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Effect of processing route on grain refinement and mechanical properties in equal channel angular pressing of aluminium alloy AA7068

Influence of different processing routes on microstructure of aluminum alloy AA7068 during equal channel angular pressing (ECAP) up to four passes is investigated. Equal-channel angular pressing is one of the best severe plastic deformation methods to achieve ultra-fine grain structure by subjecting metal to an intense plastic strain through a simple shear. A significant amount of grain refinement as occurred by passing aluminium alloy samples through ECAP split die at 200°C temperature from first pass up to four passes with two different processing routes such as route A and route Bc. The experimental results revealed that out of two different processing routes, route Bc where the samples are rotated by an angle of 90° in the same direction after each pass, is able to achieve rapid microstructure evolution with a better grain refinement and an equiaxed microstructure as compared to that of route A. Mechanical properties such as hardness and tensile strengths of the ECAP processed samples via route A and route Bc are also revealed that route Bc is the optimum process parameter which induces better mechanical properties into the metal.

Keywords: ECAP, microstructure, AA7068, microstructure, mechanical properties.

1.0 Introduction

Severe plastic deformation is the technique for the betterment of materials mechanical and tribological properties such as hardness, tensile strength, fatigue strength, wear resistance, corrosion resistance etc. There are different SPD techniques to enhance the materials properties to make them suitable for wide variety of applications. Accumulative roll-bonding, equal channel angular pressing, high pressure torsion, cyclic closed-die forging and severe torsion straining are some of the SPD techniques (Segal, 2006; Valiev, 1997). Equal channel angular pressing (ECAP) is one among the attractive grain refinement SPD methods due to its simple and cost-effective procedure introduced during 1997. ECAP results processed materials with best mechanical properties by inducing intense plastic strain into the material through simple shear which leads to achieve grain refinement to produce ultrafine grain sizes (Segal, 1981; Segal, 1995). ECAP is an effective process to reduce the macro size grains of the material into micro or up to nano size grains by pressing the material repetitively through specially designed ECAP die with plunger on a press tool with different route patterns at room temperature or elevated temperature. The ECAP die is designed with two channels of equal cross sections intersecting at an angle Φ called channel angle and corner angle ψ which is provided at the inner intersection point which also contributes to influence the strain rate as compared to the channel angle due to widening of the localized zone. The accumulated equivalent strain value was calculated using the die-channel and relief angles in Eq. (1) for N number of passes through ECAP die (Iwahashi et al., 1997).

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left(2\cot\left(\frac{\emptyset}{2}+\frac{\varphi}{2}\right)+\varphi\, cosc\left(\frac{\emptyset}{2}+\frac{\varphi}{2}\right)\right) \quad \ldots (1)$$

Where N is the number of passes, ψ is the corner angle and φ is the channel angle. The channels may be designed either circular or square to accommodate pressing of circular or square sample pieces through the die. The material of the die should be strong enough to press all stronger engineering material, hence the common material for the die is hot die steel and it should be heat treated to make it stronger after machining according to the standard die design. The parameters which influence the performance of ECAP process are channel angle, corner angle, friction, number of passes, strain hardening, pressing temperature, pressing route and pressing velocity, out of these, pressing route is one of the major criteria which impacts more on the ECAP process. In order to achieve homogeneous structure in the materials pressed through the ECAP die the samples

