

Development of Mechanical Properties by Severe Plastic Deformation Methods

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Abstract: Ultrafine Grain Materials are known for their extraordinary Mechanical Properties. Severe plastic deformation of materials produces ultrafine grains in Materials. Various Severe Plastic Deformation (SPD) techniques have been developed to produce bulk UFG material having homogenized and equiaxed structure .The extensively used SPD techniques are Equal channel angular pressing (ECAP), High pressure torsion (HPT), Accumulative roll bonding (ARB) and Multi axial forging (MAF).Various Aluminium, Magnesium and copper alloys have been converted so far to ultrafine grain material by using these techniques but there occurs a difference in shape and size of the samples obtained from each method . Refinement by SPD is based on the formation of dislocation cell or subgrains during large plastic strains and evolution of these cells into array of ultrafine grains separated by high angle boundary. This paper gives overview of the available technique along with parameters which influence these techniques. This paper also looks into the application of these techniques along with future challenges associated with techniques.

Keywords: UFG Material ,SPD Methods, ECAP, TE.

I. INTRODUCTION

Grain size plays a important role in deciding the peculiar properties of material like strength, toughness ,hardness, corrosion. A fine-grained material (one that has small grains) is harder and stronger than one that is coarse grained, because the former has a greater total grain boundary area to impede dislocation motion. For many materials, the yield strength σ_y varies with grain size according to

$$\sigma_y = \sigma_0 + k_y d^{-1/2}$$

In this expression, termed the *Hall–Petch equation*, d is the average grain diameter, and σ_0 and k_y are constants for a particular material.Two basic and complementary approaches have been developed for the synthesis of ultrafine grain materials. These are known as “bottom up” and “top down” approaches. In the bottom up approach, atoms, molecules and even nanosize particles themselves can be used as the building blocks for the formation of complex nanostructures whereas, in the “tops down” approach coarse-grained materials are refined or converted into ultrafine -grained /nanostructured materials. Ultrafine grained (UFG) materials may be defined as polycrystalline materials having average grain size less than $\sim 1\mu\text{m}$. The grain sizes of UFG materials may lie within the sub-micrometer (100-1000 nm) range

In order to convert a coarse grained solid into a material with ultrafine grains, an exceptionally high strain is imposed in order to introduce a high density of dislocations which, in turn, re-arrange to form an array of grain boundaries with increase in strain. Conventional metal forming techniques like extrusion or rolling, are restricted in their ability to produce UFG structures for

two reasons. First, there is a limitation on the overall strains that may be imposed using these techniques because for large strain to develop using these techniques there will be corresponding reduction in cross section of work pieces .Second, the strains imposed in these techniques are limited and insufficient to give rise to UFG structures because of the generally low workability of metallic alloys at ambient temperatures. As a consequence of these limitations, there is a shift in the synthesis approach for nanoscale materials and alternative processing techniques have been developed. The severe plastic deformation (SPD) technique is one of such processes, where extremely high strains are imposed at relatively low temperatures . With severe plastic deformation methods ,submicron and even nanocrystalline metals and alloys can be easily produced if the fabrication process is compared to other available techniques to manufacture nanocrystalline materials.The formal definition of SPD is given as “*Any method of metal forming under an extensive hydrostatic pressure that may be used to impose a very high strain on a bulk solid without the introduction of any significant change in the overall dimensions of the sample and having the ability to produce exceptional grain refinement*” .[1]

Structures obtained during SPD have specific features: Small size of grains down to nanolevel, low density of free dislocations, high angle misorientation of these grains, and high energy and nonequilibrium state of grain boundaries [3]. These structures lead to changes in physical and mechanical properties: a significant increase in the strength at good ductility, an increase in the wear resistance, and high-speed and low temperature superplasticity [3]

The development of the principles underlying SPD techniques goes back to the pioneering work of P.W. Bridgman at Harvard University in the 1930s. This work concerned the effects on solids of combining large hydrostatic pressures with concurrent shear deformation and it led to the award of the Nobel Prize in Physics in 1946.[3] Very successful early implementations of these principles, described in more detail below, are the processes of equal-channel angular pressing (ECAP) developed by V.M. Segal and co-workers in Minsk in the 1970s[3] and high-pressure torsion, derived from Bridgman's work. What makes SPD so popular is excellent strength and properties achievable through this technique by a factor of 50-60% in aluminium and copper alloys but despite the impressive property improvement achievable with SPD techniques, their uptake by industry has been rather slow.

