Archives of Materials Science and Engineering
Volume 36
Issue 1
March 2009
Pages 20-27
International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Structure of the copper under controlled deformation path conditions

G. Niewielski, D. Kuc*
Faculty of Materials Science and Metallurgy; Silesian University of Technology, ul. Krasińskiego 8, 40-019 Katowice, Poland
* Corresponding author: E-mail address: dariusz.kuc@polsl.pl

Received 12.02.2007; published in revised form 01.03.2009

ABSTRACT

Purpose: One of the methods of plastic deformation under complex deformation path conditions is compression with oscillatory torsion. The observable effects in the form of changing force parameters and structure changes confirm the possibility of deformation to a value many times higher than in the case of methods traditionally applied for forming. This article presents the results of the influence of compression with oscillatory torsion on structural phenomena occurring in copper deformed in such a way.

Design/methodology/approach: The examinations were conducted at a compression/oscillatory torsion test stand. The structural examinations were conducted with the use of light and electron microscopy.

Findings: In experimental investigations, a reduction of unit pressures was observed when compared to conventional compression. The structural examinations indicated substantial differences in the mechanisms of plastic deformation conducted in both conventional and combined way.

Research limitations/implications: There are premises which show that a metallic material of a nanometric structure can be obtained in this way (top-down method), by the accumulation of great plastic deformation. Metallic materials characterized by grain size below 100nm are distinguished by unconventional properties. Further examinations should focus on conducting experiments in a way that would enable grain size reduction to a nanometric size. This will enable the cumulation of greater deformation in the material.

Originality/value: The method of compression with oscillatory torsion is an original method developed at the Silesian University of Technology, owing to which it is possible to obtain high deformation values (SPD) without risking the loss of cohesion of the material. Thorough understanding of the changes taking place in the structure of metals subjected to compression with oscillatory torsion will allow the optimal choice of process parameters in order to achieve a gradual grain size reduction.

Keywords: Nanomaterials; Plastic forming; Microstructure; Compression with oscillatory torsion

Reference to this paper should be given in the following way:

G. Niewielski, D. Kuc, Structure of the copper under controlled deformation path conditions, Archives of Materials Science and Engineering 36/1 (2009) 20-27.

MATERIALS

1. Introduction

Materials with both nano- and submicrometric structure have competitive physical and mechanical properties compared to conventional alloys. There is a some of methods to obtain fine grain material, the most important being : high pressure torsion (HPT) [7,12,14,17,22,32] equal-channel angular extrusion (ECAE) [2,6,14,18,19,20,21,31,33] cumulated plastic deformation [9], cyclic extrusion compression (CEC) [16,24] or KOBO method [4,5]. Nanometric structures obtained by different methods differ from one another in many aspects: the degree of structure refinement, structure inhomogeneity, and the mechanical properties [1,8,26,28,29].

One of the methods of plastic strain under controlled deformation path conditions is compression with oscillatory torsion. The observable effects in the form of changing force parameters and a changing structure corroborate the little yet known possibility of effective strain to a value many times higher than in the case of methods traditionally applied for forming [3,15,23,27,30]. The literature shows explicitly that there is no information as regards the influence of the presented material deformation method on the structural phenomena accompanying the grain refinement processes after such deformation.

The construction of a device used for this purpose is an original solution developed at the Department of Process Modelling and Medical Engineering of the Silesian University of Technology [10,11]. The paper presents the influence of oscillatory compression parameters on the properties of submicrometric and nanometric structures of electrolytic copper. The properties of a structure being formed as a result of deformation under controlled path conditions are determined.

2. Material and methodology

The research was made on rolled samples made of copper M1E type, with an initial diameter $d_o = 10$ mm and initial height $h_o = 15$ mm ($h_o/d_o = 1.5$). The chemical composition of the electrolytic copper is shown in Table 1. Before strain, the copper was annealed at a temperature of 550°C for 2 hours, with cooling in the air. After annealing, the mean grain size of the copper was 35 μm.

The research was made on rolled samples made of copper M1E type, with an initial diameter $d_o = 10$ mm and initial height $h_o = 15$ mm ($h_o/d_o = 1.5$). The chemical composition of the electrolytic copper is shown in Table 1. Before strain, the copper was annealed at a temperature of 550°C for 2 hours, with cooling in the air. After annealing, the mean grain size of the copper was 35 μm.

