Optimization of processing conditions in equal channel angular pressing for superior structure and properties

In the past 25 years, much interest has been paid to the production of ultrafine-grained (UFG) materials due to their superior structure and properties. Equal channel angular extrusion or pressing (ECAE or ECAP) is one of the severe plastic deformation (SPD) technique used to produce UFG materials. Compared to other SPD process, higher strain per pass and higher total productive strain after 'n' number of passes is possible in this technique. ECAP process is affected by various processing conditions or parameters. These processing conditions mainly affects the structure and properties produced through this technique. In this article, an effort has been made to provide the effect of each processing parameter on the structure and properties developed through this technique. Various processing conditions like- processing route, channel angle, outer arc of curvature, pressing speed, load and friction, processing temperature and application of back-pressure are critically discussed and effect of these parameters are summarized.

Keywords: SPD, ECAP, UFG, microstructure and mechanical properties.

1.0 Introduction

Severe plastic deformation (SPD) methods are attractive because these methods result in significant grain refinement up to ultrafine grain (UFG) structure level in bulk materials. Hence, there is a considerable increase in the processing of metallic materials through SPD methods (Langdon, 2007). SPD methods are basically metal forming techniques, wherein large strain is applied on the metallic materials which leads to extraordinary grain refinement (Valiev et al., 2006). The structure developed by SPD methods possess high-angle grain boundaries (HAGB) and possess homogeneous microstructure (Valiev et al., 2000). SPD methods refine microstructures by introducing high densities of lattice dislocations. Materials developed by SPD methods possess 100% density and contamination free. Also, SPD materials possess high strength, high wear resistance, high fatigue life, good ductility, superplasticity, and good corrosion resistance (Zhu and Langdon, 2004). SPD methods were routed in from the works of Bridgman (1952), wherein simultaneous application of compression and torsion which leads to grain refinement leading to the attainment of grain refinement in metallic materials. However, major progress in SPD methods was reported by Segal et al., (1981) with the introduction of equal channel angular pressing (ECAP), a unique method for refining the grains in metals, alloys and metal matrix composites.

Various methods are introduced in SPD for refining the grains in metals, alloys and metal matrix composites. These include high pressure torsion (HPT) (Smirnova et al., 1986), equal channel angular extrusion or pressing (ECAE or ECAP) (Segal et al., 1981), accumulative roll bonding (ARB) (Saito et al., 1998), cyclic extrusion and compression (CEC) (Richert and Richert, 1986), constrained groove pressing (CGP) (Shin et al., 2002), repetitive corrugation and straightening (RCS) (Huang et al., 2001), and multi-directional forging (MDF) (Salishchev et al., 1993). Among these techniques, the ECAP processing is very attractive technique for various reasons like - it is fairly easy to prepare ECAP die set up and use in pressing machines or universal testing machines, large strains can be applied by 'n' number of passes, technique can be scaled-up to process bulk samples and this technique can be extended to deform plate samples. Several researches reported the significance of ECAP processing on various metals and alloys (Manjunath et al., 2017; Manjunath et al., 2018; Manjunath et al., 2018; Manjunath et al., 2018; Manjunath et al., 2019). In this article, an attempt is made to present the optimized processing conditions in equal channel angular pressing to develop UFG microstructure in metals and alloys.

2.0 Principle of ECAP processing

ECAP or ECAE is one of the innovative processes of SPD methods, wherein it can produce high plastic strain in metals, alloys and metal matrix composites. The terms ECAP and ECAE are adopted in the scientific literature. However, in this process, there is no reduction in the cross-sectional (c/s) area of the sample material, so the term ECAP suits more for this process. Fig.1 presents the principle involved in the ECAP process, where 'Φ' depicts the channel angle and 'ψ' depicts

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the angle where two outer parts of the channel intersect called as outer arc of curvature (Zehetbauer and Zhu, 2009). In ECAP, the deformation occurs by pure shear (Segal, 1995). The set up consists of a die containing two channels, equal in c/s area (square or circular) intersecting at an angle. The sample is pressed through these channels. As the sample moves from the plane of intersection of the two channels shear strain is induced in the sample, leads to substantial grain refinement. For smooth conduction of the process, lubrication is applied on the surface of the die channels. Also, processing can be carried out at higher temperature depending on the type of the material to be processed. As the specimen moves through the channel, von-mises equivalent strain value in the specimen depends on the 'Φ' and 'ψ' values.

