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Review
Processing of metals by equal-channel angular pressing

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Equal-channel angular pressing (ECAP) is a processing method in which a metal is subjected to an intense plastic straining through simple shear without any corresponding change in the cross-sectional dimensions of the sample. This procedure may be used to introduce an ultrafine grain size into polycrystalline materials. The principles of the ECAP process are examined with reference to the distortions introduced into a sample as it passes through an ECAP die and especially the effect of rotating the sample between consecutive presses. Examples are presented showing the microstructure introduced by ECAP and the consequent superplastic ductilities that may be attained at very rapid strain rates.

1. Introduction
There is considerable current interest in fabricating metals with ultrafine grain sizes. This interest arises for two reasons. First, it is possible in principle to increase the strength of a material at low temperatures through the standard Hall-Petch relationship [1, 2] because the strength varies with the reciprocal of the square root of the grain size. Second, the retention of an ultrafine grain size at high temperatures, in the regime where diffusion-controlled processes become important, offers the potential for achieving a superplastic forming capability at high strain rates because the rate of flow in superplastic deformation is inversely proportional to the square of the grain size [3, 4].

Several methods are now available for attaining metals with extremely small grain sizes, usually within the nanometer range, including inert gas condensation, high energy ball milling and sliding wear. However, these procedures have not been developed sufficiently to date that they are capable of producing large bulk samples which are free of any residual porosity. As a result of these limitations, attention has been devoted to the alternative processes of Equal-Channel Angular Pressing (ECAP) and High-Pressure Torsion (HPT) in which ultrafine grains are introduced into a material through intense plastic straining [5, 6]. In practice, ECAP appears to have greater utility than HPT because there is a potential for scaling-up the process for industrial applications through procedures such as the development of multi-pass facilities where high strains are attained in a single passage through the ECAP die [7]. The principle of ECAP is that a metal is deformed through a process of simple shear with the shear taking place without any concomitant change in the cross-sectional area of the sample.

This paper examines the principles underlying the ECAP process with special reference to the distortions occurring when a sample passes through a standard ECAP die containing a single shearing plane and the subsequent distortions associated with any rotation of the sample between each pass. It should be noted that the terminology in the literature associated with ECAP is not clearly defined and the same procedure has been designated Equal-Channel Angular Extrusion (ECAE) [8] and Equal-Channel Angular Forging (ECAF) [9]. Very recently, the acronym ECAP was recommended for use with this process [6] and it will be used exclusively in this report.

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2. Method of imposing an intense plastic strain in ECAP

2.1. Principle of ECAP

There are numerous well-established methods for subjecting metallic samples to an imposed strain, including through the standard industrial metal-working processes of rolling or extrusion, but all of these methods necessitate a change in the physical dimensions of the sample. By contrast, ECAP differs from these conventional procedures because the cross-sectional dimensions of the sample remain unchanged during straining. Historically, ECAP was first developed in the Soviet Union almost twenty years ago [10] but the process has received significant interest in western countries only within the last five or six years.

The principle of ECAP is depicted schematically in the three-dimensional illustration in Fig. 1. The ECAP die contains two channels, equal in cross-section, intersecting at an angle near the center of the die. The test sample is machined to fit within these channels and it is pressed through the die using a plunger. The straining imposed on the sample as it goes through the die is illustrated schematically in Fig. 2 where, for simplicity, it is assumed that the angle of intersection between the two channels is 90°. Thus, simple shear is imposed at the shearing plane between the two adjacent segments labelled 1 and 2 in Fig. 2. Three planes may be defined within the sample at the point of exit from the die, as indicated in Fig. 1 where plane x is perpendicular to the longitudinal axis of the sample and planes y and z are parallel to the side and top faces, respectively.

Fig. 3 illustrates a section through the die and defines two internal angles, Φ and Ψ, delineating the curvature associated with the two channels: there is an angle Φ between the channels and an angle Ψ at the outer arc of curvature where the two channels intersect.

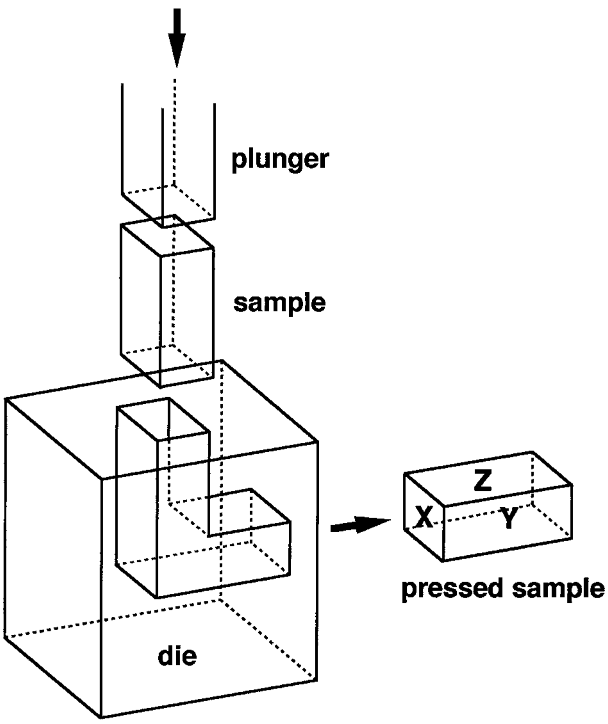

Figure 1 Schematic illustration of ECAP showing the three orthogonal planes x, y and z.

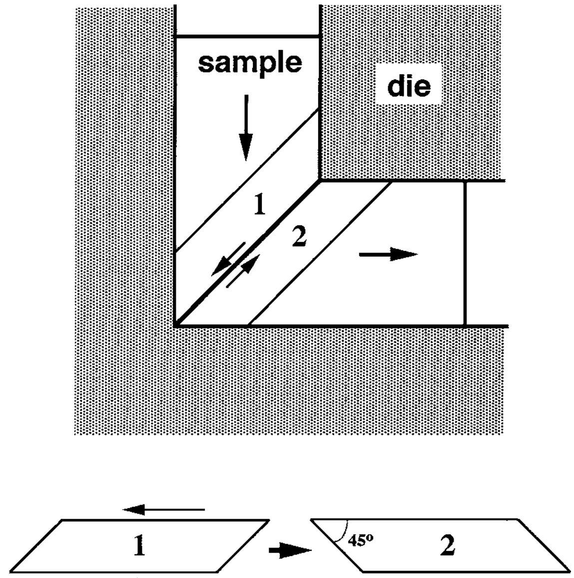

Figure 2 The principle of shearing between elements 1 and 2 in ECAP.

