Comparison of different methods of Severe Plastic Deformation for grain refinement

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Abstract. The methods of severe plastic deformation are the procedures of grain refinement of bulk crystalline materials to ultrafine or up-to Nano crystalline structure followed by improvement in mechanical properties of different metals and alloys used for various automobile, aerospace and defence applications. In this research paper, discussion has been made about the comparison of different methods of severe plastic deformation for perfection in microstructure and subsequent changes in mechanical properties for industrial applications. The results achieved by Equal Channel Angular Pressing method are more useful for grain refinement by the reason of high imposed shear strain in the material. It has also been observed that using this method strength is also increased with ductility as desired in most of the cases. It can be concluded here that detailed study is still to be explored after processing the material after severe plastic deformation.

Key words: grain refinement, severe plastic deformation, High Pressure Torsion, Equal Channel Angular Pressing, Cycling Extrusion and Compression

Abbreviation table:
Severe plastic deformation: SPD
Equal Channel Angular Pressing: ECAP
Accumulative Roll Bonding: ARB
Twist Extrusion: TE
High Pressure Torsion: HPT
Cycling Extrusion and Compression: CEC
Multidirectional Forging: MF
Repetitive Corrugation and Straightening: RCS

1. Introduction:

Practically all the mechanical components bear either static or dynamic loading during service but most of the components fail due to dynamic loading in service and that is the reason good mechanical properties has been required. So in this research paper, our focus is to gather information regarding, to produce material of desirable properties so that these failures can be minimized. The SPD methods are used for reduction of the particle size which ultimately causes improvement in the micro structure and...
mechanical properties. Some important SPD methods include HPT, ECAP, CEC, ARB, MF, etc. In the last few years, there is a lot of interest in the scientific and technological fraternity has been developed in the microstructure and mechanical properties in the size range of micro meter and sub micro meter even at nanometer level of micro crystalline, ultra-fine grained and Nano crystalline materials. It is now the established fact that the application of SPD may be used to refine the grain size to the sub micro meter or even nanometer level of different metals and alloys [1].

Out of several other factors for the improvement in mechanical and physical characteristics of any material, the mean size of the particles of the different materials play the most important role in defining the strength of the material. The well-known Hall–Petch relation explains that strength of the material can be increased by decreasing the average grain size of the material and the relation for yield stress $\sigma_y$ and friction stress $\sigma_0$ with average grain size is given in equation (1):

$$\sigma_y = \sigma_0 + kd^{1/2}$$

Where, $k$ is the constant of yielding. This relation provides the method to improve the strength which is also known as grain-boundary strengthening. It is observed that the grain boundaries restricts the dislocation among the grains which produce a counter effect on the grain boundaries and allow the movement of grains from one place to another. Thus grain size influences dislocation movement followed by mechanical properties. More the applied stress required to move the dislocation, the higher will be the yield strength. This explains that grain boundary size and yield strength can influence the inverse relationship as demonstrated by the Hall-Petch equation [1–3].

The average grain size of ultra-fine grained (UFG) material, defined by poly crystals of fine grain size, lies within the range of 1 μm or less. There are basically two methods to produce UFG materials: one is known as bottom up method and the other is known as top down method. In case of bottom up method, the UFG materials are produced or manufactured by compacting or consolidating individual atoms or Nano particulate solids, which may be in the powdered form. The material produced by this method has certain degree of porosity or cavity and low level of consolidation which is induced in the material during manufacturing. In top down method, relatively bulk sample of material with coarse grain size is used to produce the UFG size material by the application of very high strain or impact loading. This method can be used for bulk materials unlike using small samples and contamination, which is the inherent feature of the bottom up method. This method can be easily used for large range of metals and alloys, which makes the additional advantage of this method.

2. Severe Plastic Deformation (SPD)

It is an established fact that the materials with UFG structure show the improvement in microstructure and other mechanical characteristics. To obtain the UFG structure from coarse grain structure, a very high strain is necessary to impose in order to introduce a high density of dislocations which ultimately causes to re-arrange to form an array of grain boundaries. UFG material can be efficiently and economically produced by SPD methods which may be explained by the metal forming techniques in that an extreme level of strain acts on bulk samples while keeping almost the same dimensions of the material which leads to the formation of UFG material. As there is no change in the overall size of the material during the operation, the method may be applied several times to impose very high strains.
Several methods are available for attaining UFG material up to nano crystalline materials. Earlier thermo mechanical processing (TMP) methods were used for obtaining the appropriate small grain sizes. However, TMP methods are limited to produce materials with grain sizes in range of 1-10 μm and further refinement of grain sizes is achieved by heat treatments and some particular processing which is quite uneconomical. Due to this limitation TMP methods are generally not considered during the bulk production of UFG materials. Later on, numerous other methods such as high-energy ball milling, sliding wear and inert gas condensation were introduced for UFG materials. These methods were observed to be more effective to produce grain sizes even to the certain nano meter level but lacks involvement of solidification of Nano powders to attain a solid sample and hence not accomplished of producing large samples in a completely-dense condition as well as for industrial applications.

