

# Overview of Bulk Nanostructured Material by Severe Plastic Deformation(SPD)

Kanika Parkash,

Department of Materials and Metallurgical Engineering,  
PEC University of Technology  
Chandigarh , India

Dr. Mamta Sharma,

Department of Materials and Metallurgical Engineering,  
PEC University of Technology  
Chandigarh India

**Abstract** ---The quest for ever-higher performance in structural applications has resulted in the ongoing development of new processes that can improve material properties. Severe plastic deformation methods are used to convert coarse grain metals and alloys into ultrafine grained (UFG) materials. It is one of the successful top to bottom technique for obtaining UFG materials then possess improved mechanical and physical properties which destine them for a wide commercial use. This paper, in one direction, looks into historical development of SPD processes and their effect at obtaining fine crystalline structure

**Keywords** --- SPD, ultrafine/nano structure, bulk nano structure material.

## I. INTRODUCTION

For a long time in the world of science, nanoscience is topic of great intriguing. It is the next leap in science and technology. It is seen that when the grain size is reduced to ultrafine/nano size range, the physical, chemical and mechanical properties are enhanced. With this view in the mind scientists have been researching for various methods of developing bulk nanostructured materials (BNSM). In particular severe plastic deformation (SPD) is one of the easy method to develop BNSM. It is also an alternative to existing nano powder compaction. SPD also helps in overcoming number of difficulties connected with residual porosity in compacted samples, impurities from ball milling, processing of large scale billets and practical applications of the given materials. The ultrafine/nano structure produced by SPD are of high angle grain boundaries. SPD gives very large deformations at relatively low temperatures.

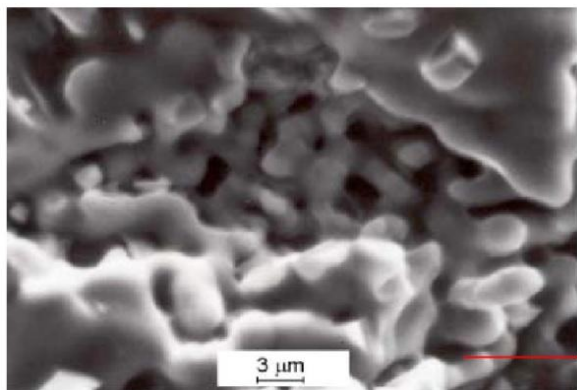


Figure 1: Scanning electron micrograph of the microstructure of a sintered vanadium carbide nanopowder. Highly dense sintered conglomerates and the free space between them with the size of up to tens of micrometers are clearly visible[1][2]

The first research and development in the area of SPD was done by Valiev and co workers in 1977 in Russia. The strength of metallic materials can be improved by grain refinement, and this has been very well explained by the Hall-Petch relationship.

Hall-Petch relationship is the mathematical relationship between the yield strength and grain size. the relation says that as the grain size decreases the strength of the material increases. The equation is as follows:

$$\sigma_y = \sigma_i + k d^{-1/2}$$

where  $\sigma_y$  = yield strength of the material,  $\sigma_i$  = yield strength for a single crystal of the same material having no grain boundaries.

In recent years , a vast development in the research has taken place and we are close to the day when BNSM will be commercialized.

## II. FORMATION OF NANOSTRUCTURES AND METHODS OF SEVERE PLASTIC DEFORMATION

R Z Valiev , R K Ismagaliev and I V Alexandrov stated that to obtain the ultrafine grained structure/nanostructured material. Three requirements are need to be fulfilled. Firstly, obtain structures(ultrafine/nano) prevailing high angle grain boundaries, since only a quantitative changes in properties of material occur. Secondly, formation of uniform structure throughout the whole volume of sample, providing stable properties of the processed material. Lastly, material that is SPD should not have any damages and cracks[3]. There are various methods are for the development of ultrafine/nanograined structures, viz., equal channel angular pressing/extrusion, accumulative roll bonding, high pressure torsion, angular twist, Cryorolling multiaxial forging, [4] constrained groove rolling (CGR), repetitive corrugation and straightening (RCS) and so on. Among them, the last two processes are especially applicable for continuous production of sheet or strip type products. These methods are based in principle on CGP method (large shear deformation in work piece is realized by its repetitive grooving and flattening between plate shaped dies) in which flat dies are replaced with grooved and flat rolls. ]

## III. EQUAL CHANNEL ANGULAR PRESSING

A lot of research has been done on ECAP. Severe plastic deformation (SPD) process has developed by Russian scientists. It is a new method of manufacturing bulk specimens having ultrafine grained (UFG) microstructure.

