etadata, citation and similar papers at core.ac.uk

brought to you by

J. ZRNIK, S. V. DOBATKIN, I. MAMUZIČ

ISSN 0543-5846
METABK 47(3) 211-216 (2008)
UDC – UDK 669.14-418:539.37:620.17=111

PROCESSING OF METALS BY SEVERE PLASTIC DEFORMATION (SPD) – STRUCTURE AND MECHANICAL PROPERTIES RESPOND

Received – Prispjelo: 2007-08-10
Accepted – Prihvaćeno: 2008-03-20
Review Paper – Pregledni rad

SPD methods are used to convert coarse grain metals and alloys into ultrafine grained (UFG) materials. Obtained UFG materials then possess improved mechanical and physical properties which destine them for a wide commercial use. This paper, in one direction, looks into historical development of SPD processes and their effect at obtaining fine crystalline structure, and on the other side also partially focuses on development of UFG structure and its stability in commercial pure aluminium as a function of strain and post-deformation annealing applied.

Key words: Severe plastic deformation, ultrafine grained materials, aluminium, microstructure evolution, annealing, recovery, mechanical properties.

Obrada metala intenzivnom plastičnom deformacijom (IPD) – odgovarajuća struktura i mehanička svojstva. IPD je postupak pretvorbe krupno u ultrafino zrnate (UFZ-a) metale i legure. Dobiveni UFZ materijal posjeduje oplemenjena mehanička i fizikalna svojstva, te su namjenjeni za široko komercijalno rabljenje. Ovaj članak s jedne strane daje osvrt na povijesni razvitak IPD-a postupka, a s druge strane djelomice ishodište za razvitak UFZ-a i njezine stabilnosti u trgovački čistom aluminiju, kao funkcija preoblikovanja i poslije deformacije primjenjenog žarenja.

Ključne riječi: intenzivna plastična deformacija, ultrafino zrnati materijal, aluminij, razvitak mikrostrukture, mehanička svojstva

INTRODUCTION

Severe plastic deformation (SPD) is one of the methods of obtaining very fine crystalline structure in different bulk metals and alloys, which possess different crystallographic structure. SPD causes the formation of micrometer and sub-micrometer sized subgrains in the initially coarse grain materials. As a result of that enhanced mechanical performance is observed. The mechanism responsible for this effect is still under investigation, however, it is believed that short and long-range intersecting shear bands produced by plastic deformation play a major role at grain subdivision and local dynamic recovery and recrystallization processes contribute to grain refinement [1,2]. Sufficiently large deformation leads to a distinct structure of dislocation-free and highly misoriented fine grains. When defining a submicron grain structure the important parameters, which are matter of concern, are average spacing of high angle grain boundaries (HAGB) and proportion of HAGB area [3,4].

The structural changes caused by SPD are reflected in improved mechanical properties of metals. The reported effects include increased hardness and yield stress, both featuring tendency to saturation. The drawback of ultrafine grained structure materials is their limited ductility [5,6]. Some other research revealed increased ductility and toughness as well as improved dumping and physical properties. The fine grained structure of UFG materials obtained by SPD leads to superplastic behaviour of these materials at lower temperatures and yet with higher deformation rates.

Various aspects of structural changes caused by SPD have been the research goals in laboratories worldwide. Hundreds of papers are published each year in distinguished journals and conference proceedings (see proceedings of two TMS conferences on UFG structure development and properties evaluation [7,8]). Today the most effort is paid to the study of the mechanics of material flow and grain subdivision when low strains ($\varepsilon_{ev} < 3$) and high strains ($\varepsilon_{ev} > 3$) are considering at the SPD. Usually, in dependence of the applied deformation methods (processing conditions) when different strain (von Mises) at deformation is developed the various structure can be found in deformed materials. At low strain the orientation splitting and microshear banding

---

J. Zrnik Comptes FHT, Ltd. Plzen, Czech Republic, S. V. Dobatkin, Moscow Institute of Metallurgy and Material Science, Moscow, Russia, I. Mamuzić, Faculty of Metallurgy University of Zagreb, Sisak, Croatia

are mechanisms, which contributes to grain subdivision and cell bands structure dominates within deformed bands. When medium and higher strain are effective the lamellar HAGB structure, ribbon grains and formation of submicron grains structure dominates in deformed materials. The repetition of the straining process is required to obtain a large strain and desired structural changes.