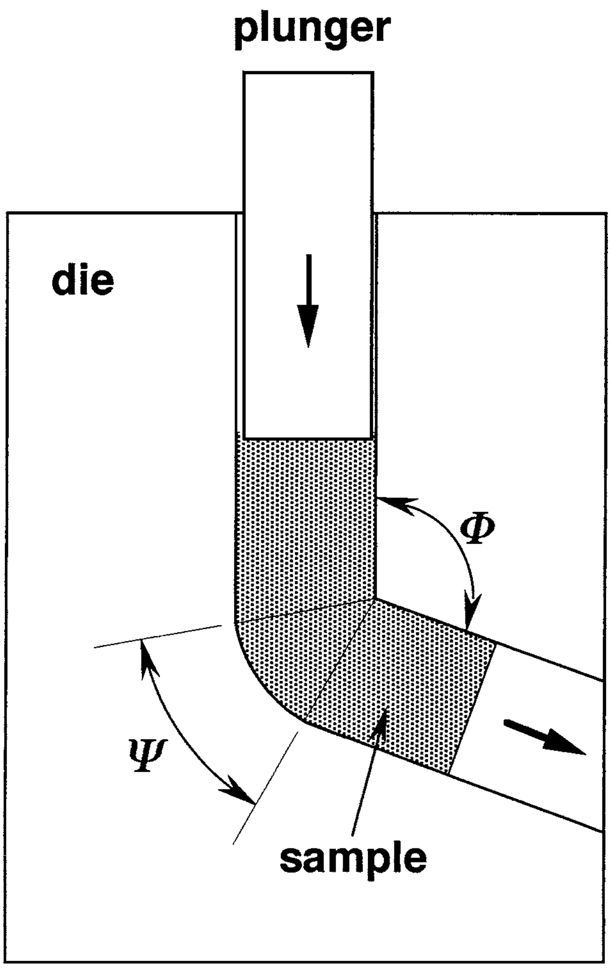

Figure 3 A section through an ECAP die showing the two internal angles Φ and Ψ.

When the sample passes through the die, the precise value of the von Mises equivalent strain is dependent upon the values of these two angles Φ and Ψ. Since the cross-sectional dimensions of the sample remain unchanged with a single passage through the die, the sample may be pressed repetitively through the die in order to achieve a very high total strain. It can be shown from first principles that the total strain accumulated through a series of repetitive pressings, ε_N, is given by the relationship [11]

$$\varepsilon_N = \frac{N}{\sqrt{3}}\left[2\cot\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right) + \Phi\,\mathrm{cosec}\left(\frac{\Phi}{2}+\frac{\Psi}{2}\right)\right] \tag{1}$$

where N is the total number of passes through the die.

Model experiments, using plexiglass dies and layers of colored plasticine, have confirmed the validity of Equation 1 except immediately adjacent to the die walls where there may be frictional effects [12]. In addition, there has been reasonable confirmation of this relationship from experiments using a sample incorporating a grid pattern [13] and, except only near the sample edges, from two-dimensional finite element modeling [14].

Very recently, an alternative expression was proposed where ε_N is given by [15]

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

However, it can be shown by calculation that Equations 1 and 2 give identical values for ε_N at the upper and lower bounds of the arc angle Ψ and they differ by $<5\%$ under all other conditions for any angle of $\Phi \geq 90^\circ$ [16].

A similar approach has been developed also to estimate the strain in situations where the two channels of the die have different cross-sectional dimensions [17] and finite element modeling has been used to examine the significance of any gap that may form between the sample and the die wall at the outer arc of curvature between the two channels [18].

2.2. Significance of the channel angle Φ

A series of detailed experiments on pure aluminum, using dies having values of Φ ranging from 90° to 157.5°, showed that an ultrafine microstructure of essentially equiaxed grains, separated by grain boundaries having high angles of misorientation, was attained most easily when imparting a very intense plastic strain with a value of Φ very close to 90° [19]. For a value of $\Phi = 90^\circ$, it follows from eqn. (1) that the strain imposed on a single passage through the die is very close to 1 for any value of Ψ. Therefore, the strain is close to $\sim N$ for a total of N passes through the die. Evidence from finite element modelling suggests a potential problem with dies having both $\Phi = 90^\circ$ and $\Psi = 0^\circ$ because of the development of a small "dead zone" which remains unfilled with material at the sharp outer corner where the two channels intersect [14, 18, 20, 21].

3. Effect of sample rotation between repetitive pressings

When a sample is pressed repetitively through an ECAP die, it has been recognized that the overall shearing characteristics within the crystalline sample may be changed by a rotation of the sample between the individual pressings [8]. It is possible to define three distinct processing routes: route A in which the sample is not rotated between repetitive pressings, route B in which the sample is rotated by 90° between each pressing and route C in which the sample is rotated by 180° between each pressing. A further possibility may be introduced when it is noted that route B may be undertaken either by rotating the sample by 90° in alternate directions between each individual pressing, termed route B_A, or by rotating the sample by 90° in the same direction between each individual pressing, termed route B_C. The principles of these four different routes are illustrated schematically in Fig. 4.

In order to understand the shearing associated with these different processing routes, it is convenient to consider the deformation of a cube passing through an ECAP die with internal angles of $\Phi = 90^\circ$ and $\Psi = 0^\circ$. The situation for a single passage through the die is illustrated schematically in Fig. 5. Thus, the cubic element in the channel on the left passes through the

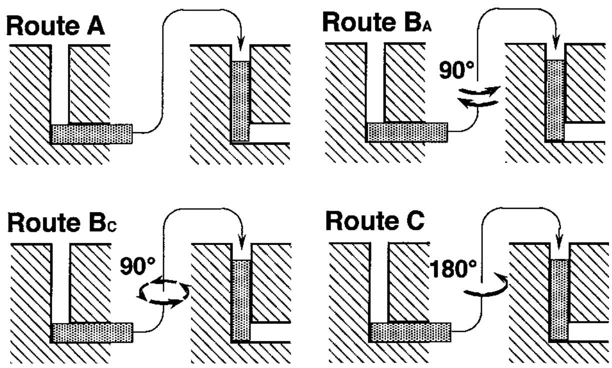

Figure 4 The four different processing routes which may be used for repetitive pressings.