So far it is the most viable and frequently used method. It is based on simple shear that takes place at the cross section of the sample. The aim is to produce large strain without changing of the cross section area of the material. It requires low tool pressure and low force as compared to high pressure torsion, that is discussed later. Its tooling is easy. The relationship between the strain applied and structure development is:

Shear strain,  $\gamma = \sqrt{(3P/Y)}$  where,  $P$  = process pressure.

In this the sample is passed repeatedly in the same die. the sample may be rotated about its axis in between each passes.

The basic rotations are  $A$ ,  $B$ ,  $B_A$ ,  $B_C$  [5] are shown in the figure 2 below.[5]

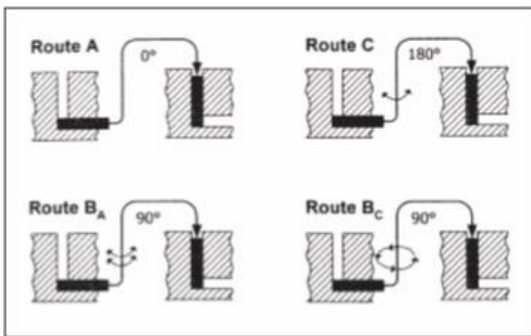


Figure2: Options for billet rotation between consecutive passes through ECAP die

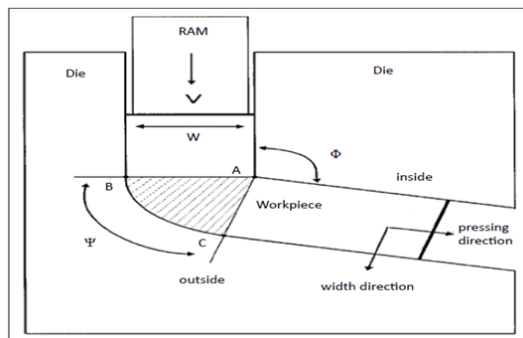


Figure 3: A schematic illustration of the equal channel angular pressing setup showing the die channel angle ( $\Psi$ ) geometry, the plunger used to press the material (RAM), the pressing direction ( $V$ ), the width of the material ( $W$ ) and the pressing and width direction.[6]

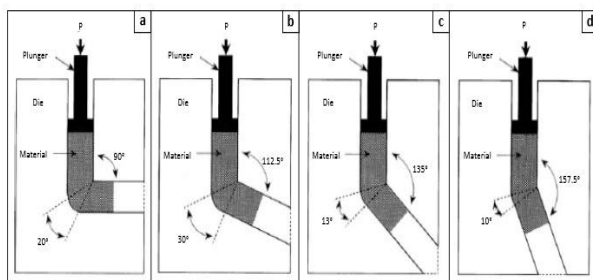


Figure 4: Schematic illustrations of the die used to evaluate the influence of the channel angle: (a) 90°, (b) 112.5°, (c) 135° and (d) 157.5°.

For obtaining homogenous microstructure of the equiaxed grains separated by high angle grain boundaries the best route is  $B_C$ . It does not involve high pressure which is advantage from the machine and tooling point. But this process is not suitable for brittle materials. Small pressure and high temperature is required. Sometimes the fracture is

inevitable in brittle material. In ductile material a bit high pressure is required to avoid accumulation of dislocation. In this system clearance between the die and the sample is necessary to avoid friction wear and heat development. The cross section diameter or its diagonals should not exceed 20mm. The length varies from 70mm- 110mm. In ECA the direction and the number of passes are very important for the microstructure refinement.

#### IV. High pressure torsion (HPT)

HPT was first investigated by Bridgeman. But Bridgeman experiment did not gave much result about the microstructure changes. Therefore this device is based on Bridgeman anvil type device[7]. Erbel carried first experiment on copper, in this an ingot is held between anvils and strained in torsion under the applied pressure of several GPa. The lower holder rotates and the ingot is deformed by shear force. The hydrostatic pressure is approximately 7 GPa.

True logarithmic strain,  $\epsilon = \ln(l_0/l)$  where  $\theta$  = rotation angle in radians,  $R$  = radius of disk,  $l$  = thickness of disk.

