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Equal channel angular pressing of pure aluminium—an analysis

M SARAVANAN, R M PILLAI*, B C PAI, M BRAHMAKUMAR and K R RAVI
Materials and Minerals Division, Regional Research Laboratory, Thiruvananthapuram 695 019, India

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Abstract. Equal channel angular pressing (ECAP) is a novel technique for producing ultra fine grain structures in submicron level by introducing a large amount of shear strain into the materials without changing the billet shape or dimensions. This process is well suited for aluminium alloys and is capable of producing ultra fine grain structures with grain sizes falling between 200 and 500 nm. The present study attempts to apply ECAP technique to 99·5% pure aluminium and characterize the resulting aluminium by optical metallography, atomic force microscopy (AFM) and hardness measurement. ECAP of 99·5% pure aluminium produces ultrafine grain structure of about 620 nm after 8 passes. Despite an increase in the hardness from 23 to 47 BHN up to 6 passes, it decreases slightly for seventh and eighth passes. The results are compared with the already existing results available on pure aluminium. Analysis of the results of this investigation with those available in the literature has revealed that the number of passes essential to achieve a homogeneous microstructure in pure Al increases, while the ultimate equilibrium grain size obtained becomes finer with decreasing purity.

Keywords. Aluminium; severe plastic deformation; ECAP; AFM.

1. Introduction

Light metals with high strength are the immediate and future requirements for aerospace as well as automotive industries. Grain refinement is one of the techniques, which provides finer grains and hence ultra high strength and ductility combination demanded for ambient and cryogenic temperature applications. On the other hand, severe plastic deformation (SPD) is an effective tool for producing bulk ultrafine grained (submicron or nanostructure) metals. Equal channel angular pressing is one of the SPD techniques developed (Segal 1995, 1999, 2004; Saravanan *et al* 2005) for producing ultra fine grain structures in submicron level by introducing a large amount of shear strain into the materials without changing the billet shape or dimensions. Extensive studies (Iwahashi *et al* 1998a; Nakashima *et al* 1998; Horita *et al* 2001; Sun *et al* 2002, 2004; Xu and Langdon 2003) carried out on aluminium have shown that ECAP is well suited for producing ultra fine grain structured aluminium. The present study attempts to apply ECAP technique to 99·5% pure aluminium and characterize the resulting aluminium by optical microscopy, atomic force microscopy (AFM) and hardness measurement as well as compare with the published data on pure aluminium.

*Author for correspondence (rmpillai@rediffmail.com)

2. Experimental

The dimensions of ECAP die for 12 mm square workpieces are designed with ϕ (intersect angle) = 90° and ψ (the angle subtended by the arc of curvature at the point of intersection) = 20°. Figures 1 and 2 show the schematic of ECAP and photograph of the fabricated ECAP die fitted to the hydraulic press of 25 tonnes capacity, respectively.

The material used in this work was commercially available 99·5% pure aluminium with a composition given in table 1. The experiments were carried out by using samples cut from an ingot and machined to a size of $12 \times 12 \times 55$ mm. The die angles, $\phi = 90°$ and $\psi = 20°$, give the maximum strain and homogeneous microstructure in the material (Wu and Baker 1997; Nakashima *et al* 1998; Saravanan *et al* 2005). The experiments were carried out by using the route, B_C (the rotation of 90° in the same direction between passes), up to 8 passes at room temperature, which is the preferred route for achieving equiaxed grains (Iwahashi *et al* 1998a; Sun *et al* 2004; Saravanan *et al* 2005).

Small samples were prepared for optical microscopic and atomic force microscopic studies. Figure 3 shows location of the specimens for optical microscopy, AFM and hardness evaluation. Samples with 10×10 mm size and thickness, 2 mm, were cut from centre of each pressed specimen. These samples were electropolished using a solution consisting of 37% orthophosphoric acid, 38% ethanol and 25% water at room temperature. Samples with 10×10 mm and 5 mm thickness were cut from centre of each pressed samples for hardness measurements.

Table 1. Chemical composition of the 99·5% pure aluminium.

