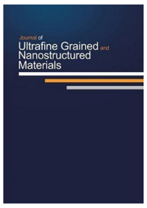

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Re-strengthening in AA6063 Alloy During Equal Channel Angular Pressing

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ABSTRACT

Equal channel angular pressing (ECAP) is carried out using two different configurations for the exit channel of the ECAP die, i.e., relieved and choked, with angles of 0.2 °. It is found that using a die with relieved exit channel, the sample was extruded for 6 passes with no surface cracks and an average cell size of 727 nm and a fraction of high angle grain boundaries of 54 % were achieved. Measured values of yield strength (YS) and ultimate tensile strength (UTS) were reported to be 201.1 and 259.5, respectively. By using the die with choked exit channel, it was possible to deform the sample for up to 14 passes with no sign of surface cracking. A relatively finer cell structure around 530 nm was achieved and the fraction of HAGBs increased to 64 %. Relative increases in YS and UTS were as well observed indicating that re-strengthening has been activated in the material after saturation at the 4th pass. In addition, the mechanism of grain refinement seemed to change to progressive lattice rotation evidenced by formation of trapped single grains within the size range of less than 100 nm.

Keywords: *Equal channel angular pressing; Die exit channel; Workability; Re-strengthening.*

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1. Introduction

Different methods of severe plastic deformation (SPD) including equal channel angular pressing (ECAP) have been used in order to produce ultrafine grained materials with high strength [1-5]. At early stages that the process of equal channel angular pressing (ECAP) was invented, effect of many processing parameters on the mode of deformation[6], effective strain [7] dead metal zone formation[8], pressing load [9] and many other mechanical characteristics of the process were investigated. The research was followed by many investigations [10-12] on the effect of the process and material parameters on the evolution of microstructure and mechanical properties. However, there are a few number of research activities focused on the effect of die design parameters on the properties of the product, e.g., evolution of microstructure and mechanical properties [13-18]. This may be due to the simplicity of the process in which there are only a few die design parameters, i.e., die angle and outer curved corner angle, which determine the level of effective strain. In recent years, the routine research procedure of severe plastic deformation has been to investigate the effect of process and/or material parameters on the evolution of microstructure, e.g., effect of number of passes[19,20], route of deformation[21,22], channel angle[23], back pressure [24-26] and initial grain size [27,28].

Although in theory, ECAP can be performed for as many number of passes as needed, however; in reality, there are two factors limiting the applicable number of passes on a material, i.e., the material workability and saturation of the product in terms of grain refinement and mechanical strengthening [29-32]. Workability of the material directly limits the application of further ECAP passes as it would lead to cracking and failure with continued deformation. Few different approaches, e.g., applying back pressure[25,26], increasing the deformation temperature [33,34] and using a die with choked exit channel [35], have been proposed and used for increasing the workability of the material during ECAP.

Saturation is a routine observation in materials with extremely high workability, e.g., pure Al [31,32] and pure Cu [30] [36] [37], when it is tried to have a large number of ECAP passes. In other words, it has been observed that after a specific number of deformation passes, negligible advancements in grain refinement and enhancement in mechanical strength are achieved and the product may be considered saturated. The level of saturation and the number of ECAP passes at which saturation occurs varies in different materials. For example, saturation of the structure in the case of AA6063 has been reported to occur after an equivalent strain of 3 to 4 [29,38]. It should be noted that the saturation equivalent strain is significantly larger for copper, i.e., 15 [30]. In the case of pure Al, a maximum is observed at equivalent strain of 2 [31,32] followed by a reduction and achieving a plateau at equivalent strain of 6.

In the current investigation, the effect of die design configuration on workability and the evolution of microstructure and tensile properties is investigated. For this purpose, two configurations, i.e., choked and relief, for die exit channel were considered. It was found that workability improves in terms of number of ECAP passes to failure which consequently affected the evolution of microstructure and tensile properties of the alloy after ECAP. It is concluded that the possibility of further deformation using the die with choked exit channel helps to overcome saturation of tensile properties and leads to re-strengthening of the alloy in addition to providing further grain refinement. In addition, the mechanism of grain refinement was observed to change to progressive lattice rotation and led to further grain refinement in AA6063 alloy.

