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DEVELOPMENTS IN THE USE OF ECAP PROCESSING FOR GRAIN REFINEMENT

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Abstract. Equal-channel angular pressing (ECAP) is one of the most efficient techniques of severe plastic deformation for the production of bulk ultrafine-grained metals. During the last decade the intensive developments of ECAP have been associated with numerous modifications of the die-set design as well as establishing new processing routes and regimes. This paper describes the various types of ECAP that have been developed recently and applied for grain refinement. Special attention is focused both on new trends in ECAP processing associated with the development of continuous processing and on its efficiency.

1. INTRODUCTION

Interest in the processing of bulk ultrafine-grained materials (UFG) through the application of severe plastic deformation (SPD) has grown significantly over the last decade [1-4]. Processing by SPD refers to various experimental procedures of metal forming that may be used to impose very high strains to materials leading to exceptional grain refinement [5]. The principles of SPD processing have been successfully demonstrated recently in several techniques such as high-pressure torsion (HPT), twist extrusion (TE), multi-directional forging (MDF) and some others where the initial dimensions of the samples are reasonably retained [5]. However, at present the most attractive SPD processing technique is Equal-Channel Angular Pressing (ECAP).

The process of ECAP, known also as Equal-Channel Angular Extrusion (ECAE), was first introduced by Segal and his co-workers in the 1970's and 1980's at an institute in Minsk in the former Soviet Union [6]. The basic objective at that time was to develop a metal forming process where high strains may be introduced into metal billets by simple shear. However, although the objective was successfully achieved, the early development of the pressing operation received only limited attention in the scientific community. This situation changed in the 1990's when reports began to appear documenting the potential for using ECAP to produce ultrafine-grained metals with new and unique properties [7,8] and these reports initiated an intense and ongoing interest in scientifically investigating, developing and ultimately utilizing the ECAP process in industrial applications. Recent developments in ECAP processing have been associated with modifications and modernizations of the die-set design and technique as well as establishing new processing routes and regimes [9].

This paper describes the various types of ECAP that have been developed recently and applied in the production of ultrafine-grained structures.

2. CONVENTIONAL ECAP WITH RODS AND BARS

There are a number of reports describing the fundamental process of metal flow during ECAP [10-13] and in practice the principle of ECAP is illustrated

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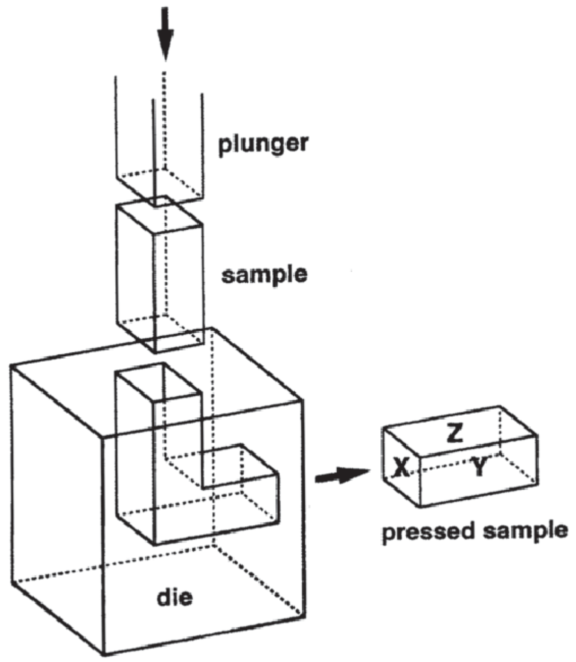

Fig. 1. Schematic illustration of a typical ECAP facility: the *X*, *Y* and *Z* planes denote the transverse plane, the flow plane and the longitudinal plane, respectively [14].

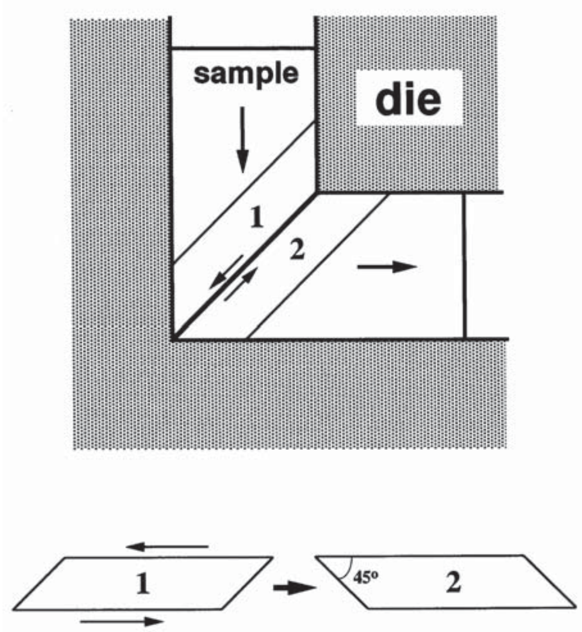

Fig. 2. The principle of ECAP showing the shearing plane within the die: the elements numbered 1 and 2 are transposed by shear as indicated in the lower part of the illustration [15].

schematically in Fig. 1 [14]. For the die shown in the illustration, the internal channel is bent through an abrupt angle, Φ, equal to 90° and there is an additional angle, Ψ, equal to 0° in Fig. 1, which represents the outer arc of curvature where the two channels intersect. The sample, in the form of a rod or bar, is machined to fit within the channel and the die is placed in some form of press so that the sample can be pressed through the die using a plunger. The nature of the imposed deformation is simple shear which occurs as the sample passes through the die as shown schematically in Fig. 2: for simplicity, the die angle in Fig. 2 is 90°, the theoretical shear plane is shown between two adjacent elements within the sample numbered 1 and 2, and these elements are transposed by shear as depicted in the lower part of the diagram [15]. Despite the introduction of a very intense strain as the sample passes through the shear plane, the sample ultimately emerges from the die without experiencing any change in the cross-sectional dimensions. This is illustrated by the pressed sample in Fig. 1. Three separate orthogonal planes are also defined in Fig. 1 where these planes are the *X* or transverse plane perpendicular to the flow direction, the *Y* or flow plane parallel to the side face at the point of exit from the die and the *Z* or longitudinal plane parallel to the top surface at the point of exit from the die, respectively. The retention of the same cross-sectional area when processing by ECAP, despite the introduction of very large strains, is the important characteristic of SPD processing and it is a characteristic which distinguishes this type of processing from other conventional metal-working operations such as rolling, extrusion and drawing.