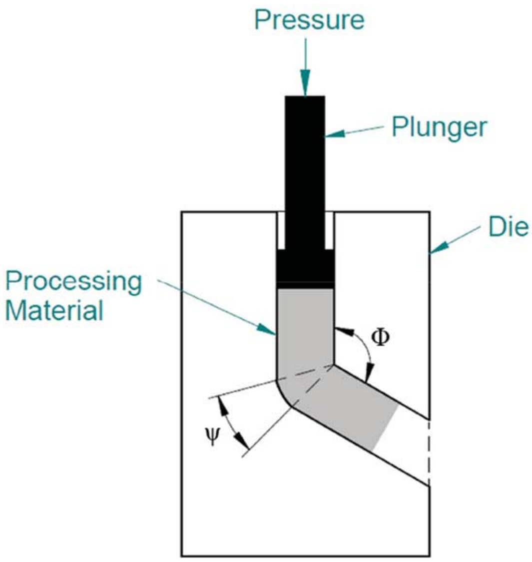

Fig.1: Principle of ECAP process

Since, the c/s area of the specimen is unaffected during pressing, the specimen can be processed repeatedly in order to attain larger strain. The total equivalent strain induced in the sample material after 'n' number of passes is accessed by the expression (Iwahashi et al., 1996),

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\operatorname{cosec}\left(\frac{\phi}{2}+\frac{\psi}{2}\right)\right] \quad \text{... (1)}$$

During this repeated processing cycles, shear strain induced in the sample leads to develop UFG microstructure. Also, distinct slip systems in the sample can be produced in the sample by rotating the sample (on its longitudinal axis) in between each successive pass. Nowadays, numerous modifications of conventional ECAP have been developed like continuous processing by ECAP for more efficient grain refinement.

3.0 Processing conditions in equal channel angular pressing

ECAP processing is a shear deformation process characterized by various operating conditions like strain imposed in each pass, slip systems introduced in the sample by rotating the specimen (on its longitudinal axis), followed by consequent shearing patterns. These operating conditions uniquely define the nature of the process. Also, these operating conditions play significant role in identifying the structure and properties developed by ECAP processing. Various factors also affect the material structure and properties when the material is processed by ECAP technique. They are- channel angle, outer arc of curvature, pressing speed, load and friction, processing temperature and application of back-pressure. These parameters dictate a dynamic role in producing homogenous microstructure, orientations in grain boundaries, textures, and improvement in the mechanical, thermal and physical properties of processed specimen. (Valiev and Langdon, 2006). In this section, these operating conditions and factors affecting the structure and properties developed by ECAP processing are discussed.

3.1 Processing Routes in ECAP

Shearing features in the specimen can be altered by rotating the specimen between each successive pass (Segal, 1995). Fig.2 depicts the four fundamental processing routes traditionally followed in ECAP. These pressing routes were established based on the turning of the sample on its longitudinal axis in between consecutive passes (Nakashima et al., 2000). These pressing routes stimulate distinct slip systems while processing, so that noticeable differences in microstructure are formed. Also, there is an interaction between the shear plane and the texture formed during ECAP. These pressing routs are identified by set of shear planes which experiences deformation while processing. In this section, these processing routes are discussed in detail. In route A, the specimen is processed repetitiously without turning between each successive pressing. In route B_A, the specimen is turned by 90° in the alternate directions in between successive pressing. In route B_C, the specimen is turned by 90° only in one direction (either clockwise or anti-clockwise) between each successive pressing. In route C, the specimen is turned by 180° between successive pressing.

