Twenty-five years of ultrafine-grained materials: Achieving exceptional properties through grain refinement

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Abstract

Twenty-five years ago, in 1988, there appeared a classic description of the application of severe plastic deformation (SPD) to bulk solids in order to achieve exceptional grain refinement to the submicrometer level. This report and later publications initiated considerable interest in materials science laboratories around the world and many experiments were subsequently performed to evaluate the principles and practice of SPD processing. The present report provides an overview of the more recent developments in this field, with special emphasis on the opportunities for achieving homogeneity in the as-processed materials and on the general characteristics of the mechanical properties achieved after SPD processing. For simplicity, special emphasis is placed on the two techniques of equal-channel angular pressing and high-pressure torsion as these are currently the most popular procedures for applying SPD processing.

Keywords: Equal-channel angular pressing; High-pressure torsion; Homogeneity; Severe plastic deformation; Ultrafine grains

1. Introduction

It is well known that the flow behavior of polycrystalline materials is readily divisible into two distinct regimes, depending primarily upon the operating temperature. These regimes are depicted most clearly using deformation mechanism maps [1,2], where there is a sharp division, occurring at \( \approx 0.5T_m \), where \( T_m \) is the absolute melting temperature, between low-temperature behavior controlled by the conservative motion of dislocation glide and high-temperature behavior in which diffusion-controlled flow is dominant through processes such as dislocation climb and diffusional creep.

Inspection shows that, for both these flow regimes, the grain size is the most important, and indeed the dominant, structural parameter in polycrystalline materials. Thus, in the low-temperature regime, the yield stress \( \sigma_y \) varies with the grain size \( d \) through the Hall–Petch relationship, which is given by [3,4]

\[
\sigma_y = \sigma_o + k_yd^{1/2}
\]

(1)

where \( \sigma_o \) is the lattice friction stress, and \( k_y \) is a constant of yielding. Conversely, in the high-temperature regime, the creep rate under steady-state conditions \( \dot{\varepsilon} \) is expressed by a relationship of the form [5–7]

\[
\dot{\varepsilon} = \frac{ADGb^p}{kT} \left( \frac{\sigma}{G} \right)^n
\]

(2)

where \( A \) is the frequency factor, \( D_o \) is the activation energy for the flow process, \( R \) is the gas constant, and \( T \) is the absolute temperature, \( G \) is the shear modulus, \( b \) is the Burgers vector, and \( k \) is Boltzmann’s constant.
constant, \( \sigma \) is the flow stress, \( n \) and \( p \) are the exponents of the stress and the inverse grain size, respectively, and \( A \) is a dimensionless constant.

It follows from inspection of Eq. (1) that a small grain size is advantageous because it leads to a significantly higher strength. Additionally, Eq. (2) shows that a small grain size leads to faster strain rates, and this provides the possibility of achieving a superplastic forming capability at rapid rates that may be readily employed in industrial forming operations. Thus, grain refinement is an important processing tool for achieving optimum properties in metallic materials. The advantage of grain refinement was recognized many years ago and led to the development of thermo-mechanical processing operations wherein materials were subjected to annealing treatments and mechanical straining in order to reduce the grain size to values that are typically within the range \(~3–10\ \mu m\).

Twenty-five years ago, in 1988, a landmark report was published demonstrating the potential for achieving even smaller grain sizes within the submicrometer range through the application of severe plastic deformation (SPD) to bulk coarse-grained solids [8]: this approach is now generally termed SPD processing. The publication of this report attracted much attention, and it led to the initiation and development of many research activities around the world devoted to processing and measuring the characteristics of materials with exceptionally small grain sizes. It is interesting to note that the research activity in this field has both continued to develop and very much expanded up to the present day.

It is often not recognized that the general concept of SPD processing has a long history which may be traced back for more than two thousand years. Thus, the pioneering concept of SPD processing, introduced 25 years ago, lay not specifically with the processing method per se but rather with the availability of new advanced analytical and microscopic tools which provided, for the first time, direct evidence that the improved mechanical properties of these processed metals was due primarily to the introduction of exceptional grain refinement.

As described elsewhere [9], the historical background of SPD processing divides readily into three separate periods spanning more than two millennia. These periods are outlined briefly in the following section, and the subsequent sections provide detailed descriptions of some of the more recent developments in this important research field.

2. The historical background of SPD processing

2.1. The ancient age

A comprehensive review of the history of SPD processing shows that the general concept dates back at least to the Han dynasty of ancient China in \(~200\ \text{BC}\) [10]. At that time, the local artisans developed a new technique for the processing of steel for use in swords, where the metal was repetitively forged and folded to form the famous Bai-Lian steels. Many archeological artifacts are now available from this area and they often have inscriptions providing a historical record of the processing operation. Thus, a high-strength 50-Lian steel sword was prepared using 50 separate forging and folding operations. Subsequently, the principles of this technology spread to Japan where it was used for the processing of samurai swords, to India where it led to the development of the wootz ultrahigh carbon steel [11], and then to the Middle East where it produced the famous Damascus steel [12]. It is important to note that, although these developments spread readily across Asia, the fundamental principles of the processing technique lacked scientific rigor and, ultimately, in about the middle of the 18th century, the principles of this technique were lost.

2.2. The scientific age

The first attempt to introduce scientific principles into the procedures now known as SPD processing lies unambiguously in the classic work of Professor P.W. Bridgman at Harvard University. Beginning in the 1930s, Bridgman conducted a remarkably comprehensive series of experiments on the application of high pressures to bulk solids [13,14] and, in 1952, the results from these many experiments were succinctly summarized in a book [15]. It is important to note that Bridgman received the Nobel Prize in Physics in 1946 with the citation reading “for the invention of an apparatus to produce extremely high pressures, and for the discoveries he made therewith in the field of high pressure physics”. A review of this work shows that Bridgman was the first to propose the processing of metals through a combination of compression and torsional straining. This approach was later further developed in the former Soviet Union [16] and ultimately evolved into the procedure now known as high-pressure torsion (HPT).

A second major influence on SPD processing may be traced to the classic work of Dr. V.M. Segal and his colleagues, conducted in the 1980s in Minsk in the former Soviet Union (now the capital of Belarus). Segal and co-workers were the first to develop the process now known as equal-channel angular pressing (ECAP) or equal-channel angular extrusion [17] and this process has now become the most important and the most used of all SPD processing techniques.

