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THE EFFECT OF ECAP PROCESSING ON HARDNESS, SURFACE MORPHOLOGY, AND CORROSION RESISTANCE OF 6061 ALLOYS

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Abstrak

Paduan aluminum Al-Mg-Si (6xxx) telah banyak digunakan sebagai material struktural untuk bangunan dan kendaraan bermotor karena memiliki kekuatan mekanik dan ketahan korosi yang baik. Proses ECAP (*equal channel angular pressing*) merupakan metode yang paling menjanjikan dengan mengaplikasikan deformasi plastis yang memproduksi material utuh dengan butir yang halus tanpa porositas sisa. Penelitian ini mempelajari tentang pengaruh jumlah pass pada proses ECAP terhadap kekerasan, struktur mikro, dan perilaku korosi pada paduan aluminum 6061. Material paduan terlebih dahulu dilakukan proses aniling di dalam tungku dengan lingkungan gas argon pada T = 530 °C selama 4 jam kemudian dicelupkan pada nitrogen cair selama 5 menit sebelum proses ECAP. Proses ECAP dilakukan melalui rute Bc dengan cetakan yang memiliki lubang dalam bersudut 120° dan variasi pass dari 1, 2, 3, dan 4. Kekerasan optimal yang diperoleh yaitu 107,58 HB pada paduan Al 6061 dengan 3 pass ECAP. Peningkatan jumlah pass pada ECAP menyebabkan adanya pengurangan ukuran butir dari ukuran 10 µm pada paduan hasil aniling menjadi ukuran 2,5 µm pada paduan dengan 4 pass. Ketahanan korosi meningkat seiring dengan peningkatan jumlah ECAP pass.

Kata Kunci: Paduan Al-Mg-Si, *ECAP*, kriogenik, struktur mikro, ketahanan korosi

Abstract

Al-Mg-Si alloys (6xxx) have been widely used as structural materials in buildings and vehicles because of their excellent strength and corrosion resistance. ECAP (equal channel angular pressing) is the most promising method to apply SPD (severe plastic deformation), producing ultra-fine grain in the bulk material without residual porosity. This study presents some experiments results on the effect of ECAP number of passes variation on the hardness, microstructure, and corrosion behavior of Al 6061 alloys. The alloy was annealed in the furnace with an argon gas environment at 530°C for 4 hours and then immersed in liquid nitrogen for 5 minutes before the ECAP process. The ECAP process was carried out via the Bc route, with dies with an internal channel angle of 120° and pass variations of 1, 2, 3, and 4. The optimum hardness was 107.58 HB in Al 6061 alloy with three passes of ECAP. The increasing ECAP number of passes leads to a significant grain size reduction from the 0-way pass; the grain size was around 10 µm, while for a 4-way pass, the grain size was around 2.5 µm. The corrosion resistance of Al 6061 alloys increased with the increasing number of passes in the ECAP process.

***Keywords:** Al-Mg-Si alloys, ECAP, cryogenic, hardness, microstructure, corrosion resistance*

1. INTRODUCTION

Aluminum alloys are widely used due to increasing demand for improving construction and automotive performance using lightweight materials [1]. The 6xxx series Al-Mg-Si alloys are heat treatable and maintain high mechanical properties. Al-Mg-Si alloys (6xxx) have been widely used as structural materials in buildings and vehicles, such as vessels, engine blocks, and pistons, because of their excellent strength and corrosion resistance [2]. The improved fine-grain microstructure, which can increase mechanical and physical properties, has become an exciting field in recent research [3]. The combination of nanostructured and sub-micrometer materials will produce high performances because of their small

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grain size [4]. The deformation process to refining the microstructure of Al 6061 (6061 aluminum alloys) has effectively improved physical-mechanical properties such as ductility, strength, toughness, strain, elongation, and corrosion resistance [5].

