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CHARACTERIZATION OF AN EQUAL CHANNEL ANGULAR PRESSED Al-Zn-In ALLOY

CHARAKTERYTYKA STOPU Al-Zn-In OTRZYMANEGO METODĄ PRZECISKANIA W KANAŁE KĄTOWYM

Equal channel angular pressing (ECAP) is a technique that creates a high accumulated strain in metals and results in ultrafine-grained structure. In this study, Al-5Zn-0.02In was processed by ECAP at a room temperature using route Bc through an ECAP die (press angle of $\Phi = 100^\circ$ and $\Psi = 20^\circ$). The samples were subjected to ECAP with 1, 2, 3 and 4 passes. The processed specimens were characterized using electron backscatter diffraction (EBSD). The results confirmed the grain refinement of the alloy after ECAP to an average grain size less than $5 \mu\text{m}$ after 4-pass ECAP. The microhardness test shows that the hardness increased with the number of passes. The hardness of the cross-sectional area of the sample was similar to that tested along the pressing direction.

Keywords: Aluminum alloys, Equal channel angular pressing, Severe plastic deformation, Electron backscatter diffraction

Metoda przeciskania w kanale kątowym (ang. Equal channel angular pressing; ECAP) prowadzi do powstania bardzo wysokich naprężeń, w wyniku czego otrzymuje się ziarna o bardzo drobnej strukturze. W niniejszej pracy, stop Al-5Zn-0,02In wytwarzano metodą ECAP w temperaturze pokojowej, wykorzystując ścieżkę Bc przez matrycę ECAP (kąty krzywizny: $\Phi = 100^\circ$ i $\Psi = 20^\circ$). Próbki poddano ECAP z 1, 2, 3 i 4 przejściami. Otrzymane próbki badano metodą dyfrakcji elektronów wstecznie rozproszonych (ang. electron back scatter diffraction; EBSD). W stopie otrzymanym metodą ECAP z 4 przejściami, średnia wielkość ziaren wynosiła mniej niż $5 \mu\text{m}$. Badania mikrotwardości wykazały, że twardość zwiększała się wraz z liczbą przejść. Twardość zmierzona w poprzek próbki była porównywalna do tej mierzonej wzdłuż osi nacisku.

1. Introduction

It has been long known by materials scientists that uniformly ultrafine-grained structure leads to higher strength of metals and alloys [1-2]. Recently, metals and alloys with ultrafine-grained (UFG) structure have becoming active research areas. Many processing techniques have been proposed to produce alloys containing UFG structure such as ball milling [3], rapid solidification [4], spray forming [5], and severe plastic deformation [6]. Among those techniques, severe plastic deformation (SPD) has gained increasingly interests as it provides many advantages e.g. high potential to produce UFG alloys in bulk and less complicated process compared to others. The main principle of SPD process is that the alloy is subjected to a high shear strain to create distortion of crystals so that high angle boundaries are formed and eventually lead to many new fine grains [7, 8]. Equal Channel Angular Pressing (ECAP) is one of the most popular SPD techniques. The process involves deformation that the sample is pressed against metallic die with a specific pressing angle and an existing channel with the same dimension as the entrance. ECAP has been applied to various engineering alloys especially alu-

minum alloys. Most of previous studies show that ECAP led to an improvement of mechanical and electrical properties of aluminum alloys [9, 10]. However, there are arguments on the effects of severe plastic deformation on corrosion behaviours of aluminum alloys. For example, Brunner et al [11] reported the effects of ECAP on corrosion behaviours of AA2024 aluminum alloys. It shows that the ultrafine-grained structures played an important roles on the intergranular corrosion by changing susceptibility and mode of intergranular corrosion. Son et al [12] indicated that better pitting corrosion resistance of an anodized aluminum-copper alloy arrived from a refinement of Al-Cu-Si-Fe-Mn precipitates. This paper investigated the effects of equal channel angular pressing on an Al-Zn-In sacrificial anode in refinement of microstructure, texture, and strain distribution.