There are significant differences in the shearing patterns developed by these processing routes. Shearing patterns developed in the material also depends the die channel angle. In 90° die channel angle, in route A, shear is developed at an angle of 45° when the specimen is moved through the shearing plane. With subsequent passes, elongation of the grains takes place only in one direction. In 90° die channel angle, route B_C is more effectual and develops shearing over wider angular span compared to other three routes. Slip tracks in route B_C ranges from 0 to 90° in the X-plane and 27 to 90° in the Y-plane and Z-plane. In route B_C, there is a rebuilding of the equal dimension microstructure after each four pressings. Also, deformation takes place in all three planes which leads to development of optimum microstructure. Whereas in 120° die channel angle,

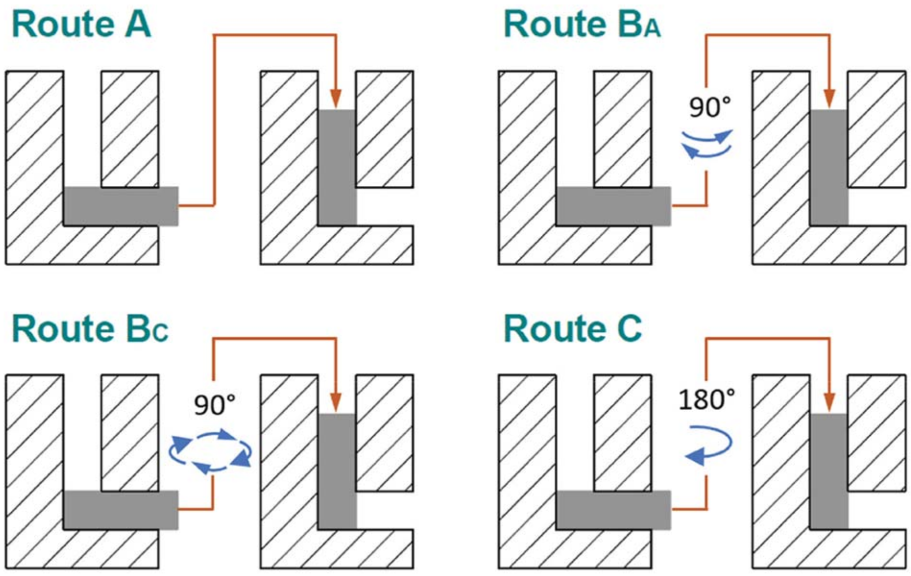

Fig.2: The four fundamental processing routes traditionally followed in ECAP

3.1.2 Shearing patterns associated with different processing routes

To develop an optimum microstructure with steady and equal dimension grains produced with high-angle grain boundaries, following circumstances should be fulfilled. (i) Attainment of slip tracks over wide angular span on all three planes. (ii) A steady and periodic reinstating of the equal dimension microstructure. (iii) Distortion happening on all three planes. Furukawa et al., (2002) studied the shearing patterns accompanying with various pressing routes in both 90º and 120º die. It was reported that, the criterions are fulfilled in using route B_C in 90° die and route A in 120° die. Even though, the route B_C is considered as the optimum procedure.

route A will be the better route to produce optimum microstructure compared to route B_A and route B_C. The least effective route in 120° die channel angle is route C. It is feasible to presume that, route B_C is more effective in 90° die channel angle and route A is more effective in 120° die channel angle. Both, route C and route B_A are not effective in both 90° and 120° die channel angle. However, route B_C is better route in 120° die channel angle because it provides wider angular span of slip tracks, deformation happens on all three planes and restoration of equal dimension microstructure after each four pressings.