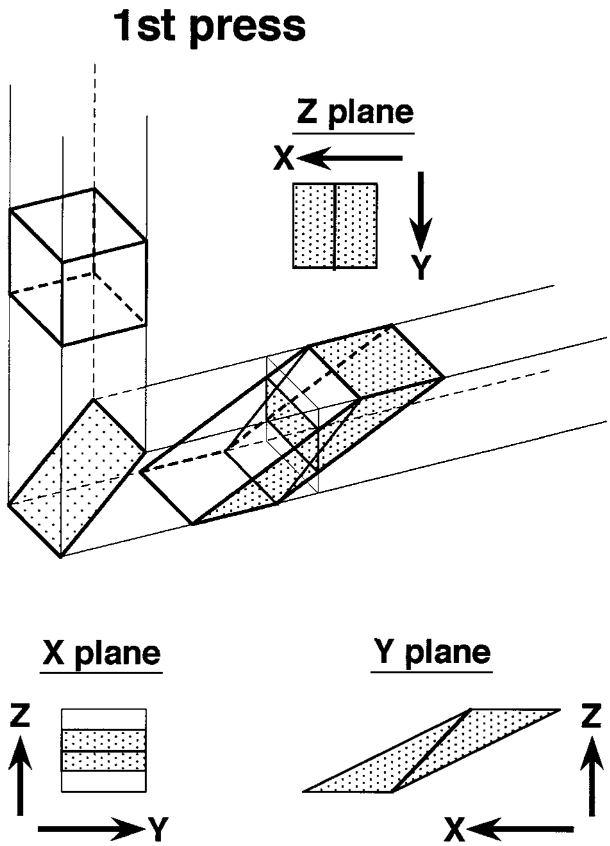

Figure 5 The deformation of a cubic element on a single passage through an ECAP die.

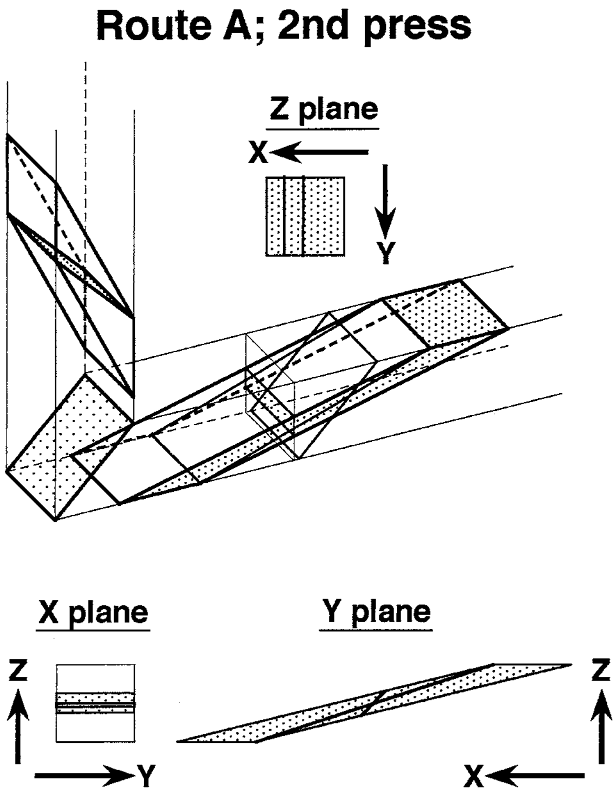

Figure 6 The second passage through an ECAP die when using route A.

theoretical shear plane, shown shaded at bottom left, and becomes sheared into a rhombohedral shape as illustrated in the exit channel of the die. Also included in Fig. 5 are illustrations of the deformation, in terms of the macroscopic grain elongation and the associated shearing planes within the individual grains, for each of the three planes of sectioning x, y and z, as defined in Fig. 1. There is direct experimental evidence supporting the implications of Fig. 5 through the use of optical microscopy with samples of pure aluminum subjected to ECAP [22].

The second passage through the die may be undertaken either with no rotation of the sample (route A) or with rotations of 90° (route B) or 180° (route C): these three situations are shown in Figs 6–8, respectively, together with illustrations of the deformation associated with each plane. Inspection shows that route A leads to an elongation of the grains in the y plane at an angle of $\sim$15° to the x axis, route B leads to elongations of the grains on each orthogonal plane, whereas the use of route C restores the cubic element: again, these predictions are consistent with experimental observations for pure aluminum [22].

For pressings in excess of two passes through the die, it is necessary to consider separately the implications for routes B_A and B_C. The predictions for the 3rd, 4th and 5th passes are illustrated in Figs 9–12 for routes A, B_A, B_C and C, respectively. It is apparent that the shearing characteristics of routes A and B_A are similar and they both lead to increasing distortions of the original cubic element, whereas routes B_C and C are also similar with a restoration of the cubic element after totals of $4n$ and $2n$ passes for routes B_C and C, respectively, where n is an integer. It is possible also to make predictions for combinations of different routes, such as

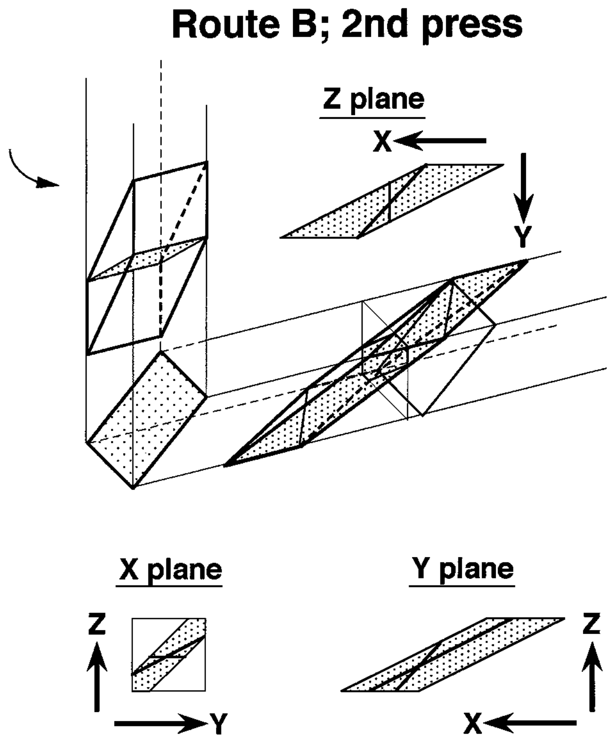

Figure 7 The second passage through an ECAP die when using route B.