II. SPD EXPERIMENTAL TECHNIQUE

Methods of severe plastic deformation should meet a number of requirements which are to be taken into account while developing them for formation of nanostructures in bulk samples and billets. These requirements are as follows. Firstly, it is important to obtain ultra fine-grained structures with prevailing high-angle grain boundaries since only in this case can a qualitative change in properties of materials occur. Secondly, the uniform formation of nanostructures within the whole volume of a sample is necessary for providing stable properties of the processed materials. Thirdly, though materials are exposed to large plastic deformations they should not have any mechanical damage or cracks. Traditional methods of severe plastic deformation, such as rolling, drawing or extrusion cannot meet these requirements. Formation of nanostructures in bulk samples is impossible without application of special mechanical schemes of deformation providing large deformations at relatively low temperatures as well as without determination of optimal regimes of material processing. Following methods /techniques are capable of producing such results.[17]

A. ECAP-(EQUI CHANNEL ANGULAR PROCESSING)

Equal-channel angular pressing (ECAP) is the most studied SPD processing technique. It is an interesting and demandable method for modifying microstructure in producing ultrafine grained (UFG) materials. A schematic diagram of ECAP is shown in figure 1(a). below. In this process a rod/square shaped billet is pressed through a die constrained within an equal channel cross section, which is bent at an abrupt angle. A shear strain is introduced when the billet passes through the point of intersection of the two parts of the channel. Since the cross-sectional dimensions of the billet remain unchanged, the pressings may be repeated to attain exceptionally high strains. The equivalent strain ϵ , introduced in ECAP, is determined by a relationship incorporating the angle (Φ) between the two parts of the channel and the angle (Ψ) representing the outer arc of curvature, where the two parts of the channel intersect. The relationship is given as[2]

$$\epsilon = \frac{N}{\sqrt{3}} \left[2 \cot \left\{ \left(\frac{\phi}{2} \right) + \left(\frac{\psi}{2} \right) \right\} + \psi \cos ec \left\{ \left(\frac{\phi}{2} \right) + \left(\frac{\psi}{2} \right) \right\} \right]$$

Where N is number of passes through die

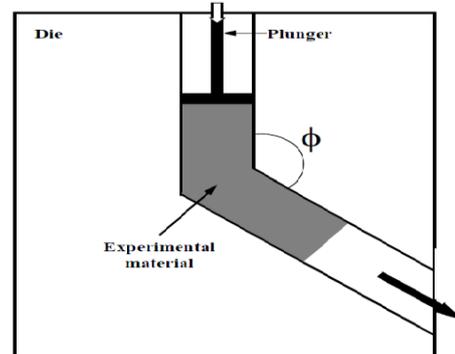
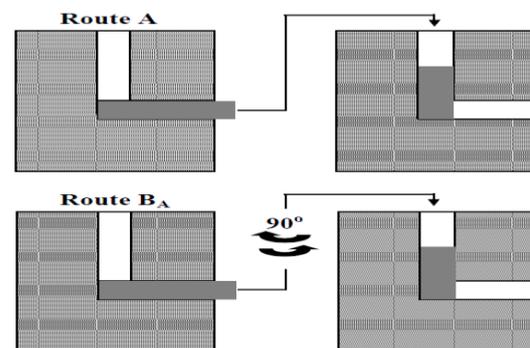


Fig.1.Schematic Diagram showing ECAP Technique

A.1 Processing routes in ECAP:

There are four basic processing routes in ECAP and these routes introduce different shear routes during the pressing operation so that they lead to significant differences in the microstructures produced by ECAP [4]. The four different processing routes are summarized schematically in Fig - 1(b). This shows that the sample is pressed without rotation (route A), the sample is rotated by 90° in alternate direction between consecutive passes (Route B_A), the sample is rotated in same sense (either clockwise or anticlockwise between each passes (Route B_C) and the sample is rotated by 180° between passes (Route C). Various combination of these routes are also possible such as combining route B_C and C by alternating rotations through 90° and 180° after every pass, but in practice the experimental evidence obtained to date suggests that these more complex combinations lead to no additional improvement in mechanical properties of the as-pressed materials. Some of the experiments performed have demonstrated that Route BC is an excellent processing route for producing equiaxed ultra-fine microstructures [3,4]