Nevertheless, a deficiency of these earlier investigations was the absence of any detailed microstructural analysis. In practice, the detailed examination of microstructure became possible only with the later development of sophisticated analytical tools, including high-resolution transmission electron microscopy (HRTEM), electron back-scatter diffraction (EBSD), orientation imaging microscopy (OIM) and modern X-ray techniques.
2.3. The microstructural age

The development of new analytical and microscopic tools provided an opportunity to evaluate the microstructures of materials processed using SPD procedures. This approach was first developed by Professor R.Z. Valiev and his colleagues, working in Ufa, Russia, in the 1980s [18], where it was shown that SPD processing produces remarkable grain refinement to give average grain sizes in the submicrometer or nanometer range even in conventional commercial alloys. For example, in the classic first paper of 1988, it was shown that processing by HPT produced a grain size of ~0.3 μm in an Al–4% Cu–0.5% Zr alloy [8], where this grain size was about one order of magnitude smaller than the grain sizes of ~3–5 μm that are generally achieved through the use of standard thermomechanical treatments. Subsequent detailed reports in the western literature described the exceptionally small grain sizes attained using processing by ECAP and HPT [19,20], and this provided the impetus for the development of many similar research activities in materials laboratories around the world. These investigations have had a major impact on the more recent literature in materials science, as documented in a very recent report [21].

3. The principles of SPD processing

Many different SPD processing techniques are now available and summaries of these various procedures are given in several recent reviews [22–25]. Nevertheless, major emphasis has been placed to date on the two techniques of ECAP and HPT and, accordingly, these procedures will be examined exclusively in this report. A comprehensive review of ECAP was published in 2006 [26] and this was followed two years later by a comprehensive review of HPT [27]. Despite these very recent lengthy reviews, research in the area of SPD processing is currently receiving much attention and accordingly the present report is designed specifically to describe some of these very new developments in the areas of ECAP and HPT. These two procedures are examined in the following sections.

3.1. Principles of ECAP

The general principles of ECAP are shown schematically in Fig. 1 [28]. Processing by ECAP uses a die containing a channel that is bent through a sharp angle near the center of the die. The sample is machined to fit within the channel, and it is then pressed through the die using a plunger and an applied pressure P. Ultimately, the sample emerges from the die as shown on the right in Fig. 1. In practice, the channel is defined by two angles: the channel angle Ψ represents the angle between the two parts of the channel (equal to 90° in Fig. 1), and the curvature angle Φ represents the angle at the outer arc of curvature where the two parts of the channel intersect. Also defined in Fig. 1 is an orthogonal X, Y, Z coordinate system, which may be used to denote specific planes and directions within the sample.

It is readily apparent from Fig. 1 that the cross-sectional dimensions of the sample are not changed during processing. This means that the sample may be pressed repetitively through the die in order to attain a very high strain. Furthermore, it is possible to initiate different slip systems by rotating the sample between each pass [29]. These various processing routes are termed route A where the sample is pressed repetitively without rotation, routes BA and BC where the sample is rotated repetitively with rotations of 90° in alternate directions or in the same direction, respectively, and route C where the sample is rotated by 180° between passes [30,31]. Experiments on face-centered cubic (fcc) metals have shown that route BC is the optimum processing route for producing an array of equiaxed ultrafine grains separated by boundaries with high angles of misorientation [32,33]. Furthermore, experiments on Cu where samples were processed for up to 25 passes using an ECAP die with a channel angle of Φ = 90° showed that samples processed by route BC exhibited the highest yield stress, the maximum ultimate tensile stress and the highest ductilities after pressing through 10 passes or more [34]. The reason for an optimization using route BC is that this processing route contains the largest angular ranges for the slip occurring on each of the three orthogonal planes within the ECAP billet [35].

The strain imposed in each pass of ECAP is dependent primarily upon the angle Φ and, to a lesser extent, on the angle Ψ. It can be shown from first principles that the shear strain εN is given by a relationship of the form [36]

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

where N is the number of passes through the die. In conventional ECAP, it is generally assumed that the billet fills the corner of the die at the intersection of the two parts of the channel, and this produces a uniform microstructure throughout the billet. Nevertheless, experiments have
demonstrated that a strain inhomogeneity generally forms near the lower surface of the ECAP billet owing to the development of a corner gap or dead zone at the outer corner when the billet passes through the die. The formation of a dead zone has been widely reported in ECAP both through direct experimental observations [37–39] and through the predictions of finite element modeling (FEM) [40–43].

It can be seen from Fig. 1 that a conventional ECAP die may have a filet radius at the outer intersection of the channels, as represented by the angle \( \Psi \), but there is no corresponding filet radius at the inner point of intersection. It is possible that the sharp inner corner may produce a stress concentration, and it was suggested that this problem may be avoided by having identical arcs of curvature at both the inner and outer corners [44]. FEM was used initially to examine this configuration [44,45]. However, subsequent detailed experiments using an ECAP die with equal arcs of curvature at the inner and outer points of intersection showed this type of die is less effective than a conventional die in producing homogeneity throughout the ECAP billets [46]. These latter experimental results are consistent with a detailed examination of the effect of an inner corner angle using finite element analysis [42].

### 3.2. Principles of HPT

Processing by HPT is generally conducted using samples in the form of thin disks, although some recent experiments have described the use of small cylindrical specimens [47,48] and samples in the form of rings [49–51].

The principle of processing by HPT is illustrated schematically in Fig. 2 [52]. The HPT disks are placed between two massive anvils, subjected to an applied pressure \( P \), and then torsionally strained through rotation of either the lower or the upper anvil. In practice, there are three different types of HPT, which depend upon the geometry of the anvils and the degree of restriction imposed on any lateral flow during the processing operation. In unconstrained HPT, the anvils are flat so that the material flows outwards in an unconstrained manner during processing. In constrained HPT, the disk is placed within a cavity in the lower anvil, a plunger from the upper anvil enters the cavity, and there is no lateral flow during processing. In practice, however, most of the HPT processing is now conducted under quasi-constrained conditions as illustrated in Fig. 2 where the disk is contained within depressions on the inner surfaces of the upper and lower anvils, the disk thickness is slightly larger than the combined depths of the two depressions, and some limited outward flow occurs during torsional straining. Quasi-constrained HPT has been described successfully using FEM [53–55]. The differences between these various types of HPT are important, because careful experiments on pure Zr have shown that the occurrence of the allotropic phase transformation is influenced by the geometry of the die used for the HPT processing [56].

The equivalent von Mises strain \( \varepsilon_{eq} \) imposed on the disk in HPT is given by a relationship of the form [57–59]

\[
\varepsilon_{eq} = \frac{2\pi N r}{h \sqrt{3}} \tag{4}
\]

where \( N \) is the number of turns of torsional straining, \( r \) is the radial distance measured from the center of the disk, and \( h \) is the initial height (or thickness) of the sample. Inspection of Eq. (4) shows that the strain varies across the disk and there is a maximum value at the outer edge and a minimum value of zero strain at the center of the disk where \( r = 0 \). This relationship suggests, therefore, that HPT processing will produce materials containing very significant inhomogeneities. Nevertheless, as described in Section 6, a microstructural evolution occurs during HPT processing, and this provides an opportunity to achieve microstructures that are reasonably homogeneous.