When studying microstructure in SPD materials the evolution and the character of the new interfaces appears as very important property with respect to evaluation their influence on the mechanical properties. Considering the deformation processing condition the heterogeneity in microstructure formation was often observed across the bulk specimen in dependence of the strain introduced [9]. Anticipating commercialisation attempts, this work addresses the processing issues. First, a choice of major SPD processes will be presented, but not analysed in details. Latter in the paper, the contribution to ductility improvement of severely deformed aluminium will be presented shortly.

SPD EXPERIMENTAL PROCESSES

Obtaining large plastic deformation is a difficult task since in most metal forming processes it is limited by either material or tool failure. Few processes such as accumulative rolling and multi-pass drawing enable large plastic deformation to be achieved; however, metal foils or micro wires produced by these processes are not necessary the billet forms required. Therefore, special metal forming processes, capable for producing SPD without a major change in the billet should follow:

a*) simple concept; b) how do you do it; c) does it really work; c) is it any use.* Among these can be included the following major SPD processes:

- Equal channel angular pressing (ECAP, Segal, 1977);
- High pressure torsion (HPT, Valiev at al., 1989);
- Accumulatibe roll bonding (ARB, Saito, Tsuji, Utsunomiya, Sakai, 1998);
- Reciprocating extrusion-compression (REC, J. and M. Richert, Zasadzinski, Korbel, 1979);
- Cyclic close die forging (CCDF, Ghosh, 1988);
- Repetitive corrugation and straightening (RCS, Zhu, Lowe, Jiang, Huang, 2001).

The major SPD processes are presented in Figure 1.

EQUAL CHANNEL ANGULAR PRESSING

Equal channel angular pressing (ECAP) was invented by Segal [10, 11] in 1977 in Russia. ECAP is based on simple shear taking place in a thin layer at the crossing plane of the equal channels. ECAP has become the most frequently used SPD process. This is due to its low force requirement (small press can be used) and the resulting low tool pressure. This together with the simple tool geometry makes laboratory tooling easily attainable. In many laboratories the researchers found ECAP method as convenient tool to investigate the relationship between the strain applied and structure development.

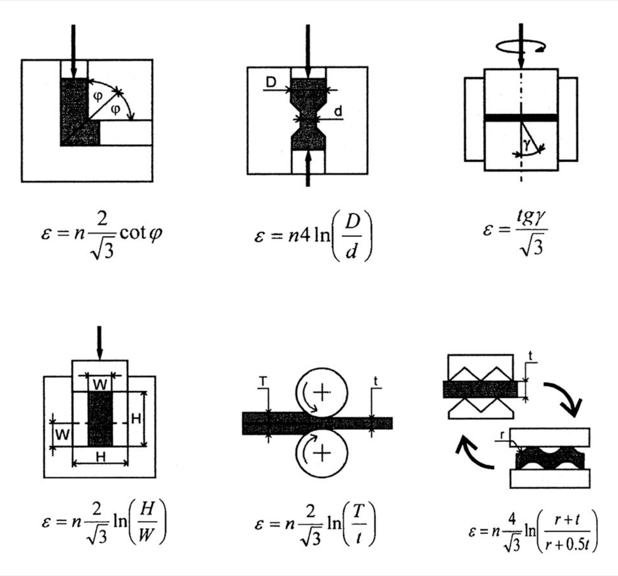

Figure 1. Schematic representation of major SPD processes.

The basic mechanics of ECAP was described by Segal in [11] where he derived an expression for shear strain tg γ and the process pressure p.

$$\mathrm{tg}\gamma = \sqrt{3}\frac{p}{Y}$$

Recently he concluded that the strain distribution depended mainly on friction uniformity in the channels and the details of the channel geometry (sharp vs. round corner). Backpressure appeared to have only small effect. In work [12], he demonstrated advantages of simple shear produced by ECAP over pure shear present in other processes.