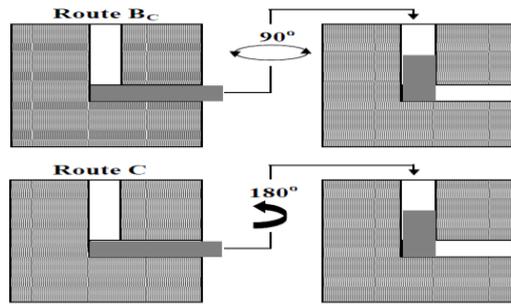


Fig.2. Four fundamental processing routes in ECAP

When materials are processed using ECAP, several different factors influence the workability and the microstructural characteristics of the as-pressed billets. These factors can be enumerated as follows: The angle between channels of the die (Φ), The angle of curvature (Ψ), The pressing speed of the punch in the die, The material temperature, Internal heating during ECAP, Back pressure on the punch.

Libor Krausa, et al. [2] studied the effect of temperature on mechanical properties of low carbon steel AISI1014 processed by ECAP at different temperatures 150°, 200°, 250°, 300° C and find that processing initially with increase in temp increases strength, at 200° shows max increase in yield strength and UTS and after that UTS and YS shows decrease. Kazeem O. Sanusi [3] studied the effect of number of passes on the hardness and microstructure on copper alloy and reported grain size refinement and increase in hardness value with increase in no. of passes. F. Djavanroodi et al. [4], studied effect of strain distribution on pure aluminium by increasing number of passes and concluded that For route A ECAP, the magnitude of strain distribution increases with increasing pass number i.e. less strain distribution homogeneity is achieved using route A by increasing pass numbers. [4]

A.2. Applications of ECAP:

- High-strength semi-finished products produced from aluminium that has undergone ECAP can be used in the aerospace, power and automotive industries. These products include fasteners like screws, screw rivets used in the assembly of aluminium components for aircraft and other structures, elements for aircraft fuselages (stringers, skin plates, etc.).
- Sections of various size and shape and sheets for assemblies operating in corrosive environments and at cryogenic temperatures, and complex-shaped parts produced by ECAP. [3]

B. High Pressure Torsion

HPT technique is based on the use of Bridgman anvil-type device. For this method, a coin-shaped sample is pressed between two anvils under hydrostatic pressure (7 GPa). During the build-up of the pressure, the sample is pressed into the cavities in the anvil and a burr is formed at the edge of the sample. Then one anvil is rotated with

respect to the other one and the rotation speed can be varied over a large range.

This leads to a deformation of the sample by almost simple shear. The burr prevents a contact between the two anvils and upholds the hydrostatic pressure. Due to the high pressure, in most metals the formation of cracks is suppressed and therefore it is possible to apply very high strain without failure of the deformed material. Up to five rotations of the anvil are usually enough to form a homogeneous microstructure with the grain size typically about 100 nm, in some metals and alloys with high melting temperature as small as 50 nm. This method enables the preparation of disc-shaped samples with diameter up to 20 mm and thickness about 0.2 mm, which are good for fundamental studies of the structure-property. The reached shear strain γ is a function of the twist angle ϕ , the radius r (of the site of investigation) and the thickness t . The strain can be expressed in terms of equivalent von Mises strain by dividing the shear strain γ by $\sqrt{3}$. The equivalent von Mises equivalent shear strain ϵ_{eq} as a function of the number of turns n is then given by [5]

$$\epsilon_{eq} = \frac{2\pi n r}{t\sqrt{3}}$$

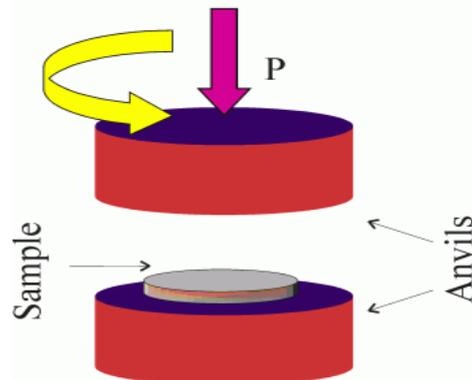


Fig.3. Schematic Diagram of Twist Extrusion

A handicap of the method is that only small coin-shaped samples, typically 10–15 mm in diameter and 1 mm in thickness, can be processed. Because of size restrictions, the samples manufactured by HPT are used primarily for research purposes.