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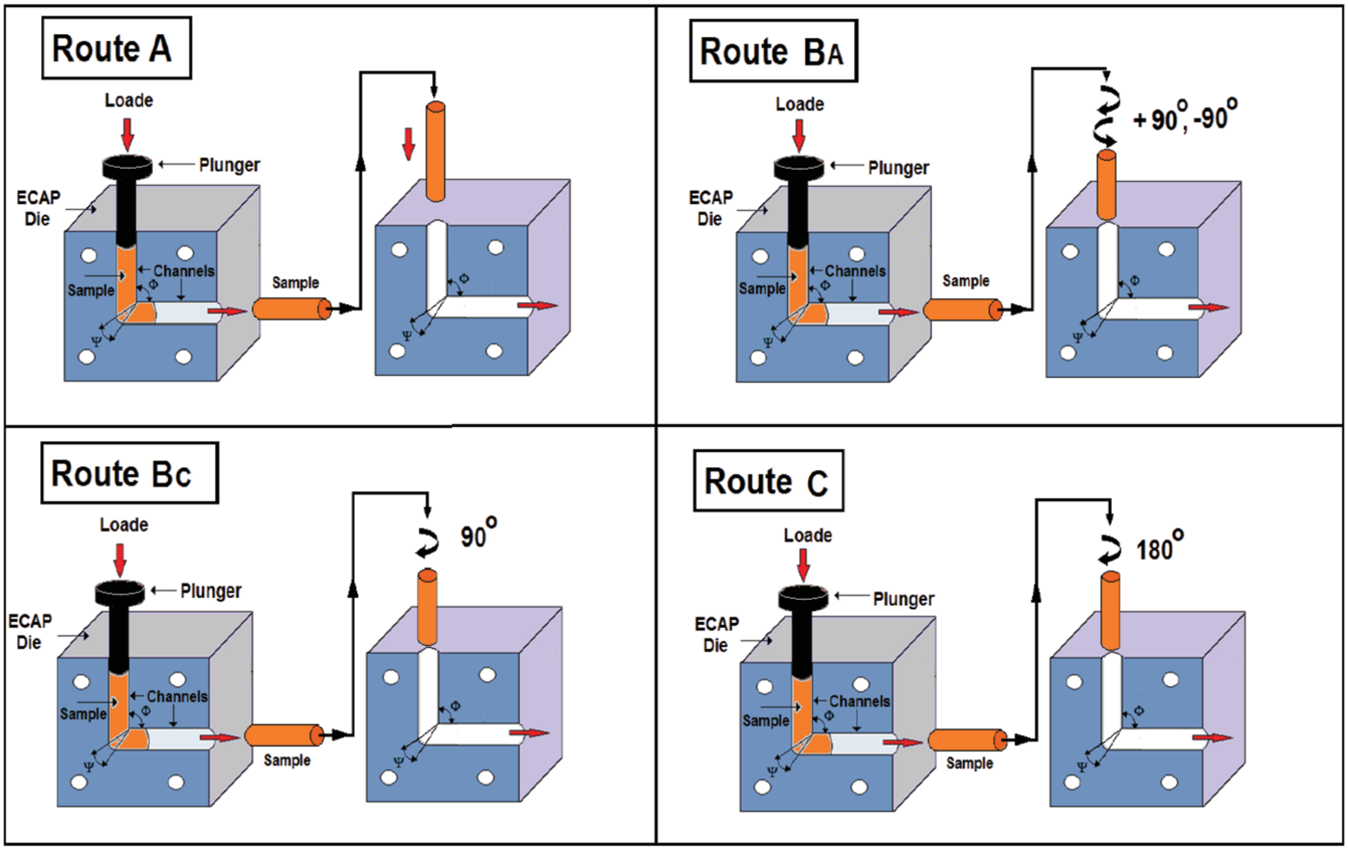

Fig.1: Pressing routes for ECAP process

have to be rotated between the successive passes about their longitudinal axis, which helps to introduce different slip systems. There are four common pressing routes which are practiced for the ECAP process called route A, route BA, route BC and route C. During route A, no rotation is given to the samples between ECAP passes. A rotational angle of 90° in the opposite direction is maintained between successive passes is called route BA, whereas 90° rotation in the same direction is followed between the passes is termed as route BC. In route C, the samples are rotated about its longitudinal axis with a rotation angle of 180º (Furukawa et al., 1998; Langdon et al., 2000).