$\gamma = 2\pi RN/l$ ,  $N$  is the number of rotations,  $l$  is the thickness of the sample

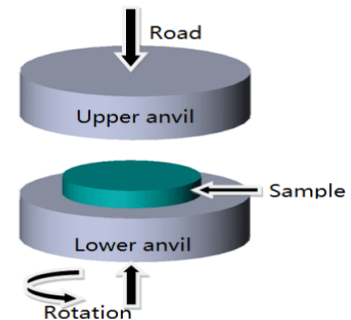


Figure 5: Schematic diagram of high pressure torsion tooling

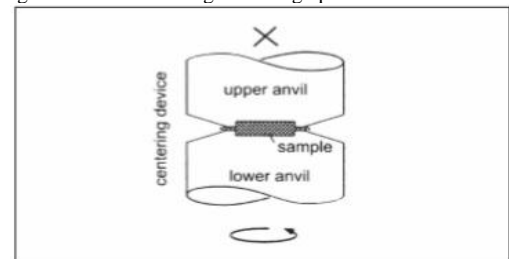


Figure 6: Principle of HPT experiment. [5]

Study shows that high pressure torsion can be used successfully for grain refinement and also for the consolidation of powder. It gives most efficient grain refinement, as close as 100% high density. It can be imposed on any type of materials or powders. It is also seen that the absence of porosity and the mean grain size after the process reached to 17nm and therefore large volume fraction of grain boundaries is present. Studies reveals that the micro hardness has improved considerably. Vorhaver and Pippan explained that the discrepancy that it is virtually impossible to realize and ideal HPT deformation due to misalignment of the axes of the anvils. Moreover the deformations are non uniform.[8]The drawbacks is that the sample size is very small typically 10-15mm in diameter and 1mm in thickness can be processed [8] therefore, samples only for research can be prepared.

## V. MULTIPLE FORGING/ MULTIAXIAL FORGING(MAF)

This method was developed by Salishchev. The process of multiple forging is associated with dynamic recrystallisation. Multiple forging is the simple process in which the free forging is repeated in three orthogonal direction, setting drawing with the change of the axis of the applied strain load. The homogeneity is lower than ECA and HPT. But in MAF it is relatively possible to obtain nanostructure in brittle materials because the processing starts at high temperature and specific loads on tooling are low[8]. It has been used for numbers of alloys like Ti, titanium alloys, Mg alloys, nickel based alloys and others. Forging is done at  $0.1-0.5T_m$  ( $T_m$  is the melting temperature of the sample) grain refinement during multiple is usually associated with dynamic recrystallisation.[3]

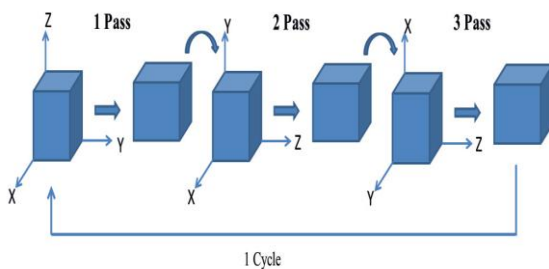


Figure 7: Schematic illustration of Multi axial forging for 1 cycle [8]

Initially large plastic strains accumulates internally and the strain distribution and accumulation of strain at the internal strain distribution is not uniform. But the refinement is possible with multiple cycles. It is a potent technique for manufacturing large size billets with ultrafine grain structures. It can be successfully applied to wide range of materials.

## VI. TWIST EXTRUSION

It based on simple shear deformation process. This process was introduced by Beygelzimer et, in late nineties[8]. In this process the billet/sample is extruded through a twist die. It also has the same problems a HPT. In this too the deformation is non uniform being lowest near the extrusion axis. It has advantage that it is highly efficient in producing ultrafine grain structure.

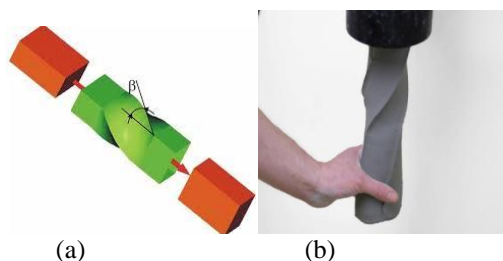


Figure 8: (a) Twist angle, (b) Tooling for twist extrusion [9]

## VII. ACCUMULATIVE ROLL BONDING

ARB was first introduced by Satio. This process is capable to overcome the limitations of the ECAP and HPT i.e. The low productivity of former and small sample size in later. It is a low cost process as it uses the conventional rolling facility. In this process the metal sheet is rolled to 50% thickness reduction[8], then, the rolled sheet is cut into half and stacked together so that the original thickness remains same. The contact faces degreased and wire brushed before stacking. This process can be applied to wide range of materials like Al, Mg Al-Mg based alloys.