C. Twist Extrusion

Under TE, a billet is extruded through a “twist die”. Each billet’s cross-section is deformed as follows: at first, it becomes twisted to some angle in one direction, and then – re-twisted to the same angle in the opposite direction. After each pass of the TE processing the billet’s form and dimensions are maintained. This achieves severe values of strain to accumulate in a billet with no form changes. In some papers it is shown that each physical cross-section of a billet is deformed in the same way as a thin disk during HPT processing, in the first approximation. Initially torsion to some angle in one direction is achieved, and then re-torsion to the same angle in the opposite direction,

i.e. the deformation is cyclic with an amplitude of a quasi-monotone part equal to half the full strain. For the dies normally used, the accumulated strain per pass is about 1.2. As the billet's form does not change during the processing, it is possible to deform it repeatedly in order to accumulate strain,

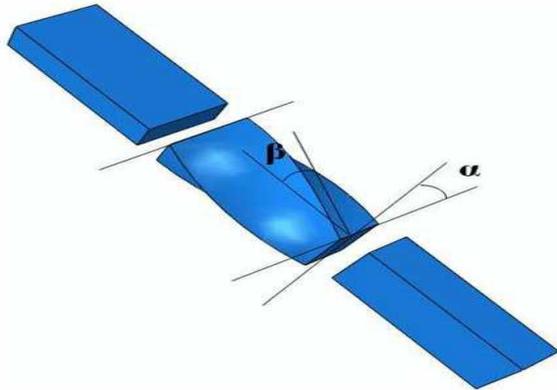


Fig.4. Schemes of Twist Extrusion Processing

Where α is rotation angle and β is slope angle. Ranjbar Bahadori et. al[6] concluded the effect of twist extrusion at room temp and thereafter cryorolling rolling on Pure aluminium and find that grain size produced after rolling on TE samples produces finer grain size of $80 \mu\text{m}$ stretched along the rolling direction than only TE sample which comprises grains of $150 \mu\text{m}$. The effects of repeated TE passes were examined in previous experiment by Ranjbar Bahadori et. al. which shows performing two and three TE passes the mean grain size decreases from $859 \mu\text{m}$ to $100 \mu\text{m}$ and $65 \mu\text{m}$, respectively. Hence, it can be concluded that the mean grain size caused by the combined process of TE + CR is the same as the one resulted by three TE passes.

D. Accumulative Roll Bonding

In this process, thin sheets of metal/alloy are taken and stacked together for roll bonding. The surfaces to be joined are roughened and cleaned; the two parts are stacked and roll bonded with approximately 50% reduction in thickness. The bonded sheet is cut into two halves and again stacked after proper surface cleaning and rolled. Several researchers have shown that the process may be repeated several times, if some precautions like removing the edge cracks are taken. Several passes of repeated roll bonding may lead to a large reduction in the initial sheet thickness. A little consideration would indicate that after six such passes a total number of 64 layers could be introduced. This means that an initial 2 mm thick sheet would be reduced to $(2/64)$ mm if a reduction of 50% is achieved during each pass. This process can be carried out either in hot condition or cold condition. However, it is beneficial to work below the recrystallisation temperature of the materials. If $T_{\text{homologous}}$ of roll bonding is close to $0.5 T_m$, sound bonding is achieved by reduction of approx. 50%. However, it may vary from materials to materials. Hence, the material can be bonded together without recrystallization. ARB can be used to introduce ultra high plastic strain without any

geometrical change, if reduction is maintained to 50% in every rolling pass, as increase in width is negligible if the aspect ratio, i.e., ratio of width to thickness is >10 . If reduction is 50% per cycle, the thickness of initial strip after n cycles is

$$t = \frac{t_0}{2^n}$$

where t_0 = initial thickness of strips

Total reduction R_t

$$r_t = \frac{t_0 - t}{t_0} = 1 - \frac{t}{t_0} = 1 - \frac{1}{2^n}$$

The von misses equivalent plastic strain is given by

$$\varepsilon = \left(\frac{2}{\sqrt{3}} \right) \ln \left(\frac{t_{\text{initial}}}{t_{\text{final}}} \right)^n$$