Since the cross-sectional area remains unchanged, the same sample may be pressed repetitively to attain exceptionally high strains. For example, the use of repetitive pressings provides an opportunity to invoke different slip systems on each consecutive pass by simply rotating the samples in different ways between the various passes [10]. In practice, many of the investigations of ECAP involve the use of bars with square cross-sections and dies having square channels. For these samples, it is convenient to develop processing routes in which the billets are rotated by increments of 90° between each separate pass. The same processing routes

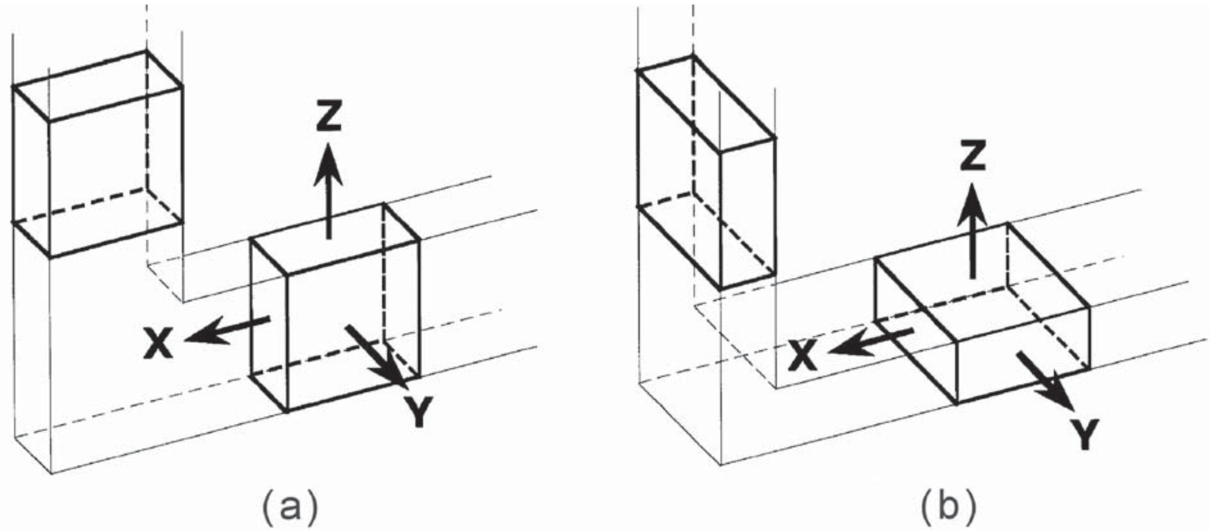

Fig. 3. Application of ECAP to plate samples: (a) vertical configuration and (b) horizontal configuration [17].

are also easily applied when the samples are in the form of rods with circular cross-sections. Four fundamental processing routes have been identified in ECAP: these are route A where the sample is pressed repetitively without any rotation, route B_A where the sample is rotated by 90° in alternate directions between consecutive passes, route B_C where the sample is rotated in the same sense by 90° between each pass and route C where the sample is rotated by 180° between passes [16].

3. THE APPLICATION OF ECAP TO PLATE SAMPLES

For some industrial applications, such as the use of ultrafine-grained materials produced by ECAP in superplastic forming operations, it is necessary that the as-pressed samples are in the form of thin metallic sheets. This requirement has prompted an interest in the possibility of applying ECAP to plate samples where the as-pressed materials can be readily prepared for use in conventional metal forming facilities. A limited number of reports are now available on the application of ECAP to plate samples [17-21].

When pressing plate samples, it is necessary to first recognize that there are two distinct pressing configurations. These configurations are illustrated in Fig. 3 where the plate is oriented either (a) in a vertical configuration or (b) in a horizontal configuration [17]: the *X*, *Y* and *Z* axes are indicated in Fig. 3 and they follow the same convention introduced in Fig. 1. Thus, these two configurations correspond to plates having their major axes either in the *X* and *Z* directions or in the *X* and *Y* directions, respectively.

Inspection of Fig. 3 shows that, unlike the bars and rods described in the preceding section, the number of possible rotations between passes is now limited. Thus, considering the vertical configuration, the sample may be rotated in several different ways. First, by rotation by 180° around the *X* axis, where this is equivalent to route C and it is designated route C_X where C denotes a rotation by 180° and the subscript X denotes a rotation about the *X* axis. Second, by rotation by 180° degrees around the *Z* axis in route C_Z. In practice, an examination of the shearing patterns associated with these two routes shows that they are identical to the conventional route C available for bar and rod samples. However, there are two additional possibilities not available for bars and rods and these are routes B_{AY} and B_{CY} where the sample is rotated by 90° around the *Y* axis after each pass either alternately in different directions or in the same sense, respectively. Routes B_{AY} and B_{CY} are not easily executed in practical situations because the plate is sheared into a parallelogram on the first and all subsequent passes so that machining must be undertaken to restores the square sections after every pass.

The horizontal orientation shown in Fig. 3b is a more practical situation and it has been used in all experiments conducted to date [17-21]. This orientation provides several potential processing routes including 180° around the *X* axis in route C_X which is equivalent to route C in bars and rods and 180° around the *Y* axis in route C_Y which is equivalent to route A. There are also two additional processing routes which are not available with rod samples or when using plates oriented in a vertical configuration as in Fig. 3a. These new routes involve rotating the horizontal plate by 90° around the *Z* axis, either in route B_{AZ} or in route B_{CZ} where there are rotations by 90° after each pass in alternate directions or in the same sense, respectively. Detailed experiments on plates of high-purity aluminum have demonstrated that route B_{CZ} is an excellent processing route for plate samples leading to excellent properties with the presence of only minor inhomogeneity after 4 passes through the die [17]. The principles of this processing route are illustrated schematically in more detail in Fig. 4 [17].

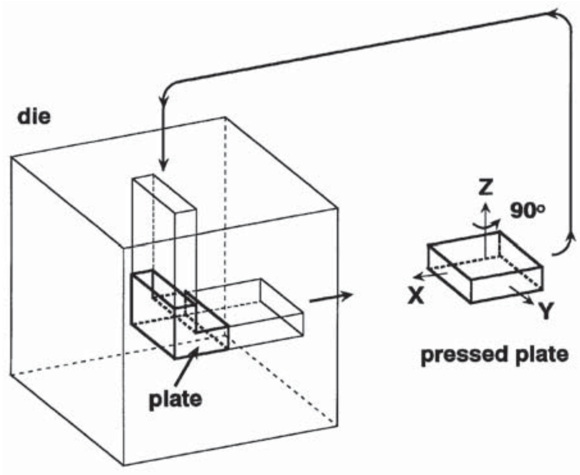

Fig. 4. The procedure adopted for pressing a plate in a horizontal configuration using processing route B_{CZ} [17].

