Effect of ECAP die angle to the microstructure and mechanical properties of bulk nanostructured Al-6061

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Abstract. In this study, the effect of die angle on the microstructure and mechanical properties of industrial aluminium alloy AA6061 produced by severe plastic deformation (SPD) through equal channel angular pressing (ECAP) is examined. The objectives of the present investigation are to evaluate the effect of ECAP die angle on the microstructure and mechanical properties of ECAP-ed AA6061. Heat treated AA6061 were divided to three conditions which is non-ECAP, ECAP-ed by 126º channel angle and ECAP-ed by 120º channel angle. The hardness evaluation and microstructural analysis were done on the samples after ECAP. The grain size of all materials was compared by applying the technique of grain size analysis while the hardness of the materials was compared by performing Vickers hardness calculation. From the hardness test, it found that 120º channel angle gives out an increment of hardness by 43.64% while for 126º channel angle the increment hardness is 40.14% compared to non-ECAP AA6061. Microstructural analysis reveals both ECAP-ed samples have elongated and refined grain size with smaller precipitate particulate compared to non-ECAP sample however no significant difference between the angles were observed. High strain induced during ECAP process increase dislocation in AA6061 and breaking the precipitate thus causing high hardness due to grain refinement. Varying the ECAP die angle may lower the pressure used during pressing without compromising the benefit of ECAP process in producing materials with improved mechanical properties.

1. Introduction

Nowadays, industries are gearing towards producing the lightweight materials with excellent mechanical properties. Equal Channel Angular Pressing (ECAP) is one of the severe plastic deformation (SPD) processing methods that can be used to produced materials with improved properties such as high strength and good ductility due to grain refinement [1]. Applying ECAP will produce a deformation with no net shape change[2]. ECAP increases the material strength by accumulating a very large plastic strain into the work-piece without changing its cross-sectional area[3]. According to the Hall–Petch relationship it is well known that refining the grain size increases the yield strength of a material. Ultrafine grains (UFG) can
result in increased strength at lower temperatures, while making the material more formable at elevated
temperatures[4]. Researchers revealed superior mechanical properties such as high strength due to the
upgrading ductility, high yield stress and improved toughness after producing ultrafine grained materials[5].
When the grain size is decreasing, it decreases the amount of possible pile up at the boundary, but increasing
the applied stress that needed to move a dislocation across a grain boundary. Therefore, the higher the
applied stress for dislocation movement the higher the yield strength is[6].

The ECAP, also known as equal-channel angular extrusion (ECAE), was first published by Segal and
his co-workers in the 1970s and 1980s at an institute in Minsk in the former Soviet Union[7]. At that time,
Segal was focusing on developing a metal forming process to introduce high strains into metal billets by
simple shear. However, even the main objective was achieved, there were only small scientific community
attended to receive the process. After a lot of reports and overviews on the potential of ECAP to produce
ultrafine-grained and submicrometer metals with new and unique properties, the situation was changing in
the 1990s[8]. As ECAP is anticipated as single way processing, a lot of attention has been given to the
ECAP die parameter such as corner angle, back pressure, strain hardening rate and friction. In this paper,
we are focusing on the effect of die angle on the microstructural and mechanical properties of AA6061. Die
angle plays an important role in determining the strain that will be imposed on the materials. The strain
accumulated in the sample after $N$ passes was proposed by Segal [9] assuming the corner angle ($\Psi$) to be $0^\circ$
is given below

$$\varepsilon_N = \frac{2N}{\sqrt{2}} \cot \left( \frac{\Psi}{2} \right)$$  \hspace{1cm} (1)

Subsequently, Iwahashi et al. [10] modified the above equation to include ‘$\Psi$’, which is given as

$$\varepsilon_N = \frac{N}{\sqrt{2}} \left[ 2 \cot \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi \csc \left( \frac{\Phi}{2} + \frac{\Psi}{2} \right) \right]$$ \hspace{1cm} (2)

This equation shows that the lower the angles, the higher the strain per pass will be imposed on the
samples. However, as strain magnitude getting higher, the distribution of the strain across the samples may
be compromised. The pressing force required also may increase with lower angle[11].