$$= 0.8n$$

$$= 4.8 \text{ (for 6 passes)}$$

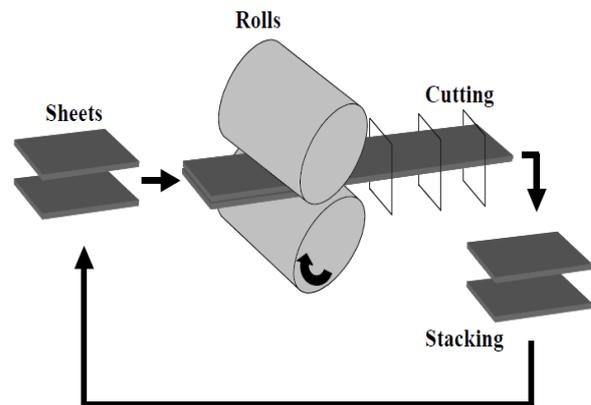


Fig.5. Schematic diagram showing ARB process

There are two possible additional mechanisms in the ARB process, which differ from other high straining processes. The first possible mechanism is the effect of severe shear deformation just below the surface. It has been reported that severe shear deformation is introduced by friction between the work piece and the roll under dry conditions. This shear deformation significantly increases the equivalent strain and promotes grain refinement[1]. Moreover, these highly deformed surface layers are further introduced into the interior of the material by repetitive folding and rolling. The other mechanism is the introduction of new interfaces. A large number of interfaces are introduced by several ARB cycles. These interfaces show a well-developed fibrous structure. The oxide films on the surfaces, as well as inclusions, are dispersed uniformly by repetition. These second phase particles contribute to strengthening and may act as obstacle for grain growth.

D.1. Phenomena Affecting Interfacial Bonding :

Accumulative roll bonding process is a cold welding process, forming a bond between participating metallic layers. The strength of bonds depends on several metallurgical and mechanical factors. And also, the bond quality reflects its presence in the mechanical properties of the ARBed materials.

Therefore, a good bonding between layers becomes of paramount importance in this process. The bond quality, in general, depends on the following material and process parameters.

- The grain size and ductility of the materials
- Cleanliness and roughness of the joining faces
- Closeness of faces and roll pressure
- Duration of the contact
- Rolling temperature
- Oxide formation on the joining faces
- Oxide fracture during rolling

D.2 Limitation due to edge cracking :

The occurrence of edge cracks on the samples after a few passes limits the application of the ARB process. One of the reasons for edge cracking behavior is due to the continuous increase in hardness and strength of the material after each pass due to which the ductility of the material decreases.

The second reason is the presence of precipitates that hinder the movement of dislocations, thus resulting in nucleation and propagation of edge cracks due to dislocation pile up. In ARB process, 99% reduction of experimental material could be achieved, which is much larger than what can be achieved in one conventional pass or by forging without edge cracking.

The large straining during reduction of material thickness is another cause of edge cracking during ARB. As the edge cracks further propagate in the material with subsequent ARB cycles, proper trimming of the edges, sometimes, becomes necessary to achieve more deformation without crack propagation

D.3 Advantages:

The major advantages of this process over other straining processes are as follows

- It has the capability of high productivity and the feasibility of bulk material production.
- It does not require any special machines, because the roll bonding is widely adopted in clad metal production
- The materials processed by this route have almost homogeneous microstructure.
- It can be applied to materials with different crystal structures and to materials ranging from precipitation-hardened alloys to intermetallics and metal-matrix composites.

E. Multi Axial Forging

MAF is a simple technique where the material is subjected to repeated forging in three orthogonal directions. After each pass, the sample is turned to an angle of 90° then the next pass is given. It is used to produce UFG structure in relatively brittle materials by operating at high temperatures. The advantage of MAF is that, the initial shape remains same with minimum distortion even after several pressings.

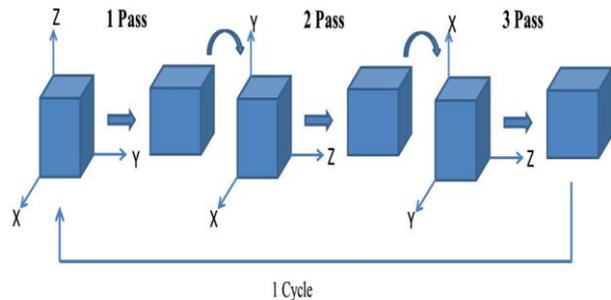


Fig.6. Schematic illustration of Multi axial forging for 1 cycle

F. Cryorolling

Deformation at cryogenic temperature has emerged as a potential route to develop UFG material with high density of dislocation for realizing the improved mechanical properties. Cryorolling suppresses the dynamic recovery during rolling at liquid nitrogen temperature there by enhancing the grain-refinement effect. It also obviates the drawback associated with SPD techniques[12]. However, suppression of the dynamic recovery preserves the high density of dislocation defects, which could act as sites for recrystallization to produce fine grain structure [12]. Cryorolling has been used by many researchers to produce UFG materials from pure metals and their alloys. A good combination of high strength and ductility was achieved by developing a bimodal grain size distribution of nanocrystalline and ultrafine grains in pure copper and aluminium through simple approach of cryorolling followed with annealing [13-15].