4. ALTERNATIVE PROCEDURES FOR ACHIEVING ECAP: ROTARY DIES, SIDE-EXTRUSION AND MULTI-PASS DIES

An important limitation in conventional ECAP is that the sample must be removed from the die and reinserted, with or without an intermediate rotation, in order to achieve large numbers of passes and a high imposed strain. These operations are both labor-intensive and time-consuming and, accordingly, several procedures are under development to avoid these limitations.

A simple procedure that effectively eliminates the need for removing specimens from the die between each pass is to make use of rotary-die ECAP [22-31]: this approach is illustrated schematically in Fig. 5 [24]. The facility consists of a die containing two channels, having the same cross-section, intersecting at the center of the die at an angle of 90°. Three punches of equal length are inserted in the lower section of the vertical channel and in the horizontal channel as shown in Fig. 5a. The sample is inserted in the vertical channel so that it rests on the lower punch and an upper punch is inserted to press the sample with a plunger. The configuration after a single pressing is shown in Fig. 5b and the die is then rotated by 90° so that the sample may be pressed again as shown in Fig. 5c. A careful inspection of the procedure shows that this type of processing is equivalent to route A where the specimen is pressed without any rotation. However, a significant advantage of this type of pressing is the simplicity of operation. For example, rotary-die ECAP has been used effectively for consecutive pressings up to a maximum of 32 passes [26,27]. However, a disadvantage of the process illustrated in Fig. 5 is that the aspect ratios of the sample are small so that end effects may lead to significant inhomogeneities [29].

An alternative but physically similar approach is the side-extrusion process illustrated schematically in Fig. 6 [32]. This process uses four punch-pull cams which are capable of generating high forces during operation. A sample is shown in place in Fig. 6 and it is pressed by punch A under a lateral pressure exerted by punch B. Repetitive pressings may be performed and, as with rotary-die ECAP, this process is equivalent to route A. This procedure has been used effectively for pressing up to 10 passes [32].

An alternative procedure, which does not require the acquisition of a complex pressing facility, is to construct a die having multiple passes. An example of a multi-pass die is shown in Fig. 7 where the die contains a channel bent through 5 separate angles of 90° [15]. Inspection shows this is equivalent to

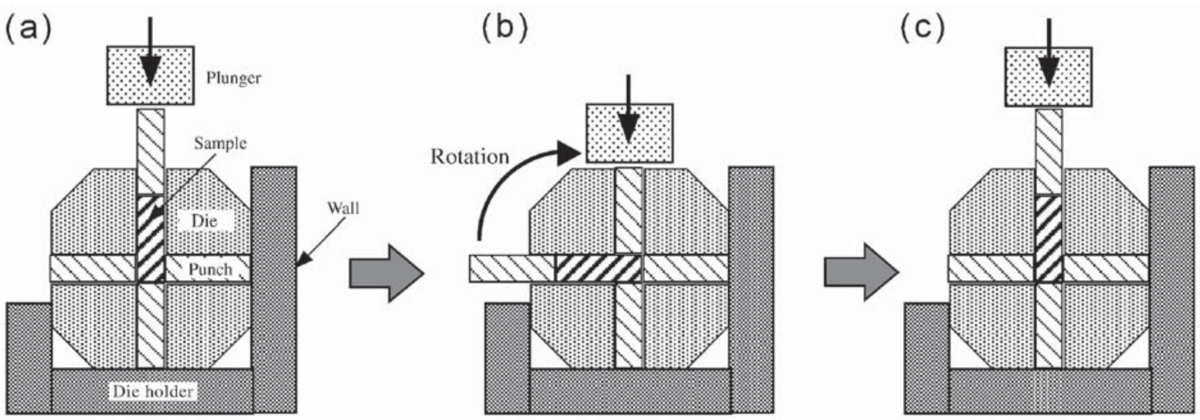

Fig. 5. The ECAP process using a rotary-die: (a) initial state, (b) after one pass and (c) after 90° die rotation [9], see also [24].

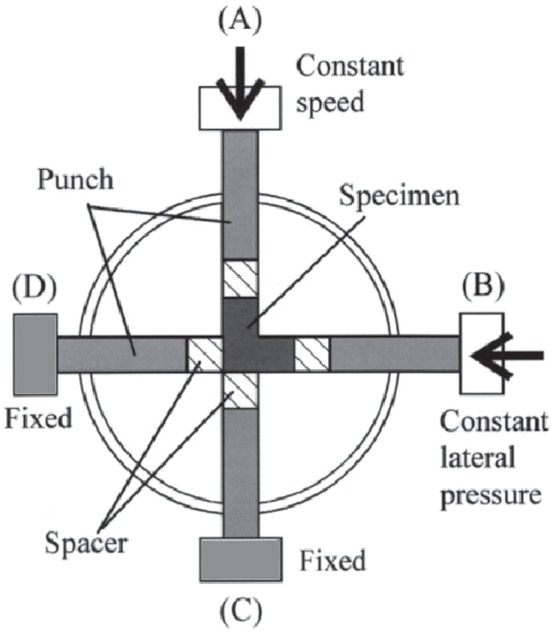

Fig. 6. A schematic illustration of the side-extrusion process for ECAP [9], see also [32].

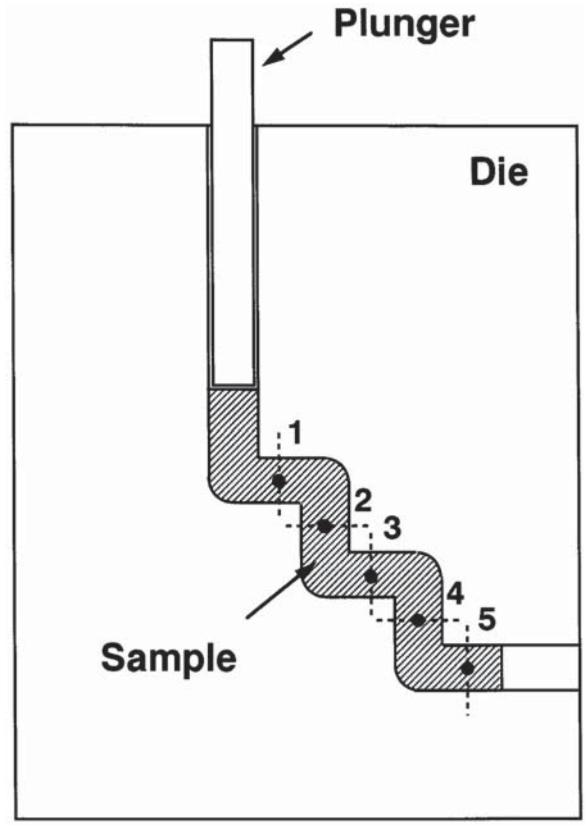

Fig. 7. A schematic illustration of a multi-pass facility for ECAP: the numbers denote positions for examining the sample after the equivalent of 1, 2, 3, 4, and 5 passes, respectively [15].