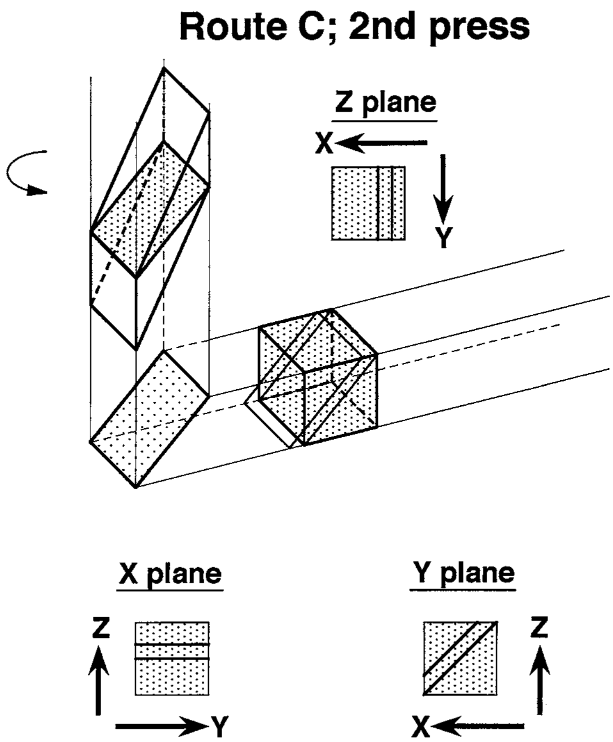

Figure 8 The second passage through an ECAP die when using route C.

B_A-A and B_C-A: these details are outside the scope of this report but further information is given elsewhere [23] together with representative experimental results when using alternative processing routes [24].

The schematic illustrations in Figs 5–12 depict the distortions associated with the cubic element as it passes through the die in repetitive pressings in ECAP. To place these illustrations in perspective, it is convenient to consider the shearing planes which are activated within the material in each of these four processing

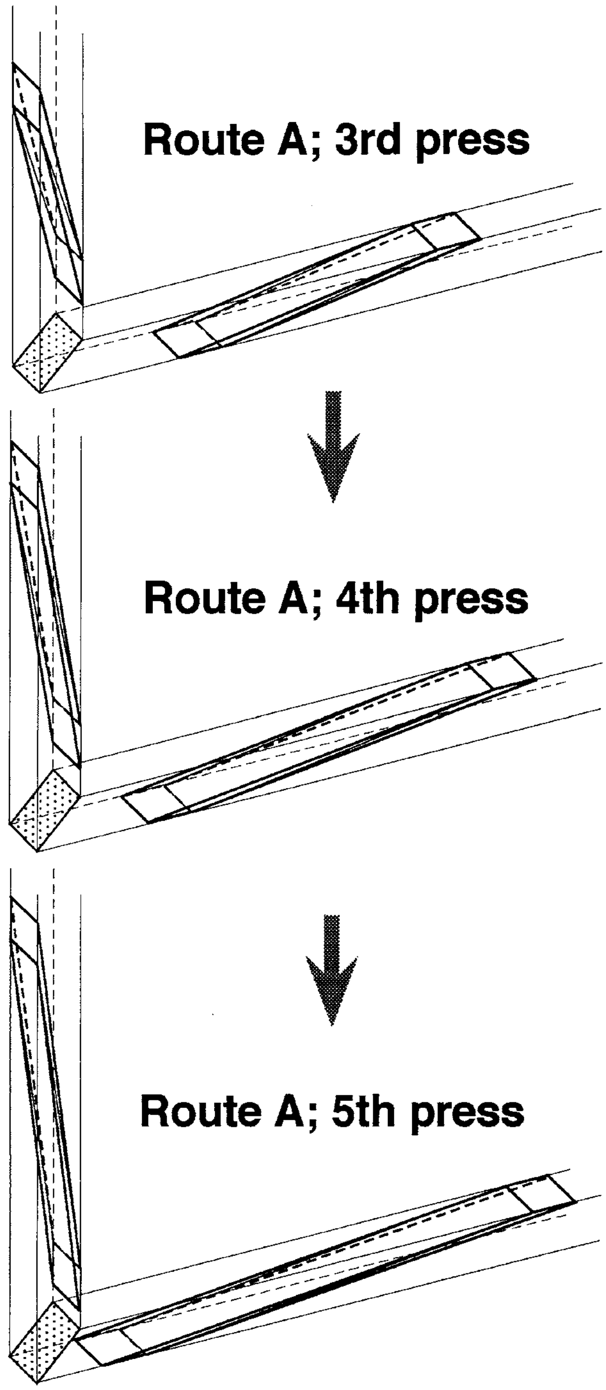

Figure 9 The 3rd, 4th and 5th passages through an ECAP die when using route A.