2. Experimental procedure

AA6063 aluminum alloy with the chemical composition shown in Table 1 is used in this investigation. Round bars of this alloy with a diameter of 100 mm were received in the form of hot extruded products. Cylindrical samples with diameter of 19.9 mm and length of 120 mm were machined from bars and annealed at 550 °C for 30 min. The samples were quenched in water followed by annealing. An initial extra coarse grain structure was generated in the samples. For this purpose, the samples were deformed for two passes using ECAP and annealed at 550 °C for 6 hrs and cooled to room temperature in furnace. The microstructure after the initial thermo-mechanical processing as indicated in [28] is composed of extremely coarse grain structure with an average grain size larger than 1000 µm. Normally metals with coarse grain structure indicate lower workability. Therefore, the aim of preparation of a sample with such a coarse grain structure was to minimize workability and promote cracking to facilitate failure of the material. This provides the possibility of distinguishing between the different designs of the ECAP die.

Two ECAP dies were used in this investigation. The channels with nominal diameters of 20 mm were connected at 90 ° with an outer curved corner of 22.5 °. The diameter of the entry channel was similar for both of the dies. However, two configurations, i.e., relieved and choked with taper angles of 0.2 °, were used for the exit channel. Deformation was performed with a constant ram speed of 1 mm/s using a 100 tons hydraulic press. ECAP deformation was continued until the first surface cracks on the samples were observed with naked eyes.

Microstructural investigation was performed on longitudinal cross section. The initial grain structure was studied by a polarized light optical microscope. The microstructures of the samples deformed using the die with choked exit channel were characterized using electron backscattered diffraction (EBSD) on the longitudinal cross section of the specimens at a depth of 5 mm beneath the surface to ensure no surface effect exist to affect the evolution of grain structure. For this purpose, samples were initially cut and grinded. Electrolytic polishing was performed at 0 °C, 17 V and flow rate 15 for 80 sec (electrolyte: 7.8 % perchloric acid, 9 % water, 73.1 % ethanoland 10 % butylcellulose). EBSD was performed using a JEOL 6500 scanning electron microscope (accelerating voltage: 25 kV; tilt angle: 70 °; working distance: 25 mm). Orientation contrast data were analyzed with HKL. Subgrains were detected by setting a critical value of 15 ° as the minimum misorientation value of high angle grain boundaries (HAGBs). The minimum misorientation value of low angle grain boundaries (LAGBs) was set to be 2 °. ImageJ analyzing software is used to extract the average grain sizes. The measurements are performed on a large area composed of more than 1000 grains to have a solid conclusion on the average numbers. By the way, these measurements are normally involved with an average error of 10 %.

For tensile testing, round 100 mm long samples were prepared. Sub-sized specimens were prepared according to ASTM B557M standard with a gage length of 45 mm and diameter of 9 mm. Tensile testing was performed using Universal Instron testing machine with 5 mm/min speed.

3. Results and discussion

3.1. Initial microstructure

The initial microstructure of the alloy is definitely effective on the workability and grain refinement procedure [27,28] during ECAP. Therefore, it is important to have a clear image of the initial grain size. Therefore, the initial microstructure of the sample after annealing at 550 °C for 30 min is shown in Fig. 1. It can be seen that the microstructure is composed of a fully recrystallized grain structure with an average grain size of 200 µm. In addition, a fraction of second phase particles of around 2-3 % is observed. These particles seem to be compounds of Al, Mg, Si and Fe and are within a size range of 1 to 5 µm.