Element wt.%	Si	Fe	Ti	V	Cu	Mn	Al	Others
	0·07	0·34	0·001	0·008	0·001	0·004	99·557	0·002

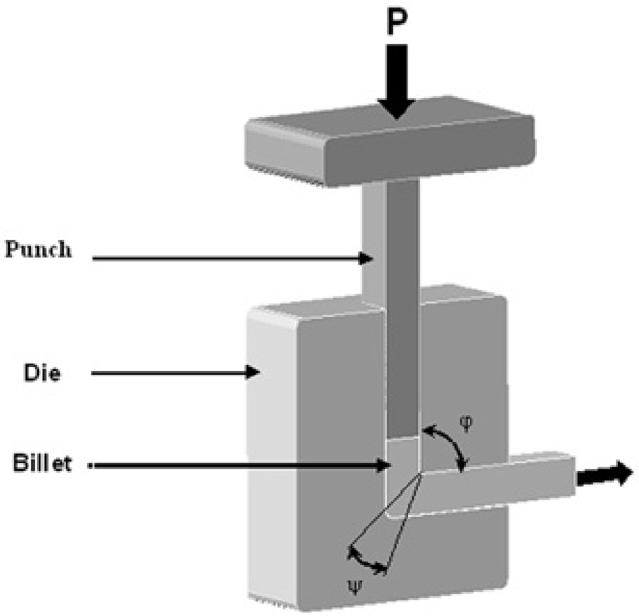

Figure 1. Schematic of ECAP.

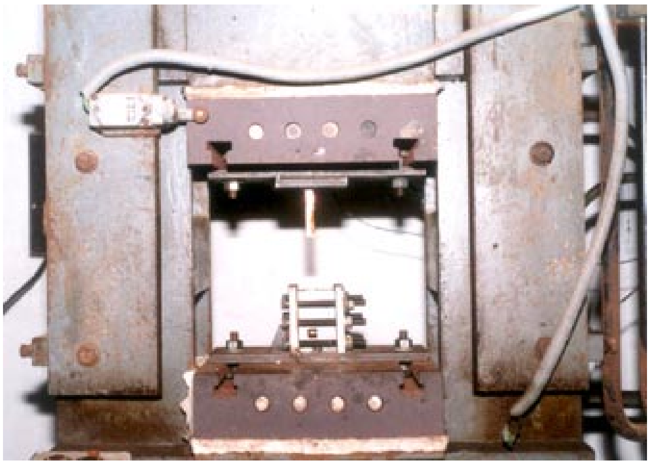

Figure 2. ECAP die assembly fitted to the hydraulic press.

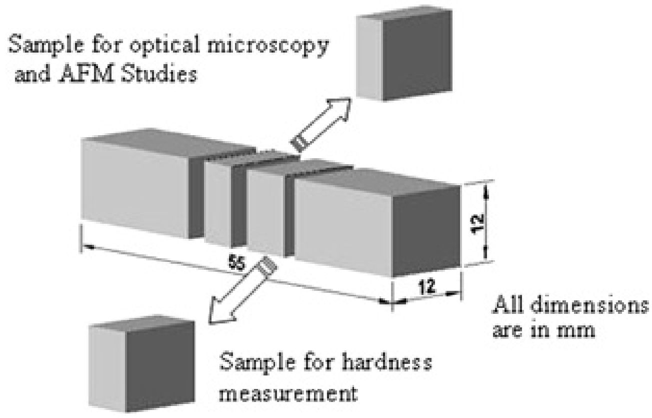

Figure 3. Location of specimens for AFM, optical and hardness evaluation.