There are two important problems associated with HPT processing that require careful attention.

First, there is a possibility that the disk may slip during processing, so that there is a difference between the measured torsional rotation of the disk and the overall rotation imposed by the HPT facility. This can be checked very easily by placing parallel marker lines on the upper and lower surfaces of a disk, torsionally straining the disk through a fraction of a rotation, such as one-half of a rotation, and then measuring the angle between the two lines. Experiments of this type show that the extent of slippage depends upon the material used for the disk, with little or no slippage for Al but some significant slippage for harder materials such as Fe [60]. These results also show that the extent of slippage increases when using faster rotational speeds or lower values for the imposed pressure \( P \).

Second, there is the problem that the two large anvils of the HPT facility may be slightly misaligned prior to the torsional straining. This is illustrated in Fig. 3a which shows the anvils in correct alignment and indicates the typical dimensions for the disk where the diameter
4. Principles of grain refinement in ECAP

4.1. Face-centered cubic metals

The processing of fcc metals by ECAP is relatively easy because of the multiplicity of active slip systems. The microstructural evolution occurring in fcc metals during processing by ECAP is now well documented, and an example is shown in Fig. 4 using OIM with samples of high-purity (99.99%) aluminum [67]. The unprocessed material is shown in Fig. 4a with an initial grain size of ~1 mm, and Fig. 4b–g shows OIM images after processing by ECAP using route BC with a die having internal angles of $\Phi = 90^\circ$ and $\Psi = 20^\circ$ through totals of 1, 2, 4, 6, 8 and 12 passes, respectively. It is important to note that Fig. 4b has the lowest magnification, Fig. 4c–f has the same magnification, and Fig. 4g has a higher magnification to display the ultra-fine-grained structure more fully. In all images, the colors within the grains correspond to the orientations of each grain as depicted by the unit triangle at upper right, the grain boundaries shown as black lines have misorientations $\theta$ between 2$^\circ$ and 15$^\circ$, and the boundaries shown as red lines have misorientations of $\theta > 15^\circ$. All these images were recorded on planes perpendicular to the pressing direction in ECAP, equivalent to the $X$ plane in Fig. 1. Except for Fig. 4g, the observations were made at points close to the central axes of the ECAP billets. For Fig. 4g, the image was recorded on the $X$ plane near the edge of the billet because of the growth of some anomalously large grains near the center of the billet after a total of 12 passes.

It is apparent from Fig. 4b that the first pass leads to an array of elongated cells or subgrains separated by low-angle boundaries and with an average width of ~2.9 $\mu$m. Small and elongated subgrains are visible after 2 passes, with an average size of ~1.8 $\mu$m in Fig. 4c, and thereafter there is a significant increase in the number of boundaries with high angles of misorientation. After 4 passes, the microstructure in Fig. 4e is reasonably homogeneous and consists of an array of essentially equiaxed grains with many boundaries having high angles of misorientation, and this equiaxed array continues up to 12 passes in Fig. 4g, except only in the central area of the billet after 12 passes where there are some isolated larger grains. The measured grain sizes were $\sim 1.1 \pm 0.6$ $\mu$m and $\sim 1.2 \pm 0.6$ $\mu$m after 8 and 12 passes in Fig. 4f and g, respectively, thereby suggesting there is no change in the grain size after 8 passes.

A similar set of OIM images, also recorded on the $X$ planes, is shown in Fig. 5 for an Al–1% Mg solid solution alloy processed by ECAP using route BC with a die having internal angles of $\Phi = 90^\circ$ and $\Psi = 20^\circ$ [68]. Fig. 5a is for the unprocessed material with an initial grain size of ~350 $\mu$m, and Fig. 5b–g shows samples processed through 1, 2, 4, 6, 8 and 12 passes, respectively; for these images, the magnification is low in Fig. 5b, higher in Fig. 5c and d, and even higher in Fig. 5e–g, with the colors in the grains again corresponding to the unit triangle shown at upper right. These results are similar to the high-purity aluminum in Fig. 4, with elongated subgrains after 1 pass and then an evolution to an ultrafine-grained structure with grain sizes of ~700 nm after 8 and 12 passes but with some larger grains remaining even after 12 passes. Comparing the microstructure of the alloy with pure aluminum in Fig. 4, it is apparent that high-purity Al evolves to a more uniform array of equiaxed grains but the alloy attains a smaller grain size. The smaller grain size in the alloy is consistent with very early results reported using different Al–Mg alloys processed by ECAP [69].

Information on the distributions of grain boundary misorientations is shown in Fig. 6a–f for the high-purity Al
processed by ECAP from 1 to 12 passes [67]. In each plot, the solid curve represents the statistical prediction for a set of random orientations [70] and information is given on the fractions of high-angle ($\theta > 15^\circ$) and low-angle ($\theta \approx 2^\circ - 15^\circ$) boundaries for each processing condition. Thus, the fraction of high-angle boundaries is initially low but increases to $\sim 50\%$ after 4 passes and continues to increase to $\sim 74\%$ after 12 passes. The distribution of boundaries after 12 passes in Fig. 6f is similar to the theoretical prediction for a random distribution, except that an excess of low-angle boundaries remains as a result of the continuous introduction of new dislocations when processing through repetitive passes.

An alternative procedure for demonstrating the development of homogeneity with increasing numbers of ECAP passes is to plot color-coded maps showing the distributions of values of the Vickers microhardness $H_v$ over cross-sectional planes of the billets. Examples are shown in Fig. 7 for a Cu–0.1% Zr alloy processed by ECAP at room temperature using route B$_C$ with a die having $\Phi = 110^\circ$ and $\Psi = 20^\circ$ through 1, 2, 4 and 8 passes [33]; for these plots, the hardness values were recorded on the $X$ cross-sectional planes, the axes $Y$ and $Z$ correspond to the two directions shown in Fig. 1, the point $(Y, Z) = (0, 0)$ corresponds to the center of the billet on the $X$ plane, and the colors represent the measured hardness values as denoted by the color key at the lower right. It can be seen from Fig. 7 that the overall microhardness values tend to increase with increasing numbers of ECAP passes and, ultimately, there is saturation after 8 passes where $H_v \approx 140$. Initially, there is a region of lower hardness near the bottom of the billet after a single pass and this is consistent with earlier reports on processing by ECAP [71–73], but the region of lower hardness is reduced in width after 2 passes and after 8 passes there is excellent homogeneity throughout the billet.