Table 1.
Chemical composition of the electrolytic copper

Cu	Fe	Bi	Pb	Ni	Sn	As
98.8	0.02	0.002	0.008	0.02	0.03	0.001

The samples were deformed at draft $\Delta h = 7.5$ mm, which corresponds to 50% of the initial height h_0, applying additional torsion at a frequency of the lower punch oscillation in the range from 0 (conventional compression) to 1.6 Hz and a torsion angle equal 6° (Table 2).

An area representative for the microstructural investigations after oscillatory compression was selected from the centre of the sample's height at a distance of ca. 0.8 of the sample's external surface radius.

Table 2.
Variants of compression tests carried out with oscillatory torsion of copper samples

No.	Torsion frequency f_{tor} [Hz]	Torsion angle $\pm \alpha$ [°]	Compression rate v_t [mm/s]
1	0	0	0.6
2	0.4	6	0.15
3	0.4	6	0.6
4	1.6	6	0.15
5	1.6	6	0.6

Metallographic investigations were determined on a Reichert Me-F2 light microscope at magnification 1000×. The copper samples were etched in a solution of the following composition: 30 ml CH_3COOH, 20 ml HNO_3 and 30 ml C_2H_6CO.

Substructure were detrmined on a transmission electron microscope, using thin film techniques. The input material were ca. 1 mm thick samples cut out by spark erosion. Initial thinning of the samples to a thickness of ca. 0.08-0.05 mm was carried out using mechanical grinding machines. Next, using a matrix, disks of a diameter of 3 mm were cut out. The so obtained samples were thinned by a two-sided stream electropolishing method in a Tenupol device manufactured by Struers. Struers' D2 electrolyte was applied, cooled down to 15ºC at a polishing voltage equal 15V. Structural investigations were conducted using a JOEL's JEM 100B transmission microscope with accelerating voltage of 100 kV.

3. Results

The dependence determined between the average unit pressure values (p), and the strain (ε), based on the measurement conducted during samples strain was presented in Figure 1. As the torsion frequency increases, a gradual fall of the mean unit pressure values is observed as compared to conventional compression.

Microstructural investigations were determined on a light microscope, using interference contrast for a detailed analysis of the morphologies of the slip and shear bands being formed. A change of the deformation method (conventional compression and compression with oscillatory torsion) at predefined process parameters does not change the basic features of the microstructure (Figures 2-4). In the case of conventional deformation or oscillatory compression, the occurrence of one system of slip was most often observed; in some cases, single deformation bands were grouped into clearly visible macrobands.

The observed structure of mutual intersection or uniformly distributed slip bands may testify to a homogeneous deformation method and lack of privileged concentrations of slips in selected planes.

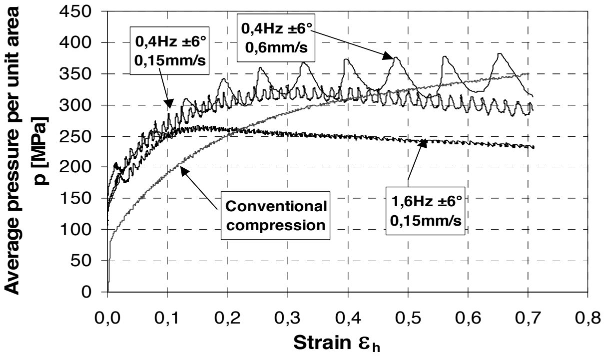

Fig. 1. Influence of the parameters of compression with oscillatory torsion on mean unit pressure values