3.1.1 Slip systems associated with different processing routes

In route C, shearing is repeated on the same plane in each successive pressing, but it changes its direction in opposite sense on each successive pressing. Thus, the route C can be called as a redundant processing route and the strain is reinstated after every even number of pressings. Route B_C is also a redundant processing route, since slip in the first pressing is neutralized by slip in the third pressing and slip in the second pressing is neutralized by slip in the fourth pressing. Route A and route B_A are not redundant strain processes. In route A, there are two distinct shearing planes crossing at an angle of 90°. In route B_A, four distinct shearing planes crossing at angles of 120°. In route A and route B_A, there is a collective growth of added strain on each pressing. The cubic element is reinstated after each 2 pressings in route C and after each 4 pressings in route B_C. However, the distortions will be more acute in the routes A and B_A. Also, there is no distortion of the cubic element on the Z plane in route A and route C. It should be noted that, route B_C imparts distortion on all three planes (Furukawa et al., 1998).

3.2 CHANNEL ANGLE ‘Φ’

Parameter channel angle ‘Φ’ is considered as a significant experimental parameter as it decides the strain introduced in each pressing, which is clearly mentioned in Equation 1. Fig.3 illustrates ECAP dies with different ‘Φ’ values. Nakashima et al., (1998) reported that, large amount of ultrafine equal sized grains and grain boundaries having high angle of misorientations could be easily achieved if the specimen is processed in a die having smaller channel angle. In smaller channel angle specimen can be subjected to die high intensity of strain. If the die angle is increased, the grain boundaries will have low angle of misorientations. It is also reported that, experimentally it is quite easy to press samples in dies having a higher channel angle, particularly for hard materials and materials with low ductility. The important factor to be considered is that, the microstructure developed is independent of total cumulative strain. It is better to induce a large strain rate on each distinct pass to form grain boundaries with high angle of misorientations and to produce more refined grains. Furuno et al., (2004) reported that, more refined microstructures could be obtained using a die having channel angle 60° in contrast to die having channel angle 90°. But high pressure is required to press the billet in die having 60° channel angle. It is feasible to presume that, die channel angle 90° results in optimum microstructure. But, to process high strength and brittle materials it is better to increase the die channel angle.

3.3 OUTER ANGLE OF CURVATURE ‘ψ’

The parameter ‘ψ’ indicates the curve (on the outer side) where the two channels of the die intersect. This parameter signifies smaller role in defining the strain induced on the specimen. Also, this parameter has smaller effect on the strain

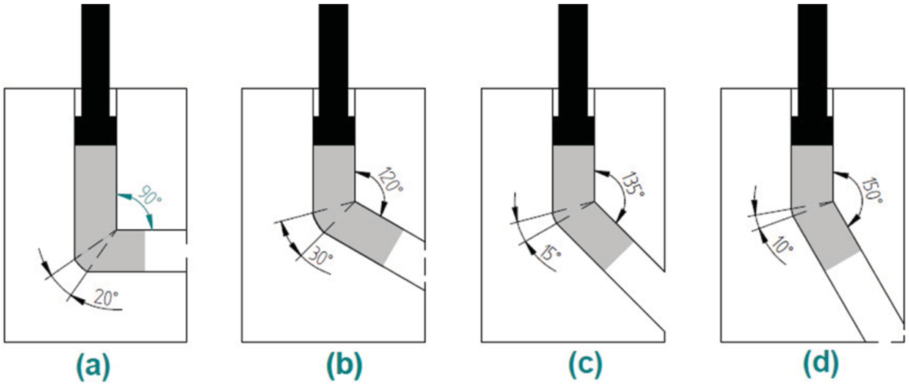

Fig.3: Schematic illustration of ECAP dies having different values of 'Ö' (a) 90°, (b) 120°, (c) 135° and (d) 150°