A mechanism for the grain refinement of fcc metals when processing by ECAP is depicted in the schematic illustrations in Fig. 8, where the microstructure is shown on the $Y$ plane, the three rows correspond to 1, 2 and 4 passes of ECAP, the three columns correspond to the processing routes A, B$_C$ and C, and the total angular range for all of the slip systems $\eta$ is indicated beneath each illustration [35]. The colors red, mauve, green and blue represent...
the slip lines introduced on the first, second, third and fourth pass, respectively, as predicted by the theoretical models for slip in each processing route [30,31] and the width of the subgrain bands is set equal to \( d \) for each pass through the die. It is readily apparent from Fig. 8 that processing using route \( B_C \) entails the largest angular range of slip, and it is reasonable to assume that the dislocations will rearrange and annihilate in a manner consistent with the LEDS (low-energy dislocation structures) theory [74,75]. After 4 passes, this will give the structures shown in Fig. 9, where there is a reasonably equiaxed array of grains in route \( B_C \) with an average size close to \( d \) but more elongated grains after processing by routes A and C [35]. The occurrence of more elongated microstructures in the early stages of processing using routes A and C is consistent with experimental observations [76].

4.2. Hexagonal close-packed metals

The processing of hexagonal close-packed (hcp) metals is more difficult than fcc metals because of the limited number of active slip systems. This problem was illustrated in an early investigation of the ECAP processing of pure magnesium and a magnesium-based alloy, where it was necessary to conduct the ECAP at high temperatures to avoid premature cracking but nevertheless it proved impossible to achieve significant grain refinement to the submicrometer level [77]. Subsequently, a two-step procedure was introduced for magnesium alloys, termed EX-ECAP, in which the grain size was initially reduced by extrusion prior to processing by ECAP [78]. This two-step process has been used effectively for achieving exceptional grain refinement in magnesium alloys [79–81] and other approaches are now also available such as using a back pressure in the ECAP die [82,83], increasing the channel angle in the die [84,85], reducing the pressing speed [86] and sequentially reducing the pressing temperature during each pass through the die [87].

Difficulties arise in the production of ultrafine grains in magnesium alloys because the refinement process occurs through the nucleation of fine grains along pre-existing grain boundaries or twins owing to the development of stress concentrations and the activation of both basal and non-basal slip [88]. This leads to a necklace-like array of new grains, and it means in practice that a critical grain size \( d_c \) is needed in order to achieve an array of equiaxed

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**Fig. 5. Microstructural evolution in an Al-1% Mg alloy processed by ECAP [68].**

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This page contains a figure illustrating the microstructural evolution in an Al-1% Mg alloy processed by ECAP, showing the changes from unprocessed to after 4 passes.
ultrafine grains. The principle of grain refinement in magnesium alloys is illustrated in Fig. 10, where the left column denotes the initial condition for grains either larger than $d_c$ or smaller than $d_c$ and the right column shows the microstructures after processing through one pass of ECAP [89]. Thus, for $d > d_c$, new grains form along the boundaries in a necklace-like manner but the grain structure is initially sufficiently coarse so that processing by ECAP produces a bimodal structure, as shown in Fig. 10b, where the centers of these larger grains are not yet consumed by the formation of the smaller grains. Conversely, if $d < d_c$, the initial grain size is sufficiently small that a single pass of ECAP produces a homogeneous array of ultrafine grains, as shown in Fig. 10d. This model is consistent with the EX-ECAP process because the preliminary extrusion is then used to produce a grain size that is smaller than the critical size $d_c$.

5. The processing of difficult-to-work alloys by ECAP

Many of the early investigations of ECAP processing were limited to the use of soft pure metals or solid solution alloys and they concentrated especially on the processing of fcc metals. More recently, much attention has been devoted to the processing of hcp metals such as magnesium-based alloys where the numbers of slip systems are limited. These so-called difficult-to-work alloys present significant challenges in processing by ECAP because of the development of plastic instabilities such as billet cracking and segmentation. Very clear examples of segmentation are shown in Fig. 11 for samples of an annealed ZK60 magnesium alloy and an extruded AZ31 magnesium alloy processed by ECAP for only one pass at different temperatures [90]. These are examples of the development of regular segmentation in which the billets become divided into
relatively uniform and discrete segments that are held together by small portions of material along the bottom surfaces.

The problem of segmentation is due to the development of an initial shear localization and local cracking which, ultimately, leads to catastrophic failure [91]. The shear localization is associated with flow softening that destabilizes plastic flow, and this may be investigated using FEM to obtain critical information on the characteristics of flow during the ECAP process. An example is shown in Fig. 12, where FEM was employed to depict billets with regular grid patterns on their side faces, equivalent to the Y planes in Fig. 1, in the exit channel of the ECAP die after a single pass using a channel angle of $\Phi = 90^\circ$ and an outer arc of curvature of $\Psi = 20^\circ$ [92]. This modeling demonstrates that the occurrence of plastic instabilities in ECAP processing is dependent upon the value of the strain rate sensitivity $m$. For low values of $m$, as in the two upper illustrations in Fig. 12, there are discontinuities in the amounts of strain introduced into the billets in the X or axial direction and these regular instabilities lead in practice to segmentation of the billets. By contrast, the two lower illustrations show that a value of $m$ of 0.05 or higher produces a steady flow in the billet and an absence of any segmentation. These calculations demonstrate the importance of measuring the strain rate sensitivity prior to undertaking ECAP.

Another important parameter in the processing of difficult-to-work alloys is the channel angle $\Phi$ within the ECAP die. It was shown by FEM that cracking and segmentation may be reduced or even eliminated by increasing the angle $\Phi$, and this effect was confirmed experimentally by pressing two billets of a ZK60 magnesium alloy through dies with different channel angles. The result is shown in Fig. 13, where billets were pressed through one pass at a temperature of 473 K with the upper billet pressed through a die with $\Phi = 90^\circ$ and the lower billet pressed through a die with $\Phi = 110^\circ$ [85]. For this material, the initial strain rate sensitivity was estimated as $m \approx 0.2$, and it is apparent that the billet passing through a channel angle of $90^\circ$ shows extensive segmentation whereas there is no significant damage from using a channel angle of $110^\circ$. These results
are consistent with the FEM calculations and they show that segmentation may be suppressed by increasing not only the strain rate sensitivity but also the channel angle.

6. Evolution towards homogeneity in HPT

6.1. The advantages and disadvantages of processing by HPT

As discussed in Section 3.2, processing by HPT provides an alternative to processing by ECAP. There are both advantages and disadvantages in using HPT. An important advantage is that, by comparison with ECAP, HPT produces materials with both smaller grain sizes and a higher fraction of grain boundaries with high angles of misorientation [93-95]. But a major disadvantage is that the samples are very small, generally in the form of thin disks, and there have been only limited attempts to extend HPT processing to larger cylindrical samples [47,48].