2. Experimental procedures

The Al-5Zn-0.02In (in wt%) alloy was used in this study. The alloy was melted and cast into a graphite mould, machined into a cylinder with a diameter of 20 mm and 100 mm long,

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and then solutionized at 530°C for 1 hour before pressing in an ECAP die with a pressing angle of $\Phi = 100^\circ$ and $\Psi = 20^\circ$ at a room temperature. The samples were pressed using Route Bc with 1, 2, 3, and 4 passes. The equivalent accumulated strain was calculated using eq. (1) [13]

$$\epsilon_N = \frac{N}{\sqrt{3}} \left[2 \cot \left(\frac{\Phi}{2} + \frac{\Psi}{2} \right) + \Psi_{csc} \left(\frac{\Phi}{2} + \frac{\Psi}{2} \right) \right] \quad (1)$$

Where ϵ_N is equivalent strain, N is number of ECAP passes, and Φ, Ψ are press angles. The as-cast and processed samples were cross-sectioned and polished using silicon-carbide papers and final polished using alumina suspension. The samples were then polished by electropolishing in a solution (83.2% ethanol, 7.8% perchloric acid, and 9% deionized water) at 50 V for 5 s. The microstructure of as-cast and ECAPed samples was characterized using Electron Backscatter Diffraction (EBSD) operating in a Hitachi S-3400N Type II at 20 kV. Microhardness of the ECAPed samples was measured using Vickers indentation with 100 g applied load and 15 s dwell time. The hardness was measured along the cross section and pressing direction of the samples at 2 mm intervals.

3. Results and Discussion

Fig. 1 shows the surface finish of a sample before and after ECAP. The samples before ECAP show machining marks. For the ECAPed samples, there was small flash along the middle of the sample that was due to a small gap from assembly of the ECAP die. The sample had a good and shiny surface finish with some porosity.

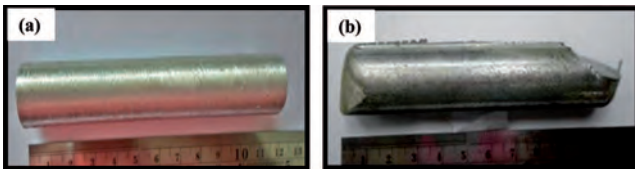


Fig. 1. Photos of (a) sample before ECAP and (b) sample after 1-pass ECAP

TABLE 1 shows the equivalent strain and the average grain size of the as-cast and ECAPed samples calculated by eq. (1). It shows that the grain size dropped dramatically with the number of passes except for 3 and 4 passes where no significant reduction of grain size observed. As seen in Fig 2 (a), there was a small fraction of high angle boundaries due to comparatively large grain size of as-cast structure. For 1-pass ECAPed sample, the IPF map (2 (b)) shows that the microstructure contained large and slightly elongated grains with contour of colors indicated small degree of distortion of crystals [14]. The transition of low angle boundaries to higher angle boundaries can be seen when subjected to 2 passes ECAP. Fig. 2 (c) shows a high proportion of low (upto 5°) misorientation angles indicated as white lines and some amount of $5-10^\circ$ misorientation angles indicated as grey lines. This was corresponding to misorientation distribution charts as illustrated in the Fig. 3 (c). The IPF maps of 3 and 4-pass ECAPed samples in the Fig 2 (d) and (e) show similar results where a large fraction of high angle boundaries and therefore finer

grains were observed. A small area of contour color indicated stored distortion and yet to form new grains. However, Fig. 2 (e) shows that within the newly formed grains, they appeared to have contour color inferring that a higher strain and more ECAP passes; more than 4 passes are required to create a finer grain structure.

TABLE 1
Equivalent strain and average grain size of different samples

	Equivalent strain	Average grain size (μm)
As-Cast	N/A	143.5
1-pass	0.71	89.2
2-pass	1.43	25.9
3-pass	2.14	4.8
4-pass	2.85	4.6