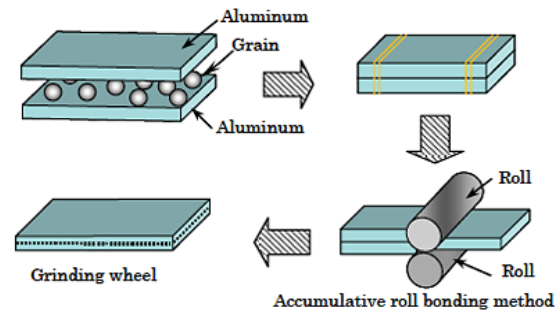


Figure 9: Schematic illustration of process of aluminum-bond grinding wheel for ELID grinding using accumulative roll bonding method ref [10] is taken from internet

## VIII. Rolling

Though this process is not widely used. But this is an effective process to produce ultrafine grain /nano structure. The process is relatively cheap as it needs conventional rolling facility. The sample/billet is passed between the rolls, and it is repeated number of times depending upon the material. Up to 90% reduction in the grain size can be achieved. This method can be applied to many materials and alloys like Al and Al alloys, Cu and Cu alloys, titanium, magnesium alloys. It is an effective process in terms of efficiency.

But brittle materials are hard to roll. For this, warm rolling is done. In warm rolling the sample is heated for 10-15mins prior to rolling, and the process the repeated for each pass.

### A. Cryorolling

cryorolling is the advanced rolling process to produce UFG/nano structures. The sample is dipped in the liquid nitrogen prior to rolling. Dipping billet in nitrogen at very low temperature leads to suppression of dynamic recovery and restoration of dislocation resulting in the accumulation of high dislocation densities in the materials. It can produce UFG structure in high stacking fault energy it has seen that there is an optimum stacking fault energy where cryogenic deformation [11] nitrogen dipped materials shows better me mechanical properties compared to normal rolling.

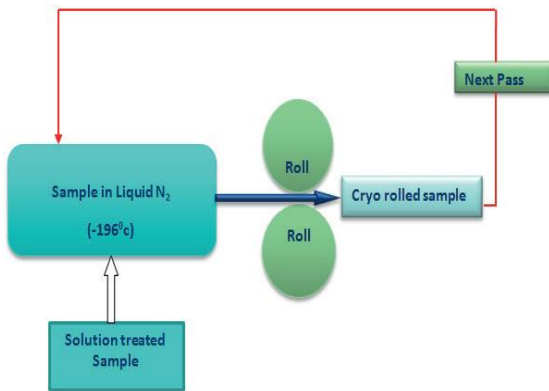


Figure 10: Schematic illustration of principle of rolling at liquid nitrogen temperature

#### IX. REPETITIVE CORRUGATION AND STRAIGHTENING METHOD(RCS)

Continuous repetitive corrugation and straightening process has been conducted by drawing of the strip through toothed rolls (corrugation) and plain rolls (straightening) set mechanism of SPD. All rolls were assembled in a die set which provided possibility for clearance control of rolling gap. The method is based on the principle of CGP method. This method induce a large shear deformation. Here instead of flat dies, grooved and flat rolls are used to ensure uniform deformation the strip/sample is turned by 180 degrees before each cycle of corrugation and straightening. This is especially applicable for continuous production of strip or sheet like product. The process has high efficiency and ultrafine/nano structure is achievable. The obtained grain size and character largely depends upon the material.[12][13]

#### CONCLUSION OF SPD

SPD was initially developed for processing bulk materials as an extension of conventional metalworking techniques can also be used for other purposes, such as efficient compaction of powders, particularly for producing alloys from blended elements[14][15]. Structural changes in materials due to SPD and their properties have been studied by various methods for long time. Many methods have been evolved in these years. A continuous efforts are going on in this direction in order to understand and control SPD effect. It is evident from the literature survey that there are a great variety possible in SPD processes. The problem and limitation of use of SPD is that still it is not applicable on fabrication of massive ingots of larger dimensions having enough homogenous structure. An new application of SPD processing, which can give scope to SPD methods, was suggested in Ref. [16]. It has been to produce simultaneous architecturing and nanostructuring of hybrid materials by using established SPD techniques. In particular, twist extrusion, HPT and some other methods.

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