The Fig.1 represents the pattern of these different pressing routes of ECAP process. Several works have been reported on ECAP process to enhance the mechanical property of different materials by introducing grain refinement mechanism into the materials. Xicheng Zhao et al. 2014 showed a significant increase in hardness and tensile strength of commercial purity titanium by conducting ECAP process by using a die having 90° channel angle at room temperature up to four passes. A.P. Zhilyaev et al. 2016 studied the effect of ECAP process on microstructure development and texture of the commercially available pure (CP) aluminum and made an investigation on impact of die relief angle and back pressure provided with the ECAP die on grain refinement mechanism. K. Regina Cardoso et al. 2013 conducted ECAP process on aluminum with 10% of Si metal matrix composite under different temperature range and able to achieve good results. Figueiredo et al., 2007 developed finite element models to explore the ECAP process on ZK60 magnesium alloy at room temperature. Gopi K.R. et al. 2017 investigated mechanical properties and microstructure enhancement of AM series magnesium alloy by applying ECAP process with different passes. Aluminum alloys are most attractive materials and are well known for different aerospace and automotive applications due to its high strength to weight ratio. AA7068 is the strongest aluminum alloy whose mechanical properties are nearly as good as steel, hence it found a wide variety of applications in automotive, medical devises, sports and so on (Joshua et al., 2017). By applying ECAP process a considerable amount of mechanical properties can further be enhanced by processing AA7068 aluminium alloys by ECAP process. The present work is an effort to improve the microstructure and mechanical properties of aluminum alloy AA7068 through ECAP process and mainly to investigate the effect of pressing route A and route Bc on grain refinement and mechanical properties of ECAP processed AA7068 and finally to arrive at which pressing route is optimal for ECAP process to achieve better results.

2.0 Material and methodology

Aluminium alloy AA7068 having a chemical composition of 8.3% Zn, 3% Mg, 2.4% Cu, 0.15% Zr, 0.15% Fe, 0.12% Si, 0.1% Ti, 0.1% Mn, 0.05% Cr and balance pure aluminum is cast by stir casting method. ECAP die is fabricated in a split die form with the high strength hot die steel which is heat treated to gain required strength after being machined to the required dimension on CNC machine tool. The split die is designed to have two equal cross-sectional channels of diameter 16 mm which are intersecting at an angle of 90º at the point of intersection, which is called channel angle Φ and corner angle ψ is maintained zero. Necessary clamping facility is provided to clamp the split die with a tight fit to retain them firm during high pressure ECAP pressing process to withstand vertical loads up to 20 tonnes applied by a plunger. Plunger with a pressing end of 15.8 mm diameter and 110 mm length with a loading head of 30 mm diameter is made of heat-treated hot die steel. ECAP process is not usually accomplished at room temperature but sometimes also performed at an elevated temperature. To furnish sample heating and hot-pressing process, the die is drilled with four through holes of 16 mm in diameter to accommodate electrical heating coils into the die. To maintain a constant pressing temperature the ECAP set up was built with a closed loop temperature control system with a continuous temperature monitoring and feedback sensor. The ECAP die is provided with a slot to hold temperature sensor to give feedback to the temperature controller. Cast rods are homogenized at 450°C for 24 hours and furnace cooled before ECAP process to make them flaw free that may be introduced during casting process. Homogenized samples are machined to a length of 100 mm and 15.8 mm in diameter to press through ECAP die. The die channels and samples are coated with Molybdenum disulfide (MoS_2) lubricant to eliminate frictional losses during ECAP process. Machined sample is placed into the die and a temperature of 200°C is set in the temperature controller. Sample was kept in the die for 20 minutes to attain 200°C then a vertical load was applied by a plunger on a universal testing machine (UTM). The sample pressed through ECAP die during its first pass was ejected from the die and placed into ECAP die without any rotation for the second, third and fourth passes successively according to route A as shown in Fig.1. Other samples are pressed through ECAP die at 200°C up to four passes by giving 90° rotation in counter clockwise direction between successive passes to perform ECAP process with a pressing route Bc as illustrated in Fig.1. To analyze the microstructure of ECAP pressed samples by route A and route Bc, the samples are sliced in the transversal direction according to ASTM E-112 standards to capture microstructure images through scanning electron microscope (SEM). To measure the tensile strength of the