3. Results

3.1 *Microstructure*

Figure 4 shows the optical microstructures of ECA pressed 99·5% pure aluminium up to 4 passes. Initially, the unpressed material (figure 4a) composed of large grains with an average grain size of 150 μm. After one ECAP pass, original grains are uniformly elongated (figure 4b). After the second pass, shear bands disappear and more fine and equiaxed grains are created by increased levels of strain and rotation of the sample. Figure 4e shows the photomicrograph of the sample after the fourth pass clearly revealing further refinement of grains. As the number of passes increases further up to 8, the dislocation density increases and grains become extremely fine. Hence, the grains and grain boundaries are not visible under optical microscope and the material is characterized by atomic force microscopy technique with contact mode operation. Figure 5 shows AFM image of pressed aluminium after 8 passes. From the AFM image, the grain size was found to be ~620 nm (figure 6).

3.2 *Hardness*

Hardness values of the unpressed and ECA pressed aluminium are shown in figure 7. ECAP leads to a substantial increase in hardness after the first pass itself. After two passes, the hardness improvement is comparatively low up to 6 passes. After 6 passes, further increase in number of passes decreases the hardness. Hardness increased with increasing number of passes due to the strain hardening mechanism in the initial stage. Further, ECAP passes promoted the formation of equiaxed grains accompanied with gradual decrease in dislocation density as already reported by several researchers (Ferrase *et al* 1997; Iwahashi *et al* 1997; Fan *et al* 2005; Han *et al* 2005; Xu *et al* 2005).