route C since the second and subsequent passes occur after effectively rotating the sample by 180°. This type of die is useful in order to compare the microstructural characteristics in the same specimen after different numbers of passes. For example, the positions labeled 1, 2, 3, 4, and 5 in Fig. 7 correspond to pressing through 1, 2, 3, 4, and 5 passes of ECAP, respectively. In experiments using a multi-pass facility with a two-piece die that was easily separated to permit access to the specimen, it was shown that the microstructural evolution and the values of the local hardness were identical after the same numbers of passes whether using a multi-pass die or a conventional die containing a single shearing plane [15].

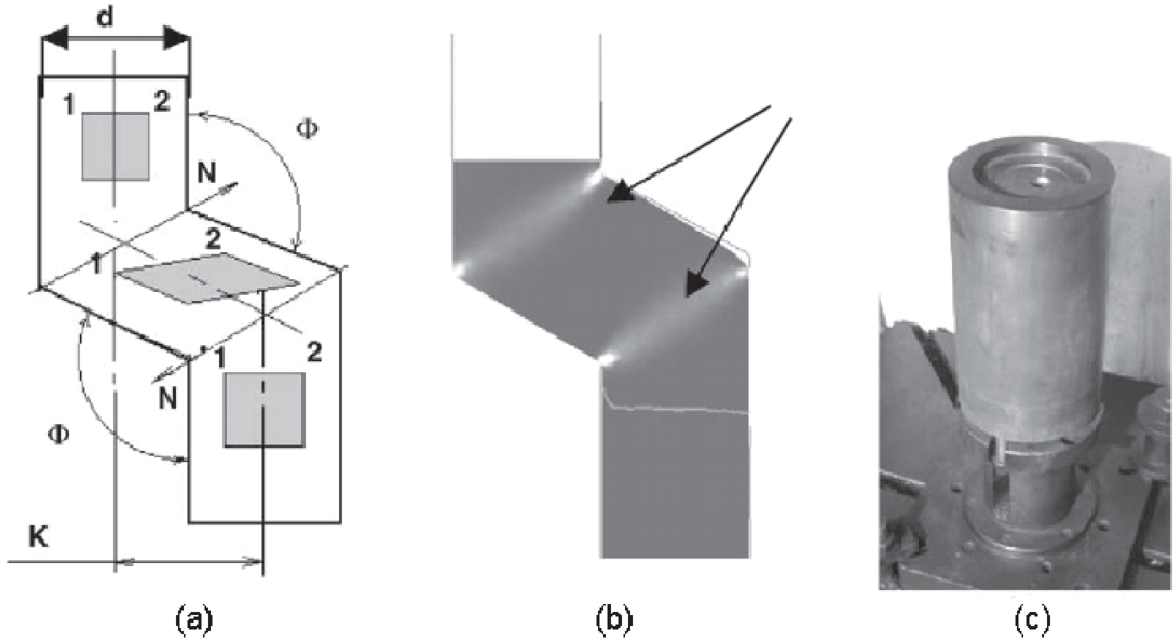

Fig. 8. The principle of ECAP with parallel channels: (a) A schematic illustration where *N* is in the shear direction*,* *K* is the displacement between the two channels, ϕ is the angle of intersection between the two parts of the channel and the internal shaded areas depict the shearing as the sample traverses the shearing zone, (b) a view of the deformation zones obtained by 2-D FEM simulation for ECAP with parallel channels and (c) a general view of the experimental ECAP die-set where ϕ = 100° and *K* is equal to the channel diameter of 18 mm [9], see also [37].

5. DEVELOPING ECAP WITH PARALLEL CHANNELS

There is an important new development showing the potential for conducting ECAP using a facility containing two parallel channels. Some early results made use of this approach [33-36] but the most recent approach, combining a two-dimensional finite element method (2D-FEM) simulation and direct experiments, provides a very clear demonstration of the advantage of pressing with two parallel channels. The principles of this procedure are illustrated schematically in Fig. 8 [37] where ϕ is the angle of intersection between the parallel channels and *K* is the channel displacement.

A distinctive feature of ECAP with parallel channels is that, during a single processing pass, two distinct shearing events take place [35,36]. This means in practice that there is a considerable reduction in the number of passes required for the formation of an ultrafine-grained structure. The values for the displacement between the two channels, *K*, and the angle of intersection of the channels, ϕ, are the main parameters of the die geometry which influence both the flow pattern and the strain-stress state of the ECAP process.

The influence of the ϕ and *K* parameters on the flow pattern and strain homogeneity of a copper specimen was investigated using 2-D FEM simulation during ECAP with parallel channels [37]. It has been established that the optimal values of these parameters, leading to the largest strain homogeneity in the cross-section of the pressed billet, are ϕ = 100° and a displacement value of $K \approx d_c$, where d_c is the channel diameter. This means in practice that the optimum condition is achieved when the measured lateral displacement between the two channels is approximately equal to the dimension of the channel. Under these conditions, the accumulated strain for one pass is approximately equal to 2. It is important to note also that the simulation results have been confirmed experimentally using a grid method.

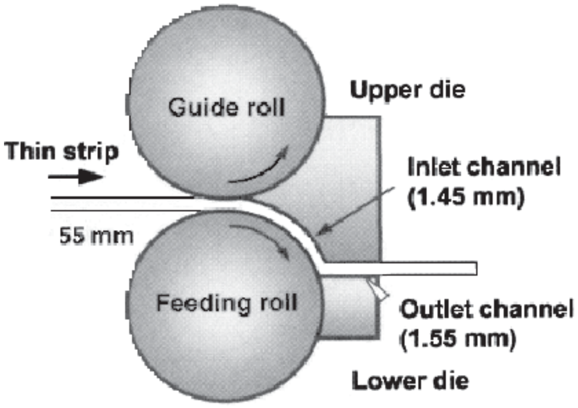

Fig. 9. The principle of the DCAP process for use in continuous production [9], see also [42].

