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ULTRAFINE-GRAINED MATERIALS PRODUCED BY SEVERE PLASTIC DEFORMATION

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ABSTRACT

In recent years, ultrafine-grained materials have attracted increasing interests for their good combinations of high strength and ductility. To prepare these materials, Various SPD processing techniques for refining the structure have been developed. This work highlights the recent achievements and new trends in the production of bulk ultrafine-grained (UFG) materials using severe plastic deformation (SPD). Some of those methods permit grain refinement to a Nanometric level. These methods include, among others, high pressure torsion (HPT), equal channel angular pressing (ECAP) and hydrostatic extrusion (HE). In this work, special attention is given to the principles of the various SPD processing techniques as well as the applications of the ultra-fine grained (UFG) metals.

KEYWORDS

Sever plastic deformation (SPD), ultrafine-grained materials (UFG), ECAP, HPT, ARB

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INTRODUCTION

The development of the principles of 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 [4]. Very successful implementations of these principles are the high pressure torsion (HPT) technique introduced in the mid-1980s and the process of equal-channel angular pressing (ECAP), also known as equal-channel angular extrusion, developed by V.M. Segal and co-workers in Minsk in the early 1980s [5]. The applications of these SPD techniques for the production of ultrafine-grained metals, pioneered by R.Z.Valiev and co-workers in Ufa in the late 1980s and subsequently continued by many investigators around the world, led by about 2000 to the systematic development of a range of nano-structured materials with exceptionally favorable properties [6] . The last decade has seen the advent of new SPD techniques [3] that are now under development for use in a range of practical applications.

Based on the extensive research conducted to date, applications of SPD techniques are now starting to emerge for use in manufacturing industries and several commercial products, such as sputtering targets, fasteners and dental implants, are already available. An up-scaling of the SPD processes, which are already proven viable at the laboratory scale, and the development of continuous processing techniques will further allow for the development of large-scale applications. The compaction of powders through SPD processing is also an additional area of opportunity for utilization in large-scale manufacturing.

Ultrafine-grained (UFG) materials are defined as polycrystalline materials having a homogeneous and reasonably equiaxed microstructures with average grain sizes less than 1 µm and with a majority of grain boundaries having high angles of disorientation [1]. Although these materials may be produced through the assembly of individual atoms in gas condensation or by the consolidation of nano-particles after high-energy ball milling in the so-called "bottom- up" approach, the production of large bulk fully-dense UFG solids requires the use of a "top-down" approach in which high dislocation densities are introduced into materials with coarse grain sizes by imposing severe plastic deformation (SPD) [2].

Processing by severe plastic deformation (SPD) may be defined as those metal forming procedures in which a very high strain is imposed on a bulk solid without the introduction of any significant change in the overall dimensions of the solid and leading to the production of exceptional grain refinement so that, typically, the processed bulk solids have 1000 or more refined grains in any section [3].

Numerous techniques for SPD processing are now available. The major methods already established for the fabrication of UFG materials include equal-channel angular pressing (ECAP) [7-8], high-pressure torsion (HPT) [9-10], multi-directional forging [11], twist extrusion [12], cyclic-extrusion–compression [13], reciprocating extrusion[14], repetitive corrugation and straightening (RCS) [15], constrained groove pressing (CGP) [16], cylinder covered compression (CCC) [17], accumulative roll-bonding (ARB) [18], friction stir processing (FSP) [19] and submerged friction stir processing (SFSP) [20], severe torsion straining (STS) [21], cyclic closed-die forging

(CCDF) [22] and super short multi-pass rolling (SSMR) [23]. All of these procedures are capable of introducing large plastic straining and significant microstructure refinement in bulk crystalline solids.

EQUAL-CHANNEL ANGULAR PRESSING

Equal-channel angular pressing is at present the most developed SPD processing technique. As illustrated in Figure 1, a rod-shaped billet is pressed through a die constrained within a channel, 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 between the two parts of the channel, Φ, and the angle representing the outer arc of curvature where the two parts of the channel intersect, Ψ. The relationship is given by [1]:

$$\varepsilon = \frac{N}{\sqrt{3}}\left[2cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right) + \Psi\, cosec\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right]$$

where N is the number of passes through the die.