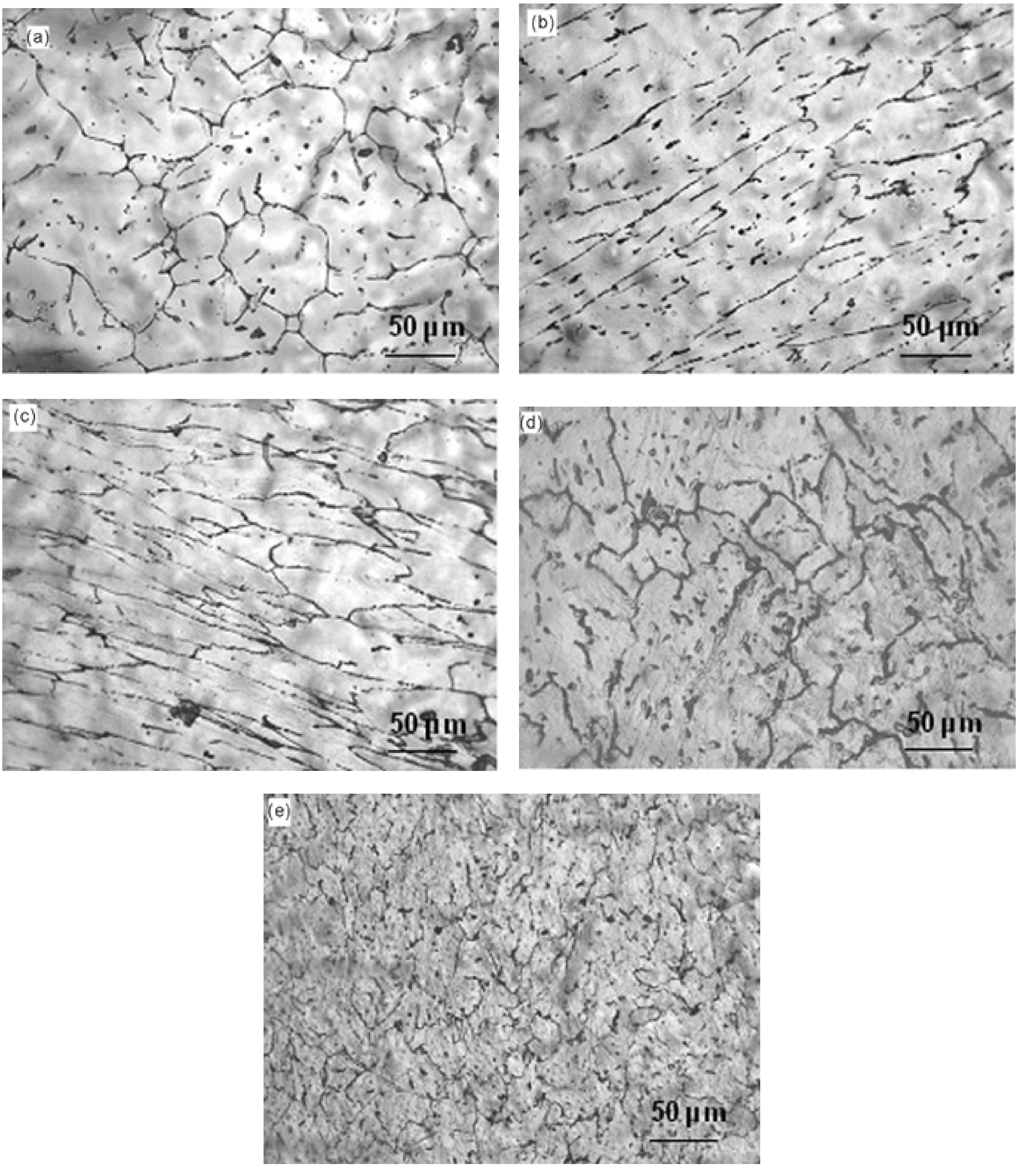

Figure 4. Optical microstructures of 99·5% pure aluminium: (**a**) unpressed, (**b**) after first pass, (**c**) after second pass, (**d**) after third pass and (**e**) after fourth pass.

4. Discussion

Table 2 compares results of the present investigation with various other studies conducted on pure aluminium. In the present study, the equiaxed grain size obtained is 620 nm after the eighth pass. This result is only 10–15% above those reported by Sun *et al* (2002, 2004) and well below the results of others (Iwahashi *et al* 1997, 1998a;

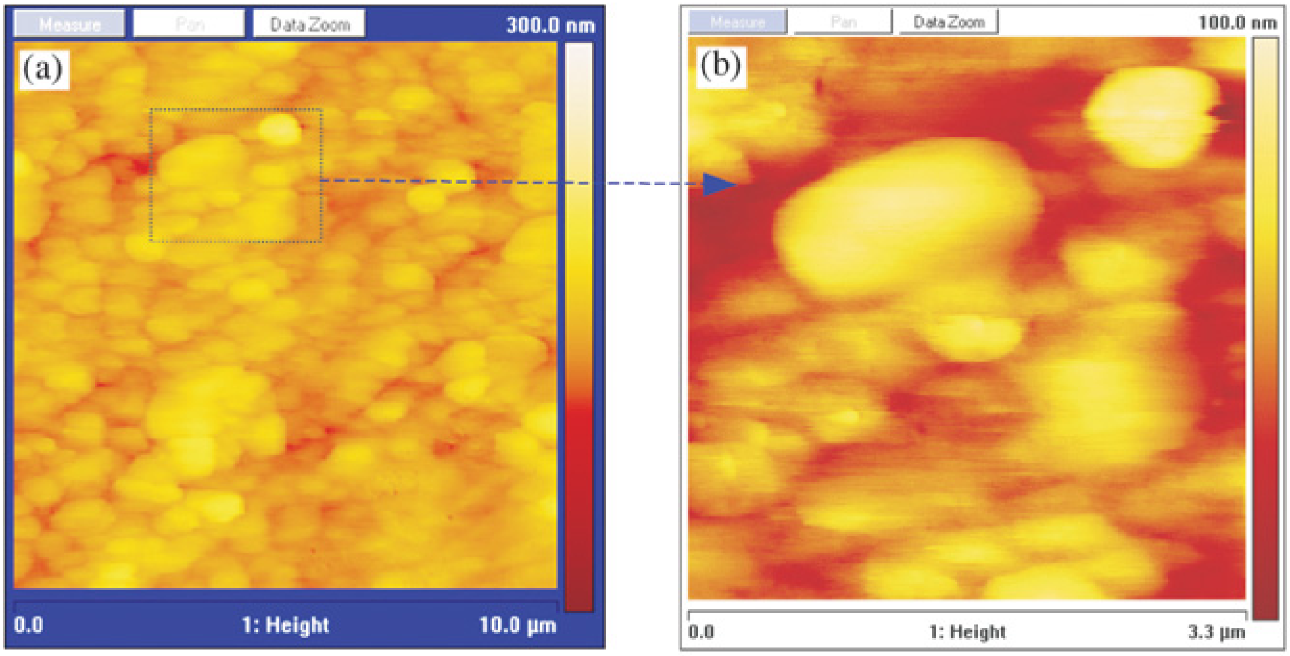

Figure 5. AFM microstructures of 99·5% pure aluminium after 8 passes.