Aluminum alloys, like AA 6061 can be considered as the most widely used materials today in industry
which span the entire range of industries such as in transportation, piping and aircraft[12]. That is why the
material was chosen in this project to be investigated the mechanical properties and microstructure. It easy
to be shaped, decent variety of shape, erosion protection, low cost, and accessibility make it a decent material
for some applications[13]. However, various study has shown that nanostructured Al alloys produced via
SPD showing increased static and cyclic strength, larger strength to weight ratio along with improved
service and multifunctional properties making it more desirable as structural and functional materials in
various engineering sectors[14].

ECAP is found as an attractive processing method in producing AA6061 with superior mechanical
properties. However, high pressure required during ECAP process may imposed higher manufacturing cost.
The high cost of manufacturing is depending on the electricity which produces power for the pressing
machine to press. The higher the power needed for the process, the higher the electricity needed. Therefore,
the suitable angle need to be determined in order to reduce the pressure needed. Therefore, this paper is
focusing on the effect of ECAP die angle on the microstructure and mechanical properties AA 6061.

2. Methodology
2.1. Material
Commercially available AA6061 rod with 10 mm diameter and 15 cm, solution treated using heat treatment T6 (parameter as shown in Table 1) was used for ECAP. The AA6061 samples composition were checked using spectrometer and presented in Table 2.

Table 1. Solution heat treatment requirement and those required in for AA 6061-T6.

| Process                          | AA6061            |
|----------------------------------|-------------------|
| Solution Treat Temperature       | 526 °C +/- 5.6 °C  |
| Solution Treat Time              | 3 hours +/- 15 minutes |
| Max Quench Delay                 | 15 seconds        |
| Quenchant Material               | Water or glycol   |
| Quenchant Temperature            | 27 °C             |
| Artificial Age Temperature       | 204°C +/- 2.4°C    |
| Artificial Age Time              | Naturally aged     |

Table 2. Chemical composition of this AA6061 in percentages.

| Element | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Ti  | Zn  | Al  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Weight percent (%) | 0.65 | 0.2 | 0.42 | 0.014 | 0.93 | 0.09 | 0.15 | 0.009 | 97.5 |

2.2. ECAP
The ECAP die was built in-house with two channels, equal in cross-section, intersecting at an angle near the center of the die. To fit within these channels, the specimens were machined and use the plunger to press it through the die. Two magnitudes of channel angles were tested in this project, which are 120° and 126°. The higher the channel angle, the lower the pressure needed to successfully complete the process[15]. The samples were pressed by one pass of ECAP at 80 bars. To reduce the friction between the rod sand the die walls, a lubricant with molybdenum disulfide (MoS2) was used. A hydraulic press machine was used to carry out ECAP process at room temperature. Figure 1 show the schematic illustration of ECAP with 120° of angle.
2.3. Hardness test
The ECAP-processed specimens’ hardness was investigated using Wilson Vickers hardness machine. The specimens were subjected to load of 100 g for 10 seconds. The testing taken from 7 point horizontally and 3 point vertically to check the hardness distribution across the samples. All values were divided to 3 groups which were top, middle and bottom. For every line, the average was calculated to be representing as groups’ value. All groups for all samples were tested on the samples’ surfaces from left to right, from top bottom and horizontally. The value can be taken randomly, but to avoid the parallax error, the testing was standardized for all samples. Each point in each group located 1mm from other point horizontally. Figure 2 shows the hardness testing distribution points used in this study.