induced on the specimen, if the channel angle is greater than 90° (Furuno et al., 2004). But it is necessary to understand the effect of outer angle of curvature in the development of UFG materials. Split design dies can be machined with $\psi = 0°$, but for solid design dies it is necessary to incorporate outer angle of curvature. Even though, outer angle of curvature has relatively less effect on the strain induced on the specimen, it is necessary to provide outer angle of curvature. If the outer angle of curvature is not provided, the processing material will not be in surface contact with the channel of the die at the outside corner, which leads to develop dead zone at the outside corner once the sample moves through the die (Shan et al., 1999). This dead zone will lead to develop uneven deformation in the processing specimen. Strain hardening rate in the processing material also depends on the corner gap provided in the die. Corner angle can also be provided at the inside corner, but it is not recommendable because of practical difficulties. It is feasible to machine the ECAP dies having 20° to 30° outside angle of curvature achieve optimum processing conditions.

3.4 Pressing Speed, Load and Friction

Pressing speed is one of the variable parameters in the ECAP process. Pressing speed in ECAP process varies from 1mm to 5mm per second. Generally, pressing is carried out in hydraulic press. But it is feasible to carryout pressing in conventional mechanical testing machines, because mechanical testing machines provide the wide range in pressing speeds. However, pressing speed has less significance on the formation of equal size ultra-fine grains. Pressing at less speed develops more equal size grains, but recovery happens quite easily when processing is carried at less speeds. But there is rapid rise in the temperature if the processing is carried at higher speed. Ductile materials can be processed at higher speeds, but brittle materials have to be pressed at lower speeds to avoid catastrophic fracture in the processed materials.

Generally, load necessary for ECAP processing is identified by the specimen material strength. Pressing load requirement increases with rise in the strength of the specimen material. Pressing load requirement also increases with enlarge in the c/s area of the sample (Horita et al., 2001). But sample size will not affect the microstructure and mechanical properties achieved after ECAP processing. During processing, surface contact exists in between die channel and processing material, these surface contact led to develop friction between the die channel and processing material. This friction will lead to increase the pressing load and internal heating during processing. For smooth processing of the material, this friction has to reduced. Lubrication is applied between the die channel and surface of the processing material to reduce friction. Generally, two types of lubricants are used in ECAP processing. They are: semi-solid and solid lubricants. Commonly used semi-solid lubricant is Molybdenum disulphide (MoS_2), which is stable in room temperature and remains stable upto 400°C. Commonly used solid lubricants are fine graphite powder (applied with base grease) and zinc stearate, preferred for processing high strength materials and at high temperature. The selection of the lubricant depends on the processing temperature.

3.5 Processing Temperature

The processing temperature plays crucial role in ECAP process by directly affecting the grain size achieved in ECAP. Selection of processing temperature depends upon the nature of material being processed. Generally brittle materials are difficult to process at lower temperatures. Processing of brittle materials at high temperature is necessary to avoid significant cracking in the samples. Processing at high temperature can be achieved in various ways. Simple way to process the material at high temperature is by heating the processing material in external furnace for sufficient time and at required temperature. After that, the processing material is moved to the die from the furnace. The heating time in the furnace depends on the size and cross-section of the processing material. In this case, there is no heating of the die set up and the die set up is maintained to room temperature. Since, the processing material has to be transferred from the furnace to the die set up, there will be heat loss in the processing material. Also, the die set up is kept in room temperature, there will be heat loss also during processing. Therefore, in this case, it is difficult to estimate the processing temperature. Another way to process the material at high temperature is by heating the processing material in the die set up itself instead heating in the furnace. In this case, entire die set up is heated along with the processing material. The heating time of the die set up depends on the size and cross-section of the processing material. In this case, there is no transfer of processing material for heating purpose, there will be no heat loss. Also, the die set up is heated continuously during processing; hence it is easy to estimate and control the processing temperature.