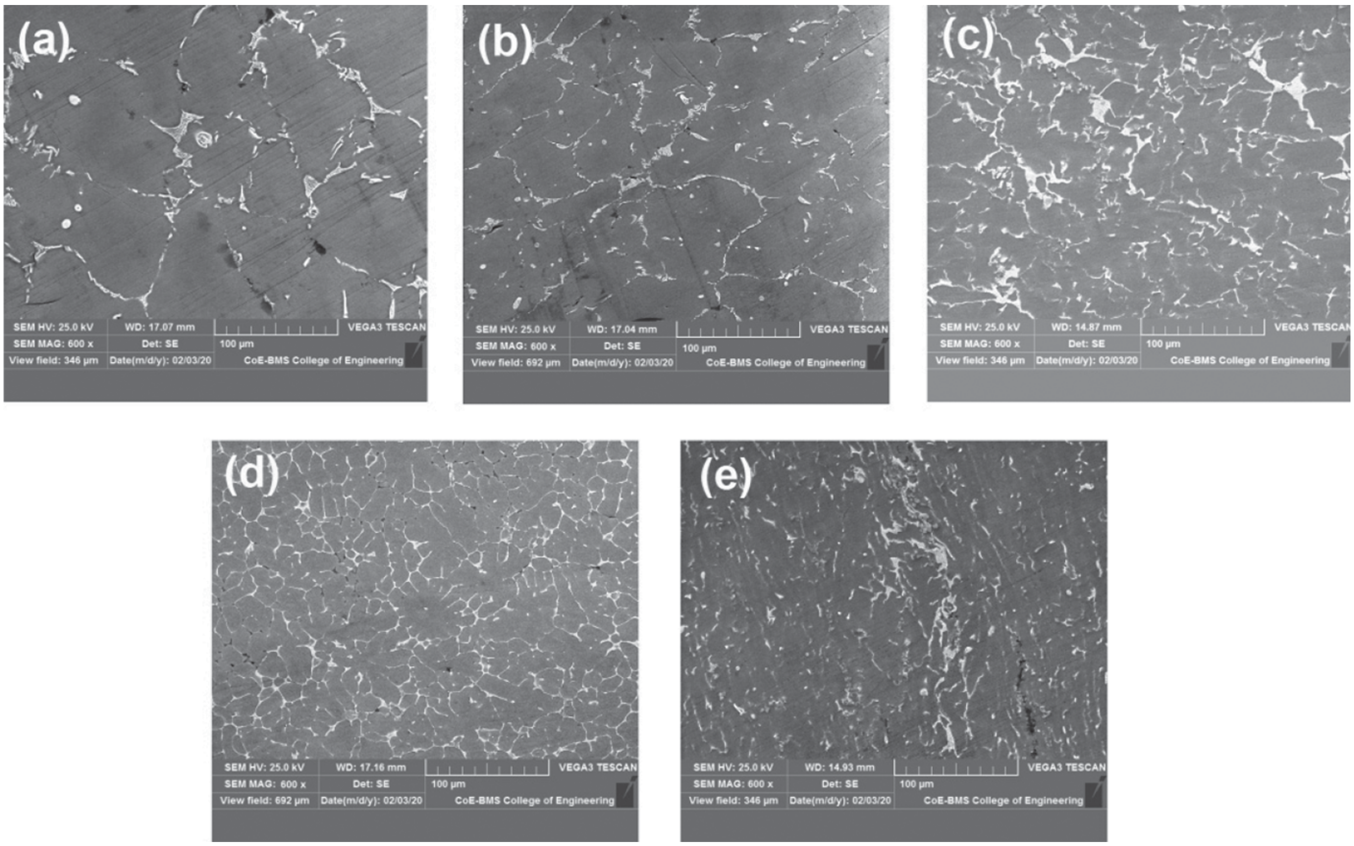

Fig.2: Microstructures of AA7068 ECAP pressed samples by route A (a) homogenized (0 pass), (b) 1 pass, (c) 2 pass, (d) 3 pass, and (e) 4 pass