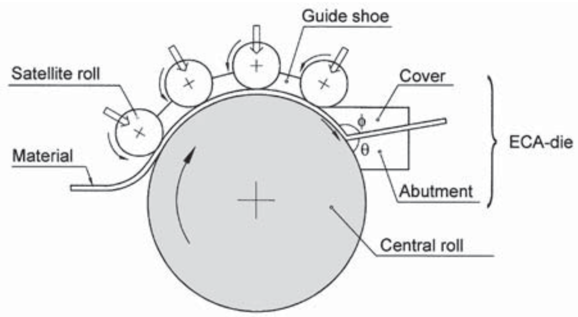

Fig. 10. The principle of the conshearing process [9], see also [50].

An important feature has been revealed concerning the nature of metal flow during ECAP in parallel channels. After one full pass, a mesh on the sample appears undistorted [37], thereby demonstrating that a uniform strain distribution is achieved including in the tail-pieces. Thus, unlike conventional ECAP, the sample shape after pressing remains identical to the initial sample.

The calculations obtained with parallel channels were used for the fabrication of an ECAP die-set for operation at temperatures up to 500°C as shown in Fig. 8c. Samples of Cu and Ti were produced by pressing through 4 passes and observations by transmission electron microscopy (TEM) showed that the structural refinement in these samples corresponded to the formation of an ultrafine-grained structure observed after conventional ECAP when pressing through 8 passes. Furthermore, the UFG structure was rather homogeneous along the length of the bulk sample including up to the ends. Such a high microstructural uniformity is of much practical importance because of the increasing potential for utilizing the material after ECAP [35].

6. CONTINUOUS PROCESSING BY ECAP

6.1. Continuous confined strip shearing, equal-channel angular drawing and conshearing

Processing by ECAP has attracted considerable attention because it produces ultrafine-grained materials with unique physical and mechanical properties and these materials may have important applications in industry. Nevertheless, the ECAP process as currently used in the laboratory is labor-intensive because it requires much manual effort to add and remove the billets from conventional dies. Accordingly, it is now recognized that any extensive industrial application will require the development of some form of continuous processing technique that can be used efficiently in the production of relatively large volumes of material. Some initial progress has been made in developing continuous ECAP procedures for the processing of long metal strips.

First, a process was developed using a rolling facility combined with the principles of ECAP [38-44]. This process was variously designated continuous confined strip shearing (C2S2) [38,39], dissimilar-channel angular pressing (DCAP) [40,42,43] and equal-channel angular rolling (ECAR) [41,44] and the principles of the process are illustrated schematically in Fig. 9 [42]. Thus, the material is in the form of a thin strip and it is fed into the facility between two rolls, extruded slightly to reduce the thickness from 1.55 to 1.45 mm, and then it flows into the outlet channel where the original thickness of 1.55 mm is restored. The terminology DCAP arises therefore because of the small difference in the thickness associated with the passage into the outlet channel.

Second, equal-channel angular drawing (ECAD) was proposed as a potential route for the processing of rod samples [45,46] but subsequent calculations, combined with experiments, demonstrated that ECAD entails a reduction in the cross-sectional area of the sample by >15% so that it cannot be used effectively for multi-pass processing [47].

Third, the conshearing method was proposed for use with metal strips [48-50] and this process, which employs a continuous rolling mill, is illustrated schematically in Fig. 10 [50]. In this procedure, the material is fed into the mill between satellite rollers and a large central roller and all of these rollers rotate at the same peripheral speed in order to generate a large extrusion force. The strip passes be-

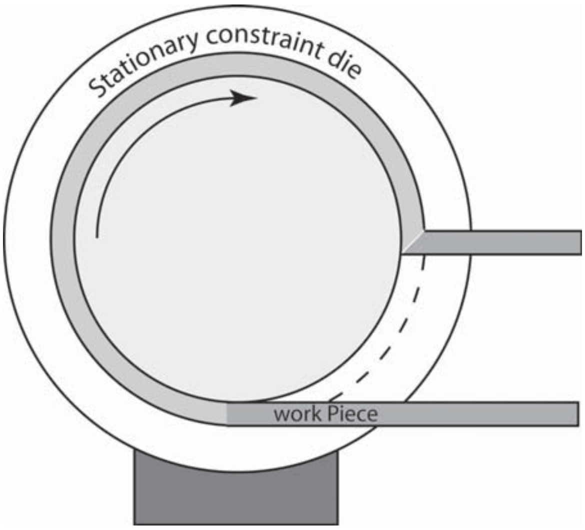

Fig.11. A schematic illustration of the ECAP-Conform process [53].

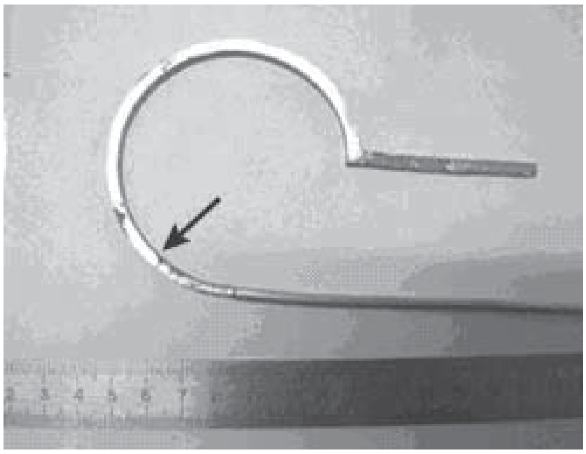

Fig. 12. An Al work-piece undergoing processing by ECAP–Conform: the arrow marks the transition to a rectangular cross-section [53].

tween the rollers and ultimately passes from the mill through an abutment where it is displaced through an angle ϕ. Detailed experiments using commercial purity aluminum strips showed that optimum conditions were achieved for ECAP when the angle within the abutment was given by $\phi = 65°$ [50].

Despite the apparent success associated with these various procedures, the results obtained to date cover only a very limited range of materials and they deal also with the processing of very small batches of each alloy. More work is now needed to provide a detailed assessment of the potential for using these techniques to produce large quantities of materials in a continuous process that is both fast and economically viable.

6.2. The ECAP-Conform process

The conform extrusion process was developed over thirty years ago for the continuous extrusion of wire products [51,52] but very recently it has been conveniently combined with ECAP in the ECAP-Conform process [53].

In this process, the principle used to generate the frictional force to push a work-piece through an ECAP die is similar to the Conform process [51] while a modified ECAP die design is used so that the work-piece can be repetitively processed to produce UFG structures.