An important advantage in the processing of hcp metals is that HPT provides an opportunity for avoiding some of the difficulties associated with processing by ECAP. In
practice, the ECAP processing of magnesium alloys is usually undertaken at temperatures of \( \sim 473 \, \text{K} \) or higher in order to avoid the problems of cracking at lower temperatures. However, the imposition of a high hydrostatic pressure in HPT provides an opportunity for processing these same alloys at much lower temperatures. An example is the AZ31 magnesium alloy which was successfully processed by HPT at room temperature to give a grain size of \( \sim 0.9 \, \mu\text{m} \) [96-98].

As noted earlier, Eq. (4) predicts the development of gross homogeneities in HPT processing because the strain varies across the disk from zero at the center to a maximum at the edge. In practice, however, many experiments have revealed a gradual evolution with increasing strain towards a homogeneous distribution of the measured hardness values. Furthermore, it is reasonable to anticipate that the degree of homogeneity in the hardness measurements provides a reasonable indication of the degree of homogeneity in the microstructure within the disk. The significance and variations in the evolution towards homogeneity in HPT are examined in the following sections.

### 6.2. Development of homogeneity on the horizontal planes of disks processed by HPT and the concept of a minimum grain size

There are now many examples showing an evolution in the values of hardness in the plane of the HPT disks...
towards an essentially saturated condition. An example of a typical evolution with additional straining is shown in Fig. 14 for a commercial purity (99.5%) aluminum Al-1050 alloy where the hardness values are denoted by colors shown in the color key at the lower right [99]. It is readily apparent that the hardness values are higher around the periphery of the disk after a 1/4 turn and there are lower values of hardness in the central region extending over a diameter of ~5 mm. This region of lower hardness is reduced to a diameter of ~3 mm after one turn, and essentially it disappears after five turns to give a reasonable level of hardness homogeneity of Hv ≈ 65 throughout the disk. This type of evolution has been demonstrated for numerous materials processed by HPT [52,100–108].

It was suggested in early experiments on HPT that the hardness measurements recorded after different numbers of revolutions may be conveniently correlated by plotting the individual hardness values as a function of the predicted equivalent strains calculated using Eq. (4) [109]. An example of this approach is shown in Fig. 15 for an Al–0.6% Mg–0.4% Si alloy after processing by HPT at room temperature (298 K) using a pressure of 6.0 GPa and a rotation speed of 1 rpm through total numbers N, from 1 to 20 turns: the point for N = 0 denotes the initial material without processing [110]. This plot shows that the values of Hv increase rapidly in the early stages of processing and, ultimately, there is a reasonable saturation in hardness at Hv ≈ 158 at equivalent strains above ~100. There are now many similar reports showing the variation of hardness with equivalent strain for a number of different materials [50,51,106,108,111–127].

An important and essentially unresolved question concerns the maximum level of grain refinement that may be achieved in processing by HPT [128,129]. Some preliminary information on the minimum grain size may be obtained using a dislocation model that was initially developed to predict the minimum grain size attainable by ball-milling [130]. The predictions of this model are generally consistent with results obtained for samples processed by HPT [131], and a new dislocation model was recently proposed specifically to predict the minimum grain size when processing by HPT [132]. A similar approach was also developed to predict the minimum grain size attainable in ECAP [133].

6.3. Variations in the development of homogeneity on horizontal planes during processing by HPT

Most metals tend to give results that are reasonably consistent with those shown in Figs. 14 and 15: specifically, the hardness is initially higher at the edge of the disk and lower in the center, but the hardness in the center increases until ultimately there are similar hardness values throughout the disk. Nevertheless, two important differences have been observed in some materials.

First, early results on the HPT processing of high-purity (99.99%) aluminum at room temperature with an imposed pressure of 1.25 GPa showed that the hardness was initially higher in the center of the disk and lower around the edge, whereas with increasing numbers of turns the hardness values in the center were reduced and became similar to those at the edge [134]. An example of this effect is shown in Fig. 16 for high-purity Al processed through 1, 3 and 5 turns [134]. Inspection shows the hardness values after 1 turn are exceptionally high in the center of the disk over an area with a diameter of ~4 mm, but this region is reduced to a diameter of <0.5 mm after 3 turns and disappears after 5 turns. The hardness in the initial stages of processing may be characterized in greater detail by taking
hardness measurements after processing through fractional values of one complete revolution. This result is shown in Fig. 17 where the values of Hv are plotted against the equivalent strain after processing for various numbers of revolutions up to a total of 1 turn [135].

It is readily apparent from Fig. 17 that all the datum points tend to cluster around a single line which shows a peak at an equivalent strain of ~2 and at higher strains, above ~7, the hardness attains a steady-state condition and remains constant to even higher strains. The occurrence of a saturation condition at higher strains is analogous to the data shown in Fig. 15 but the appearance of the overall curve is different because of the initial high values of hardness. Careful observations by transmission electron microscopy (TEM) showed the presence of smaller grains with highly deformed structures in the central region of a disk processed through one pass, whereas near the edge of the disk the grains were larger and appeared similar to a fully annealed condition after extensive recovery [134]. These microscopic observations are consistent with the hardness measurements shown in Fig. 16 and they demonstrate that the very high stacking fault energy in aluminum leads to easy recovery by cross-slip around the edge of the disk in the very earliest stages of processing. By contrast, the recovery in the center of the disk occurs more slowly so that the hardness remains high in the early stages, but it is reduced ultimately to the same level as at the disk periphery. Essentially similar trends were reported recently for pure (99.9%) Mg [136] and high purity (99.99%) Zn [119].

Second, there are examples for some materials where the hardness is lower, rather than higher, after processing by HPT. An example is shown in Fig. 18 for a Zn–22% Al eutectoid alloy processed at room temperature using an applied pressure of 6.0 GPa [137]. Now the hardness values are higher in the center of the disk and lower around the periphery, and this is similar to Fig. 16 for high-purity Al except that all the measured values of hardness are now lower than in the annealed and unprocessed material. This can be seen by plotting the values of the Vickers microhardness against the equivalent strain for various testing conditions as shown in Fig. 19 where the initial annealed and unprocessed condition is denoted by the upper dashed line at Hv ≈ 68 and the values of the equivalent strain were estimated using Eq. (4) and taking the thickness as h ≈ 0.70 mm to allow for the small reduction in thickness that occurs during processing [138]. Inspection of Fig. 19 shows that all datum points are in excellent agreement, and there is again a gradual transition to a saturated condition but now the results are essentially the inverse of those obtained in most alloys, as depicted in Fig. 15, because the hardness values decrease, rather than increase, to the final saturation hardness.