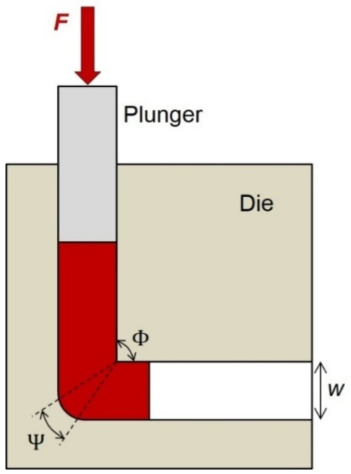

Figure 1. Principle of ECAP.

During repetitive pressings, the shear strain is accumulated in the billet, leading ultimately to a UFG structure. In practice, different slip systems may be introduced by rotating the billet about its longitudinal axis between each pass and this leads to four basic processing routes: there is no rotation of the billet in route A, rotations by 90° in alternate directions or the same direction in routes B_A and B_C, respectively, and rotations by 180° in route C [24]. When using a die with a channel angle of Φ = 90°, route B_C is generally the most expeditious way to develop a UFG structure consisting of homogeneous and equiaxed grains with grain boundaries having high angles of disorientation. There have also been numerous recent modifications of conventional

ECAP that are designed to yield more efficient grain refinement including the incorporation of a back-pressure, the development of continuous processing by ECAP, and others.

High Pressure Torsion HPT

The scientific origin of processing by HPT may be traced to a classic paper, written by Bridgman and appearing in the Journal of Applied Physics in 1943, entitled "On Torsion Combined with Compression". In this early report, Bridgman succinctly set out the basic tenets of this type of testing by stating: "If a bar is twisted while a longitudinal compressive load is simultaneously applied, it is possible to twist the bar through much greater angles without fracture than is possible without the compressive load. At the same time the magnitude of the torque which the bar can support without fracture is increased." [25].

The principles of modern HPT processing are shown in Figure 2. The sample, in the form of a disk, is located between two anvils where it is subjected to a compressive applied pressure, P, of several GPa at room temperature or at an elevated temperature and simultaneously it is subjected to a torsional strain which is imposed through rotation of the lower anvil. Surface frictional forces therefore deform the disk by shear so that deformation proceeds under a quasi-hydrostatic pressure. The strain in torsion is given by:

$$\gamma = \frac{2\pi N.r}{h}$$

where: N…. is the number of revolutions

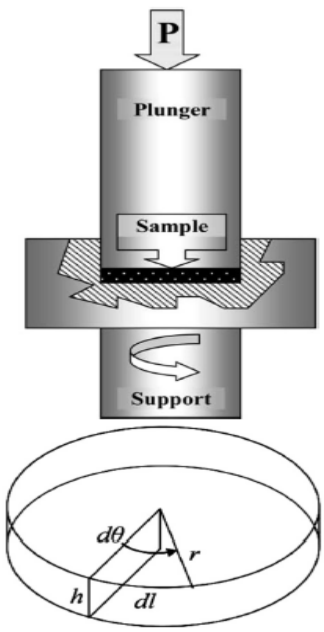

Figure 2. the thin disc-HPT process [25].

This method has the disadvantage that it utilizes specimens in the form of relatively small discs and is not available for the production of large bulk materials. Another disadvantage is that the microstructures produced are dependent on the applied pressure and the location within the disc. The severe plastic torsion straining (SPTS) process can be used for the consolidation powders using a similar apparatus as shown in Fig. 2 [26]. By using this process at room temperature, the disk type samples with a high density close to 100% were developed. The SPTS consolidation of powders is an effective technique for fabricating metal–ceramic nano-composites with a high density, ultra-fine grain size and high strength.

ACCUMULATIVE ROLL-BONDING (ARB) PROCESS

The principle of the ARB process is represented systematically in Figure 3, where stacking of sheets and conventional roll-bonding are repeated in the process. First, a strip is neatly placed on top of another strip. The interfaces of the two strips are degreased and wire-brushed before placing them in contact in order to enhance the bonding strength. The two layers are joined together by rolling, as in the conventional roll-bonding process. Then, the length of the rolled material is sectioned into two halves.