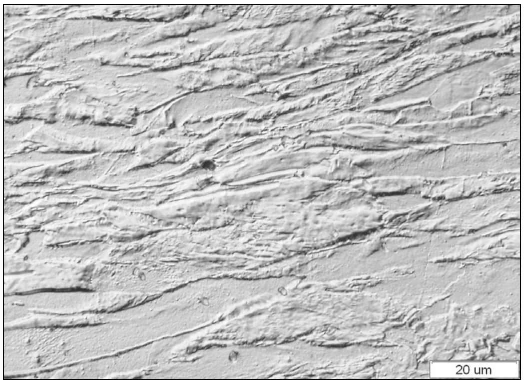

Fig. 2. Cu microstructure after conventional compression

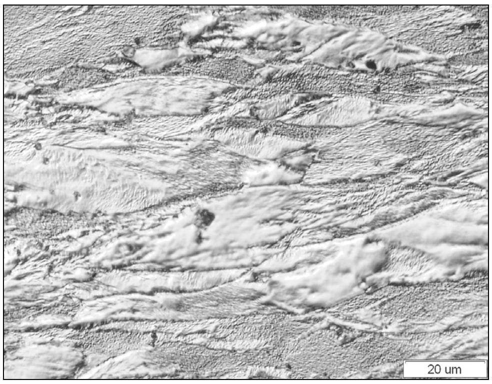

Fig. 3. Cu microstructure after oscillatory compression at the following parameters: f = 0.4 Hz, α = 6° and v = 0.15 mm/s

A change of the deformation method into oscillatory compression in the investigated range of parameters' change does not intensify the process of slip bands or shear bands formation. No deformation localization phenomena were observed at this stage of structural investigations.

A full picture of the structural changes which accompany the deformation process under conventional and oscillatory compression conditions is provided by substructure investigations. They shoved the formation of two types of dislocation structure - flat elongated systems of dislocation boundaries (most often, dislocation cells are visible between elongated systems of boundaries) and often, equiaxial cell dislocation structures.

Conventional deformation, apart from the accompanying cellular structure and dislocation bands, is accompanies by a system of dislocation bands' intersections,, which indicates the initiation of deformation in two systems of slip (Figure 5).

The microstructure of Cu samples deformed at f = 0.4 Hz, α = 6° and v = 0.15 mm/s, is characterized by the presence of a cellular dislocation structure. Such dislocation systems prevailed during the thin foil observations.

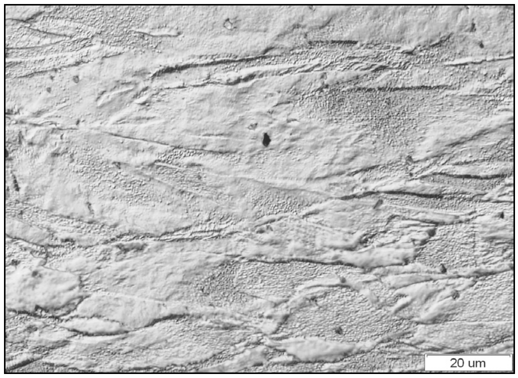

Fig. 4. Cu microstructure after oscillatory compression at the following parameters: f = 1.6 Hz, α = 6°and v = 0.15 mm/s

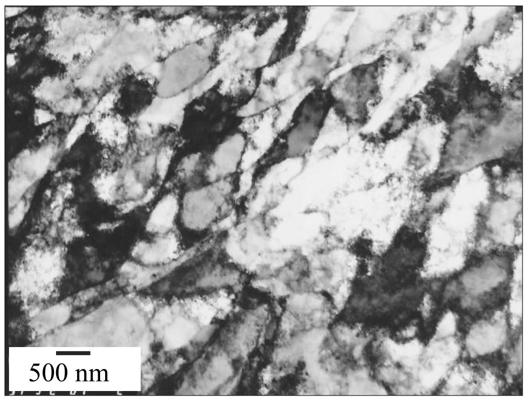

Fig. 5. Cu substructure after conventional compression. Cellular structure and dislocation bands

Most frequently, dislocation cells are visible between elongated systems of dislocation boundaries (Figure 6). In addition, narrow bands of considerable disorientation occur as well (Figure 7). The phenomenon of dislocation bands' intersection was sporadically observed.

The deformation of copper at a higher rate of the process, 0.6 mm/s, with maintaining the other parameters (f = 0.4 Hz, α = 6°), is accompanied by the formation of a cellular structure, banded structure, dislocation bands' intersections (Figure 8), as well as recrystallization processes with subsequent recovery during another deformation.