3.2. Workability of the alloy

3.2.1. Number of passes to failure in the die with relief exit channel

Number of ECAP passes to surface cracks or failure are used in order to evaluate workability of the materials. In fact, surface cracks are considered as indications that the deformation must be stopped if a sound flawless product is expected. Shapes of

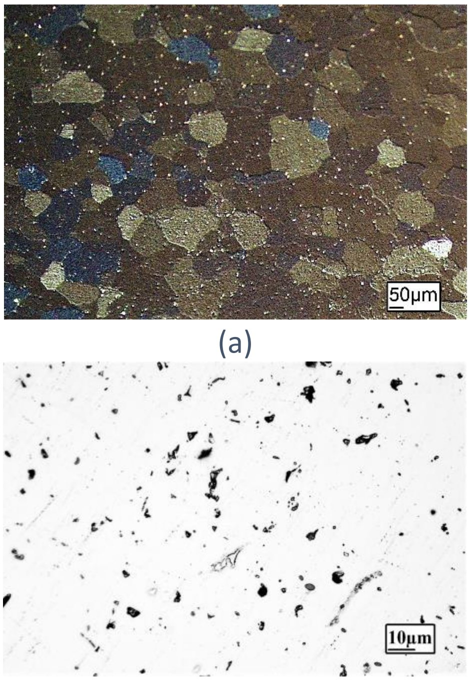

Fig. 1- Initial microstructures of the samples after solution treatment at 550 C and water quenching and prior to ECAP, (a) grain structure and (b) distribution of the second phase particles.

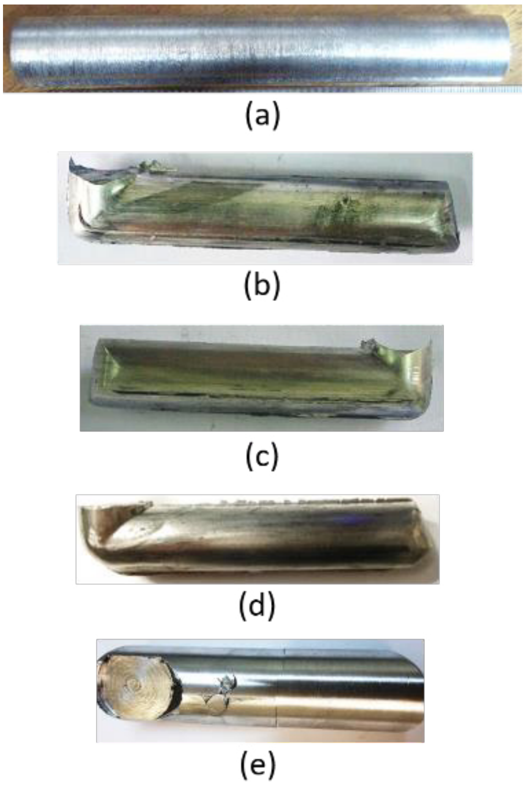

Fig. 2- Shapes of the round samples (a) before deformation and after (b) two, (c) four, (d) six and (e) seven passes ECAP.

the round samples, without deformation and after 2, 4, 6 and 7 passes ECAP are shown in Fig. 2. It can be seen that up to the 6th ECAP pass, no surface cracks have formed. However, surface cracks appear in the 7th ECAP pass. Obviously, if the deformation is continued, the surface cracks grow further and fracture occurs. Therefore, the workability of this material in the ECAP process is considered 6 passes. Such a material can be considered one with good workability. Poor workability with surface cracking after one pass ECAP has been reported in the literature[40].

3.2.2. Number of passes to failure in the die with choked exit channel

Shape of a deformed sample in the die with choked exit channel is shown in Fig. 3. The sample has been soundly deformed for 14 passes without surface cracks in the die with choked exit channel. This indicates a significant improvement in the workability of the material by utilization of a die with a choked exit channel with respect to one with relieved exit channel. The improvement of workability is attributed to the compressive maximum principal stress which is caused due to the opposite pressure acting on the deformed samples against its movement in the exit channel [35]. This has been extensively discussed elsewhere [35].