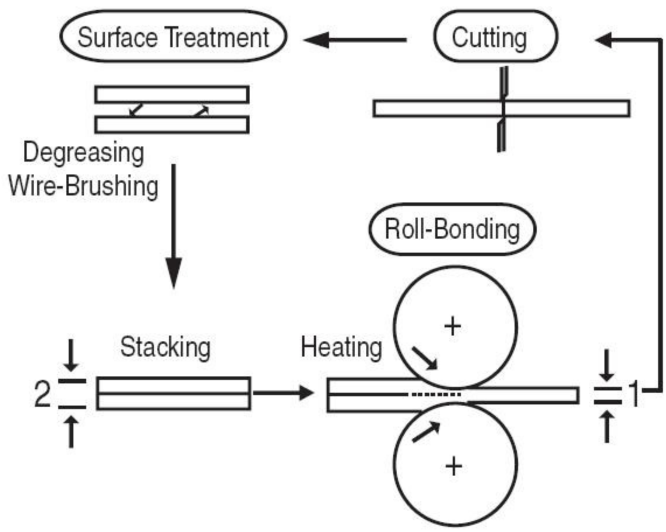

Figure 3. Diagrammatic representations of the accumulative roll-bonding (ARB) process [27].

The sectioned strips are again surface-treated, stacked and roll-bonded. These procedures can be repeated limitlessly in principle, so that very large plastic strain can be applied to the material [27]. It is possible to heat the sheet when rolling but at a temperature where there is no recrystallization. The ARB process has been used by many researchers in order to create ultra-fine grained metals [28]. The strain (ε) after (n) cycles of the ARB process can be expressed as,

$$\varepsilon = \frac{\sqrt{3}}{2}\ln(r), \quad r = 1 - \frac{t}{t_o} = 1 - \frac{1}{2^n}$$

where t_o is the initial thickness of the stacked sheets, t is the thickness after roll-bonding and r is the reduction in thickness per cycle.

Table 1 summarizes the geometrical changes of the specimen thick sheets are stacked and roll-bonded by a 50% reduction per cycle. The number of the initial sheets included in the specimen processed by (n) cycles of ARB becomes 2n. After 10 cycles of the ARB process, the number of layers becomes 1024 so that the mean thickness of the initial sheet is smaller than 1 mm.

Table 1 summarizes the geometrical changes of the specimen during the ARB process where roll-bonded by 50% reduction per cycle [27].

Table 1. geometrical changes of the specimen during the ARB process.

Number of cycles (n)	1	2	3	5	10
Number of layers (m)	2	4	8	32	1024
Total reduction r (%)	50	75	87.5	96.9	99.9
Equivalent strain (ε)	0.8	1.6	2.4	4	8

MULTIPLE FORGING (R16)

One more method of formation of nanostructures in bulk billets is multiple forging developed by Salishchev et al. [29]. The principle of multiple forging is schematically illustrated in Figure 4 assumes multiple repeats of free forging operations: setting drawing with a change of the axis of the applied strain load. The homogeneity of strain provided by multiple forging is lower than in the case of ECA pressing and torsion straining. However, the method allows one to obtain a nano-structured state in rather brittle materials because processing starts at elevated temperatures and specific loads on tooling are low. The choice of appropriate temperature-strain rate regimes of deformation leads to a minimal grain size.

CYCLIC EXTRUSION COMPRESSION (CEC)

Cyclic extrusion and compression (CEC) “hourglass pressing” is performed by pushing a sample from one cylindrical chamber of diameter D to another with equal dimensions through a die with diameter d, and with a diameter ratio of typically $d/D \approx 0.9$, the principle is illustrated in Figure 5 [30]. Thus, the processing induces extrusion and the chambers provide compression so that, during one cycle, the material is pushed to first experience compression, then extrusion, and finally compression again. In the second cycle, the extrusion direction is reversed, leading to the same sequence of deformation modes. The process can be repeated N times by pushing the sample back and forth to give an accumulated true strain of $\Delta\varepsilon = 4n \ln (D/d)$. The strain imposed on the material in one cycle is $\Delta\varepsilon \approx 0.4$. Although the strains reached with this method are much higher than those with any unidirectional SPD technique, the microstructure

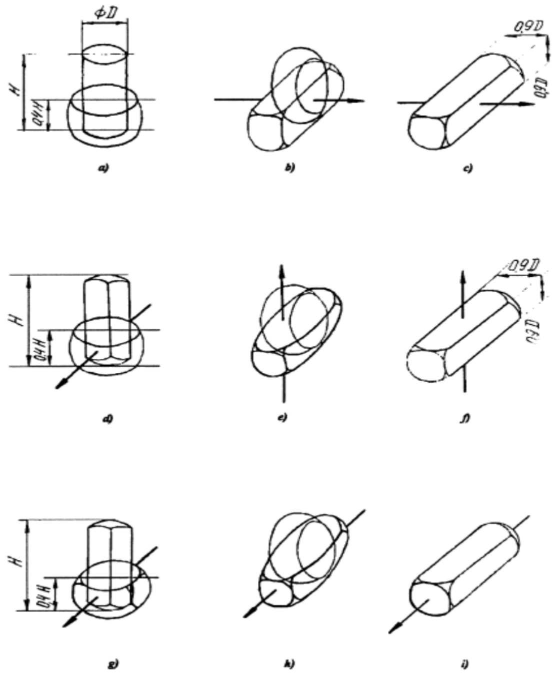

Figure 4. Principle of multiple forging [29].