![Figure 1: Schematic illustration of ECAP.](image1)

![Figure 2: Hardness testing distribution points.](image2)

2.4. Microstructure test
For microstructure test, a metallurgical microscope was used. After the image of the microstructure appears, the shape and size of the metal crystals, metal damage after deformation and heat treatment process, and compositional differences could be observed. The microstructure of AA6061 is a small structure of the material, as it could only observed by a microscope above 5x magnification. Hence, the microstructure or called as nanostructure was small, the microscope was used to check the non-ECAP and ECAP-ed samples. 3 condition of samples’ grain size could be measured from this test. Only one of each condition was tested. ImageJ software was used to further the analysis on grain size and particles area.
3. Result and discussion

Figure 3 shows the condition of samples before and after the ECAP process. The dimension of both samples is according to the dimension of the ECAP dies which are less than 130mm in length and less than 10mm in diameter as stated in Figure 1. After the hardness test run on the 3 samples, the results shown in a graph as shown in Figure 4.

![Figure 3](image1.jpg)

Figure 3. AA 6061 rods (a) before ECAP process and (b) after ECAP process.

![Figure 4](image2.jpg)

Figure 4. Vickers hardness profile across sample between Angle of 120°, 126° and non-ECAP.

From the graph in Figure 4, it found that 120° gives out an increment of hardness by 43.64% while for 126° the increment hardness is 40.14% compared to non-ECAP AA6061. It is proved that both of angles show the improvement of hardness. All the ECAP-ed conditions will give the improvement to the materials especially to the hardness properties of the materials. Standard deviation for non-ECAP condition is 3.21,
for 126° ECAP-ed condition is 5.05 and for 120° ECAP-ed condition is 5.43. The highest value is at point -3mm from 120° ECAP-ed condition which is 133.43 HV. The lowest is from non-ECAP is 80.23 also at point -3mm. The closest values of 120° ECAP-ed condition and 126° ECAP-ed condition are 122.07 HV and 124.17 HV respectively at point 2mm from the centre. Meanwhile, the farthest values of 120° ECAP-ed condition and 126° ECAP-ed condition are at point -3mm which are 133.43 HV and 121.33 HV respectively. The percentage of 120° ECAP-ed condition is higher than 126° ECAP-ed condition.

Optical micrographs of AA6061 before and after ECAP were shown in Figure 5.

**Figure 5.** Grain sizes microstructure analysis for (a) non-ECAP (b) 120° ECAP-ed condition and (c) 126° ECAP-ed condition.

Based on Figure 5, non-ECAP condition, typical equiaxed coarse grain with some scattered precipitates was observed. The grain sizes of the non-ECAP samples are markedly larger at about ~20μm. The precipitates were observed to be randomly distributed on the grain. However, for both ECAP-ed samples, it was observed that the grains have elongated and some sub-grain boundary was observed. The high strain induced during ECAP processing also leads to the formation of slip bands. Their grain sizes also decreases significantly; for 126° is at about ~5μm while the grain size for 120° is at about ~2.5μm. Higher distribution of precipitates that segregated along the grain boundaries was also observed. The increase in precipitates may be due to the fragmentation on these precipitates during ECAP processing. The fragmented precipitates were then sheared into several pieces, which inhibits growth phenomena[16].

The high stress distribution was observed more at material pressed at 120° compare to the 126° based on the hardness value and microstructural evidence. This is due to the higher residual stress that was produced from the material that pressed at lower die channel angle as stated by Iwahashi[10]. Lower die angle also caused higher magnitude of effective strain that is imposed on the samples. However, as die angle decreases, higher pressing force is required. But as observed in Figure 4, hardness distribution of 120° sample was less homogenous compared to 126, even though its average hardness is higher. Increasing die angle leads to decreases in strain magnitude but the strain induced is more homogeneous throughout the samples [17]. This explains the homogeneity of the hardness value across the samples as shown in Figure 4.

**4. Conclusion**

The effect of die angle on the mechanical and microstructural properties of AA6061 has been successfully realized in this study.

- Both ECAP-ed samples has shown improvement in their hardness with 120° channel angle had the highest magnitude about 43.64% increments compared to the non-ECAP-ed sample while the ECAP-ed sample for 126° channel angle had 40.14% increments compare to the non-ECAP-ed sample. This is due to the grain refinement that is shown in the microstructural evaluation.
- Lower die angle requires more forces during processing, which leads to higher strain magnitude. However, by increasing die angle, more homogeneous strain distribution can be achieved at lower pressing force.

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