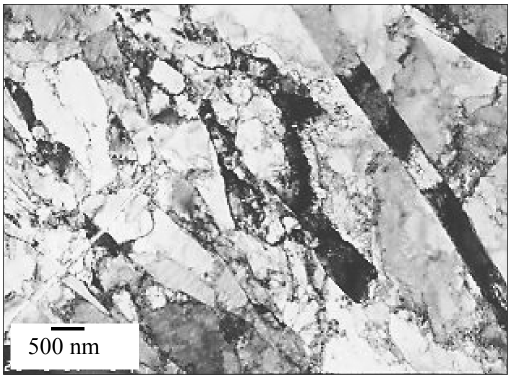

Fig. 6. Cu substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6^\circ$, v = 0.15 mm/s. Dislocation cells are visible between elongated systems of dislocation boundaries

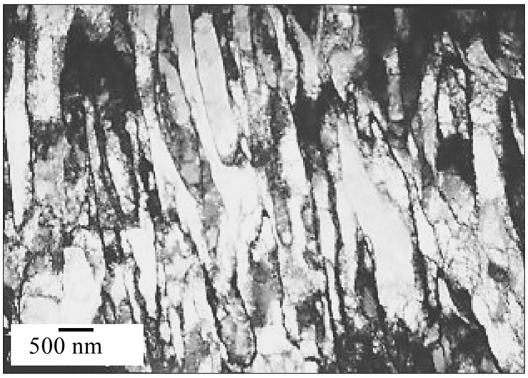

Fig. 7. Cu substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6^\circ$, v = 0.15 mm/s

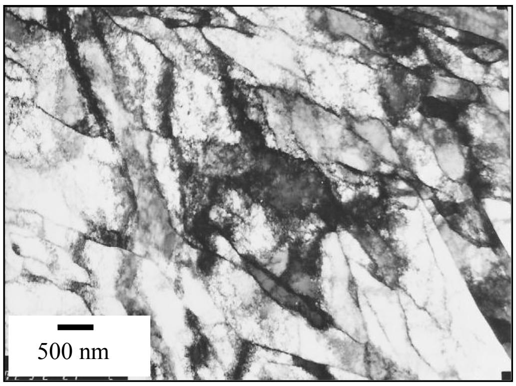

Fig. 8. Substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6^\circ$ and v = 0.6 mm/s. Dislocation bands' intersections

An increasing torsion frequency intensifies the formation of microbands which differ from one another with mutual disorientation. Most frequently, microbands were formed in the areas of cellular dislocation structures (Figure 9).

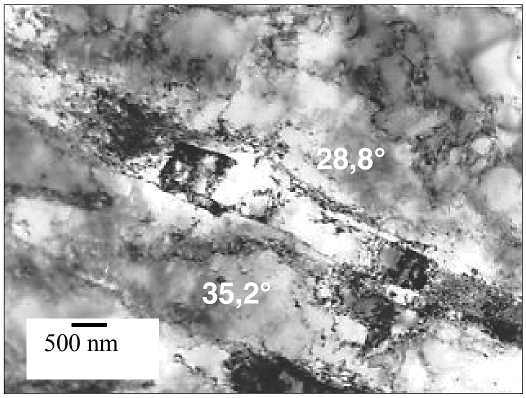

Fig. 9. Substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6^\circ$ and v = 0.6 mm/s. Microbands were formed in the areas of cellular dislocation structures

The image of the banded dislocation structure was different than that of typical long bands characteristic of conventional compression. Systems built in major part from well-shaped, elongated subgrain prevailed.

The microbands were detected in the vicinity of dislocation cells. The presence of shear microbands testifies to inhomogeneity of the dislocation structure observed in samples subjected to oscillatory compression at f = 1.6 Hz, $\alpha = 6^\circ$ and v 0.6 mm/s. The microbands were detected in the vicinity of dislocation cells (Figure 10).

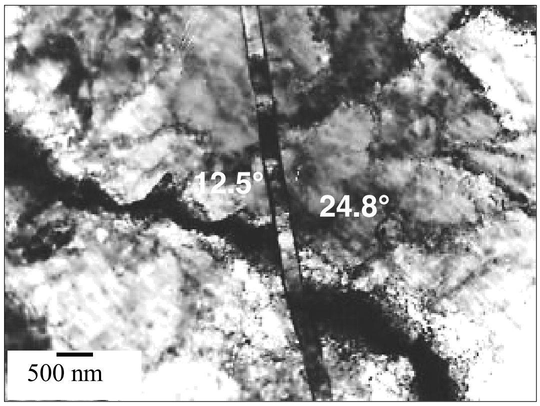

Fig. 10. Substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6^\circ$ and v = 0.6 mm/s. Microbands were formed in the areas of cellular dislocation structures

In other regions of copper subject to strain at f = 1.6 Hz, $\alpha = 6^\circ$ and v = 0.6 mm/s, numerous shear bands were observed (Figure 11). The width of the bands was in the range: from ca.