In order to achieve the required strain in ECAP, the billet is processed repeatedly in the same die. The billet can rotate about its axis between each pass. The four basic options for this rotations are called A, C, B_A, B_C, Figure 2. These options have been then classified and assessed in terms of their ability to control the structure and texture of processed materials. From these tests Langdon et al. [13] established that for obtaining homogeneous microstructures of equiaxed grains separated by high angle boundaries the best route is B_C.

ECAP method does not involve high pressure which is advantageous from the machine and tooling point of view. This may turn into a disadvantage when processing brittle materials. Even ductile materials may require a bit higher pressure to avoid damage accumulation and substantially reduced ductility in further metal forming operations. It is possible to process brittle materials at a smaller pressure provided the temperature is high

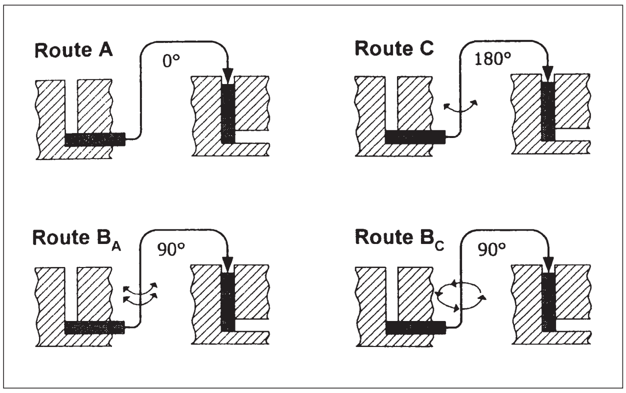

Figure 2. Options for billet rotation between consecutive passes through ECAP die.

enough. This, however, may change the material behaviour by making it prone to unstable flow and fracture. What is observed resembles a serrated or cyclic chip sometimes produced in metal cutting, Figure 3.

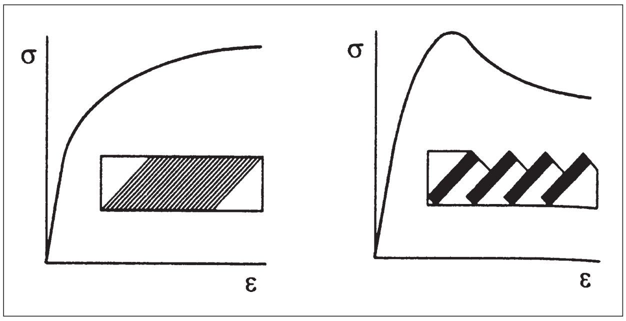

Figure 3. Schematic representation of stable and localised flow in ECAP depending on the material hardening – softening [14].

The classical ECAP inspired many further developments in ECAP tool in order to process difficult-to-work alloys described in [15]. There are also attempts of designing a continuous system in which workpiece by friction or by rolls support is fed to the die [16-19]. In order to avoid buckling the force required to feed the billet has to be low. ECAP is low-force process by nature. Nevertheless, reducing the amount of deformation in one pass, reducing friction by leaving a clearance between the billet and the die and increasing process temperature may be necessary.

RECIPROCATING EXTRUSION COMPRESION

J. Richert et al. came with the idea of cyclic reciprocating extrusion compression (RE) [20, 21]. RE involves the cyclic flow of metal between the alternating extrusion and compression chambers, Figure 4. The deformation effect could obviously be achieved with the frame/die fixed and the movable punches or vice versa.

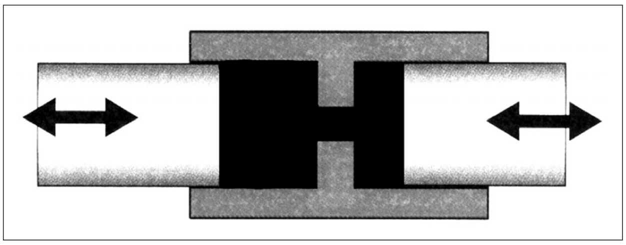

Figure 4. Reciprocating extrusion compression method.