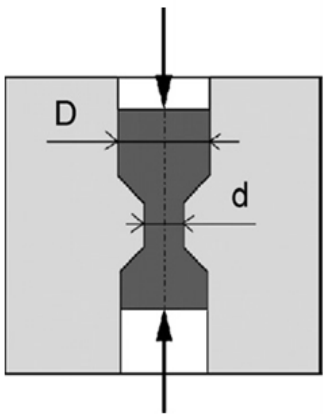

Figure 5. Schematic illustrations of cyclic extrusion and compression (CEC) [30].

and/or mechanical properties are similar because of the extra annihilation of dislocations due to the cyclic character of the straining.

REPETITIVE CORRUGATION AND STRAIGHTENING (RCS)

Repetitive corrugation and straightening was introduced recently and the principle is illustrated in Figure (6). In a repetitive two-step process, the work piece is initially deformed to a corrugated shape and then straightened between two flat platens using a processing cycle that may be repeated many times.

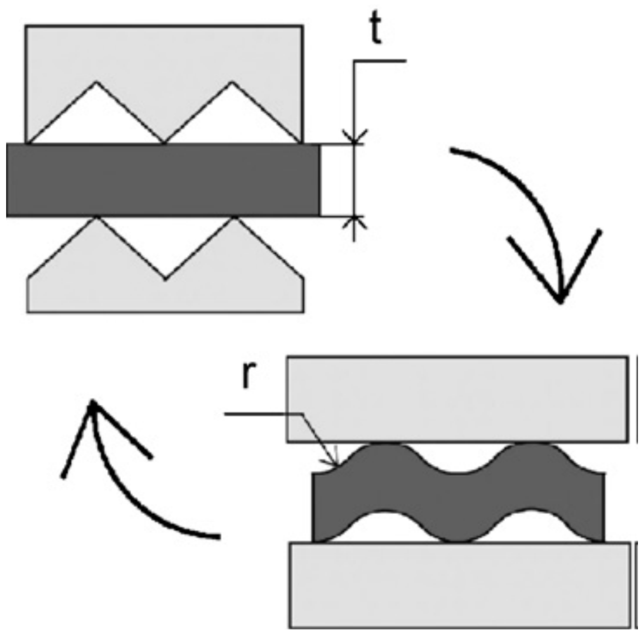

Figure 6. Principle of repetitive corrugating and straightening [31].

In RCS the work piece is subjected to both bending and shear, which promotes grain refinement. An advantage of RCS is that it can be adapted easily to current industrial rolling facilities. It is not difficult to machine a series of corrugating teeth into the rollers of a conventional rolling mill, thus enabling the RCS process, and this has the potential of producing nano-structured materials in a continuous and economical way [31]. The RCS technique is currently in the early stages and further research is needed to develop the process to a mature SPD technique for producing nano-structured materials. One critical issue is the need to design equipment and processing schedules for improving micro structural homogeneity.

LINEAR FLOW SPLITTING (LFS)

Linear flow splitting developed by Groche et al. is another possibility to obtain ultra-fine grained metal [32]. The principle of this process is shown in Figure 7. A sheet metal is compressed between the splitting roll and the supporting rolls. Under this state of stress two flanges are formed into the gap between the splitting and the supporting rolls. The material flow is mainly associated by a surface enlargement of the band edge. Several hundred percent of plastic strain occur. As a consequence, the outer surface areas of the flanges consist of ultra-fine grained metal. The properties of the metal in this state can be used for an increase of load bearing capability, e.g. bearings for rollers.