In the recent past several other methods were accepted for developing ultra-fine grained materials via SPD. The advantage of using SPD is that it yields refined grains at very low temperature. The final product thus obtained shows high angled boundaries grains with improved mechanical characteristics. Moreover SPD also yields components with high densification and therefore can be used in various automobile, aerospace and defence applications. For the conversion of coarser grains to UFG, it is important to implement a high value of strain so as to obtain highly dense dislocated particles which form an agglomeration at the grain boundaries. SPD produces UFG and thus is defined as a forming technology in which a high value of strain is implemented on a bulk sample without much change in the overall dimension of the workpieces that also leads to the refinement of grains. Since there is no change in dimensions of components during SPD technique, it can be used in very high strain rates. The important severe plastic deformation methods which are already in usage for the production of UFG materials are ARB, MDF, HPT, ECAP, TE, etc. The concept and process of different SPD methods are discussed in the following sections.

3. High Pressure Torsion

High pressure torsion (HPT) method is considerably more recent method used for grain refinement in metal processing, first introduced by Percy Bridgman.

![Concept of HPT Tool with material disc placed in the cavity](image)

Fig.1 Concept of HPT Tool with material disc placed in the cavity [1,2]

Process of this method involves, the material in the form of disc is placed between the two anvils, the lower one is known as support anvil and upper one is known as top anvil, and a very large torsional
stress in the range of several GPa is applied under high hydrostatic pressure, where the top anvil is rotated to create a torsion force. In this method, the sample in the form of disc may be located within a cavity of support anvil or in the cavities of both lower support and anvils [4–9]. Fig. 1 shows the concept of HPT where the sample is located within a cavity of the tool, where plastic torsional straining is achieved by rotation of one of the anvils through high hydrostatic pressure.

4. Accumulative Roll-Bonding

In this technique, founded in late 90’s, the two rolled sheets of the same material are stacked, heated below the recrystallization temperature, rolled and bonded together to form a single sheet. These sheets are then divided in two halves and then are stacked, followed by a number of attempts. In comparison to the other technique, this technique proves to be more beneficial as it does not involve any specific equipment but only a tool room rolling mill is required. In this technique the two counter parts which are to be joined should be cleaned and mirror polished before rolling so as to give better bonding strength. Fig. 2 shows the ARB technique where sheets are cut, stacked and rolled [10-14].

Fig. 2 The concept of Accumulative Roll-Bonding where sheets are cut, stacked and rolled [3]

5. Multi-Directional Forging

Fig. 3 illustrates the MDF which was initially used in 1990’s with an objective of grain refinement for bulk materials. In order to get large strain with less variation in original dimension this technique makes use of bulk material. In This technique, continuous setting and drawing is carried out in three mutually orthogonal directions. Deformation temperature is an important factor for the refinement of grains. MDF with high temperature is used for the grain refinement in brittle samples. In earlier study, it was found that multi axial forging (MAF) on aluminum alloy yields similar properties as compared to ECAP technique. It was also found that the values of micro hardness decreased with an increase in values of accumulated strain. However, the homogeneity in strain is developed by MDF is found to be less as compares to ECAP and HPT [15-18].
6. Twist Extrusion

This is a recent SPD technique. In this, the billet of bulk material formed generally in the shape of a square or rectangular rod is allowed to pass through an extrusion die whose dimension will not change when it is twisted through a desired angle along its entire longitudinal axis. It is carried out under high values of hydrostatic pressure in the center of deformation. Fig. 4 shows the idea of twist extrusion where the shape of workpiece before entering, inside the die, after exiting the die is illustrated. In this method, the sample attains its original dimension after passing through TE technique which allows the repetition of the process several times in order to get superior refinement in grains. By using this technique various cross sectional shapes can be achieved apart from a circular geometry [19-23].

7. Cyclic Extrusion and Compression

CEC technique, also called as hourglass processing, is one of recently developed technique used for producing ufg and nano materials. In CEC, billet material is pushed from one chamber to another chamber having equal dimension via a die with a diameter \( d_{in} = d_{0}^{0.23} \) which is smaller than \( d_{0} \). This technique reduces the grain size of the billet from micrometer to nanometer level. Fig. 5 shows the
concept of CEC where the material is pushed through the cylindrical chamber. As the name suggests the complete processing is done in two steps, first extrusion followed by second compression [24-28].

Fig. 5 The concept of CEC where the material is pushed through the cylindrical chamber [2]

8. Repetitive Corrugation and Straightening

It is a relatively easy technique of SPD which is used for manufacturing of ufg sheets in large quantities. In this technique, the workpiece is deformed to a corrugated form and later on straighten between two parallel plates which imparts high value strain to the samples which leads to refinements of grain, model of continuous RCS can easily be adopted from any manufacturing industry which can yield metallic sheets of fine grains. The RCS facility subjected to both shear and bending, which results grain refinements. Merit of using RCS is that it can easily be used for present industrial rolling technique to yield high amount of fine grained sheets of metals. This technique has strong potential of developing nano structured materials in a continuous and economical manner. Fig. 6 shows the RCS technique where the material is corrugated and straightened [26].