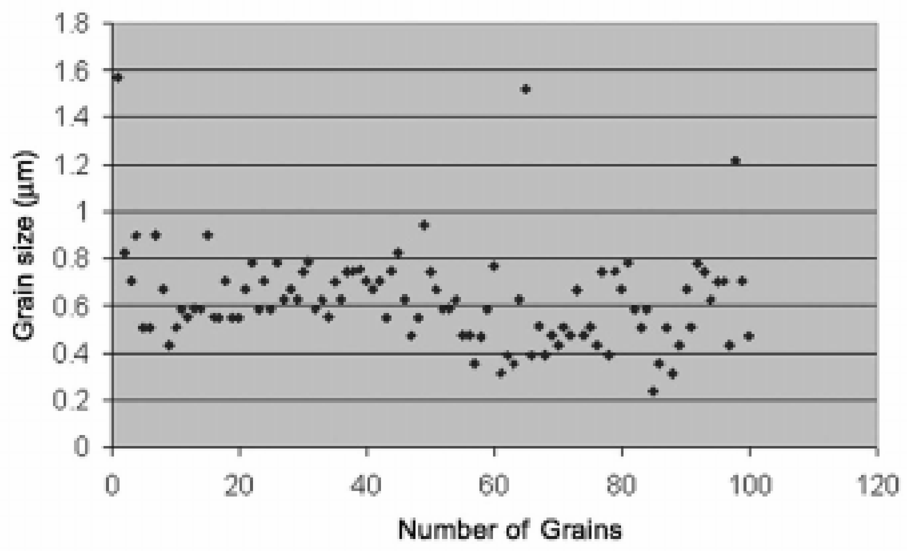

Figure 6. Grain size distributions of 99·5% pure aluminium after 8 passes.

Nakashima *et al* 2000; Horita *et al* 2001; Xu *et al* 2005). Iwahashi *et al* (1997) and Nakashima *et al* (2000) studied ECAP of pure aluminium using route C. Since grain refinement efficiency of route C is comparatively less, there exists a large difference between the results of the above and those of the present investigation and Sun *et al* (2002, 2004). The present investigation and that of Sun *et al* have used 99·5% pure aluminium having 0·34 and 0·2 wt% iron contents, respectively. Since the solubility of iron in solid state aluminium is very low (0·04 wt%), the iron appears as intermetallic second phase causing heterogeneities in plastic flow and more grain refinement during ECA processing. Similar observation has been reported (Apps *et al* 2003) in ECA processed (route A, $\phi = 120°$) Al–1·3%Fe–0·09%Si alloy containing $Al_{13}Fe_4$ second phase particles with a finer grain size (~0·5 µm) compared to that of Al–0·13%Mg alloy (~0·64 µm). Horita *et al* (2001) studied ECAP of 1100 aluminium

Table 2. Comparison of hardness and grain size obtained in the present investigation with those obtained by other researchers in ECA pressed pure aluminium.