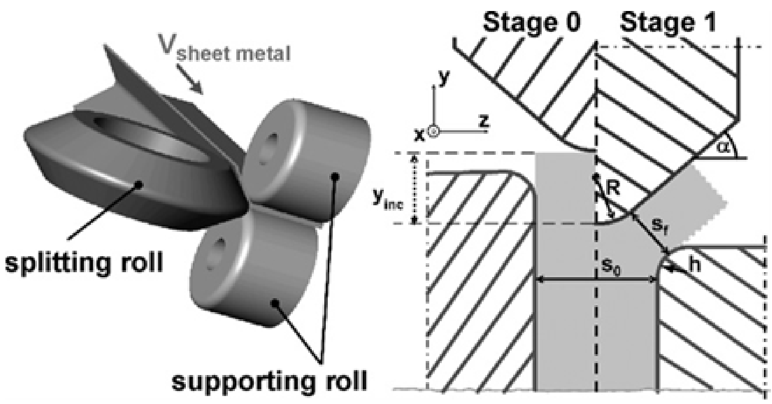

Figure 7. Principle of linear flow splitting [32].

TWIST EXTRUSION (TE)

The illustration shows the shapes of a work piece before entering a TE die, inside the die, and after exiting the die where the workpiece is deformed by twisting within the TE die [33]. The use of Twist Extrusion (TE) for grain refinement was introduced in 2004 [33] and the principles are illustrated in Figure 8. During TE, a work piece is pushed through an extrusion die whose cross section maintains its shape and size while it is twisted through a designated angle around its longitudinal axis. As a result, the work piece regains its shape and size after each TE pass and thus it is possible to repetitively process a sample for excellent grain refinement. A variety of cross-sectional shapes, but not circular geometries, are possible with this technique. In practice, and by analogy to HPT, the plastic strain is not uniform across the cross section but the plastic strain increases with the distance from the axis so that the more distant regions have a finer grain size. This micro structural heterogeneity leads to inhomogeneous mechanical properties with the cross-sectional center having the lowest strength. It is anticipated that the micro structural homogeneity may improve with increasing numbers of TE passes.

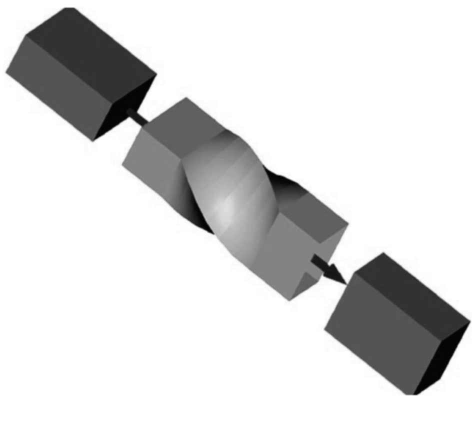

Figure 8. The principle of TE.

TORSION EXTRUSION (TE)

The principle of the torsion extrusion process developed by Mizunuma et al [34] is represented schematically in Figure 9. This process is characterized by rotation of a die or a container during an extrusion process for introducing a very large strain in to the metal. As high hydrostatic pressure involved in the extrusion raises the ductility of the metals, a very large torsion straining can be introduced to the work piece. The mean value of representative strain on a cross-section of a column can be calculated as follow:

$$\varepsilon = \frac{4\pi RN}{3\sqrt{3}H}$$

where R is radius of column, H is the height of the column and N is the number of rotation.

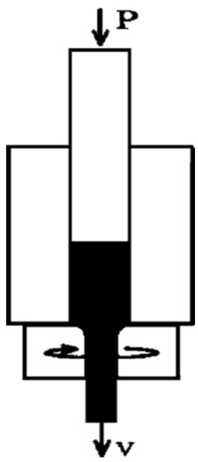

Figure 9. Principle of the torsion extrusion process.

APPLICATIONS

The properties of the metals processed by SPD exhibit high strength, ductility and fatigue characteristics. UFG metals are used as a structural material due to these properties. Bolts are also manufactured with titanium alloys processed by ECAP and are widely used in the automobile and aircraft industries [35]. Micro bolts using the UFG carbon steel processed by cold ECAP have also been manufactured [36]. Long carbon steel bars, of over several kilometers, with ultrafine grains are manufactured by the warm continuous caliber rolling and cooling process, from which the micro bolts are manufactured. Recently, sheets of low carbon steel of 2 mm thickness with ultra-fine grains were manufactured by the Thermo-Mechanically Controlled Processed (TMCP) process. The deep drawing ratio of each sheet was 1.9 and the parts were used in sheet metal forming [37].

It is well known [38] that super plastic forming is a highly efficient method of processing complex shape articles. An example of a possible practical application for nano structured Al alloys presents a complex shape article of ‘Piston’ type which was fabricated from the nano structured Al1420 alloy by super plastic forming using the high strain rate super plasticity.