Equal channel angular press is the most promising method to apply severe plastic deformation (SPD), producing ultra-fine grain in the bulk material without residual porosity [6]. The large bulk sizes of ECAP processing materials become an advantage to obtain nano and ultra-fine structured mechanical parts and offer the opportunity to scale up the process to an industrial level [7]. The ECAP process utilized a sample that pressed through a die with two intersecting channels equal in the cross-section. The ECAP process uses shear force to deform the materials through the intersection of the angular channels [8]. The other advantage of the ECAP process is that the sample holds a similar cross-sectional area after pressing so that it is feasible to repeat the pressing a few times [9]. The improved material properties are the result of the ECAP process, which is dependent on the geometry of the channels and the number of passes. [10]. Furthermore, some parameters such as die geometry and channel angle of ECAP can influence the induced equivalent plastic strain, which improves mechanical properties in the materials [11].

Aluminum alloys are well known for their high corrosion resistance because of their oxide film in the atmospheric environment [12]. Uniform, localized and pitting corrosions are the most common corrosion founded in aluminum alloys with a halide ion environment. Recently, the effect of grain refinement on the hardness and corrosion properties has attracted much attention. Some researchers revealed that decreasing grain size using ECAP could increase the corrosion resistance of Al-Mg and pure Mg alloys [13]. It also reported that the decreasing hardness properties resulted from the increasing number of ECAP passes [14]. In this paper, the effect of ECAP number of the pass in the cryogenic environment has been investigated on hardness, microstructure, and corrosion properties of Al 6061.

2. MATERIALS AND METHODS

The element percentage in the Al 6061 was determined using OES (optical emission spectroscopy) on a commercial billet of 6061 aluminum alloys. For the ECAP process, the Al 6061 billets were machined into cylindrical specimens with dimensions of 65 mm length and 13.55 diameters. Figure 1 showed the flow diagram of the ECAP (equal channel angular pressing) process and the characterization after the ECAP process. Before the ECAP process, the samples were annealed in an argon gas environment for 4 hours at 530 °C to homogenize the microstructure and remove internal stress [15]. The samples were immersed in liquid nitrogen for 5 minutes before the ECAP process to obtain the cryogenic temperature in the samples. The immersion of samples in liquid nitrogen was done before the addition of the ECAP pass. The immersion of sample in liquid nitrogen was done in every each ECAP pass addition.

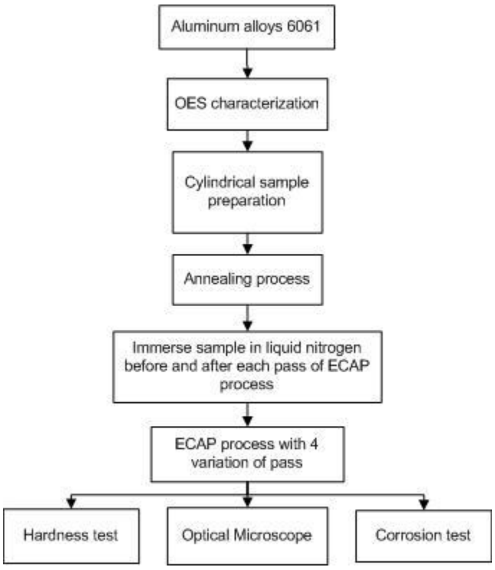


Figure 1. The flow diagram of ECAP process and characterization

After the nitrogen immersion of the sample, the ECAP process was done using the Bc route using 120° of internal channel angle with pass variations of 1, 2, 3, and 4. The Bc deformation was done by rotating the sample orientation of 90° before each new ECAP pass. After the ECAP process was done, the hardness properties were characterized using a hardness test, and the microstructure of the ECAP-processed sample was also evaluated using an optical microscope. The corrosion properties such as open circuit potential and Tafel polarization were characterized using CMS (corrosion measurement system). The morphology of corrosion products was also investigated using an optical microscope.

The hardness test was held using Hardness Brinnel in 5 varied points and the values obtained are averaged to obtain the hardness number. The hardness test was done on annealed samples and ECAP-processed samples. The ECAP-processed pieces were cut along their longitudinal cross-section, ground, polished, and etched using

Poulton’s reagent. After that, the samples were characterized using an optical microscope to analyze the microstructure which has been formed.