Material	Tooling parameters		No. of passes	Hardness	Grain size	
	Die angle	Route			Before ECAP (μm)	After ECAP (nm)
Pure Al (99·99%) (Iwahashi *et al* 1997)	$\phi = 90°$; $\psi = 20°$	C	8	38 Hv	1000	1000
Pure Al (99·99%) (Nakashima *et al* 2000)	$\phi = 90°$; $\psi = 30°$	C	5	38 Hv	–	–
Pure Al (99·99%) (Xu *et al* 2005)	$\phi = 90°$; $\psi = 20°$	B_C	4	42 Hv	1000	1300
Pure Al (99·99%) (Xu and Langdon 2003)	$\phi = 90°$; $\psi = 20°$	B_C	4	43·2 Hv	–	–
Pure Al (99·99%) (Iwahashi *et al* 1998a)	$\phi = 90°$; $\psi = 20°$	B_C	4	–	1000	1300
AA1050 (99·5%) (Sun *et al* 2004)	$\phi = 90°$; $\psi = 20°$	B_C	8	–	330	450
AA1050 (99·5%) (Sun *et al* 2002)	$\phi = 90°$; $\psi = 20°$	B_C	8	62 Hv	330	550
Pure Al (99·5%) (Present study)	$\phi = 90°$; $\psi = 20°$	B_C	8	44·1 BHN	150	620
1100 Al (99%) (Horita *et al* 2001)	$\phi = 90°$	B_C	6	60 Hv	30	1000

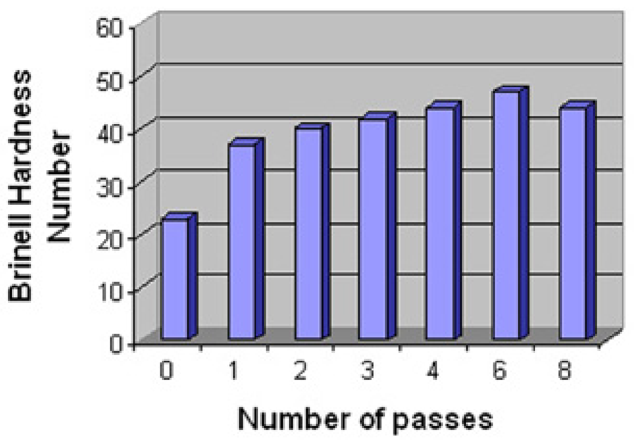

Figure 7. Variation of hardness with the number of passes in 99·5% pure aluminium.

(99% pure) using route B_C and six number of passes only. Iwahashi *et al* (1998a) and Xu *et al* (2005) studied ECAP of pure aluminium (99·99% pure) using route B_C and four passes only. On the other hand, studies of Sun *et al* (2002, 2004) resemble the present study (Die angle, $\phi = 90°$; $\psi = 20°$, route B_C and 8 passes). Hence, the equiaxed grain size obtained is almost the same in the present results and that reported by Sun *et al* (2002, 2004). The marginal difference existing may be due to the use of different microscopic techniques for characterization. Even, marginal difference has been found in the results reported by Sun *et al* (2002, 2004) for the same purity Al.

It is to be noted that the number of passes required to achieve a homogeneous microstructure in pure Al subjected to ECAP (B_C route with $\phi = 90°$ and $\psi = 20°$) increases with decreasing purity or increasing impurity content. It is 4 and 8 passes for 99·99 (Iwahashi *et al* 1998a; Xu *et al* 2005) and 99·5% (Sun *et al* 2002, 2004) purity Al. Similar observation has been reported (Iwahashi *et al* 1998b) with 1 and 3% Mg addition to 99·99% pure Al processed by ECAP (B_C route) and the number of passes required to achieve a homogeneous microstructure is 4, 6 and 8 for 99·99% pure Al, Al–1%Mg and Al–3%Mg, respectively. Further, the ultimate equiaxed equilibrium grain achieved is finer with Mg addition (~1·3, 0·45 and 0·27 μm) for 99·99% pure Al, Al–1%Mg, Al–3%Mg, respectively. It is again interesting to observe similar behaviour in pure Al with increasing impurities: 1300 and 450–620 nm for 99·99 and 99·5% purity Al, respectively.

5. Conclusions

ECAP of 99·5% pure aluminium produces ultrafine grain structure of about 620 nm after 8 passes. Despite the increase in the hardness from 23 to 47 BHN up to 6 passes, it decreases slightly for seventh and eighth passes. Analysis of the results of this investigation with those available in the literature has revealed that the number of passes essential to achieve a homogeneous microstructure in pure Al increases, while the ultimate equilibrium grain size obtained becomes finer with decreasing purity.

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