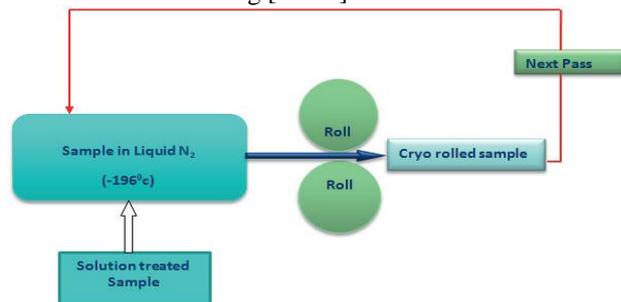


Fig.7. Schematic diagram showing cryorolling cycle

Maruff Hussain et.al [18] compared microstructural and mechanical properties of Al 6063 processed through MAF and CR and reported that mechanical properties of samples processed through MAF-4 and CR 92% are nearly same which are shown in the following table

Table .1. Mechanical properties of Al 6063 alloy after MDF and CR

State	σ_y	σ_{uts}	%elongation	Vickers hardness
ST	56	106	30	42
CR	239	248	4.5	88
MDF-4	241	252	6	92

S.K. Panigrahi, R. Jayaganthan [16] severely rolled Aluminium alloy (6063) upto 92% thickness reduction at liquid nitrogen temperature and room temperature to study the effect of rolling temperature on its mechanical properties and microstructural as compared to room temperature rolled (RTR) material with the same deformation strain.

An improved strength (257MPa) of cryorolled 6063 Al alloy was observed as compared to the room temperature rolled alloy (232MPa). The tensile properties of cryorolled alloy and the alloy subjected to different annealing treatments were measured. The cryorolled alloy subjected to annealing treatment at 300 °C for 5 min exhibits an ultrafine-grained (UFG) microstructure with improved tensile strength and ductility.

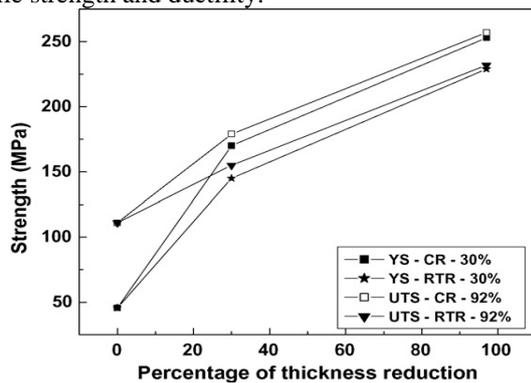


Fig.8. Yield strength (YS) and ultimate tensile strength (UTS) of CR and RTR 6063Al alloy at different percentage of thickness reductions.

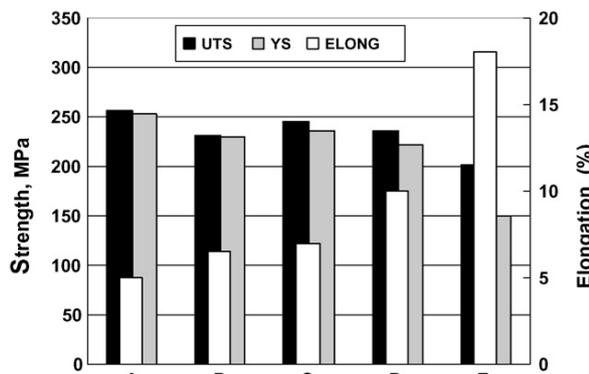


Fig.9. Tensile properties of CR and RTR 6063 Al alloy at 92% thickness reduction subjected to different annealing conditions: (A) CR material; (B) RTR material; (C)CR material, annealed at 200 °C for 5min; (D) CR material F.1. Effect Of Post Cryorolling Treatment on Mechanical Properties:

a. Effect of warm rolling afterwards cryorolling

Dharmendra Singh et. al[12] subjected Aluminium–Magnesium (Al 5083) alloy to cryorolling (CR) and cryorolling followed by warm rolling (WR) in order to investigate the changes in mechanical behaviour and microstructure evolution alloy specimens were first cryorolled up to 50% thickness reduction followed with warm rolling at 100 °C, 145° C, 175° C and 200 °C till to achieve total 90% thickness reduction. The final microstructure of all conditions were analysed. The mechanical behaviour of the processed samples were evaluated through hardness and tensile tests performed at room temperature.