The corrosion testing of Al 6061 samples was held using a 3-electrode flat corrosion cell in a 3.5% NaCl with Gamry Reference potentiostat. The platinum electrode was utilized as the counter electrode, the saturated calomel (SCE) was used as the reference electrode, and the Al 6061 sample was used as the working electrode. The samples were wet ground using 240, 600, 800, 1000, 1200 SiC abrasive paper followed by degreasing with distilled water and acetone and then blow-dried with compressed air. The OCP characterization was done by immersed the sample in the NaCl solution in 1 hour. Tafel polarization was then investigated against the OCP at a scan rate of 0.6 m/s.

3. RESULTS AND DISCUSSIONS

3. 1. Chemical Composition Characterization

Optical emission spectroscopy was done to characterize the percentage of elements contained in the samples. Table 1 presents the chemical composition of 6061 aluminum alloys which are characterized using OES (optical emission spectroscopy).

Tabel 1. Chemical composition of comercial 6061 aluminum alloys (wt.%)

Si	**Fe**	**Cu**	**Mn**	**Mg**	**Zn**	**Ti**
0.643	0.497	0.227	0.106	0.871	0.039	0.01
Cr	**Ni**	**Pb**	**Sn**	**V**	**Cd**	**Al**
0.085	0,01	0.002	0.002	0.06	0.01	Bal

The dominants of alloying elements are Mg with 0.871 wt.% value and Si with 0.643 wt.%. Other alloying elements such as Fe, Cu, and Mn are detected in this alloy.

3. 2. Hardness Test

The hardness test was utilized to investigate the effect of number pass on hardness properties of the Al 6061 alloy. Figure 2 presents the hardness of the ECAP (equal channel angular pressing) processed of the alloy under different variations of passes.

The ECAP process increases the hardness number of Al 6061 alloy, and it is also noted that further hardness number improvement is proportional to the number of passes increased. It can be concluded that the improvement in hardness number is caused by grain refinement concerning the standard of the Hall-Petch relationship predicts that the yield strength would increase as grain size decreases [16].

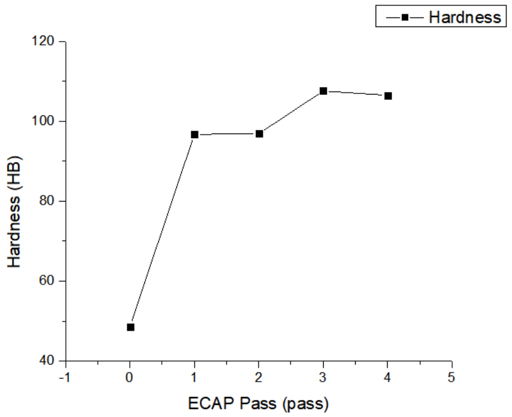


Figure 2. Hardness characteristics of the Al 6061 alloy under different processing conditions

Besides that, the strengthening mechanism such as fragmentation and homogeneous distribution of precipitates and the high number of dislocation density also could improve hardness number. Figure 2 depicts the hardness characteristics of the examined alloys under various processing settings. The hardness of the annealed Al 6061 alloy was 48.6 HB and increased to 107.58 HB in 3 passes number of ECAP. The ECAP processed alloy improves grain refinement and the number of dislocation networks at grain boundaries and within grains significantly.

Furthermore, the presence of smaller strengthening phase particles within the matrix of the ECAP processed alloy creates barriers to unrestricted dislocation migration inside the matrix. Slip or dislocation movement occurs across these grain boundaries, and it can also aid in the cross slip of screw dislocations obstructed by precipitates or dislocation locks during plastic deformation [17]. Dislocation changes direction as it passes from one grain to the next because the grain borders of polycrystalline grains have different crystallographic orientations. Dislocation entanglement occurs because of such variations in dislocation direction, preventing dislocation mobility. The increases hardness of materials by strain hardening due to mutual restriction of dislocation glide on the intersecting system [18].