The weakening of this alloy by HPT processing appears unusual but it may be understood by noting that earlier observations by TEM showed that the processing of the Zn–22% Al eutectoid alloy by HPT at room temperature led to a significant reduction in the presence of rod-shaped precipitates of stable hcp Zn which were visible within the Al-rich grains in the annealed condition [139,140]. This means, therefore, the high pressures associated with HPT

![Fig. 14. Evolution of microhardness in an Al-1050 alloy processed by HPT [99].](image)

![Fig. 15. Evolution to a saturation hardness in an Al–0.6% Mg–0.4% Si alloy processed by HPT for up to 20 turns [110].](image)
processing lead to an absorption of many of the Zn precipitates by the Zn-rich grains. It is interesting to note that a similar weakening by HPT processing was reported also for the Pb–62% Sn eutectic alloy [141] but conversely it appears that the weakening effect is not an inherent feature of all two-phase eutectic or eutectoid alloys because experiments on an Al–33% Cu eutectic alloy gave increasing values of hardness around the peripheries of the disks as in conventional alloys [142].

The overall conclusions from these experiments is that the hardness values incurred in HPT processing, and therefore the microstructural features within the material, tend consistently towards a saturation condition at high strains and therefore with increasing numbers of torsional revolutions. Although this is contrary to the general inhomogeneity that is anticipated by a direct application of Eq. (4), it is consistent with a theoretical approach for HPT processing based on the application of strain gradient plasticity modelling [143]. Nevertheless, the evolution towards a condition of saturation may occur in three different ways, as depicted earlier in Figs. 15, 17 and 19 and now illustrated schematically in Fig. 20 where the initial annealed and unprocessed conditions are indicated by the horizontal lines on the vertical axes [144]. In Fig. 20a, the hardening occurs in the absence of any significant recovery as in a wide range of
metallic alloys; in Fig. 20b, the high stacking fault energy in a material such as high-purity aluminum leads to easy cross-slip and rapid recovery, giving an initial bell-shaped relationship between the hardness values and the equivalent strain; and in Fig. 20c, there is a weakening effect, as in the Zn–22% Al eutectoid alloy, owing to the high pressure imposed in the HPT processing.

6.4. Variations in the development of homogeneity on internal planes during processing by HPT

The results described in the preceding sections relate exclusively to hardness measurements taken on the horizontal upper or lower planes of HPT disks but it is important also to determine the variations in hardness occurring throughout the disks. The first investigation of the hardness values on sections within HPT disks was conducted using high-purity (99.99%) Al by measuring the hardness values on horizontal planes located at distances of ~200 μm from the top and bottom surfaces of the disks, henceforth denoted as the upper and lower positions, and on a plane cut at the mid-section parallel to the upper and lower surfaces and denoted as the center position. Fig. 21 shows color-coded contour maps recording the hardness values across the disks for these three positions after processing at room temperature using a pressure of 6.0 GPa for 1/4 turn in the upper row and 1/2 turn in the lower row, where the coordinate system X and Y denotes two randomly selected orthogonal axes with coordinates (0,0) at the center of each disk [145]. Inspection shows that the results are essentially identical for each plane after 1/4 turn, with a doughnut-like pattern in which there is a ring of higher hardness and a small central region of much lower hardness. The results after 1/2 turn are similar and, again, all planes are essentially identical but now the widths of the rings of higher hardness are reduced by comparison with the results after 1/4 turn.

To determine the variations in hardness at the upper, center and lower positions within the disks for different numbers of torsional revolutions, Fig. 22 shows the variations in the Vickers microhardness across randomly selected diameters within each disk, where the lower line corresponds to the annealed condition at Hv 20, the error bars represent the 95% confidence limits, and the upper points are recorded for 1/4, 1/2, 1, 5 and 20 turns [146]. These results demonstrate conclusively that there are no significant variations between the different sectional planes for disks of high-purity aluminum and, in addition, the results after 20 turns, shown in Fig. 22e, display a very high level of homogeneity throughout the disk.

The evolution into a material with a high degree of homogeneity is further illustrated in Fig. 23 by taking hardness measurements on vertical planes cut through the disks to reveal one-half of each cross-section [146]. These five samples correspond to the disks shown in Fig. 22 with the centers of each disk lying at the left axes. Thus, although there are some regions of higher hardness in these disks in the early stages of processing, these regions extend throughout each disk from the upper to the lower surfaces. After 5 turns, there is good homogeneity in the high-purity aluminum and, after 20 turns, the hardness values are
Fig. 20. Three different examples of the evolution to a saturation hardness when processing by HPT for conditions (a) without recovery, (b) with recovery and (c) with weakening [144].

Fig. 21. Evolution of microhardness on different planes of sectioning in high-purity Al processed by HPT for 1/4 and 1/2 turn [145].
homogeneous in the through-thickness direction throughout the disc. These results contrast with the processing of magnesium alloys, as shown in Fig. 24 for the AZ31 alloy, where there are higher hardness values adjacent to the lower surfaces of the disks after 1 and 5 turns [147]. Similar heterogeneity was reported also in an AZ91 magnesium alloy after processing by HPT [148].

7. Mechanical properties after SPD processing

The preceding sections confirm the ability to achieve exceptional grain refinement in bulk metals through the application of SPD processing and to attain good homogeneity in materials processed using ECAP and also when using HPT provided the processing is continued to a sufficiently high strain. In order to examine the mechanical properties of materials subjected to SPD processing, it is necessary to consider separately the behavior at low temperatures (typically <0.5\(T_m\)) where diffusion is not important and at high temperatures (typically ≥0.5\(T_m\)) where flow is controlled by the rate of diffusion.

7.1. Mechanical properties in the low-temperature regime

Processing through the application of SPD, whether by ECAP or HPT, leads to significant grain refinement to the
submicrometer or even the nanometer level. This means that, as a consequence of the Hall–Petch relationship in Eq. (1), these materials are anticipated to exhibit exceptional strength. There are now many reports documenting the high strengths of ultrafine-grained materials processed using SPD techniques [149–151], but there is evidence also for a deviation from the Hall–Petch relationship at grain sizes smaller than \( \sim 500 \, \text{nm} \), which may be due to the easy movement of extrinsic dislocations in the non-equilibrium grain boundaries introduced during the SPD processing [110].

Examples of the mechanical testing of materials after processing by ECAP are shown in Fig. 25 [152]. For these tests, samples were prepared by processing by ECAP using a die with a channel angle of \( \Phi = 90^\circ \) and an outer arc of curvature of \( \Psi = 20^\circ \), and with the processing conducted using route B\(_C\) through 1, 2 and 6 passes corresponding to imposed strains of \( \sim 1 \), \( \sim 2 \) and \( \sim 6 \), respectively. The Al-6061 alloy in Fig. 25a was pressed at room temperature, and the Al-7034 alloy in Fig. 25b was processed at 473 K. Following ECAP, compression samples in the form of small rectangular parallelepipeds were cut from the ECAP billets with their longest axes lying parallel to the \( X \), \( Y \) or \( Z \) directions as defined by the orthogonal axes shown in Fig. 1. All compression testing was conducted at room temperature (298 K) using an initial strain rate of \( 5.5 \times 10^{-4} \, \text{s}^{-1} \) and with identical samples also cut and tested from the as-received and unprocessed alloys. The testing showed similar results for the stress–strain curves in all three orthogonal directions for both alloys and therefore, for simplicity, this report will document only the data obtained using compression samples cut in the \( Z \) direction.