200 nm to 8.5μm. Fine grain structures, often elongated were formed inside the shear bands formed (Figures 11a, b).

The presence of shear microbands testifies to inhomogeneity of the dislocation structure observed in samples subjected to oscillatory compression at f = 1.6 Hz, α = 6° and v = 0.6 mm/s (Figure 10). The microbands were detected in the vicinity of dislocation cells. A banded structure was also characteristic for such deformation parameters.

Based on diffraction investigations, it was proved that the structures being formed were separated from one another with wide angular boundaries (Figures 11b, c).

In samples deformed at the parameters: f = 0.4 Hz, α = 6°and v = 0.6 mm/s, recovery and recrystallization processes were observed (Figures 12 and 13).

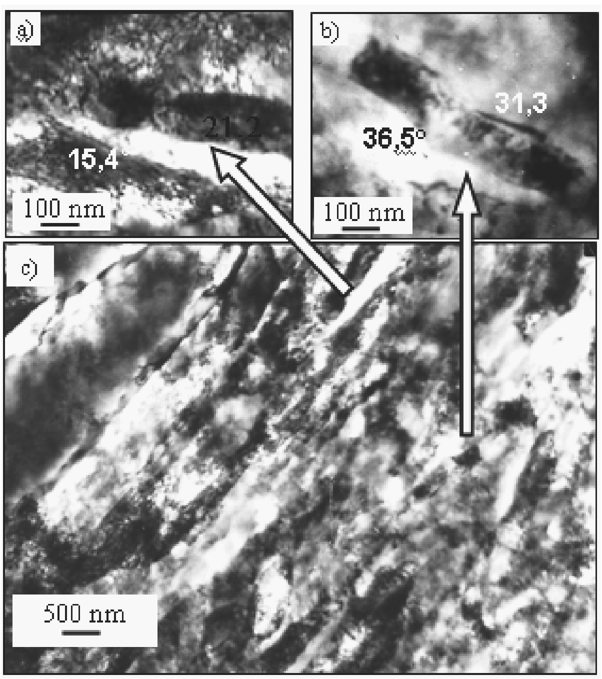

Fig. 11. Cu microstructure after oscillatory compression at the following parameters: f = 1.6 Hz, α = 6° and v = 0.6 mm/s. The structure inside wide shear bands is composed of crystallites differing with respect to high crystallographic disorientation

The measurement results of the mean diameter of dislocation cells and bands width are presented in Figures 14, 15. The largest mean diameter of dislocation cells and dislocation bands' width were shown by Cu samples deformed when deformed to compression with oscillatory torsion at the parameters: f = 1.6 Hz, α = 6°and v = 0.15 mm/s.

The lowest values of the mean diameter of dislocation cells and dislocation bands width were noted for samples subject to conventional compression and for samples subject to oscillatory compression at the parameters: f = 0.4 Hz, α = 6°and v = 0.15 mm/s.

Comparable measurement results of the analyzed geometric features were obtained for Cu samples deformed with parameter: f=1.6 Hz, α = 6°, v = 0.6 mm/s and f = 0.4 Hz, α = 6°, v = 0.6 mm/s.

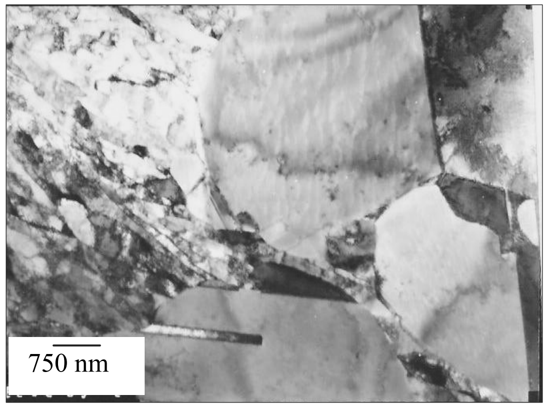

Fig. 12. Substructure after oscillatory compression at the following parameters: f = 0.4 Hz, α = 6°and v = 0.6 mm/s. Recovery and recrystallization processes