Figure 3 reveals that optical microscope results showing the grain structure after ECAP processing. The shear strain affects their dislocation density and realignment to produce new cells and grains. The effect of cryogenic treatment caused a higher density of dislocation in the grain and cell interiors. The increasing ECAP number of passes leads to a significant grain size reduction to the sub-micrometer scale of 1-2μm. It can be seen in the sample with four passes in Figure 2€ had a smaller grain size than the sample with a smaller number of passes in the ECAP process. The microstructure of the ECAP processed sample consists of elongated and rounded grain shapes surrounded by dislocation walls. The effect of ECAP pass number in cryogenic treatment could increase dislocation density inside cells and grain. In other words, the sample with four passes of ECAP has more dislocation density than the sample with a smaller number of passes and annealed sample. The dislocation movements are also affected by low-temperature pressing. The Al 6061 with four passes of ECAP had much free dislocation of grain because of dislocation deposition in cell and grain boundaries. Slip bands are formed as the result of the high strain caused during ECAP processing. Their grain sizes also fall dramatically; for a 0-way pass, the grain size is around 10 µm, while for four-way passes, the grain size is approximately 2.5 µm. There was also a high distribution of precipitates that segregated along grain boundaries. The fragmentation of these precipitates during ECAP processing could explain the rise in precipitates. Furthermore, the shattered precipitates were sheared into multiple pieces, which prevented growth [19]. At the interface between the particles and the matrix, there is no sign of deformation. According to the literature, significant dislocation density and lattice misorientation can develop during particle deformation, resulting in inhomogeneous deformation and grain size discrepancies in the particle's immediate surroundings [20].

3. 3. Open Circuit Potential Measurement

Two electrochemical techniques, such as OCP and Tafel polarization, are used to investigate the corrosion behavior of Al 6061 after ECAP is processed. Figure 4 represents the open circuit potential that studied the passive film’s characteristic on the surface of Al 6061. All ECAP-deformed Al 6061 alloys have a higher free corrosion potential than annealed Al 6061. This phenomenon explained that the increasing ECAP number of passes could improve the oxide film on the surface, decreasing electrochemical reactions. It can be seen from Figure 4 that there is some fluctuation in the potential time curve in all samples, which reflects dissolution and repassivation events, which are connected to the activity of different impurities contained in the samples.

Open circuit potential is an important variable to understand the corrosion resistance of materials. The higher value of free corrosion potential indicated the difficulty of material to corrode [21]. According to Table 2, the free corrosion potential value of Al 6061 with four passes has the highest value of all samples. This result indicates that Al 6061 with four passes of ECAP was the noblest of all samples.

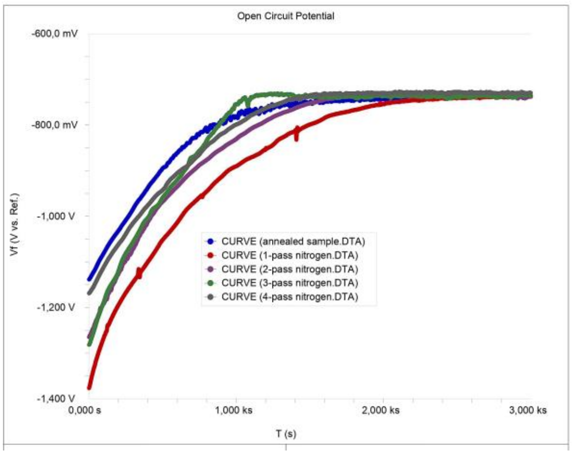


Figure 4. Potential time curve of annealed and ECAP processed Al 6061 with the variation of passes in 3.5% NaCl solution

Table 2. Free corrosion potential for 1 hour of Al 6061 alloy

Sample	Vmin (mV)	Vmax (V)	Eoc (mV vs SCE)
As-annealed	-733.4	-1140	-733.4
1-pass nitrogen	-733.5	-1380	-733.5
2-passes nitrogen	-728.9	-1269	-728.9
3-passes nitrogen	-727.7	-1284	-727.7
4-passes nitrogen	-725.4	1170	-725.4