The ultimate tensile strength (UTS) and yield strength(YS) of starting solution treated material significantly improved after cryorolling upto 90% reduction (UTS- 277 -478 MPa,YS-168 to 460 MPa) but its elongation to failure has decreased from 22% to 3% (Fig. 2 b) which is distinctive behaviour of cold worked samples. Whereas warm rolling of 50% cryorolled sample at 100 ° C increased not only tensile strength but also ductility (Fig. 2b). With increasing warm rolling temperature from 100 ° C to 175° C simultaneous improvement in strength and ductility was observed. The increase in strength and ductility may be due to formation of fine precipitates and dynamic recovery effect. The further increase in warm rolling temperature to 200°C led to increase in elongation to failure but decrease in tensile strength. After CR and WR at175° C annealing of samples showed increase in ductility but decrease in hardness. It is due to formation of fully recrystallised equiaxed fine grain structure

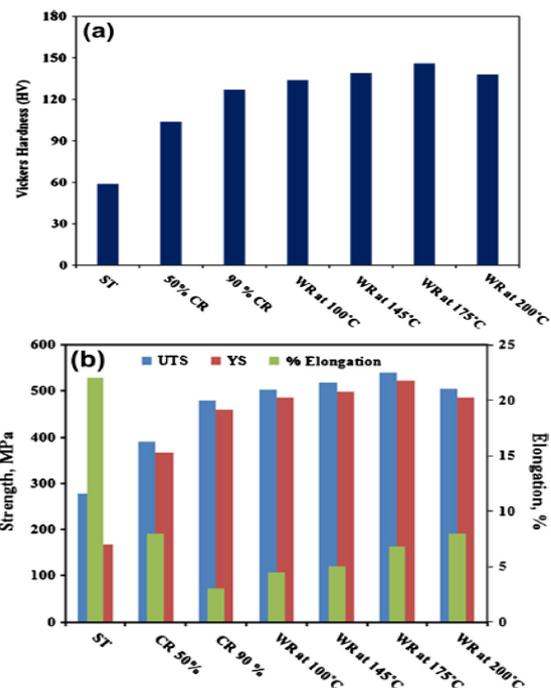


Fig.10. Variation in UTS, YS, elongation and hardness with respect to rolling condition: (a) Vickers hardness values, (b) UTS, YS and elongation values.

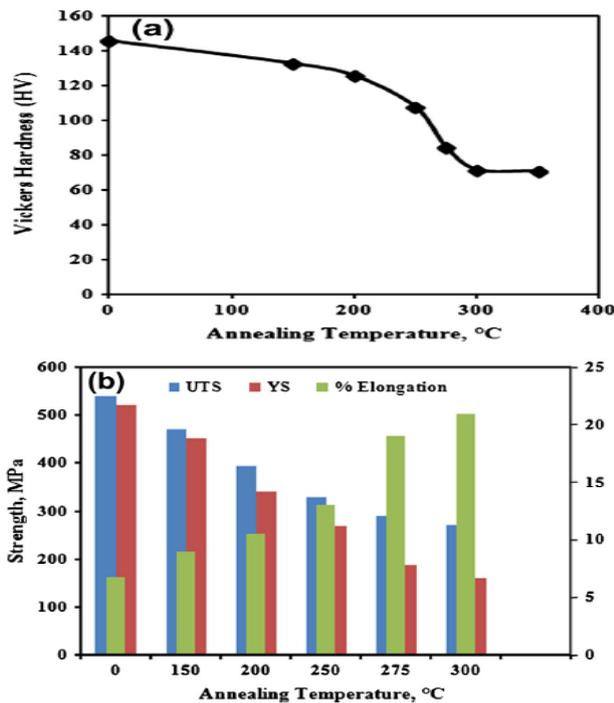


Fig.11. Variation in hardness, UTS, YS, and elongation with respect to annealing temperature for 1 h: (a) Vickers hardness values, (b) UTS, YS and elongation values.

b. Effect of annealing afterwards cryorolling

Dharmendra Singh et al [13] studied the effect of annealing after cryorolling on hardness and impact toughness on 30% and 50% cryorolled samples. With increasing annealing temperature, the hardness decreases slightly up to 250°C and suddenly drops to nearly the same value for the cryorolled samples irrespective of cryorolling strain from 250 to 350°C. The sudden drop in hardness within the range of 250-350 °C is due to nucleation and growth of dislocation free recrystallised grains during annealing treatment.