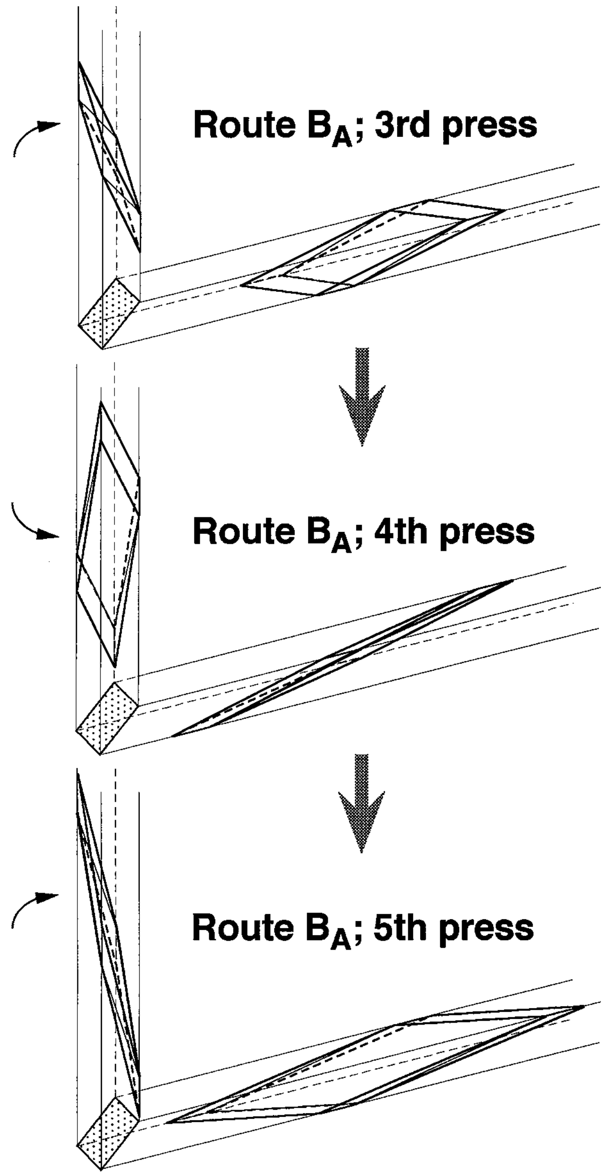

Figure 10 The 3rd, 4th and 5th passages through an ECAP die when using route B_A.

routes. This information is given in Fig. 13 where the planes labelled 1 through 4 denote the shearing which occurs on the first four pressings through the die and the planes x, y and z are indicated. Processing through route A therefore leads to shearing on two planes intersecting at 90°, processing through route C leads to repetitive shearings on the same plane, and processing through routes B_A and B_C leads to shearing on a set of planes intersecting at 120°.

4. Characteristics after ECAP

4.1. Microstructural characteristics

Several experiments have been conducted to investigate the characteristics of the microstructures produced in polycrystalline materials through the use of ECAP, including the effect of rotation of the sample using different processing routes [22, 25–37]. The most significant consequence of ECAP is a very substantial refinement in the average grain size after only a single passage through the die. Furthermore, this grain refinement appears also to be essentially independent of the speed used for the pressing [38]. It is now well established that the precise microstructural characteristics after ECAP are dependent upon the processing route and in practice it is found that, at least for the condition where the internal angle Φ within the die is 90°, the optimum pressing condition is via route B_C where the as-pressed microstructure evolves most rapidly into an array of equiaxed grains separated by high angle grain boundaries [39]. There is also evidence suggesting route A may be more effective when using a die with an angle of $\Phi = 120°$ [35] and a possible explanation for this difference has been proposed by examining the interaction of the shearing plane with the deformation texture and the crystal structure [40].

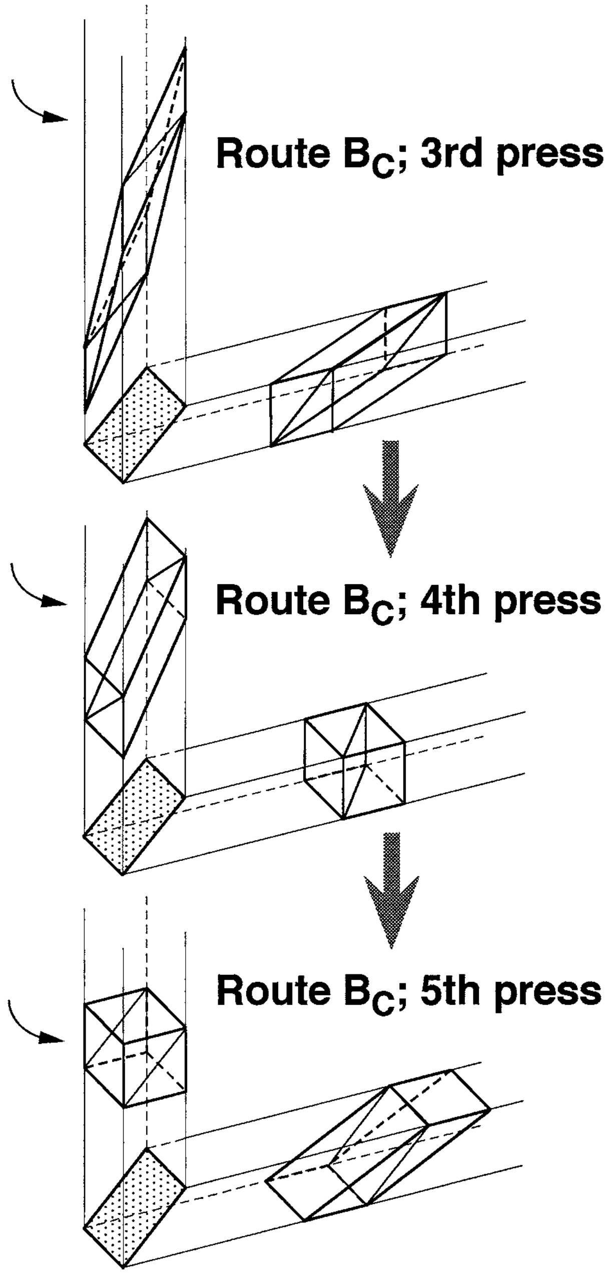

Figure 11 The 3rd, 4th and 5th passages through an ECAP die when using route B_C.

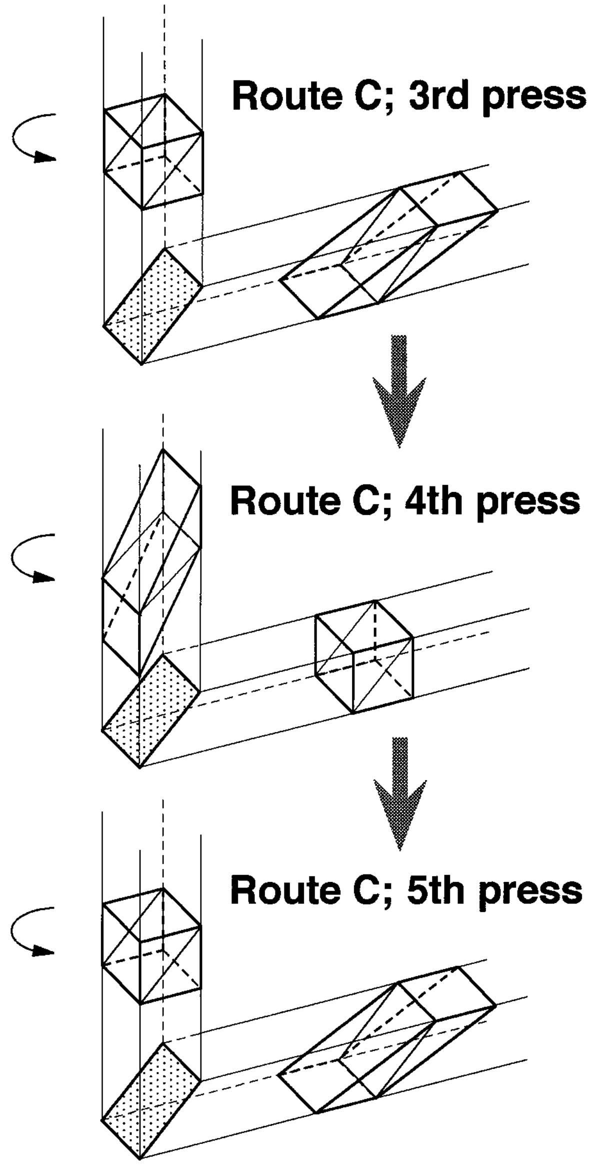

Figure 12 The 3rd, 4th and 5th passages through an ECAP die when using route C.