The designed and constructed ECAP-Conform set-up is illustrated schematically in Fig. 11 [53]. As shown in the diagram, a rotating shaft in the center contains a groove and the work-piece is fed into this groove. The work-piece is driven forward by frictional forces on the three contact interfaces with the groove so that the work-piece rotates with the shaft. However, the work-piece is constrained within the groove by a stationary constraint die and this die also stops the work-piece and forces it to turn at an angle by shear as in a regular ECAP process. In the current set-up, the angle is close to 90° which is the most commonly used channel intersection angle in ECAP. This set-up effectively makes the ECAP process continuous. Other ECAP parameters, such as the die angle and the strain rate, can also be incorporated into the facility.

In recent work [53], a commercially-pure (99.95%) coarse-grained long Al wire with a diameter of 3.4 mm and more than 1 m in length was used for processing at room temperature with 1-4 passes using ECAP via route C in which the sample was rotated by 180° between the ECAP passes. The initial grain size of the Al wire was ~5-7 μm. Figure 12 shows an Al work-piece at each stage of the ECAP-Conform process, from the initial round feeding stock to the rectangular Al rod after the first ECAP pass [53]. As indicated, the rectangular cross-section was formed shortly after the wire entered the groove (marked by an arrow in Fig. 12). The

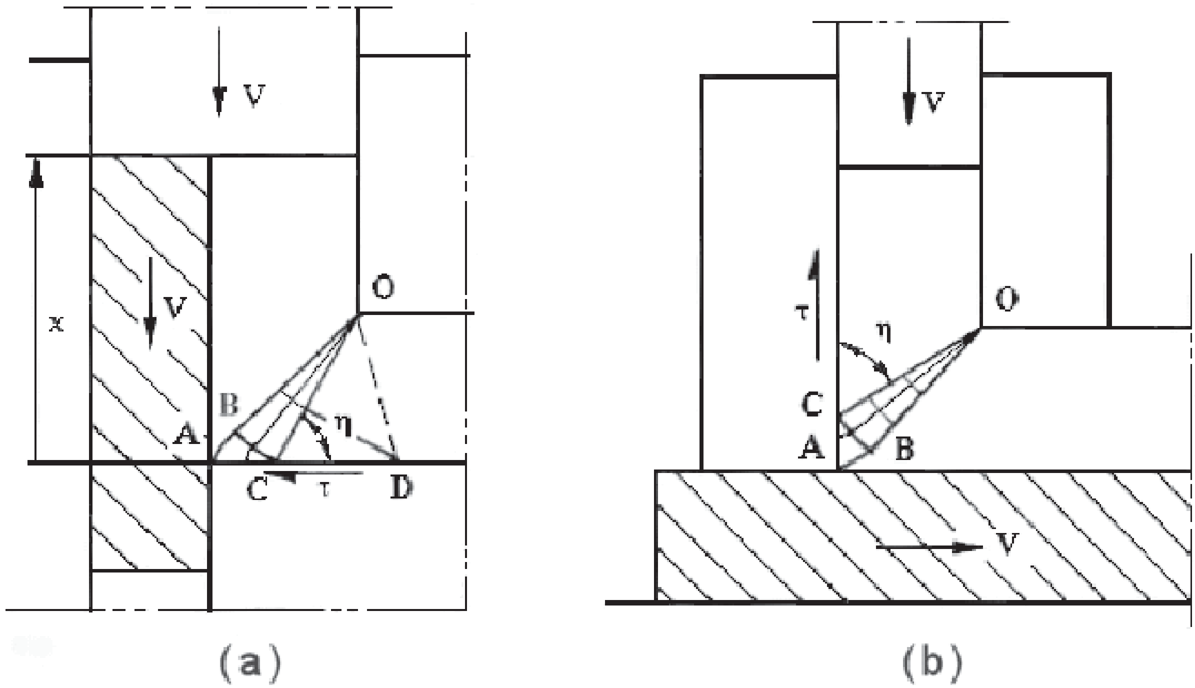

Fig.14. The principle of ECAP with movable die walls (shown shaded): (a) in the entrance channel and (b) in the exit channel [9], see also [13].

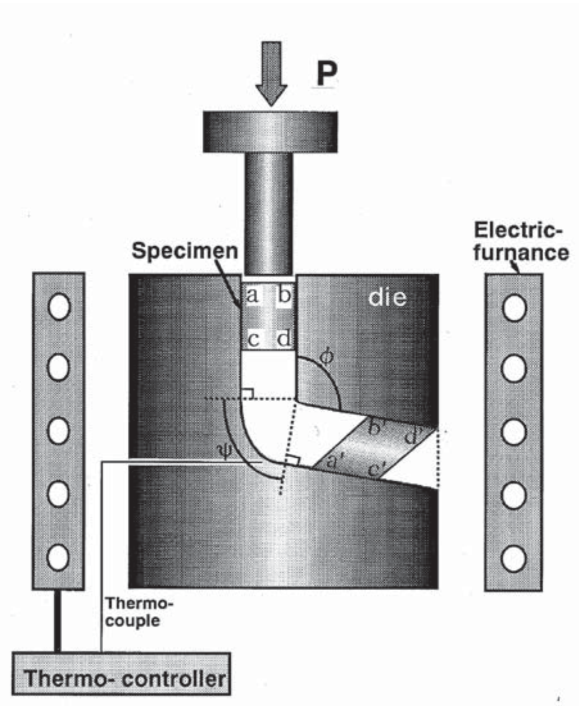

Fig.13. The ECAP principle used for the consolidation of aluminum alloy powder: the powder is inserted into a tight-fitting outer jacket labeled *abcd* [9], see also [55].

change was driven by the frictional force between the groove wall and the Al work-piece. Thus, the frictional force pushed the wire forward and deformed the wire to make it conform to the groove shape. After the wire cross-section changed to the square shape, the frictional force per unit of wire length became larger because of the larger contact area between the groove and the wire. The total frictional force pushed the wire forward from the groove into the stationary die channel which intersected the groove at an angle of 90°. This latter part of the straining process is therefore similar to the conventional ECAP process. Observations by TEM have shown that the ECAP-Conform process leads to a microstructural evolution that is typical of the ECAP process and thus this new technique can effectively refine grains and produce UFG microstructures.

7. CONSOLIDATION BY ECAP

Although ECAP is generally associated with the processing of solid metals, it may be used also for the consolidation of metallic powders [54-60].