The next highly anticipated application is in the area of medical implants. These include hip, knee and dental implants as well as various screws, plates and meshes used in orthopedic applications. Popular materials usually used in these applications are cobalt-chrome alloys, stainless steel and titanium alloys. Titanium alloys are used for implants because of their strength, low modulus of elasticity (better matching that of bones), corrosion resistance and good biocompatibility. Commercial pure (CP) titanium has better compatibility than titanium alloys but it is not used for load bearing implants because it is not strong enough. However, when nano structured by SPD and subjected to further thermo-mechanical treatment, CP titanium can be strengthened to achieve the yield stress of 1100 MPa, which is comparable with the yield strength of titanium alloys [39]. Traditional titanium implants do not perform well with respect to wear resistance and fatigue life. Therefore, improvements in these properties, reported for UFG titanium, will be appreciated. Some Russian [40] and USA laboratories report that the UFG CP titanium implants are being already tried.

The defense industry could benefit from two large scale applications of UFG metals, which are armor plates and armor penetrators. Lighter armor for military vehicles is crucial for the reduction of fuel consumption, higher speed, better maneuverability, longer operation range and air-transport of vehicles to remote locations. At the same time the ballistic performance must not be reduced. This can be achieved by the nano structuring of aluminum or titanium alloys traditionally used for light armored vehicles. A good example is a UFG Al 5083 plate, which was obtained by cryogenic ball milling, consolidation by HIP, forging or extrusion and finally rolling [41].With the yield strength of 600–700 MPa and elongation of 11%, the material exhibited a 33% improvement in the ballistic performance or a similar mass reduction compared to the standard plate. Armor structures are usually fabricated by welding of plates. However, traditional welding based on melting is destructive to the UFG material. An alternative technique is a solid state process of friction stir welding, which has the ability to refine grain structure. This results in the weld hardness being only marginally reduced compared to the initial hardness of a UFG material [42].

Health issues surrounding the use of depleted uranium for armor penetrators resulted in a search for alternative materials with similar performance characteristics. One of those characteristics is an inherent ability of depleted uranium to self-sharpen on impact which is due to the generation of adiabatic shear bands. Tungsten, sometimes considered as a replacement for depleted uranium because of its high density, does not have this ability; thus penetrators made of tungsten undergo mushrooming on impact, which results in less penetration.

UFG metals are known to have reduced strain hardening capacity, which promotes localized plastic deformation; at high deformation rates this leads to adiabatic shear banding. This was confirmed by producing UFG tungsten (by ECAP with a die angle of 1208 at 1100–1000 ^{o}C and subsequent rolling at 600–700 ^{o}C), which exhibited adiabatic shear banding when subjected to a dynamic load [43]. There has only been limited information published so far on the potential use of UFG metals by the aerospace industry; Boeing, filed a few patents on friction stir welding used as a means of nano structuring metals for fasteners and other parts [44] while EADS is interested in UFG aluminum sheets.

Users of sports equipment will also benefit from UFG metals, particularly where high strength and low weight is required. UFG metals could find applications in high performance bicycles, sailing equipment, mountaineering equipment, golf, tennis, hockey, etc. One example is NanoDynamics high performance (NDMX) golf balls, which have a hollow nanostructured titanium core. Another example of using UFG metals in sporting goods is the commercial activity of Power Metal Technologies, a company with an exclusive license to use Integran's electrode position technology in consumer products. They cooperate with HEAD in the production of their new Metallix and Airflow racquets, which use a composite of carbon fibers and Nano crystalline metal.

The above applications of UFG metals are only a fraction of the possible uses. Since the SPD technology can convert all CG metals into UFG metals, it is only a matter of time when new, sometimes unexpected, applications will be discovered. For this to happen, information dissemination among industrial engineers, transfer of reliable SPD technologies to industry and commercialization effort is required [45].

CONCLUSION

Processes of severe plastic deformation, defined as metal forming processes in which an ultra-large plastic strain is imposed on a bulk material in order to make ultra-fine grained metals, and is reviewed in this paper. As processes used for this purpose, various methods such as, ECAP, HPT, ARB, MDF, CEC, RCS, LFS, TE…. etc. were developed, and combined SPD processes with conventional processes were also proposed. The UFG metals could be used as structural materials due to these properties, but the area of application is limited now because the available size of billet is small. Since SPD technology can convert all metals into UFG metals, it is expected that new methods of producing larger billets will enlarge the area of applications.

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