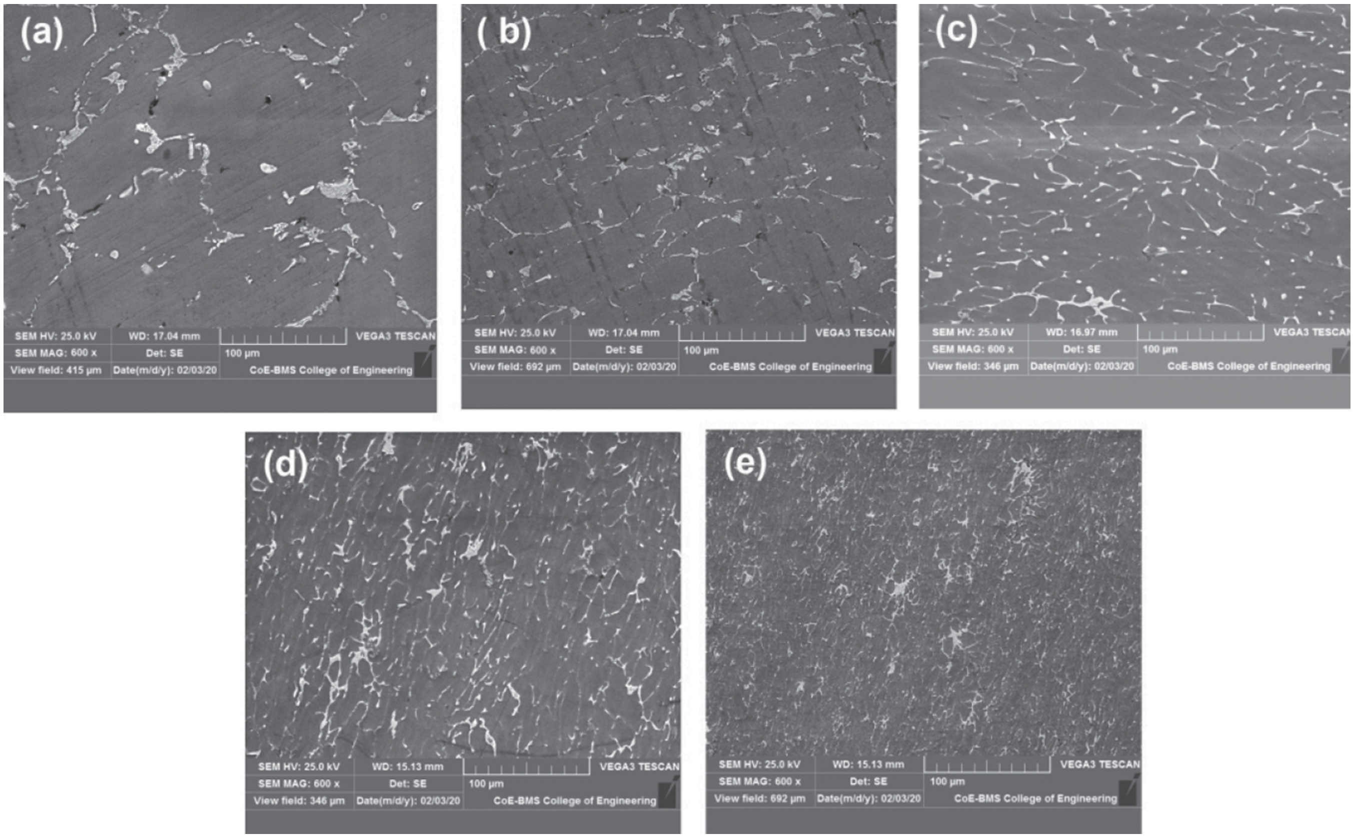

Fig.3: Microstructures of AA7068 ECAP pressed samples by route Bc (a) homogenized (0 pass), (b) 1 pass, (c) 2 pass, (d) 3 pass, and (e) 4 pass

ECAP processed samples on tensile tester, the test specimens are prepared as per ASTM-E8 standards in which a gauge length of 16 mm is maintained in its longitudinal direction. On Vickers hardness tester micro hardness tests are conducted on ECAP pressed samples as per ASTM-E384 by applying a load of 25g for 15s.