3.3. Evolution of microstructure and tensile properties

It has been previously discussed [28,38] that the alloy under investigation in this study, starts to be saturated after 4 passes ECAP. In other words, after 4 passes ECAP, no further strengthening evidenced by negligible variation in hardness, YS and UTS occurs [28,38]. As the workability limit of this alloy in a normal ECAP die is around 6 passes without surface cracking, routinely the deformation is stopped at this level and the 6 mentioned passes would be reported as the saturation limit [28,38]. However, it was possible to deform the sample up to 14 passes using the new designation of the die. It is of great interest to find out the effect of this improvement in workability on the microstructure and tensile properties of the alloy.

Fig. 3- Shape of the sample after 14 passes ECAP in the die with choked exit channel.

3.3.1. Re-strengthening by further ECAP processing

Tensile test results on the samples in as-annealed conditions and after 2, 4, 6 [28] and 14 passes ECAP are shown in Fig. 4. It should be noted that the samples which are deformed for 2, 4 and 6 passes ECAP are prepared by the die with relieved exit channel and the one deformed for 14 passes is deformed using the die with choked exit channel. One can clearly see the considerable increase in the yield (YS) and tensile strengths (UTS) of the alloy by application of the first two passes of ECAP. This is due to work hardening and significant increase in dislocation density. The sample which is deformed for 4 passes shows a considerable increase in YS and UTS with regard to the 2 pass deformed sample. However, such increase is not observed when the sixth pass of ECAP is performed. This indicates the occurrence of saturation in mechanical properties after 4 passes ECAP which has led to negligible change in tensile properties of the sample. This is in line with what has been previously observed in this alloy [29,38]. However, when the number of passes is increased to 14, the material starts to re-strengthen evidenced by increasing, YS and UTS. This may be attributed to further grain refinement which has occurred by increasing the number of passes and will be discussed later. In addition, no reduction in elongation by increasing the number of passes is observed. This is due to the increasing the fraction of HAGBs which indicates the occurrence of more intensive dynamic recrystallization during grain refinement leading to stress relived microstructure with an acceptable elongation.

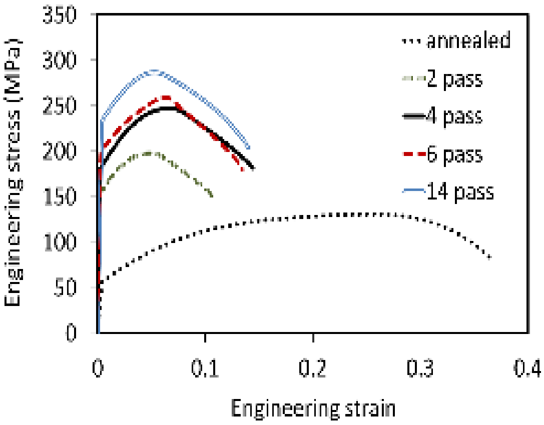

Fig. 4- Tensile test results on samples in as-annealed condition and after 2, 4, 6 [28] and 14 passes ECAP.

3.3.2. Microstructure

EBSD maps of the AA6063 sample used in this investigation after 2, 6 and 14 passes ECAP are shown in Fig. 5. Results of image analysis show that the average cell size reduces from 895 to 727 and ultimately to 530 nm by increasing number of passes from 2 to 6 and 14, respectively. In addition, the fraction of high angle grain boundaries has increased from 37 to 54 and 64 % with increasing the number of passes from 2 to 6 and 14, respectively. Gradual reduction of the average cell size and increasing the fraction of HAGBs indicate that the refinement of the microstructure has not stopped after a few number of passes and has continued till the 14th ECAP pass. However, the EBSD results indicate that the mechanism of grain refinement is slightly different with the traditional mechanism of grain refinement during SPD processing. In order to understand the difference, the traditional mechanism of grain refinement [41] must be explained. During SPD, significant increase in dislocation density occurs, the grains become thinner, the fraction of grain boundary area increase and cells or subgrains form with rearrangement of dislocations [41]. At this stage, most of the cell boundaries are surrounded by low angle ones. With further increasing strain, the boundaries become closer to each other and form new grains by impingement and a fine grain structure forms. By increasing dislocation density with further deformation, the matrix would not accommodate further increase in the dislocation density and consequently the excess dislocations would be consumed for increasing the missorientation angle of boundaries. However, the sizes of the cells and grains do not reduce. This is why a saturated microstructure in terms of grain size forms. In fact, there should be a second mechanism involved with reduction of the cells and grains sizes in the sample which is deformed for 14 passes. Magnified EBSD image of the grain structure of the sample after 14 passes ECAP is shown in Fig. 6. This image is used to indicate the formation of a number of extremely fine new grains, within the size range of less than 100 nm inside the microstructure. In fact, this indicates that after the activation of the first mechanism of grain refinement [41], a second mechanism, i.e., progressive lattice rotation [42], activates. The image in Fig. 6 clearly indicates the occurrence of dynamic recrystallization by the mechanism of progressive lattice rotation which is not observed in the case of the sample after 6 passes ECAP. Therefore, increasing the number of passes to 14 has clearly helped for further grain refinement in this alloy and has possibly led to re-strengthening as observed in tensile properties presented in Fig. 4.