While the microstructural results of RE have been published widely, the mechanics of the process received less attention. Some results for RE of cylindrical billets are available in [22], where a simplified stress analysis as well as closer to real conditions Finite Element Method (FEM) simulation shed some light on the deformation process. Results obtained from these analyses revealed that some sections with a hydrostatic stress state (section 0 and 3) and other sections (1-2 and 4-5) where the yield condition is met, Figure 5. This means that there is an elastic unloading in the transition zone between the two chambers of the die.

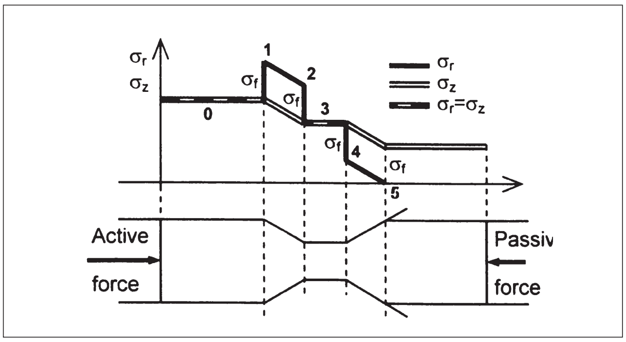

Figure 5. Schematic distribution of radial and axial stresses in reciprocical extrusion process [22].

In the stress area, the stress path comprises primary yielding of the material due to extrusion, unloading into the elastic domain and secondary yielding by compression on the opposite side of the yield surface.

The forming force as well as tool pressure depends very much on friction. In Figure 6 are documented the active force and die pressure for low carbon steel obtained at simulation with friction and without friction. When adding small friction (μ=0.06) the active force increased essentially. A similar effect was observed for the die pressure. The consequence of a high forming force is just a bigger, more expensive press and use of special materials for tools.

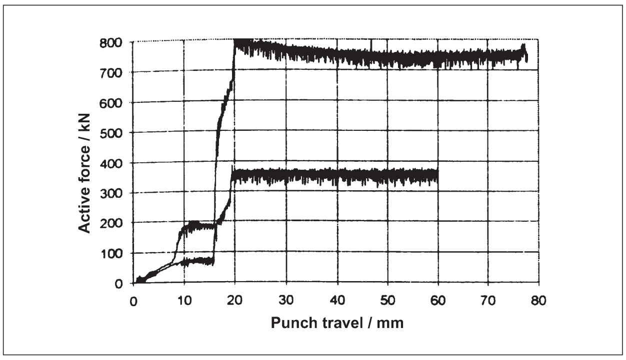

Figure 6. The effect of friction on the forming force after the first cycle of RE with passive force simulation of 200 kN [22].

HIGH PRESSURE TORSION

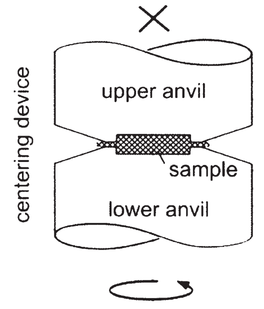

Figure 7. Principle of HPT experiment.

High pressure torsion was (HPT) first investigated by Bridgman [23]. Bridgman's experiment did not bring much light on the microstructural changes taking place in severely deformed metals. It was Erbel seems to be the first who carried out HPT experiments for copper [24].

He descriebed and interpreted the grain structure evolution towards a structure of dislocation free subgrains with high angles of misorientation and submicrometer size. He also directed out the increase and eventual saturation of the mechanical properties of severely deformed materials.

Numerous papers prove capability of HPT to achieve UFG structure [25, 26, 27]. For this method, a coin-shape sample is pressed between two anvils under hydrostatic pressure (~ 7 GPa). During the build-up of the pressure, the sample is pressed into the cavities in the anvil and a burr is formed at the edge of the sample. Then one anvil is rotated with respect to the other one and the rotation speed can be varied over a large range. This leads to a deformation of the sample by almost simple shear. The burr prevents a contact between the two anvils and upholds the hydrostatic pressure. Due to the high pressure, in most metals the formation of cracks is suppressed and therefore it is possible to apply very high strain without failure of the deformed material.