Various observations have been reported to understand the consequence of processing temperature on ECAP process. Yamashita et al., (2000) reported that, grain size after processing increases with rise in the processing temperature. It was also reported that, with the rise in the processing temperature, grain boundaries will have low angle of misorientations, recovery happens in faster rate and density of dislocation reduces. To develop grain boundaries with high angle of misorientations it necessary to process the material at least possible temperature. Optimal UFG microstructure will be achieved by processing the material at least possible temperature. Mainly, higher strength in the material is achieved if processing is conducted at least possible temperature and as the processing temperature is rises the strength achieved in the material declines. It can be concluded that, maintaining low processing temperature, both the UFG microstructure and large quantity of grains with high angle of misorientations could be achieved. Increase in the processing temperature also alters the deformation mechanism from parallel shear bands to twinning bands.

There will be additional heat generation (ΔT) in the specimen during processing of the material. Few works have been reported to measure the magnitude of additional heat generation during processing. There will be a rapid increase in the temperature of the processing specimen as it moves from the shear plane of the die, but this temperature subsequently decreases within short span. Pei et al., (2003) reported the analytical approach to forecast the increase in the temperature during ECAP processing; the observations are consistent with the experimental results. The effect of internal heating in ECAP processing is more while processing high strength materials. Internal heating increases with increase in strength of the material and decrease in channel angle 'Ω'. Internal heating also increases with increase in pressing speed.

3.6 Back Pressure

Back pressure refers to reverse pressure applied on the specimen at the departure end of the channel after it crosses the shear plane. Fig.4 illustrates the principle of ECAP with back pressure. Stolyarov et al., (2003) stated that workability of the processing material can be enhanced by applying back pressure. Also, applying back pressure reduces cracking in the intermetallics and enhances the ductility of the processed material. Application of back pressure is generally used to process hard and brittle materials. Frictional effects, dead zone formation and deformation zone shape change are reduced by applying back pressure during processing (Lapovok, 2005). Application of back pressure will also have considerable effect in grain refinement. Number of passes can also be increased by the application of back pressure. Significant benefit of back pressure is the visibility of the specimen movement while processing. Back pressure can be applied in different methods. An improved and more controlled method is to use secondary plunger from the departure end of the channel as shown in Fig.4. Back pressure can also be applied by increasing the friction in the departure end of the channel by using viscous-ductile material.

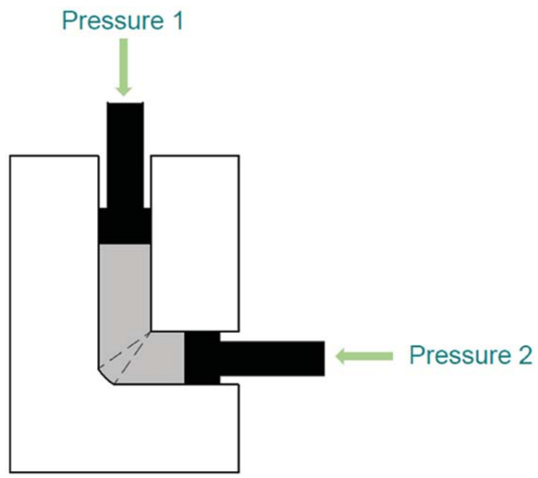

Fig.4: Principle of ECAP with back pressure

4.0 Summary

ECAP processing is a shearing process used to refine the microstructure of the metallic materials. In this technique, it is feasible to produce essential structures and textures by ensuring strict control over the processing conditions. This technique can be adopted to process different metallic materials, intermetallics and metal matrix composites. In this review article, a complete overview of the processing conditions in ECAP are discussed. The consequence of each parameter on the grain refinement and mechanical properties are discussed in detail. The parameters discussed are processing route, channel angle, outer arc of curvature, pressing speed, load and friction, processing temperature and application of back-pressure. The final summary of this article can be concluded as follows. Equiaxed grains can be produced in route B_C and it develops shearing over larger angular ranges compared to routes. Superior grain refinement is possible by reducing the die channel angle. Optimal grain refinement can be attained by pressing the specimen as least possible temperature. Proper lubrication is necessary for smooth processing. Workability of the processing specimen can be enhanced by the application of back pressure.

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