Fig. 25a shows plots of the engineering stress against the engineering strain for the as-received Al-6061 alloy and for samples of the alloy processed by ECAP through 1\( p \), 2\( p \) and 6\( p \), where \( p \) denotes the numbers of passes. Thus, the as-received alloy shows a gradual yielding and then a significant rate of strain hardening, whereas the samples processed by ECAP show higher strength, an abrupt and reasonably well-defined yield point and no significant hardening. It is also apparent that the yield stresses increase with increasing numbers of ECAP passes. The results shown in Fig. 25a are generally similar to those reported...
for several different commercial aluminum-based alloys processed by ECAP and then tested in tension at room temperature [153].

A similar set of stress–strain curves is shown in Fig. 25b for an Al-7034 alloy but these results are unusual because the alloy is now weaker, rather than stronger, than the unprocessed material. Specifically, the yield stress is lower for the alloy processed through 1p by comparison with the as-received alloy, and the yield stress also continues to decrease with increasing numbers of passes. Although these results are unusual, they match results obtained in tensile testing, where it was shown that the alloy exhibited a significant loss of strength when testing at room temperature [154]. This loss of strength is due to a transformation occurring at the processing temperature of 473 K when the semi-equilibrium η′-phase, which is the major strengthening phase in the Al-7034 alloy, transforms into the η-phase (MgZn2) and also the rod-like η-phase precipitates are fragmented by the pressure applied in the ECAP processing. It is interesting to note that the weakening of the Al-7034 alloy introduced by ECAP processing in Fig. 25b has similarities to the weakening of the Zn–22% Al eutectoid alloy when processing by HPT, as documented in Fig. 19, with the weakening in both materials occurring as a direct consequence of the high pressures imposed in the processing operation.

7.2. Mechanical properties in the high-temperature regime after processing by ECAP

In the high-temperature regime, where diffusion is important, the rate of flow is controlled by Eq. (2) and therefore the flow rate increases with decreasing grain size. Superplastic flow is an important process at elevated temperatures and it is of considerable technological importance because it forms the basis for the superplastic forming industry which plays a major role in the processing of metallic parts for use in the aerospace, automotive and other industries [155]. Superplasticity refers specifically to the ability of a material to pull out to a tensile elongation of at least 400% with a measured strain rate sensitivity of ~0.5 [156] and it is well known that the process requires a very small grain size which is typically smaller than ~10 µm [157]. Thus, provided the ultrafine grains produced by SPD processing are reasonably stable at elevated temperatures, there should be an opportunity for using SPD processing to produce materials exhibiting exceptional superplastic elongations. Furthermore, the inverse dependence on grain size in Eq. (2) suggests that these high elongations may occur at unusually rapid strain rates that may lie even within the regime of high strain rate superplasticity which is defined formally as superplastic elongations occurring at strain rates at and above a strain rate of $10^{-2}$ s$^{-1}$ [158]. In fact, there was a very early demonstration of the occurrence of high strain rate superplasticity in two commercial aluminum alloys processed by ECAP to produce elongations of up to ~1000% at strain rates of $10^{-2}$ s$^{-1}$ [159].

There are now a large number of reports documenting the occurrence of superplastic elongations in materials processed by ECAP. Thus, a detailed tabulation, published in 2007, recorded more than 60 reports of superplasticity in materials processed by ECAP with an emphasis on Al and Mg alloys [160]. Fig. 26 shows an example of superplasticity in a ZK60 magnesium alloy (Mg–5.5% Zn–0.5% Zr), where the extruded alloy was processed by ECAP through 2 passes at a temperature of 473 K using a die with angles of $\Phi = 90^\circ$ and $\Psi = 20^\circ$ and then tested in tension at 473 K using an initial strain rate of $1.0 \times 10^{-4}$ s$^{-1}$ [161]. The elongation at failure is $\Delta L/L_0 = 3050\%$, where $\Delta L$ is the change in length and $L_0$ is the initial gauge length. This superplastic elongation of 3050% is the highest reported to date in any magnesium alloy and it is also the highest elongation recorded for any alloy processed by ECAP [162]. In addition, the absence of any visible necking within the gauge length in Fig. 26 demonstrates conclusively that this is true superplastic flow [163].

It is now well established that superplasticity occurs through the process of grain boundary sliding (GBS) in which the small grains slide over each other in response to the applied stress [164]. GBS is now well documented for conditions of both high-temperature creep and superplasticity [165] and it has been established that superplastic flow is achieved when the grain size is sufficiently small that it is not possible to form subgrains within the grains [166]. Accordingly, a theoretical model for superplasticity was developed in which GBS is accommodated by slip within the adjacent grains so that the dislocations in the slip process move across the grains and then pile up and climb into the opposing grain boundaries [167]. This model leads to a relationship of the form shown in Eq. (2) with $p = 2$, $n = 2$, $D = D_{gb}$ for the coefficient for grain boundary diffusion and with a dimensionless constant of $A \approx 10$. Sufficient results are now available that it is instructive to check the validity of this model in describing superplastic flow in ultrafine-grained materials processed by ECAP.

These analyses are shown in Figs. 27 and 28 for Al and Mg alloys, respectively [168], where the experimental data are taken directly from extensive reports for Al [159,169–175] and Mg [84,161,176–185] alloys processed by ECAP. In each plot, the data are presented as the temperature and grain size compensated strain rate, $\left\{ 6kT/D_{gb}Gb \right\}(d/b)^2$, against the normalized stress $\sigma/G$, and the lines with a slope of $n = 2$...
represent the predicted theoretical strain rates for superplastic flow \( \dot{\varepsilon}_{sp} \) using the model for GBS accommodated by intragranular slip [167]. It is readily apparent that, for both sets of materials, the experimental datum points cluster around the predicted lines to within about one order of magnitude of strain rate thereby showing excellent agreement with the theoretical model and demonstrating that superplastic flow in ultrafine-grained materials occurs by the same dislocation process as in conventional superplastic alloys.