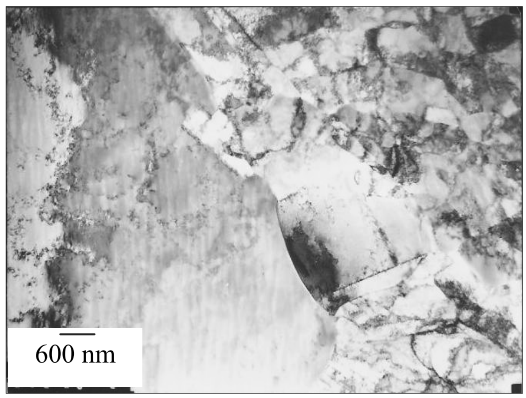

Fig. 13. Substructure after oscillatory compression at the following parameters: : f = 0.4 Hz, α = 6°and v = 0.6 mm/s. Recovery and recrystallization processes

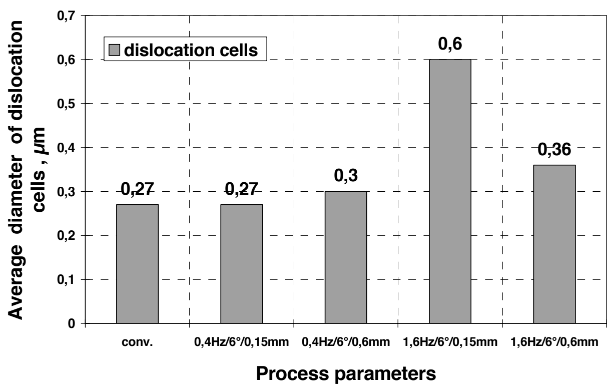

Fig. 14. Diagram of the mean diameter of dislocation cells for Cu samples deformed under oscillatory compression at different process parameters

As results from the measurements of width of the fine-grain structure (crystallites) for samples subject to oscillatory compression at the parameters: f=1.5 Hz, $\alpha = 6°$, v = 0.6 mm/s and f = 1.6 Hz, $\alpha = 6°$, v = 0.16 mm/s being formed inside shear bands, as in Figure 16.

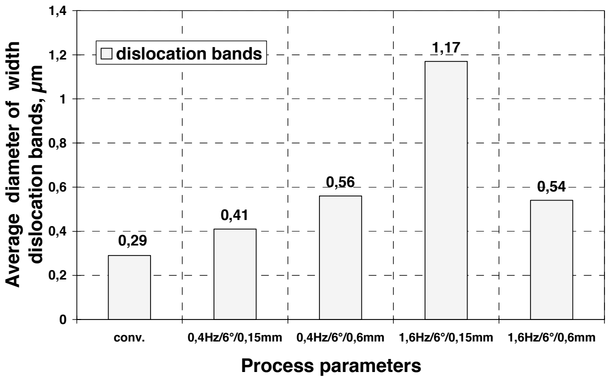

Fig. 15. Diagram of the mean diameter of bands' width for Cu samples deformed under oscillatory compression at different process parameters

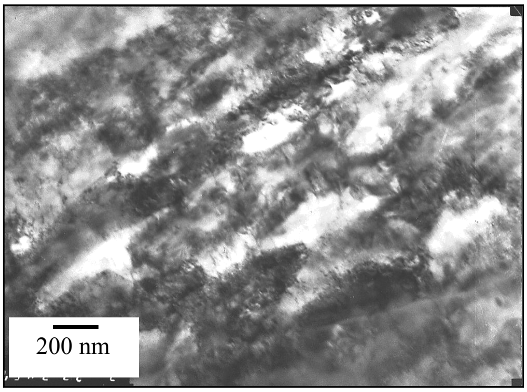

Fig. 16. Substructure after oscillatory compression at the following parameters: f = 0.4 Hz, $\alpha = 6°$and v = 0.6 mm/s