3.0 Results and discussion

3.1 Microstructural analysis

Figs.2 and 3 show microstructural images captured through scanning electron microscope (SEM) of ECAP pressed samples of aluminium alloy AA7068 with different pressing routes such as route A and route Bc. The initial average grain size before ECAP process has been measured to be ~80μm. The average grain size values after being pressed through ECAP die using route A and route Bc are found to be ~21μm and ~7μm respectively at the end of four ECAP passes. This evidences that route Bc is the most effective pressing route to achieve rapid grain refinement. Fig.2 shows SEM images of AA7068 samples pressed using route A, which are sliced in transversal direction, where figure 2(a) shows the microstructure of unprocessed sample which measures a mean grain size of ~80μm, Figs 2(b), 2(c), 2(d) and 2(e) represents SEM images extracted from first pass, second pass, third pass and fourth pass ECAP pressed samples respectively by applying route A, which measures mean grain sizes of ~75μm, ~50μm, ~35μm, ~21μm from first to fourth passes ECAP pressed samples respectively. Fig.3 shows SEM images of AA7068 samples pressed using route Bc, where Fig.3(a) shows the microstructure of unprocessed sample which measures a mean grain size of ~80μm, Figs.3(b), 3(c), 3(d) and 3(e) represents SEM images extracted from first pass, second pass, third pass and fourth pass ECAP pressed samples respectively, which measures mean grain sizes of ~60μm, ~35μm, ~16μm, ~7μm from first pass up to fourth pass samples respectively. These images shows a remarkable amount of grain refinement along with homogenized grain structure obtained in the samples pressed with route Bc. Rapid evolution of microstructure with route Bc is due to high level of recoverable shearing mechanism took place during ECAP passes, whereas in case of route A evolution of microstructure is inhibited due to the occurrence of distortion only in a single plane (y-plane) during repetitive ECAP passes especially during initial passes. In route-A shearing directions during consecutive passes were perpendicular to one another, the amount of shearing was equally divided between two sets of orthogonal planes which makes route A as a least effective method for grain refinement where no rotations are given between repetitive ECAP passes. Rotations given to the samples during successive ECAP passes in route Bc (0°–90°–180°–270°), causes the shearing directions of each passes to lie on planes which intersect at 120° and shear strain operates