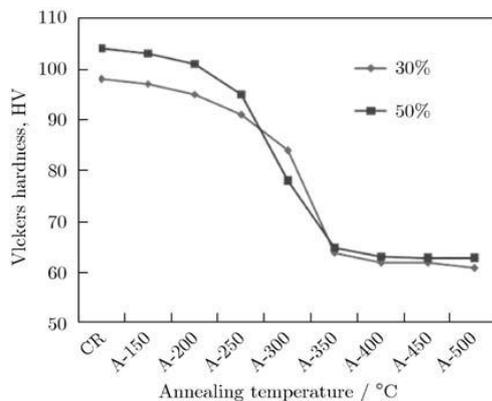


Fig.12. variations in vicker hardness of al 5089 alloy after cryorolled and cryorolled+ annealed at various temperatures

After cryorolling Annealing of sampled shows remarkable increase in impact toughness through recovery and recrystallization. With increasing annealing temperature,

the energy absorption capacity of the material gradually increased up to 250 °C due to the availability of enough rooms created by annihilation of dislocations during recovery in both the cryorolled samples to accept dislocations further. After annealing at 300 °C 1 h, the absorbed impact energy suddenly increased to about 78.5% in the 50% cryorolled sample, whereas it is 36.8% in the 30% cryorolled sample. The sudden rise in impact energy of the 50% cryorolled sample upon annealing is due nucleation and growth of dislocation free recrystallised grains that can absorb more amounts of dislocations before fracture. The observed impact energy of the cryorolled samples after annealing at 350 °C is slightly lower than that of the initial solution-treated sample.

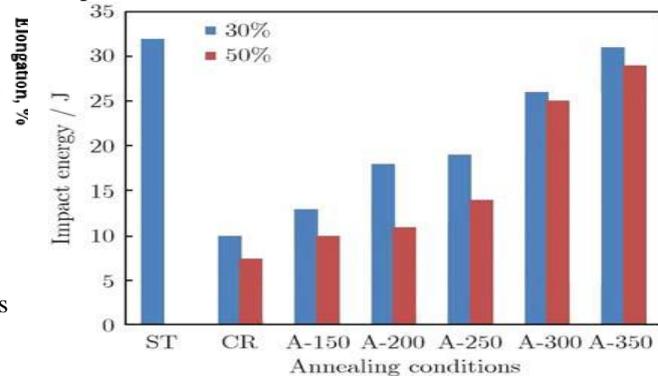


Fig.13. Variation of impact toughness with respect to annealing temperatures

c. Effect on annealing on grain boundaries

With increase in annealing temperatures the fraction of high angle grain boundaries increases whereas fraction of low angle grain boundaries decreases.

III. LIMITATIONS OF SPD

1. Severe plastic deformation techniques requires severe plastic strain
2. Expensive tooling
3. Design difficulties
4. Less production rates due to which less industrial interest
5. High cost of the products developed by SPD

IV. CHALLENGES

Material Processing by SPD is still in Laboratory Scale. Up scaling of the process has not taken place still to large extent. Though the research activities in this area are enormous yet industrial application are very few. The situation is changing, and there are real and tangible applications of SPD processing ready to be picked up by industry. Manufacturing of biomedical implants, where commercial products have emerged recently, is a market-ready area par excellence. The main lines of substructure and fine grain formation are more or less understood, although several details need some further clarifications. Besides improving strength and ductility there are other properties like Fatigue behaviour, corrosion resistance, diffusion properties, electrical properties biocompatibility, bio-corrosion, properties (bio-corrosion is corrosion of

UFG material implanted in human body due to bodily fluids, hydrogen sorption capacity of UFG material are very hot topic of research now a days .[1] so by further increase in research activities in these areas we can produce multifunctional UFG material by SPD

V. CONCLUSION

There are a number of methods of SPD that can be used to refine the structure of Metal and Alloys. Over the past years this Knowledge has increased manifold. Scientists have worked a lot in this Field using various materials and techniques. However, for commercialization the cost to convert the lab scale work to large scale application is an important decisive factor.

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