Fig. 6 The concept of RCS where the material is corrugated and straightened [1]

9. Equal Channel Angular Pressing

The ECAP, also recognized as equal channel angular extrusion (ECAE), is one of first methods of SPD, was first introduced by Segal and his team members in early 80’s at an institute in Minsk in the former Soviet Union. In this process a well lubricated sample of material is pressed by plunger, to pass it through two crossing channels meeting at an oblique angle, which is known as die channel angle $\Phi$,
in a special die and plastic strain is imposed by simple shear at the intersection of the channels. As the billet passes through the point of intersection of the two channels a shear strain is introduced. Unlike conventional deformation processes such as rolling, forging, drawing, etc., in this method, as the billet dimensions remain unchanged, the pressings may be repeated, in order to achieve exceptionally high strains with homogeneous equiaxed grains. The Fig. 7 (a) below gives an idea about how the sample is being passed and Fig. 7 (b) depicting the plane of shear within the die through which the sample is being passed and Fig. 8 Showing the concept of channel angle $\phi$ and curve angle $\Psi$ through the die [24].

![ECAP Concept](image)

Fig. 7 Concept of ECAP (a) Schematic of ECAP facility (b) plane of shear within the die: as indicated in the lower part 1 and 2 numbered element is shear transposed [9]

![ECAP Concept](image)

Fig. 8 concept of ECAP showing channel angle $\phi$ and curve angle $\Psi$ [1, 9]

This method exhibits four basic processing routes consisting of different slip systems all through the pressing operation resulting in different microstructures. Fig. 9 illustrates the four different processing routes available in ECAP. In route A the sample is pressed without rotation, while in route $B_x$ the rotation of sample occurs by $90^\circ$ in alternate directions (clockwise and counter clockwise) between
successive passes. The sample is rotated by 90° in the same sense between each pass in route B, and finally in route C the sample is rotated by 180° between passes [13, 16, 17, 18].

Fig. 9 the four fundamental processing routes in ECAP [30]

For grain refinement through ECAP, the billet material is pressed through a special die in which two channels of equal cross-section intersect at channel angle Φ and corner angle Ψ, of the intersection of the two channels [27-31]. In the frictionless situation between the billets and the die walls, the strain accumulated after N pressings through the die will be given by the following relationship in equation (2):

$$\varepsilon_N = \frac{N}{3} \left[ 2\csc \frac{\phi}{2} + \frac{\Psi}{2} \right]$$

(2)

During the pressing operation since the cross-section of the pressed billet remains constant after pressing that permits to press the same billet through the die a number of times in order to achieve a high total strain [31]. The above relationships can be directly applied to the center of the billets but not valid at areas away from the center of the billets due to friction [28].

10. Results and Discussions:

From the above study and contributions of other researchers, following advantages of ECAP over other SPD techniques are observed: (a) ECAP may also be applied to sufficiently large size billets for fabricating samples which can be used for wide engineering applications. (b) It is comparatively simple procedure which can be straightforwardly accomplished on a wide range of alloys apart from die construction. Moreover, ECAP technique uses equipment that is readily available in most of the laboratories. (c) This technique is used for large variety of crystalline materials having different crystalline structures. (d) As the material billet can be processed repetitively in the process, a sufficiently high strain of reasonable homogeneity can be obtained. (e) This technique can be used for manufacturing of a large size samples. (f) Strain can be imposed in this process without changing the dimensions of the sample.

Figure 10 (a-d) showed the optical microstructure of ECAPed experimental Aluminium Alloy AA6063 material up to 3 passes with magnification at 500X. The testing samples were prepared and micrographs were observed at room Temperature of 27°C. The etchant used was Keller’s Reagent which is a mixture of nitric acid, hydrochloric acid and hydrofluoric acid, used to etch aluminum alloys to reveal their grain boundaries and orientations. The average grain size was reduced to very high level after 3 passes to 7-8 at 100x magnification as per ASTM standards (from 456nm to 361nm
after three ECAP Passes). Figure 10(a) showed the micrograph of as received material where dark spots were visible as alloying elements and Figure 11(b) explained that the grains were refined after single pass of ECAP. Further Figure 10(c) illustrated that the grains were elongated in one direction after second pass and Figure 10(d) showed the further refinement of grains with homogeneous and equiaxed grain structure. The die used for ECAP technique was made up of H13 tool steel with channel section d = 20 mm, channel angle Φ=135°, corner angle Ψ=20° and route Bc in which the specimen was rotated 90° in clockwise direction in succeeding number of passes at room temperature of 27°C. The size of sample used was 20 mm diameter and length 100 mm.

![Figure 10](image1.png)

![Figure 11](image2.png)

Fig. 10 Microstructure of aluminium alloy after (a) 0, (b) 1, (c) 2 and (d) 3 ECAP passes

11. Conclusions:

The study of different methods of SPD was concise in this research paper and it has been concluded that the ECAP is the utmost efficient and economical method which can be utilized for refining the grains in bulk industrial materials. The attractiveness of this technique is based on its aptness for wide range of shapes and sizes of the billet material and the only limitation of this technique is initial high
initial cost involved for Die design and fabrication. The final effect produced by ECAP technique is homogeneous and equiaxed grains in the billet material.

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