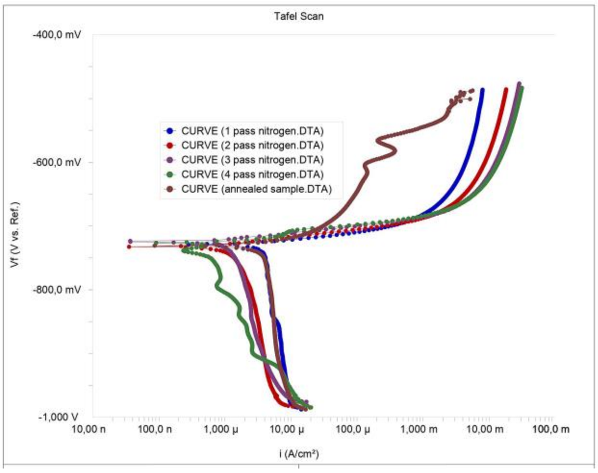


Figure 5. Tafel polarization curve of as-annealed and ECAP processed Al 6061 in 3.5% NaCl solution

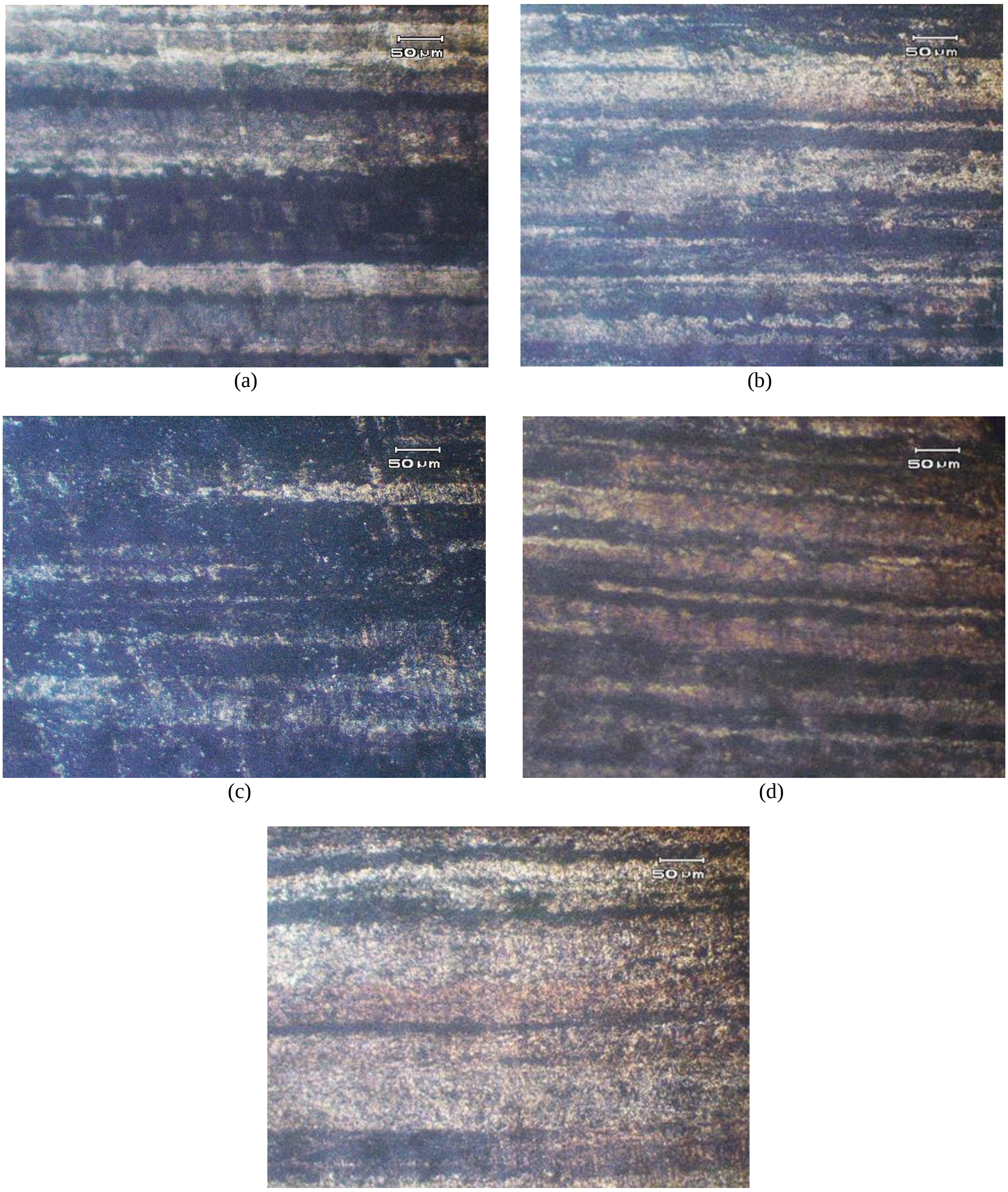
Table 3. Electrochemical parameters obtained from Tafel polarization of annealed and ECAP processed Al 6061 alloys