There are two fundamental characteristics associated with the development of a homogeneous microstructure through ECAP. These characteristics are related to the ultimate stable grain size which is achieved by the pressing process and the numbers of passes needed to attain a homogeneous microstructure. In general terms, it appears that metals exhibiting low rates of recovery are especially attractive for the production of extremely fine grain sizes but the numbers of passes needed to achieve homogeneous arrays of grains in these materials is then increased. For example, experiments have shown that, for materials subjected to ECAP at room temperature using route B_C, the ultimate stable grain sizes are $\sim$1.3, $\sim$0.45 and $\sim$0.27 μm for samples of pure aluminum, an Al-1% Mg alloy and an Al-3% Mg alloy, but homogeneity of the microstructure requires pressing through, respectively, 4, 6 and 8 passes for these three materials [29].

Fig. 14 shows an example of the ultrafine microstructure produced in pure aluminum after 4 passes through an ECAP die at room temperature using processing route B_C [28], where the x, y and z planes are as defined in Fig. 1. In this condition, the average grain size after ECAP is $\sim$1.3 μm and this compares with an initial grain size after annealing but prior to ECAP of $\sim$1.0 mm.

4.2. Mechanical characteristics

Detailed experiments have confirmed that ECAP may be used to attain high superplastic elongations at very rapid strain rates provided particles are present, as in many commercial alloys, to restrict the growth of these ultrafine grains at the high temperatures required for superplastic flow. The first example of this effect was achieved using commercial Al-Mg-Li-Zr and Al-Cu-Zr alloys where the elongations to failure were as high as >1000% at the rapid strain rate of $\sim 10^{-2}$ s^{-1} [41]. There have been numerous recent reports of the occurrence of high strain rate superplasticity in materials processed by ECAP [42–48].

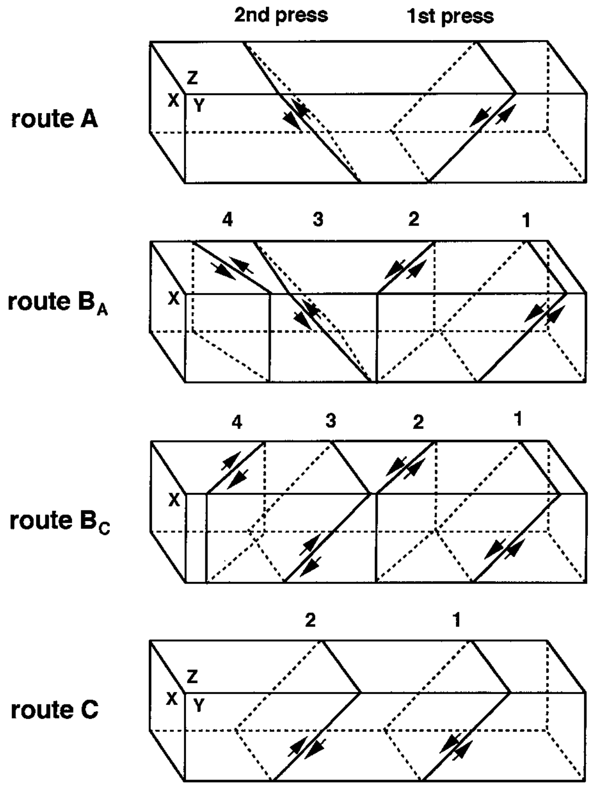

Figure 13 The shearing planes associated with the four different processing routes.

The beneficial effect of ECAP is illustrated in Fig. 15 where the measured elongations to failure are plotted as a function of the imposed strain rate for samples of an Al-5.5% Mg 2.2% Li-0.12% Zr alloy tested in tension. This is a commercial Russian alloy with the designation Al-1420 and it exhibits modest superplastic elongations after appropriate thermomechanical processing. The three lines at the lower left show typical results after an optimum thermomechanical treatment to obtain a grain size of $\sim$8 μm and subsequent testing in tension at temperatures from 768 to 793 K [49]. The remaining points show the dramatic increases in ductility which are achieved through ECAP where the grain size is reduced to $\sim$1.2 μm. For the lower points at 603 K, the sample was pressed at a temperature of 673 K for 4 passes to a total strain of $\sim$4 [50] whereas the remaining two sets of data on the right in Fig. 15, for temperatures of 573 and 623 K, respectively, relate to samples pressed for 8 passes at 673 K and 4 additional passes at 473 K to a total strain of $\sim$12 [42]. It is apparent from Fig. 15 that there is a very substantial increase in the ductilities achieved at the faster strain rates after ECAP and, in addition, the superplastic effect is further enhanced when ECAP is performed to higher total strains. This latter effect is attributed to the need for the grain boundaries in the ultrafinegrained material to attain high angles of misorientation [43].