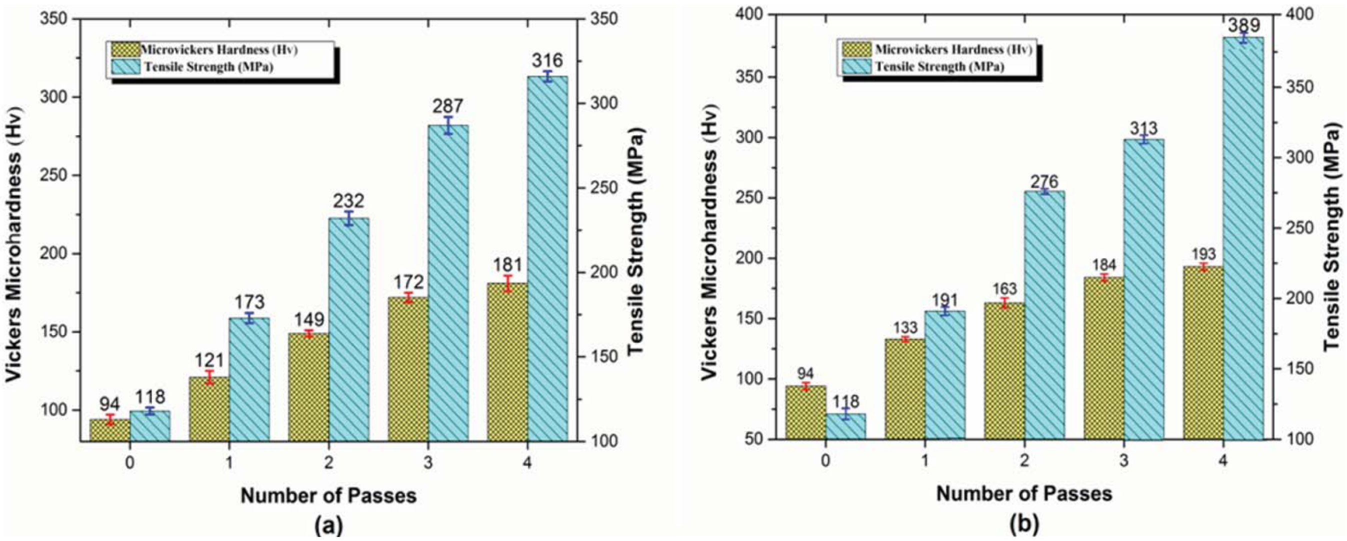

Fig.4: Vickers microhardness and tensile strength of AA7068 processed by ECAP through (a) route A and (b) route Bc.

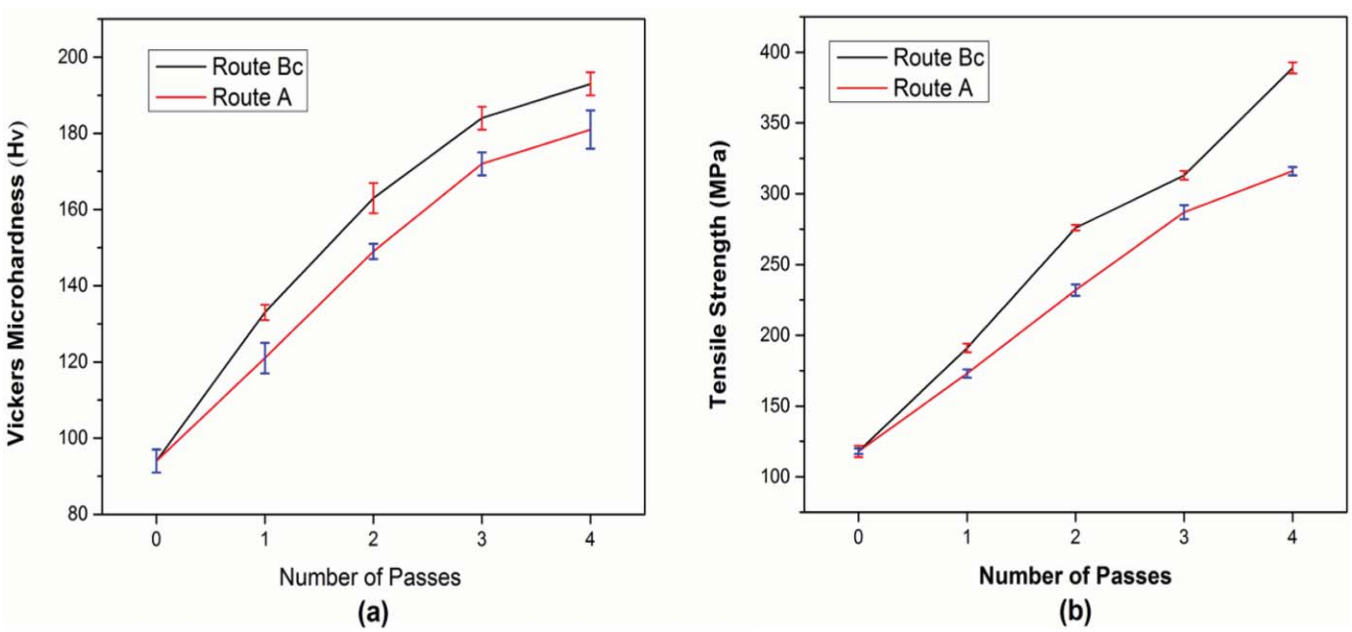

Fig.5: Comparison of (a) vickers microhardness and (b) tensile strength, of ECAP processed AA7068 through route A and route Bc