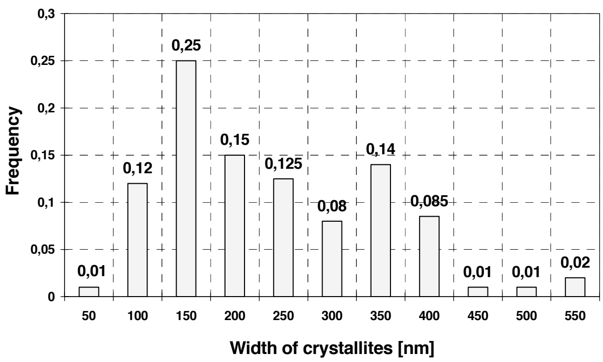

Fig. 17. Bar chart of Cu crystallites' width after oscillatory compression at the following parameters: f = 1.6 Hz, $\alpha = 6°$ and v = 0.6 mm/s

The grain structures have a mean width of 227 nm, with the highest occurrence frequency noted for crystallites of 150 nm in width (0.25) for samples deformed at at the parameters: f=1.6 Hz, $\alpha = 6°$, v = 0.6 mm/s (Figure 17). For samples deformed at at the parameters: f=1.6 Hz, $\alpha = 6°$, v=0.15 mm/s the grain structures have a mean width of 220 nm, with the highest occurrence frequency noted for crystallites of 150 nm - 0.22 (Figure 18).

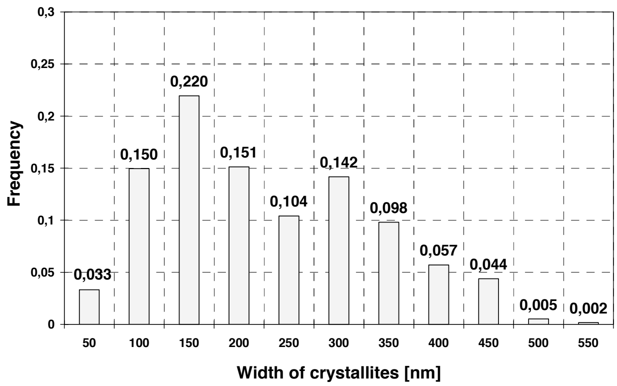

Fig. 18. Bar chart of Cu crystallites' width after oscillatory compression at the following parameters: f = 1.6 Hz, $\alpha = 6°$ and v = 0.16 mm/s

Table 3.
Microhardeness of cooper samples after deformation process

Torsion frequency f_{tor} [Hz]	Torsion angle ± α [°]	Compression rate v_t [mm/s]	Microhardness μHV
0	0	0.6	180
0.4	6	0.15	205
0.4	6	0.6	196
1.8	6	0.15	210
1.8	6	0.6	198

Average microhardness of samples after conventional compression was 180μHV, and after compression with oscillatory torsion the samples were characterised by average hardness from 196 to 210 μHV (Table 3).

4. Conclusions

The examinations carried out have made it possible to observe an intensive influence of the change of the deformation path on the power related parameter and the structure of the examined material. Compression with oscillatory torsion leads to a considerable decrease of plastic flow resistance of the metal.

The course of average unit stresses obtained in the experimental investigations has shown that the course of the deformation path has a influence on their values. A corroboration of such influence is the value of unit pressures obtained for deformation processes characterized by parameters: f= 1.6 Hz and v_t = 0.6 mm/s, and f = 0.4 Hz and v = 0.15 mm/s.

There are some differences in the process of Cu structure changes during compression with oscillatory torsion and during normal compression.

The differences refer to the mechanism of plastic flow, which changes from a multi-system one, typical of conventional deformation, to a mechanism of plastic flow located in shear bands in the case of oscillatory compression .

As results from the measurements of width of the fine-grain structure (crystallites) being formed inside shear bands the grain structures have a mean width of 227 nm, with the highest occurrence frequency noted for crystallites of 150 nm in width.

The number of microbands and their sizes (width) are determined by the parameters of the oscillatory compression process. The typical image of cellular dislocation structures and next, banded structures with a considerable concentration of dislocation bands observed for the samples subject to conventional deformation, changes during the oscillatory compression process applied.

The dislocation cells occupy a relatively large area in the investigated microregions, with their diameters clearly growing as the strain frequency increases from 0.4 Hz to 1.6 Hz. The frequency of narrow dislocation bands' occurrence falls with the strain frequency increase.

The dislocation bands, distinct for samples subjected to conventional deformation, gradually pass into systems of elongated subgrain as the strain frequency increases. No process of lamellar structure formation is observed, which would be the effect of dislocation bands concentration, as in the case of conventional deformation.