Fig. 13 shows an ECAP facility used effectively for the pressing of an aluminum powder of the Al-2024 alloy [55]. The powder had an initial size of <45 μm and it was cold isostatically pressed to a billet size with a diameter of 20 mm and length of

70 mm. As illustrated in Fig. 13, the channels within the die met at an angle of 105° and there was an outer arc of curvature of 75°. Repetitive pressings of the billet were undertaken at a temperature of 573K up to a maximum of three passes through the die. An important difference in the pressing of powders is that cracking occurs readily on the surface of the pressed samples. To avoid cracking during ECAP consolidation, the Al-2024 powder was machined and inserted into a tight-fitting outer jacket of the Al-2024 alloy and the pressing was conducted at a temperature of 573K: the jacketed specimen is marked as *abcd* in Fig. 13. The results from this research demonstrated the production of a fully-dense alloy without the presence of any surface cracking [55]. It was shown recently that the propensity for cracking may be significantly reduced, or even eliminated, by pressing under a confining back-pressure and, in addition, a grain size of ~1 μm was achieved when consolidating pure aluminum powder [60]. These and other similar results confirm, therefore, the potential for using ECAP as a tool in powder consolidation for the production of UFG microstructures.

8. CONSTRUCTION OF AN ECAP FACILITY

It is a relatively simple task to establish a facility for conventional ECAP by machining a two-piece split die consisting of a highly polished smooth plate bolted to a second polished plate containing a square-sided channel. This type of die works well in the laboratory and can be used for multiple passes provided care is taken to manually tighten the bolts between each separate pass. A suitable lubricant such as MoS_2 is generally used to minimize frictional effects at the die walls. However, an alternative approach for minimizing friction is to make use of more complex configurations incorporating moving die walls [13,61-64]. Two examples of movable die walls are shown in Fig. 14 where there is a movable wall shown shaded (a) in the entrance channel and (b) in the exit channel: as illustrated, these two configurations lead to different slip line solutions at the theoretical shear plane [13].

An alternative approach is to construct a solid die from tool steel. Solid dies have an advantage because they avoid any problems associated with the extrusion of slivers of material between the separate parts of a die. However, solid dies require the use of a channel having a circular cross-section and, in addition, the die must be constructed with a finite outer arc of curvature at the point of intersection of the two parts of the channel so that $\Psi \neq 0°$. In practice, experiments have shown that little or no inhomogeneity is introduced into the pressed samples by using solid dies having arcs of curvature of $\Psi \approx$ 20° [65]. Furthermore, model experiments with billets made of plasticine have revealed no significant differences when using samples with either square or circular cross-sections [66]. When working with solid dies, it is important to note that it is necessary to remove each specimen from the die by pressing the next specimen into the die. In practice, therefore, the final specimen is generally removed using a dummy specimen which then remains within the die.

The scaling of ECAP processing to incorporate large billets [67-69] and the pressing of hard-to-deform materials require more complex construction of the ECAP facilities in order to maintain enhanced loading during the pressing operation. This is true also for the development of ECAP processing for commercial use and for these conditions the construction of an optimal die requires special technical solutions.

9. SUMMARY AND CONCLUSIONS

Processing by ECAP is very effective in producing bulk metallic samples with ultrafine grain sizes. Recently, there has been considerable progress in modifying the fundamental ECAP process in order to achieve a continuous and effective processing procedure. These modifications are designed to incorporate the principles and advantages of ECAP with more conventional and well-established processing routes such as rolling and the conform process.

REFERENCES

[1] R.Z. Valiev, R.K. Islamgaliev and I.V. Alexandrov // *Prog. Mater. Sci*. **45** (2000) 103.
[2] *Investigations and Applications of Severe Plastic Deformation*, ed. by T.C. Lowe and R.Z. Valiev (Kluwer: Dordrecht, The Netherlands, 2000).
[3] *Nanomaterials by Severe Plastic Deformation*, ed. by M.J. Zehetbauer and R.Z. Valiev (Weinheim, Germany: Wiley-VCH, 2004).
[4] *Nanomaterials by Severe Plastic Deformation*, ed. by Z. Horita (Uetikon-Zürich, Switzerland: Trans Tech Publications, 2005).