The reached shear strain γ is a function of the twist angle φ, the radius r (of the site of investigation) and the thickness t. The strain can be expressed in terms of and equivalent von Mises strain by dividing the shear strain by $\sqrt{3}$. The equivalent von Mises strain $\varepsilon_{\mathrm{eq}}$ as a function of the number of turns n is then given by

$$\varepsilon_{eq} = \frac{2\pi rn}{t\sqrt{3}}$$

The expression was criticized as giving unreasonable results for higher shear angles γ and therefore replaced in some publications by a logarithmic measure ($\varepsilon = 2\ln(\mathrm{tg}\gamma / 3^{0.5})$). On the other hand in a paper [28] the authors claim that the logarithmic measure is inappropriate for simple shear (present in torsion) because id does not take into account the incremental rotations of the principal axes of the strain tensor. A logarithmic measure is the right one for pure shear though.

HPT of pure metals leads to a grain refinement until an equilibrium between the fragmentation of large structural elements and grain restoration processes leads to a saturation of the refinement process. Parallel to the refinement of the structure, the mechanical strength increases until it saturates, too. UTS of ~ 1500 MPa for pure iron and ~ 450 - 500 MPa for pure copper were measured after HPT. Fromcomparison of the in-situ measured torque of nickel with iron and copper, the UTS of nickel can be estimated to be ~ 1300 MPa [29].

CONSTRAIN GROOVE PRESSING

In 2001, Zhu et al. described an SPD method based on the repetitive corrugating and straightening now know as CGP [30, 31]. This method involves bending a straight slab with corrugated tools and then restoring the straight shape of the slab with flat tools. The details of the whole CGP process when material is pressed in asymmetrically positioned grooves and then straighten between a set of flat die is illustrated in Figure 8. Repetition of the process is required to obtain large effective strain $\varepsilon_{\mathrm{ef}}$ needed to refine the coarse grain structure [32]. Unlike the widely used ECAP process for structure refinement, the CGP process has an advantage that large plastic deformation can be applied to the metal in sheet or plate form. However, one drawback of this process is the presence of deformation heterogeneity in microstructure across the bulk specimen depending on the strain introduced [33].

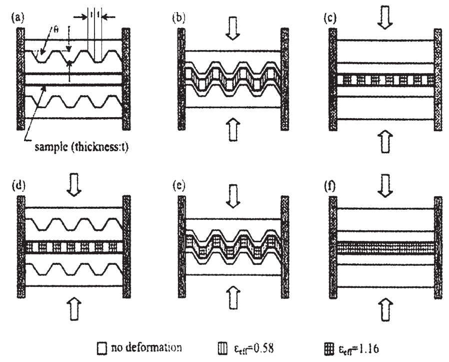

Figure 8. Schematic process of CGP deformation cycle.

The results of microstructure analysis of pure aluminium using TEM carried out after different effective strain introduced ($\varepsilon_{\mathrm{eff}}$ ranging from 1,16 to 9,6) confirmed that by applying CGP the microstructure refinement at room temperature is sluggish process [34]. As results of different straining applied the banded elongated subgrain structure is present due to dominant shear strain. The formation of new equiaxed polygonized grains with high angle boundaries strongly depends on effective strain introduced. However, the

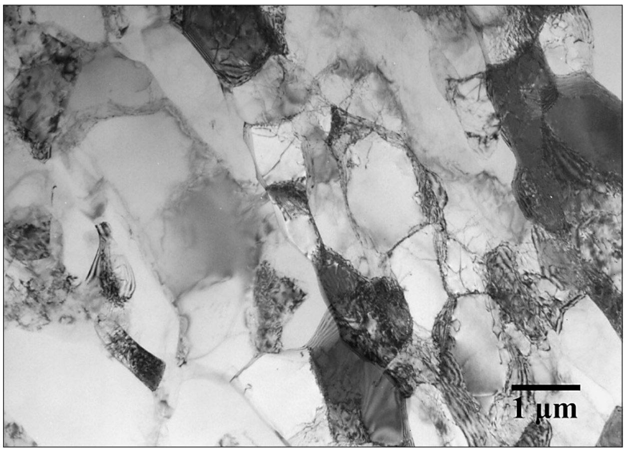

Figure 9. Fragmentated subgrain structure showing different progress of polygonization in banded structure experienced 4 passes, $\varepsilon_{ef} \sim 4{,}6$.