Sample	Ecorr (mV)	Icorr (µA/cm^2)	Corr rate (mmpy)
As-annealed	-730.9	4.05	133.3e-3
1-pass nitrogen	-729.4	3.60	118.6e-3
2-passes nitrogen	-732.6	1.52	50.17e-3
3-passes nitrogen	-725.6	1.38	45.43e-3
4-passes nitrogen	-726.4	0.005	16.04e-3

3. 4. Tafel Polarization

Tafel polarization curves of the annealed and ECAP processed alloy are shown in Figure 5. The alloy was immersed in 3.5% NaCl solution for 1 hour to obtain their stable OCP values.

It can be observed from Fig. 5 that the cathodic current density slope shifted left with the increased ECAP number of passes. Table 3 also shows that the corrosion potential (Vcorr) of the annealed alloy decreased from -730.9 mV to -726.4 mV after four passes. The corrosion rate also decreased with the increased ECAP number of passes from 133.3e-3 mmpy of annealed alloy to 16.04e-3 mmpy after four passes. The effect of grain refinement is caused by the increase in the ECAP number of passes, which affects a more uniform corrosion attack and lower corrosion rate [22]. The Tafel slope in the anodic curve of Al 6061 decreased with the higher number of passes in the ECAP process. This phenomenon explains that the ECAP number of passes has a noticeable effect on the anodic reaction.