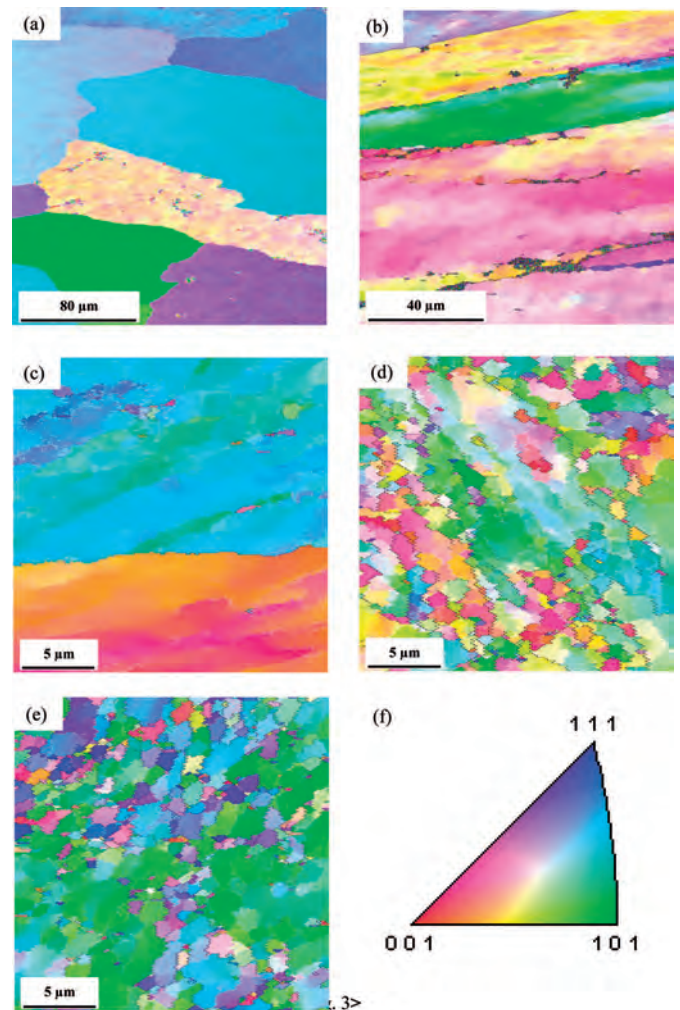


Fig. 2. A representative EBSD orientation map of (a) as-cast (b) 1-pass ECAP (c) 2-pass ECAP (d) 3-pass ECAP and (e) 4-pass ECAP with a misorientation angle of $\geq 15^\circ$ shown as a black line

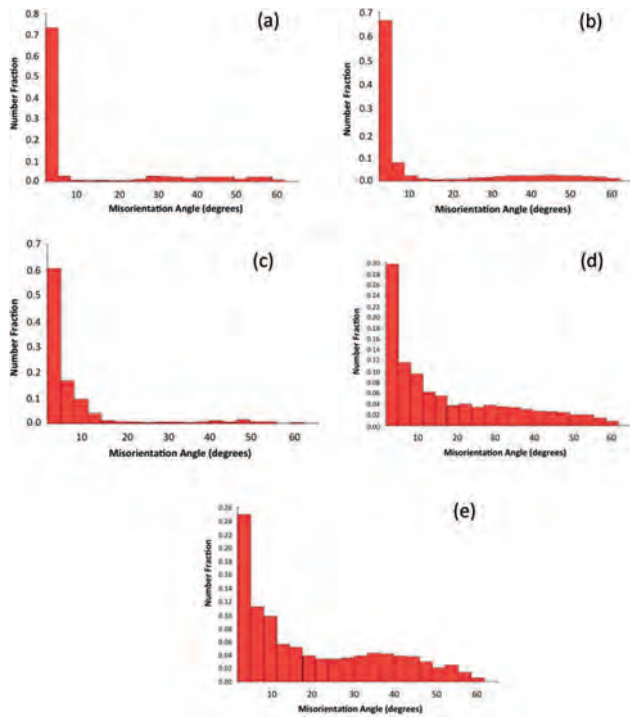


Fig. 3. Misorientation distribution of α -Al grains

Fig. 4 (a) to (e) show (001) and (111) pole figures of as-cast and ECAPed samples. The as-cast grain structure in Fig. 4 (a) contained comparatively random textures. The stronger textures derived from an increase in number of ECAP passes.

