP.

In this manner, the grains of the substance may be greatly refined. Because the dislocation's inner energy and density are increased by the second pass-pressing process, grain refinement proceeds [44, 45]. Meanwhile, the grain refining process is rapidly reduced after a few pressing cycles because the expansions of internal energy encourage crystalline recoveries and recrystallizations. Moreover, the grain refinements process cycle of the specimen changes depending on the pressing process. Route C dislocations, accumulation, and balance are likewise altered. An examination of the deformations and dislocations evolution reveals that nanostructured materials may be generated utilizing the ECAP method. Grain refining may be represented as a dynamical recovery and crystallization process that occurs continuously. From the standpoint of microstructure analysis, the grains refinements technique governs the dynamic equilibrium of dislocations production and annihilations [46].

Table 7 Comparisons between the previous studies with the current study in terms of Ultimate tensile strength, elongation to failure, and micro-hardness as tabulated below.

Table 7: A comparison of UTS, elongation to failure, and Micro-hardness between previous studies and the current study

| Material | Ultimate tensile strength MPa | Elongation to failure % | Micro- hardness HV | Year | Methodology | Authors | Ref. |
|------------------------|-------------------------------------|-------------------------------|--------------------------|------|--|---------------------|------|
| Al-6063 | 176 MPa | 10.95% | - | 2019 | As a received ECAP | S. Kadiyan et al. | [17] |
| Al-3030 Al-Mn-Fe-Si | 240 MPa | - | 70 HV | 2020 | Plates as a received I-ECAP | M. Ciemiorek et al. | [18] |
| Al-6063 | - | 27 % | - | 2019 | As a received ECAP | O. Abioye et al. | [34] |
| Al-5083 | 263 MPa | - | - | 2021 | As a received heat treated at 500 °C ECAP | M. Baig et al. | [47] |
| Al-6061 | 254 MPa | 11.9 % | 84 HV | 2021 | Boron carbide composite ECAP at 500°C | S. Al-Alimi et al. | [13] |
| Al-6063 | 250 MPa | - | 85 HV | 2021 | As received ECAP at 250°C | A. Gupta et al. | [15] |
| Al-8176 Al–Fe alloy | 186 MPa | 10.5 % | - | 2021 | As a received heat treated at 550 °C ECAP | G. Shuai et al. | [19] |
| Al-6061 | 265 MPa | 46% | 86 HV | 2023 | S.S.R Al chips hot extrusion followed by ECAP at RT | Current study | |

4. Conclusion

Commercial Aluminum Grade AA6061 Chips are recycled by using hot extrusion followed by SPD processing using both BC and C routes via the die 90° channel angle and 20° corner angles (best die designing characteristics). The following conclusions can be drawn based on this research:

- 1) With four runs, the tensile strength increased dramatically from 159 to 265 MPa. The stress-strain graph indicates that the tensile strength rises for each cycle.
- 2) Ductility decreased from 27% to 13% for one pass. However, ductility increased dramatically after four passes for both routes BC and C, reaching 46% and 41% after four passes, respectively.
- 3) Elongation increased from 33% for the extruded sample to 46% after a fourth run via die with route BC. While elongation increased from 33% for the extruded sample to 41% after the fourth pass through the ECAP die via Route C.
- 4) The ultimate tensile strength increased significantly as the number of passes increased. Both routes achieved 265 MPa for route BC and 238 MPa for route C after four passes through the ECAP, respectively.
- 5) Improvements in tensile strength and yield strength are reported for the ECAPed Al-6061 alloys. an increase of around (67% enhancement of UTS) for route BC while, an increase of around (50% enhancement of UTS) for route C.
- 6) The results demonstrated using Route BC exhibited that it is the most effective route for producing UFG materials with a homogenous microstructure. In addition, grain refinement can be accomplished by recycling aluminum 6061 alloy chips.
- 7) The micro-hardness increased from 47.55 HV to 79.36 HV, an increase of about 67% after four passes. However, the value of micro-hardness was increased to 62.82 HV second passes, and 61.16 HV one passes for route BC.

- 8) The micro-hardness increased from 47.55 HV to 86.06 HV, an increase of about 81% after four passes. However, the values of micro-hardness were increased to 68.34 HV second passes, and 61.16 HV one pass for route C.
- 9) Improvements in yield strength are reported for the ECAPed Al-6061 alloys. an increase of around (42% enhancement of YS) for route BC while, an increase of around (30% enhancement of YS) for route C.
- 10) The relationship between YS and hardness was direct proportion,microhadness increased with increasing the yield strength.

| Abbreviation's List | Description |
|---------------------|--|
| ECAP | Equal channel angular pressing |
| RT-ECAP | Room Temperature –Equal channel angular pressing |
| I-ECAP | Incremental - Equal channel angular pressing |
| (Φ) | The angle of intersection of two-channel Die (Die inner angle) |
| (Ψ) | Outer arc angle of Die (Die corner angle) |
| 3 | Strain |
| R | Extrusion ratio |
| BC | Type of route of ECAP process |
| C | Type of route of ECAP process |
| SPD | Severe plastic deformation |
| UTS | Ultimate tensile strength |
| Y.S | Yield strength |
| S.S.R | Solid State Recycling |
| H.V | Unit of microhardness |
| °C | Unit of temperature (Celsius) |
| MPa | Unit of strength (pressure) |
| m/min | Cutting speed |
| mm | Depth of cut |
| mm/rev | Feed |
| bar | Uint of pressure (hydrostatic compressive stress) |
| Φ | Diameter of the bar in mm |
| MoS_2 | Molybdenum disulfide |
| UTM | Universal testing machine |
| EDM | Electro Discharge Machine |
| ASTM | American Society for Testing and Materials |

Author contributions

All authors contributed equally to this work.

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Data availability statement

Not applicable.

Conflicts of interest

The authors of the current work do not have a conflict of interest.

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