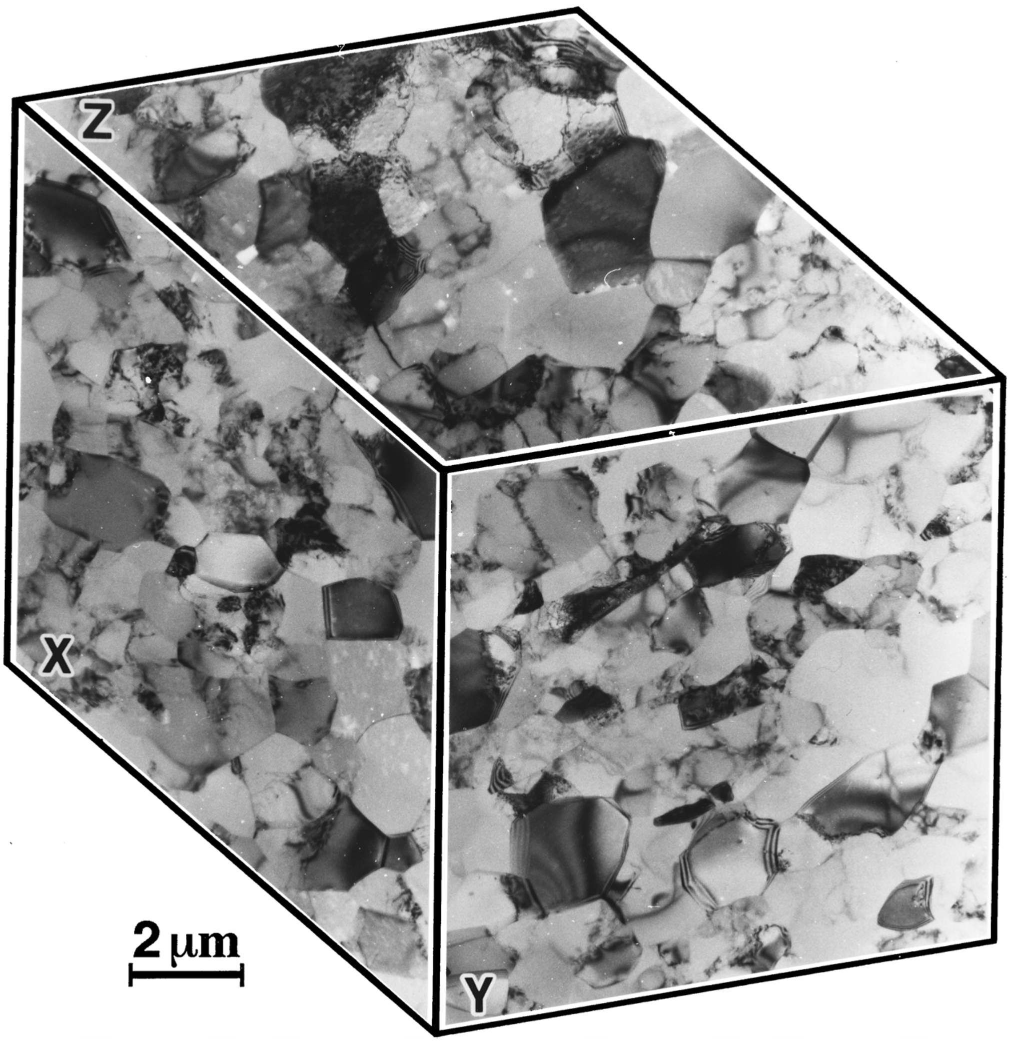

Figure 14 The microstructures produced on the *x*, *y* and *z* planes in pure Al after ECAP at room temperature to a total of 4 passes using route B_C [28].

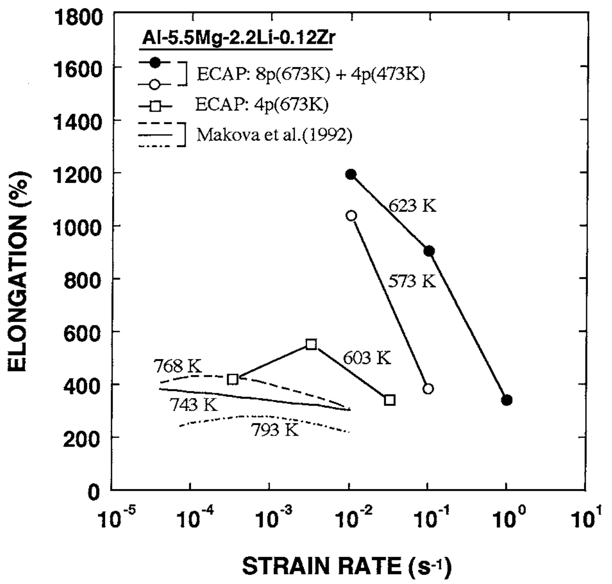

Figure 15 Elongation to failure versus strain rate for samples tested in tension after thermomechanical processing to produce a grain size of $\sim$8 μm [49], after ECAP at 673 K to a strain of $\sim$4 to produce a grain size of $\sim$1.2 μm [50] and after ECAP at 673 and 473 K to a total strain of $\sim$12 to produce a grain size of $\sim$1.2 μm with high angle grain boundaries [42].

5. Summary and conclusions

1. Equal-channel angular pressing (ECAP) imparts an intense plastic strain to a sample through simple shear but without any change in the cross-sectional dimensions. Thus, repetitive pressings may be conducted to achieve very high total strains.

2. The resultant microstructure introduced by ECAP is dependent both upon the processing route, in terms of the rotation of the sample between repetitive pressings, and the angle between the two channels within the ECAP die. Experiments show that an optimum homogeneous microstructure of equiaxed grains, separated by high angle grain boundaries, is attained most readily when the angle between the two channels is close to 90° and the sample is rotated by 90° in the same direction between repetitive pressings (termed processing route B_C).

3. The ultrafine grain sizes introduced by ECAP provide the potential for achieving superplasticity at very rapid strain rates provided particles are present to retain these ultrafine grain sizes at the high temperatures required for superplastic flow.