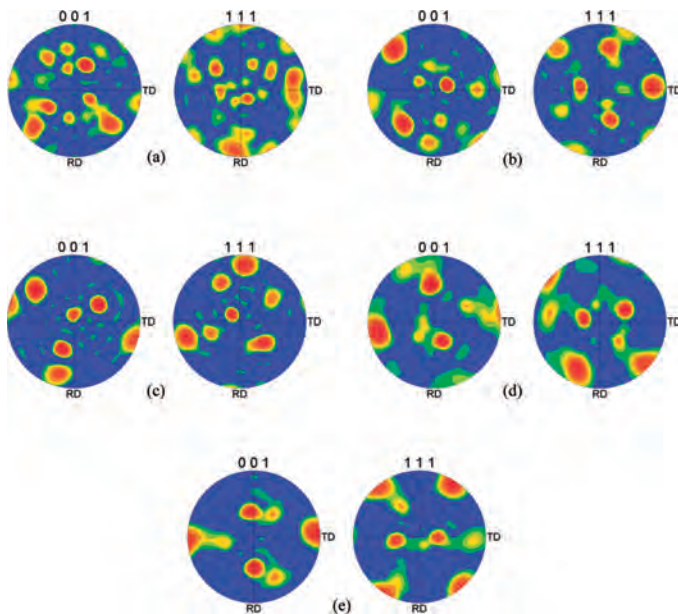


Fig. 4. (001) and (111) pole figures of (a) as-cast, (b) 1-pass ECAP, (c) 2-pass ECAP, (d) 3-pass ECAP, and (e) 4-pass ECAP

The microhardness of the as-solutionized and ECAP samples is shown in the TABLE 2 while Fig. 5 and 6 shows microhardness measured perpendicular to and along the pressing direction. The results show slightly inhomogeneous hardness along the ECAPed samples which can be normally seen in ECAPed aluminum alloys. It was also shown that the hardness sharply increased after 1-pass ECAP with small increment of

hardness after 2-4 pass ECAP similar to that reported by Prell et al [15]. This may infer that the residual strain inside the α -Al matrix had strong contribution to the strengthening of the sample.

TABLE 2
Hardness (cross-sectional and along pressing direction)

	Average cross-sectional hardness (Hv)	Average hardness along pressing direction (Hv)
As-cast and solutionized	29.52	29.52
1-pass	57.47	55.85
2-pass	63.08	62.68
3-pass	63.41	65.56
4-pass	63.54	64.68

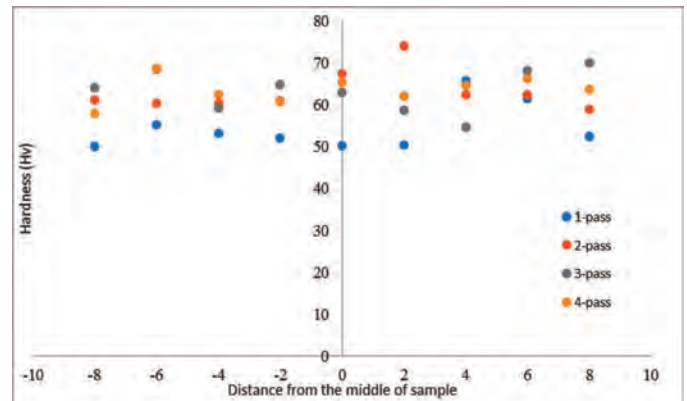


Fig. 5. Hardness measurement of cross-sectional area of the 1-4 pass ECAPed samples

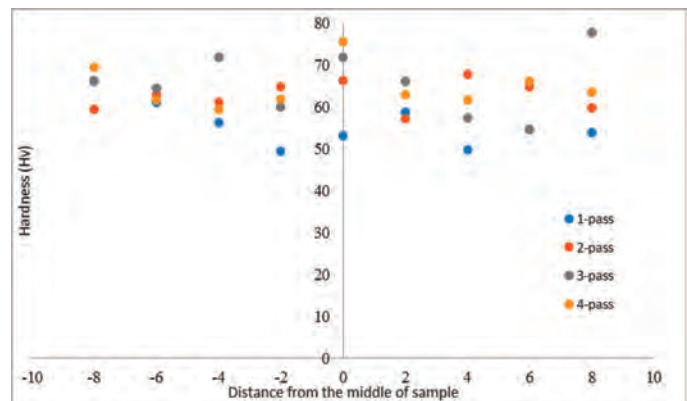


Fig. 6. Hardness measurement of the 1-4 pass ECAPed samples along the pressing direction

4. Conclusions

This research investigated the equal channel angular pressing of an Al-Zn-In alloy. It shows that good surface finish can be produced after several ECAP passes. The average grain size of the alloy reduced dramatically during the ECAP due to an increase of number of passes from ~ 144 to $\sim 5 \mu\text{m}$. The EBSD IPF map revealed the formation of low angle and

high angle boundaries. Increasing number of passes resulted in reducing fraction of low angle misorientation and increasing high angle misorientation. The pole figures maps clearly show that preferred misorientation/textures were developed during ECAP. The hardness of ECAPed samples increased sharply after 1-pass ECAP but slowly increased for the next passes. This referred that more passes can be applied to increase the hardness.

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