banded structure, where mixture of deformed subgrain having low angle boundaries (LAGB) and polygonized subgrains and eventually grains with high angle boundaries (HAB) result from local dynamic recovery persisted till high straining, Figure 9. The local formation of grains with high angle boundaries was confirmed by SAE diffraction.

More precise information to clarify the efficiency of CGP in structure refining was obtained from SEM/EBSD analysis. From the orientation mapping data evaluation, the boundary misorientation maps (EBSD data) confirmed that in deformed structure, experienced different strain in range of $\varepsilon_{ef} - 1{,}16 - 9{,}3$, the heterogeneous mixture of subgrains with LAB (misorientation angle around $\theta < 15°$) and new grains with transformed HAB (misorientation angle $\theta > 15°$) can be found. The results confirmed that the fraction of high angle boundaries in severely deformed aluminium ($\varepsilon_{ef} \sim 9{,}3$), comparing to results employing ECAP method, is lower. Evaluating the efficiency of CGP method the formation of UFG structure is less effective. The EBSD misorientation map of deformed aluminium after 4 passes ($\varepsilon_{ef} \sim 4{,}6$), is shown in Figure 10. The values from the tensile and hardness tests also confirmed that there is not major difference between the specimens subjected to different number of pressing [29]. The structure and tensile results showed the concord effect, and confirmed the hardening results from deformed structure is still dominating.

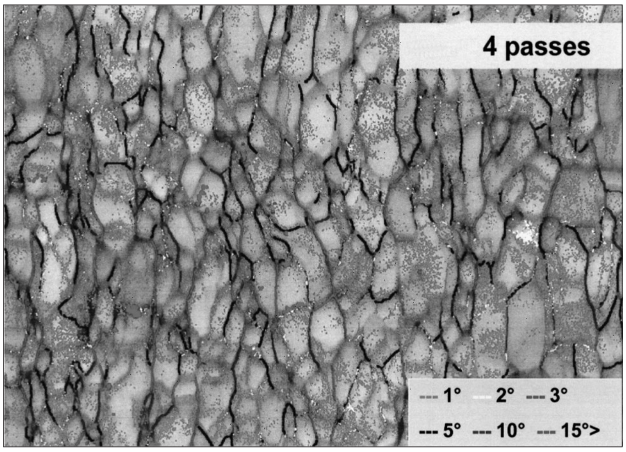

Figure 10. EBSD misorientation map of CGP deformed aluminium to effective strain $\varepsilon_{ef} \sim 4{,}6$.

CONCLUSIONS

Structural changes in materials subjected to SPD and their effect on properties have been investigated for more then twenty years. In the last ten years, our knowledge of the governing phenomena has been largely extended. However, there is still work to be done in order to understand and control SPD effect.

It is clear from literature survey of the SPD technology that there are a great variety of possible SPD processes. No doubt new and improved processes will still be developed. The main technical problems are the same as in traditional metal forming operations. Thus one of them is the integrity of material deformed. Different materials show different deformation ability. The increased temperature may negate the structural effects of SPD by recovery and recrystallization and help in deformation of more brittle materials. Another problem related with increased processing temperature is flow softening of the material which may lead to plastic flow localization and to fracture.

Acknowledgement – This paper contains results of investigation conducted as part of the MSM2631691901 project funded by the Ministry of Education of the Czech Republic.