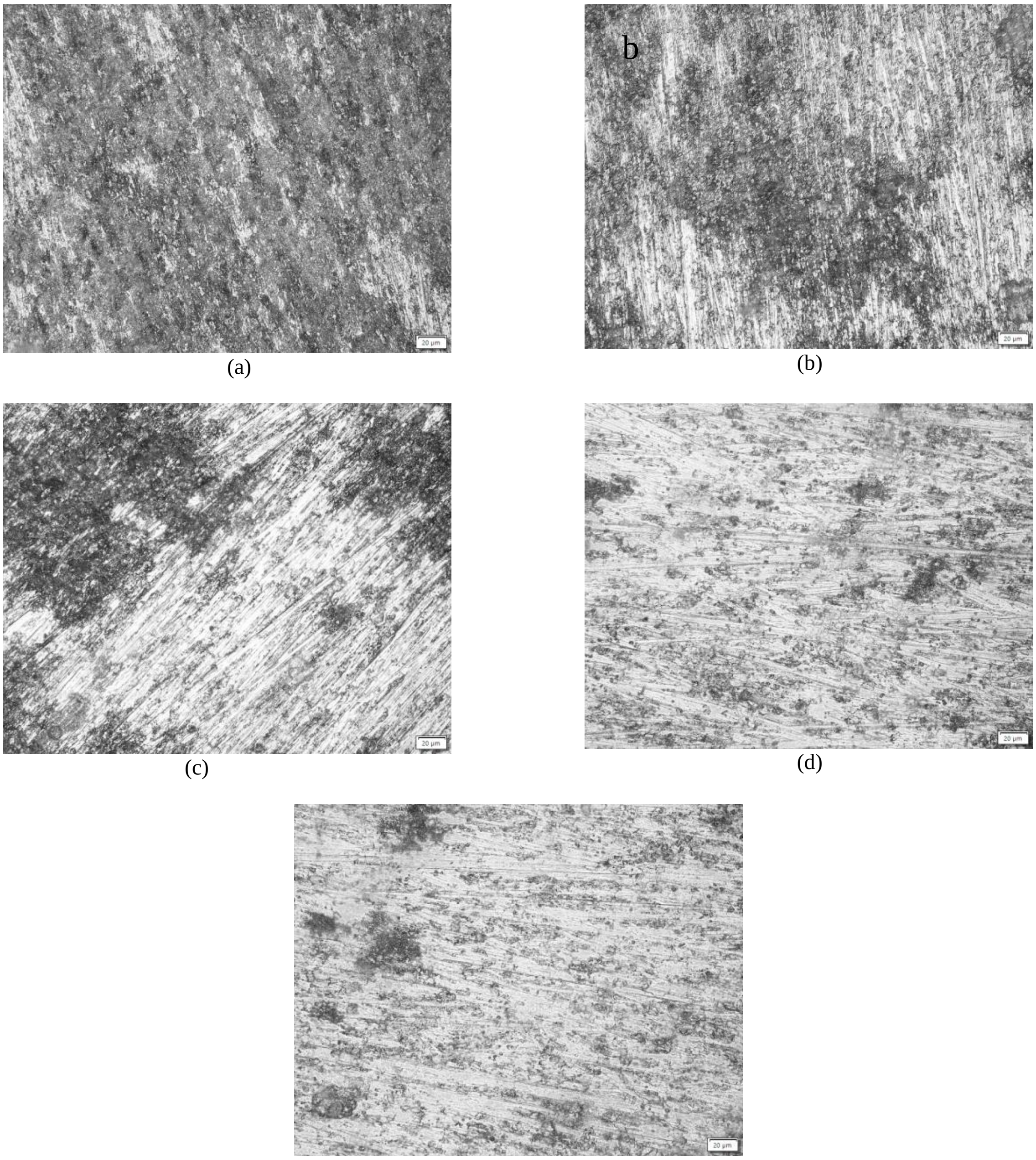


(a) (b)

(c) (d)

(e)

Figure 3. The optical microscope results of Al 6061 alloys with the variation of (a). As-annealed, (b). 1-pass ECAP, (c) 2-pass ECAP, (d) 3-pass ECAP and (e). 4-pass ECAP



(a) (b) (c) (d) (e)

Figure 4. The optical microscope results of Al 6061 alloys with the variation of (a). As-annealed, (b). 1-pass ECAP, (c) 2-pass ECAP, (d) 3-pass ECAP and (e). 4-pass ECAP after corrosion test in 3.5% NaCl

Figure 4 shows the optical microscope of corroded alloy in 3.5% NaCl solution. It can be observed that the high amount of corrosion attacks on the surface of the as-annealed alloy after the Tafel polarization test in 3.5% NaCl solution. The ECAP-processed of Al 6061 alloys exhibit lower localized corrosion than the as-annealed Al 6061. The ECAP processed samples of Al 6061 alloys after the Tafel polarization test illustrates less localized corrosion on the surface of alloy with three and four passes in Figs. 4(d) and 4(e). This phenomenon is caused by the improvement of grain refinement and secondary phases distribution, which lower the corrosion rate of Al 6061 alloy. Furthermore, an alloy with a higher Ecorr value indicates more passivated oxide film, which caused a lower corrosion rate because of the slow dissolution rate of fine grain structure [23]. This fact also supported the corrosion morphology of the alloy in Figs. 4(a), 4(b), and 4(c) are more corroded than the others because of their coarse grain.

4. CONCLUSIONS

In the current study, the effect of ECAP processing on the hardness, microstructure, and corrosion behavior of Al 6061 was successfully done. The hardness of the sample before aging was 48.6 HB and increased to 107.58 HB in three passes number of ECAP. The effect number of passes was in line with grain refinement, which affects the hardness. The Al 6061 annealed and ECAP processed microstructure revealed the elongated and rounded shapes of grain bounded by dislocation walls. Besides that, the increased ECAP number of passes led to a significant grain size reduction from 10 µm in the Al 6061 annealed to 2.5 µm after four passes of the ECAP process. The corrosion behavior of the Al 6061 deformed by ECAP had nobler free corrosion potential than the annealed Al 6061. The corrosion rate also decreased with the increased ECAP number of passes from 133.3e-3 mmpy of Al 6061 annealed to 16.04e-3 mmpy after four passes.

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