[5] R.Z. Valiev, Yu. Estrin, Z. Horita, T.G. Langdon, M.J. Zehetbauer and Y.T. Zhu // *JOM* **58(4)** (2006) 33.
[6] V.M. Segal, V.I. Reznikov, A.E. Drobyshevskiy and V.I. Kopylov // *Russian Metall* **1** (1981) 99.
[7] R.Z. Valiev, N.A. Krasilnikov and N.K. Tsenev // *Mater. Sci. Eng*. **A137** (1991) 35.
[8] R.Z. Valiev, A.V. Korznikov and R.R. Mulyukov // *Mater. Sci. Eng*. **A168** (1993) 141.
[9] R.Z. Valiev and T.G. Langdon // *Prog. Mater. Sci*. **51** (2006) 881.
[10] V.M. Segal // *Mater. Sci. Eng.* **A197** (1995) 157.
[11] V.M. Segal // *Mater. Sci. Eng.* **A271** (1999) 322.
[12] M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon // *J. Mater. Sci.* **36** (2001) 2835.
[13] V.M. Segal // *Mater. Sci. Eng.* **A386** (2004) 269.
[14] P.B. Berbon, M. Furukawa, Z. Horita, M. Nemoto and T.G. Langdon // *Metall. Mater. Trans.* **30A** (1999) 1989.
[15] K. Nakashima, Z. Horita, M. Nemoto and T.G. Langdon // *Mater. Sci. Eng*. **A281** (2000) 82.
[16] M. Furukawa, Y. Iwahashi, Z. Horita, M. Nemoto and T.G. Langdon // *Mater. Sci. Eng*. **A257** (1998) 328.
[17] M. Kamachi, M. Furukawa, Z. Horita and T.G. Langdon // *Mater. Sci. Eng*. **A361** (2003) 258.
[18] S. Ferrasse, V.M. Segal, S.R. Kalidini and F. Alford // *Mater Sci Eng* **A368** (2004) 28.
[19] S. Ferrasse, V.M. Segal and F. Alford // *Mater. Sci. Eng*. **A372** (2004) 44.
[20] S. Ferrasse, V.M. Segal and F. Alford // *Mater. Sci. Eng*. **A372** (2004) 235.
[21] M. Kamachi, M. Furukawa, Z. Horita and T.G. Langdon // *Mater. Trans*. **45** (2004) 2521.
[22] Y. Nishida, H. Arima, J.C. Kim and T. Ando // *J. Japan. Inst. Metals* **64** (2000) 1224.
[23] Y. Nishida, H. Arima, J.C. Kim and T. Ando // *J. Japan. Inst. Light Metals* **50** (2000) 655.
[24] Y. Nishida, H. Arima, J.C. Kim and T. Ando // *Scripta Mater*. **45** (2001) 261.
[25] J.C. Kim, Y. Nishida, H. Arima and T. Ando // *Mater. Lett*. **57** (2003) 1689.
[26] A. Ma, K. Suzuki, Y. Nishida, N. Saito, I. Shigematsu, M. Takagi, H. Iwata, A. Watazu and T. Imura // *Acta Mater*. **53** (2005) 211.
[27] A. Ma, K. Suzuki, N. Saito, Y. Nishida, M. Takagi, I. Shigematsu and H. Iwata // *Mater. Sci. Eng*. **A399** (2005) 181.
[28] Z.M. Sun, H. Hashimoto, N. Keawprak, A.B. Ma, L.F. Li and M.W. Barsoum // *J. Mater. Res*. **20** (2005) 895.
[29] A. Ma, Y. Nishida, K. Suzuki, I. Shigematsu and N. Saito // *Scripta Mater*. **52** (2005) 433.
[30] A. Watazu, I. Shigematsu, A. Ma, K. Suzuki, T. Imai and N. Saito // *Mater. Trans.* **46** (2005) 2098.
[31] A. Ma, M. Takagi, N. Saito, H. Iwata, Y. Nishida, K. Suzuki and I. Shigematsu // *Mater. Sci. Eng*. **A408** (2005) 147.
[32] A. Azushima and K. Aoki // *Mater. Sci. Eng.* **A337** (2002) 45.
[33] Z.Y. Liu, G.X. Liang, E.D. Wang and Z.R. Wang // *Mater. Sci. Eng.* **A242** (1998) 137.
[34] Z. Liu and Z. Wang // *J. Mater. Proc. Tech*. **94** (1999) 193.
[35] G.I. Raab, R.Z. Valiev, G.V. Kulyasov and V.A. Polozovsky, *Russian Patent* N 2181314 (2002).
[36] G.I. Raab // *Fizika i Tekhnika Vysokikh Davleniy (Physics and Engineering of High Pressures)* **14(4)** (2004) 83.
[37] G.I. Raab // *Mater. Sci. Eng*. **A410-411** (2005) 230.
[38] J.C. Lee, H.K. Seok, J.H. Han and Y.H. Chung // *Mater. Res. Bull.* **36** (2001) 997.
[39] J.H. Han, H.K. Seok, Y.H. Chung, M.C. Shin and J.C. Lee // *Mater. Sci. Eng*. **A323** (2002) 342.
[40] J.C. Lee, H.K. Seok, J.Y. Suh, J.H. Han and Y.H. Chung // *Metall. Mater. Trans.* **33A** (2002) 665.
[41] C.Y. Nam, J.H. Han, Y.H. Chung and M.C. Shin // *Mater. Sci. Eng*. **A347** (2003) 253.
[42] J.C. Lee, J.Y. Suh and J.P. Ahn // *Metall. Mater. Trans.* **34A** (2003) 625.
[43] J.Y. Suh, J.H. Han, K.H. Oh and J.C. Lee // *Scripta Mater.* **49** (2003) 185.
[44] J.W. Park, J.W. Kim and Y.H. Chung // *Scripta Mater.* **51** (2004) 181.
[45] U. Chakkingal, A.B. Suriadi and P.F. Thomsen // *Scripta Mater.* **39** (1998) 677.
[46] U. Chakkingal, A.B. Suriadi and P.F. Thomsen // *Mater. Sci. Eng.* **A266** (1999) 241.
[47] J. Alkorta, M. Rombouts, J. De Messemaeker, L. Froyen and J. Gil Sevillano // *Scripta Mater.* **47** (2002) 13.
[48] H. Utsunomiya, Y. Saito, T. Hayashi and T. Sakai // *J. Mater. Eng. Perform* **6** (1997) 319.

[49] Y. Saito, H. Utsunomiya, H. Suzuki and T. Sakai // *Scripta Mater.* **42** (2000) 1139.
[50] H. Utsunomiya, K. Hatsuda, T. Sakai and Y. Saito // *Mater. Sci. Eng*. **A372** (2004) 199.
[51] D. Green // *J. Inst. Metals.* **100** (1972) 295.
[52] C. Etherington // *J. Eng. Indust.* **96** (1974) 893.
[53] G. J. Raab, R.Z. Valiev, T.C. Lowe and Y.T. Zhu // *Mater. Sci. Eng.* **A382** (2004) 30.
[54] S. Xiang, K. Matsuki, N. Takatsuji, M. Tokizawa, T. Yokote, J. Kusui and K. Yokoe // *J. Mater. Sci. Lett.* **16** (1997) 1725.
[55] K. Matsuki, T. Aida, T. Takeuchi, J. Kusui and K. Yokoe // *Acta Mater.* **48** (2000) 2625.
[56] A. Parasiris and K.T. Hartwig // *Intl. J. Refract. Metals & Hard Metals* **18** (2000) 23.
[57] I. Karaman, J. Robertson, J.T. Im, S.N. Mathaudhu, Z.P. Luo and K.T. Hartwig // *Metall. Mater. Trans.* **34A** (2003) 247.
[58] M. Haouaoui, I. Karaman, H.J. Maier and K.T. Hartwig // *Metall. Mater. Trans.* **35A** (2004) 2935.
[59] O.N. Senkov, S.V. Senkova, J.M. Scott and D.B. Miracle // *Mater. Sci. Eng.* **A393** (2005) 12.
[60] K. Xia and X. Wu // *Scripta Mater.* **53** (2005) 1225.
[61] S.L. Semiatin, D.P. DeLo and E.B. Shell // *Acta Mater.* **48** (2000) 1841.
[62] S.L. Semiatin and D.P. DeLo // *Materials and Design* **21** (2000) 311.
[63] A. Shan, I.G. Moon and J.W. Park // *J. Mater. Proc. Tech.* **122** (2002) 255.
[64] V.M. Segal // *Mater. Sci. Eng.* **A345** (2003) 36.
[65] C. Xu and T.G. Langdon // *Scripta Mater.* **48** (2003) 1.
[66] R. Manna, P. Agrawal, S. Joshi, B.K. Mudda, N.K. Mukhopadhyay and G.V.S. Sastry // *Scripta Mater.* **53** (2005) 1357.
[67] Z. Horita, T. Fujinami and T.G. Langdon // *Mater. Sci. Eng.* **A318** (2001) 34.
[68] V.V. Stolyarov, Y.T. Zhu, G.I. Raab, A.I. Zharikov and R.Z. Valiev // *Mater. Sci. Eng*. **A385** (2004) 309.
[69] R. Srinivasan, B. Cherakari and P.K. Chaudhury // *Mater. Sci. Forum* **503-504** (2006) 371.