REFERENCES

1. S. V. Dobatkin, "Severe Plastic Deformation of Steels: Structure, Properties and Techniques" in *Investigation and Applications of Severe Plastic Deformation*, Ed. by T. C. Lowe and R. Valiev (Kluwer, Netherlands), 3 (2000) 13-22.
2. R. Z. Valiev, I. V. Alexandrov, Nanostructured Materials Produced by Severe Plastic Deformation, Logos, Moscow, 2000.
3. R. Z. Valiev, Y. V. Alexandrov, Y. T. Yhu, T.C. Love, J. Mater. Res. 17 (2002) 5.
4. R. Z. Valiev, R. K. Islamgaliev, Y. V. Alexandrov, Progr. Mater. Sci. 45 (2000) 103
5. N. Tsuji, R. Ujei, Y. Ito, H. W. Kim, Ultrafine Grained Materials IV., Ed. by Y. T. Zhu, T. Langdon, Y. Horita, M. J. Zehetbauer, S. I. Semiatin, T. C. Lowe, TMS (The Minerals, Metals & Materials Society) 2006, 81
6. H. Miyamoto, K. Ota, T. Mimaki, Scripta Materialia, 54 (2006) 1725
7. T. C. Lowe, R. Z. Valiev (Eds.), Investigation and Applications of Severe Plastic Deformation (Kluwer, Dodrecht, 2000)
8. TMS zbornik
9. J. Zrnik, Metallic Materials, 44 (2006) 5, 243
10. V. M. SegaL, Patent No. 575892, 1977
11. V. M. Segal, V. I. Rezhnikov, A. E. Dobryshevsky, V. I. Kopylov, Russ. Metall. (Engl. Transl.) 1 (1981) 99
12. V. M. Segal, Mat. Sci. Eng. A 345 (2003) 36

13. T. G. Langdon, M. Furukawa, M. Nemoto, Z. Horita, Jom, 52 (2000) 4, 30
14. D. P. Delo, S. L. Semiatin, Metall. Mater. Trans. A, 30 (1999) 2473
15. S. L. Semiatin, D. P. Delo, U.S. Patent No. 5904062,1999
16. V. M. Segal, U.S. Patent No. 5400633, 1995
17. M. V. Markusev, V. N. Sloboda, O. A. Kaibyshev, Russian Patent No. 2146571, 2000
18. Y. Saito, H. Utsunomiya, H. Suzuki, in M. Geiger (Ed.), Advanced Technology of Plasticity, (Springer, 1999) 2459
19. J. C. Lee, H. K. Seok, J. H. Han, Y. H. Chung, Mater. Res. Bull., 36 (2001) 997
20. J. Richert, M. Richert, J. Zasadzinski, A. Korbel, Patent PLR No. 123026, 1979
21. A. Korbel, M. Richert, J. Richert, Proc. 2nd Riso Int. Symp. on Metall. and. Mat. Sci. Roskilde, 1981, 445
22. A. Rosochowski, R. Rodiet, P. Lipinski, Proc. of the 8th Int. Conf. Metalforming 2000, Krakow, Sept. 2000, 235
23. P. W. Bridgman, Physical Review, 48 (1935) 825
24. S. Erbel, Met. Technol., (1979) 482
25. R. Z. Valiev, N. A. Krasilnikov, N. K. Tsenev, Mat. Sci. Eng. A, 137 (1991) 35
26. R. Z. Valiev, Mat. Sci. Eng. A, 234-235 (1997) 59
27. O. N. Senkov, F. H. Froes, V. V. Stolyarov, R. Z. Valiev, J. Liu, Scripta Mater., 39 (2001) 1511
28. C. Shrivastava, J. J. Jonas, G. Canova, J. Mech. Phys. Solids, 30 (1982) 75
29. A. Vorhauer, R. Pippan, Scripta Mater., 51 (2004) 921
30. Y. T. Zhu, T. C. Lowe, H. Jiang, J. Huang, U.S. Patent No. 6197129 B1, 2001
31. J. Y. Huang, Y. T. Zhu, H. Jiang, T. C. Lowe, Acta Mater. 49 (2001) 1497
32. J. Zrnik, J. Drnek, M. Cieslar, Z. Novy, Metallic Materials, 44 (2006) 267
33. J. Zrnik, T. Kovarik, M. Cieslar, L. Kraus, J. of Mat. Sci. 2008 (in review process)

Note: The Author J. Zrnik is responsible for English language.