All these indicate further grain refinement has occurred by increasing the number of ECAP passes which is against what has been

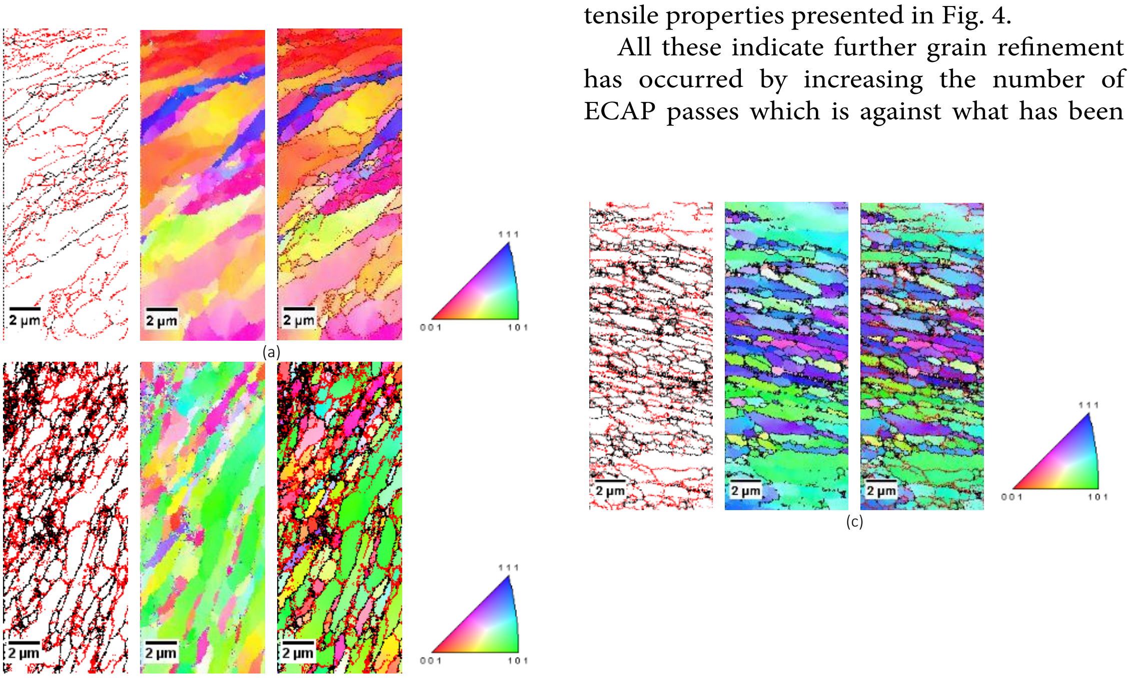

Fig. 5- EBSD images of the samples deformed for (a) 2, (b) 6 and (c) 14 passes ECAP.