both in y-plane as well as in z-plane, this further leads to the enhancement of subdivision effect, hence the route Bc is the most effective grain refinement method (Iwahashi et al., 1998; Su et al., 2006; Tong et al., 2010).

3.2 Mechanical properties

The effect of pressing routes on mechanical properties such as tensile strength and hardness of aluminium alloy AA7068 are illustrated in Fig.4(a) and (b). The samples of aluminum alloy AA7068 processed through ECAP die using pressing route A shows an increase in its vickers micro hardness value from 94 Hv (zero pass) to181 Hv (fourth pass) and tensile strength increases from 118 MPa (zero pass) up to 316M Pa (fourth Pass). For pressing route Bc from one pass to four ECAP passes through split die got a sensible increase in its hardness and tensile strength measured 94 Hv (zero pass) to193 Hv (fourth pass) and Tensile strength increases from 118 MPa (zero pass) up to 389 MPa (fourth pass). It is observed that both hardness and tensile strength value were increased with increase in number of ECAP passes (Venkatachalam et al., 2010). Fig.5 explains that route Bc is able to introduce high values of hardness and tensile strength into the ECAP processed samples as compared to the values achieved through route A. In route Bc sample rotation of 90^o practiced between each successive passes helps to result cross hardening due to the interaction taken place between mobile dislocation and geometrically necessary dislocation (Divya et al., 2017).

Table 1: Grain size and strain induced during ECAP process by route A and route Bc

ECAP Passes	Equivalent plastic strain	Grain size (in µm)	
		Route A	Route Bc
0	0	80 ± 10	80 ± 10
1	1.15	75 ± 13	60 ± 12
2	2.31	50 ± 8	35 ± 10
3	3.46	35 ± 6	16 ± 5
4	4.62	21 ± 4	7 ± 2

These interactions strongly resists reverse dislocations, which results in a good amount of dislocation accumulations which leads to the formation of high angle gain boundary. Whereas in route A active slip planes are almost parallel to one another, which promotes dislocations to get annihilated. Table 1 clearly maps all the achieved values of grain sizes, and equivalent plastic strain introduced into the samples from first pass to fourth ECAP pass separately for route A and route Bc (Venkatachalam et al., 2012).

4.0 Conclusions

AA7068 is subjected to ECAP process through a die angle of 90° with processing routes A and Bc. The achieved results were concluded as follows.

Different processing routes during ECAP process result with different microstructure in aluminum alloy AA7068, as is evidenced by their respective SEM images. Route Bc emerged as most effective ECAP pressing route than route A to achieve rapid grain refinement because, during route Bc the shear strain operates both in y-plane as well as in z-plane, this further leads to the enhancement of subdivision effect which does not happen in route A due to no rotation given to the samples between ECAP passes.

Route Bc is able to obtain a better hardness and tensile strength of the samples as compared to route A due to the occurrence of cross hardening of the samples as the grain refinement proceeds with each pass. An overall hike of 271 MPa is achieved by pressing the samples four times using route Bc from 118 MPa (without ECAP) to 389 MPa (fourth pass) with a billet rotation of 90º between each pass.

The route A is proved to be least effective pressing route which lags behind to promote rapid grain refinement with number of ECAP passes, because of no rotation given to the samples between consecutive passes and hence active slip planes are almost parallel to one another, which promotes dislocations to get annihilated. Hence route A is only able to obtain a total hike of 198 MPa after four ECAP passes which is less as compared to that of route Bc, from 118 MPa (without ECAP) to 316 MPa (after successful four ECAP passes).

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