Two-phase alloys often exhibit superplasticity because the small grain sizes are retained through the presence of two separate phases. However, in practice the sliding contributions may differ between the various types of interfaces. For example, in the Zn–22% Al eutectoid alloy it was shown in early experiments that the sliding contributions were high on the Zn–Zn and Zn–Al interfaces but relatively low on the Al–Al interfaces [186,187]. Later, the same trend was reported also for the Zn–22% Al alloy after processing by ECAP [188,189]. In a recent detailed analysis of the role of GBS in the superplastic flow of the Zn–22% Al alloy processed by ECAP, it was noted that the differences between these various interfaces may influence the total extent of superplasticity in the alloy [190]. Thus, it is well established that agglomerates are formed in the Zn–22% Al alloy after processing by ECAP [140] or HPT [139] but these agglomerates may be removed in HPT processing by torsionally straining through larger numbers of turns [137]. The nature of these agglomerates is illustrated in Fig. 29, where Fig. 29a shows the annealed condition consisting of a homogeneous distribution of Zn and Al grains so that there is a low fraction of Al–Al interfaces, Fig. 29b shows the agglomerates of small grains formed after SPD processing where there is now a high fraction of Al–Al interfaces, and Fig. 29c shows the microstructure after SPD processing to a high imposed strain where the fraction of Al–Al interfaces is now reduced [190]. Thus, this microstructural evolution, and the presence of an excess number of Al–Al boundaries in the early stages of SPD processing, affects the measured contributions of GBS.
and this is in agreement with experimental measurements [190].

7.3. Mechanical properties in the high-temperature regime after processing by HPT

Processing by HPT also produces samples that have a potential for exhibiting superplastic elongations. An example is shown in Fig. 30 for a Zn–22% Al alloy processed by HPT through 5 turns at room temperature under an applied pressure of 6.0 GPa and then tested in tension at 473 K using strain rates from $1.0 \times 10^{-3}$ to $1 \text{s}^{-1}$ [191]. To avoid any microstructural inhomogeneities that may exist in the centers of the HPT disks, these tensile specimens were produced by electro-discharge machining, so that two parallel samples were cut from off-center positions within each disk [192]. It is evident from Fig. 30 that very high elongations may be achieved at strain rates in the vicinity of $10^{-1} \text{s}^{-1}$ whereas there is clear evidence for necking in the two samples pulled at the slowest strain rates. A tabulation of superplastic data for samples prepared by HPT shows that the elongation of 1800% visible in Fig. 30 is the highest elongation reported to date for any material processed by HPT [191].

Deformation mechanism maps are a useful tool for providing a visual display of the rate-controlling flow processes occurring in ultrafine-grained materials produced by SPD processing and a recent report reviewed the types of maps currently available [2]. There are now several examples of deformation mechanism maps for metals processed by ECAP or HPT [2,190,191,193–197] and an example is shown in Fig. 31 for the Zn–22% Al eutectoid alloy at a temperature of 473 K [2]. This map plots the normalized grain size $d/b$ against the normalized stress $\sigma/G$, and the various fields denote areas in grain size-stress space within which the designated mechanism is rate controlling. The map was constructed using the theoretical models for Nabarro–Herring [198,199] and Coble [200] diffusion creep and using creep data for the three regions of flow associated with superplasticity in the Zn–22% Al alloy [201,202], where region II represents superplasticity and regions I and III are regions of non-superplastic behavior [203]. The dashed line at $d/b = 20(\sigma/G)^{-1}$ shows the condition where the sample grain size is equal to the subgrain size [5], so that subgrains are not formed below this line in the superplastic region II. The experimental points shown in Fig. 31 are for the four specimens shown in Fig. 30, where the grain size is 610 nm after processing by HPT. It is readily apparent that these points lie in the correct regions on the map and they show the gradual transition to region I at the slowest strain rate. This type of map is easily constructed and provides very useful information on the anticipated transitions between different flow processes.

8. Potential for future trends in SPD processing

There are several recent reports documenting the innovation potential for bulk materials processed using SPD techniques [204–208]. However, a first important requirement in establishing ECAP as a viable processing technique is to scale-up the processing facility for the preparation of larger billets. In practice, this can be performed fairly easily by fabricating ECAP dies with larger channel dimensions [209–211] and in this way there is a potential for the ECAP process to become commercialized [212].

There are also modifications to the processing techniques that offer improvements over the traditional procedures. For example, processing by ECAP in the conventional form shown in Fig. 1 leads to distortions at both ends of the billets and to an estimated final usage ratio of only $\sim 0.5–0.7$ [213]. In practice, this wastage of 30% or more may be avoided by using an ECAP die in which the channel is bent twice in order to give two parallel channels, since it has been shown experimentally that this produces little or no wastage at the ends of the billets [213]. The approach of using parallel channels has been adopted successfully in several investigations [214–216]. Another approach is to make use of the conform process which
was developed many years ago for the continuous extrusion forming of metal wires [217,218]. By incorporating an ECAP step into the conventional conform process, an early investigation demonstrated the potential for achieving a continuous processing capability [219] and this procedure was later used in several investigations [220,221]. Although traditional HPT uses samples in the form of very thin disks, the process may be developed to give continuous HPT (CHPT) and this procedure was used successfully for the continuous processing of wire samples [115,122].

Processing by ECAP has been used successfully for the production of micro-gears [222,223] and for the production of net-shaped microelectro-mechanical system (MEMS) components [224,225]. It is important to note also that the grain size has a strong influence on the local flow behavior during micro-forming and it is believed to be the limiting factor that dictates the minimum size for the production of geometrical features [226]. This means in practice that nanocrystalline materials exhibit the best formability in micro-imprinting [227] and the use of ultrafine-grained materials may improve the flow behavior and the filling quality since size effects, such as process scatter and uneven shape evolution, are significantly reduced in comparison with the conventional coarse-grained materials [228–230]. Very recent results have demonstrated the potential for making use of ECAP processing in micro-forming applications [231].

Finally, it should be noted that considerable attention is now focused on the development of ultrafine-grained materials for use in specific applications. For example, magnesium alloys are under consideration for use in hydrogen storage [232–235] and ultrafine-grained titanium is an excellent candidate material for use in dental and surgical implants [236–240]. Descriptions of these developments are beyond the scope of this paper, but nevertheless these topics provide exciting challenges for new and meaningful research that will have scientific rigor and yet provide valuable information for the future use of these materials in technological applications.

9. Summary

1. Processing through the application of SPD, as in ECAP and HPT, provides an excellent opportunity for producing bulk solids with submicrometer or even nanometer grain sizes.
2. Good homogeneity can be achieved in both ECAP and HPT provided the samples are processed to sufficiently high strains.
3. These processing methods generally produce high strength at ambient temperature, but in some materials there may be a weakening due to the high pressure imposed during the processing operation.
4. If the ultrafine grains are reasonably stable during tensile testing at elevated temperatures, it is possible to achieve excellent superplastic properties.

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