normally reported in the literature [29]. In fact, it is well-known that the microstructure acquires a saturated form after a limited strain [29]. The number of ECAP passes to acquire saturation depends on the alloy and its alloying elements [29-32]. For example, the saturation limit for pure Al is 6 passes [31,32] while 3-4 passes for the case of AA6063 alloy [29] and 15 passes for pure Cu [30]. In addition, presence of some alloying elements. e.g. Mg, may promote grain refinement and postpone saturation [43,44]. However, in the current investigation, it is found out that although saturation was observed in tensile properties after 4-6 passes ECAP, however, when the number of passes increased to 14 by utilization of the new die design, the microstructure continued to refine and the tensile strength to increase. This observation, i.e., re-strengthening, may be attributed to the new possibilities after increasing the number of passes provided by utilization of the die with choked exit channel. In fact, in the current investigation, the number of passes has been tremendously increased, i.e., from 6 to 14, by application of the new configuration of die while in the reported literature, the effect of increasing a few number of passes has been investigated.

4. Conclusions

In this research, the effect of a modification in the design of the exit channel of the die on the workability of the material and its effect on the evolution of microstructure and tensile properties during equal channel angular pressing (ECAP) are investigated. Two different die designs are utilized with constant die angles and diameters of the entry channels with different configurations for exit channel, i.e., choked and relieved. According to the results of this investigation, the following conclusions are made;

1) Application of a die with choked exit channel results in improvement of workability compared to using a die with relieved exit channel.

2) Significant improvement in tensile properties and grain refinement is achieved by increasing the number of deformation passes. In fact, increasing the number of passes has helped the alloy to re-strengthen after it acquires a saturated

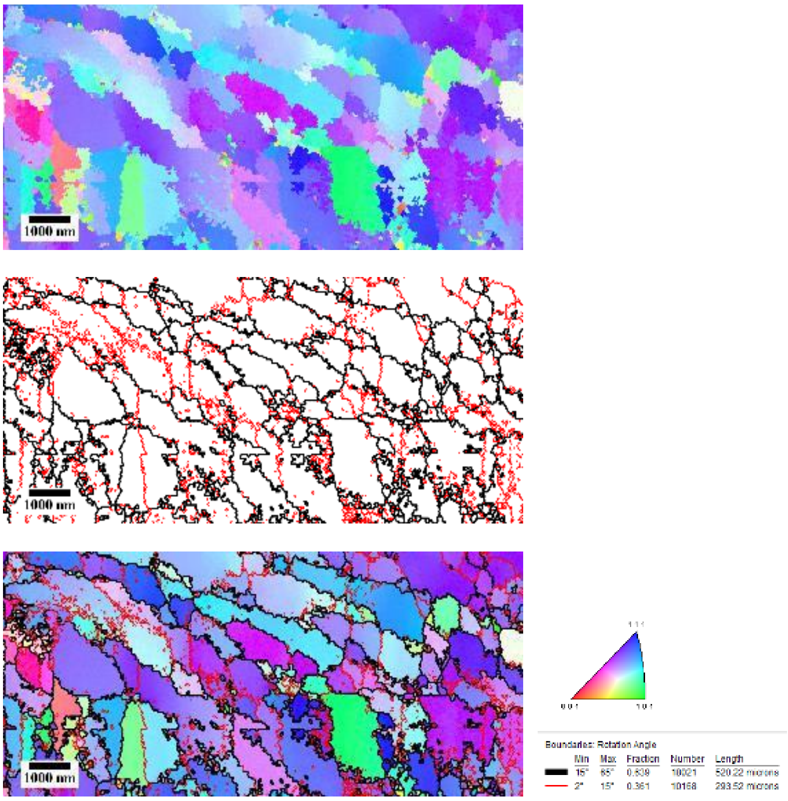

Fig. 6- High magnification grain boundary map showing progressive lattice rotation in AA6063 alloy after 16 passes ECAP.

level of hardness and tensile strength.

3) By significant increase in the number of passes due to utilization of the die with choked exit channel, a second mechanism of grain refinement, i.e., progressive lattice rotation, has been activated leading to extremely fine grains within the size range of less than 100 nm.

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