Acknowledgements

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References

1. E. O. HALL, *Proc. Roy. Soc. B* **64** (1951) 747.
2. N. J. PETCH, *J. Iron Steel Inst.* **174** (1953) 25.
3. A. BALL and M. M. HUTCHISON, *Metal Sci. J.* **3** (1969) 1.
4. T. G. LANGDON, *Acta Metall. Mater.* **42** (1994) 2437.
5. T. C. LOWE and R. Z. VALIEV, *JOM* **52**(4) (2000) 27.
6. T. C. LOWE and R. Z. VALIEV (ed.), "Investigations and Applications of Severe Plastic Deformation" (Kluwer, Dordrecht, The Netherlands, 2000).
7. K. NAKASHIMA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Mater. Sci. Eng. A* **281** (2000) 82.
8. V. M. SEGAL, *ibid.* **197** (1995) 157.
9. S. R. AGNEW, U. F. KOCKS, K. T. HARTWIG and J. R. WEERTMAN, in "Modelling of Structure and Mechanics of Materials from Microscale to Product," edited by T. Leffers, T. Lorentzen, O. B. Pedersen, B. F. Sørensen and G. Winther (Risø National Laboratory, Roskilde, Denmark, 1998), p. 201.
10. V. M. SEGAL, V. I. REZNIKOV, A. E. DROBYSHEVSKIY and V. I. KOPYLOV, *Russian Metall.* **1** (1981) 99.
11. Y. IWAHASHI, J. WANG, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Scripta Mater.* **35** (1996) 143.
12. Y. WU and I. BAKER, *ibid.* **37** (1997) 437.
13. A. SHAN, I.-G. MOON, H.-S. KO and J.-W. PARK, *ibid.* **41** (1999) 353.
14. D. P. DELO and S. L. SEMIATIN, *Metall. Mater. Trans. A* **30A** (1999) 1391.
15. R. E. GOFORTH, K. T. HARTWIG and L. R. CORNWELL, in "Investigations and Applications of Severe Plastic Deformation," edited by T. C. Lowe and R. Z. Valiev (Kluwer, Dordrecht, The Netherlands, 2000), p. 3.
16. T. AIDA, K. MATSUKI, Z. HORITA and T. G. LANGDON, *Scripta Mater.* **44** (2001) 575.
17. D. N. LEE, *ibid.* **43** (2000) 115.
18. H. S. KIM, M. H. SEO and S. I. HONG, *Mater. Sci. Eng. A* **291** (2000) 86.
19. K. NAKASHIMA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Acta Mater.* **46** (1998) 1589.
20. P. B. PRANGNELL, C. HARRIS and S. M. ROBERTS, *Scripta Mater.* **37** (1997) 983.
21. S. L. SEMIATIN, D. P. DELO and E. B. SHELL, *Acta Mater.* **48** (2000) 1841.
22. Y. IWAHASHI, M. FURUKAWA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Metall. Mater. Trans. A* **29A** (1998) 2245.
23. M. FURUKAWA, Y. IWAHASHI, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Mater. Sci. Eng. A* **257** (1998) 328.
24. S. LEE and T. G. LANGDON, *Mater. Res. Soc. Symp. Proc.* **601** (2000) 359.
25. Y. IWAHASHI, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Acta Mater.* **45** (1997) 4733.
26. S. FERRASSE, V. M. SEGAL, K. T. HARTWIG and R. E. GOFORTH, *Metall. Mater. Trans. A* **28A** (1997) 1047.
27. *Idem.*, *J. Mater. Res.* **12** (1997) 1253.
28. Y. IWAHASHI, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Acta Mater.* **46** (1998) 3317.
29. *Idem.*, *Metall. Mater. Trans. A* **29A** (1998) 2503.
30. M. FURUKAWA, Y. MA, Z. HORITA, M. NEMOTO, R. Z. VALIEV and T. G. LANGDON, *Mater. Sci. Eng. A* **241** (1998) 122.
31. S. KOMURA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *J. Mater. Res.* **14** (1999) 4044.
32. H. HASEGAWA, S. KOMURA, A. UTSUNOMIYA, Z. HORITA, M. FURUKAWA, M. NEMOTO and T. G. LANGDON, *Mater. Sci. Eng. A* **265** (1999) 188.
33. Z. HORITA, T. FUJINAMI, M. NEMOTO and T. G. LANGDON, *Metall. Mater. Trans. A* **31A** (2000) 691.
34. A. YAMASHITA, D. YAMAGUCHI, Z. HORITA and T. G. LANGDON, *Mater. Sci. Eng. A* **287** (2000) 100.
35. A. GHOLINIA, P. B. PRANGNELL and M. V. MARKUSHEV, *Acta Mater.* **48** (2000) 1115.
36. C. P. CHANG, P. L. SUN and P. W. KAO, *ibid.* **48** (2000) 3377.
37. T. G. LANGDON, M. FURUKAWA, M. NEMOTO and Z. HORITA, *JOM* **52**(4) (2000) 30.
38. P. B. BERBON, M. FURUKAWA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Metall. Mater. Trans. A* **30A** (1999) 1989.
39. K. OH-ISHI, Z. HORITA, M. FURUKAWA, M. NEMOTO and T. G. LANGDON, *ibid.* **29A** (1998) 2011.

40. Y. T. ZHU and T. C. LOWE, *Mater. Sci. Eng. A* **291** (2000) 46.
41. R. Z. VALIEV, D. A. SALIMONENKO, N. K. TSENEV, P. B. BERBON and T. G. LANGDON, *Scripta Mater.* **37** (1997) 1945.
42. P. B. BERBON, N. K. TSENEV, R. Z. VALIEV, M. FURUKAWA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Metall. Mater. Trans. A* **29A** (1998) 2237.
43. P. B. BERBON, M. FURUKAWA, Z. HORITA, M. NEMOTO, N. K. TSENEV, R. Z. VALIEV and T. G. LANGDON, *Phil. Mag. Lett.* **78** (1998) 313.
44. S. KOMURA, P. B. BERBON, M. FURUKAWA, Z. HORITA, M. NEMOTO and T. G. LANGDON, *Scripta Mater.* **38** (1998) 1851.
45. S. LEE, P. B. BERBON, M. FURUKAWA, Z. HORITA, M. NEMOTO, N. K. TSENEV, R. Z. VALIEV and T. G. LANGDON, *Mater. Sci. Eng. A* **272** (1999) 63.
46. Z. HORITA, M. FURUKAWA, M. NEMOTO, A. J. BARNES and T. G. LANGDON, *Acta Mater.* **48** (2000) 3633.
47. S. KOMURA, Z. HORITA, M. FURUKAWA, M. NEMOTO and T. G. LANGDON, *J. Mater. Res.* **15** (2000) 2571.
48. *Idem.*, *Metall Mater. Trans. A* **32A** (2001) 707.
49. O. B. MAKOVA, V. K. PORTNOY, I. I. NOVIKOV, N. I. KOLOBNEV and L. B. KHOKHLATOVA, in "Aluminium-Lithium," Vol. 2, edited by M. Peters and P.-J. Winkler (DGM Informationsgesellschaft, Oberusel, Germany, 1992), p. 1133.
50. M. FURUKAWA, P. B. BERBON, Z. HORITA, M. NEMOTO, N. K. TSENEV, R. Z. VALIEV and T. G. LANGDON, *Metall. Mater. Trans. A* **29A** (1998) 169.

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