Another structural process which distinguishes oscillatory compression from conventional compression is the formation of shear bands. A significant development of shear bands is observed at the parameters: f = 1.6 Hz, $\alpha = 6°$ and v=0.6 mm/s.
The application of lower strain rates and lower frequency initiates the processes of inhomogeneous deformation. Higher hardness of samples after the combined process is also observed when compared with samples compressed in a conventional way.

The initiation of recrystallization processes in the investigated samples should be attributed to local gradients of reinforcement and temperature. The bands transfer considerable strain and show a reinforcement gradient and therefore, they may constitute the place of new grain nucleation. In order to stop the structure reconstruction during compression with oscillatory torsion, intensive heat abstraction should be applied.

The data obtained indicate the possibility of producing ultrafine materials from copper using the method of compression with oscillatory torsion.

Acknowledgements

This work was supported by the Ministry of Education and Science of Poland under grant No. PBZ 096/T08/2003.

References

[1] I.V. Alexandrov, R.Z. Valiev, Nanostructures from severe plastic deformation and mechanisms of large-strain work hardening, Nanonstructured Materials 12 (1999) 709-712.

[2] B.S. Altan, G. Purcek, I. Miskioglu, An upper-bound analysis for equal-channel nagular extrusion, Journal of Materials Processing Technology 168 (2005) 137-146.

[3] A. Belyakov, R. Kaibyshev, Structural changes of ferritic stainless steel during severe plastic deformation, Nanostructured Materials 6 (1995) 893-896.

[4] W. Bochniak, A. Korbel, Type Forming: forging of metals under complex conditions of the process, Journal of Materials Processing Technology 134 (2003) 120-134.

[5] W. Bochniak, K. Marszowski, A. Korbel, Theoretical and practical aspects of the production of thin-walled tubes by the KOBO method, Journal of Materials Processing Technology 169 (2005) 44-53.

[6] U. Chakkingal, P.F. Thomson, Development of micro-structure and texture during high temperature equel channel angular extrusion of aluminium, Journal of Materials Processing Technology 117 (2001) 169-177.

[7] Z.H. Chen, L.C. Chan, T.C. Lee, C.Y. Tang, An investigation on the formation and propagation of shear band in fine-blanking process, Journal of Materials Processing Technology 138 (2003) 610-614.

[8] S.V. Dobatkin, A.M. Arsenkin, M.A. Popov, Production of bulk metallic nano- and submicrocrytalline materials by the method of severe plastic deformation, Metal Science and Heat Treatment 47 (2005) 188-192.

[9] D. Ferguson, W. Chen, R. Kuziak, S. Zając, New developments in the field of physical simulation of thermomechanical processing, Proceedings of the 5th Scientific International Conference "Materials Forming", Kraków, 2002, 599-602.

[10] F. Grosman, J. Pawlicki, Concepts of technological applications in controlled deformation of materials, Acta Metallurgica Slovaca R 8/1 (2002) 178-182.

[11] F. Grosman, J. Pawlicki, Method of testing materials sensitivity on the strain path changes, Proceedings of the 10th Scientific International Conference "Plasticity of Materials", Podlesice, 2003 (CD-ROM).

[12] K. Hyoung Seop, Finite element analysis of high pressure torsion processing, Journal of Materials Processing Technology 113 (2001) 617-621.

[13] K. Hyoung Seop, S. Min Hong, H. Sun Ig, Plastic deformation analysis of metals during equal channel angular pressing, Journal of Materials Processing Technology 113 (2001) 622-626.

[14] K. Hyoung Seop, H. Sun Ig, L. Young Shin, A. Dubravina, I. Alexander, Deformation behaviour of copper during a high pressure torsion process, Journal of Materials Processing Technology 2 (2003) 334-337.

[15] O.A. Kaibyshev, Grain refinement in commercial alloys due to high plastic deformations, Journal of Materials Processing Technology 117 (2001) 300-306.

[16] K.J. Kurzydlowski, M. Richert, On the mechanisms of nanograins formation in cold–plastic deformation conditions, Materials Science and Engineering 4 (2005) 189-191.

[17] Y. Lee, B.M. Kim, K.J. Park, S.W. Seo, O. Min, A study for the constitutive equation of carbon steel subjected to large strains, high temperatures and high strain rates, Journal of Materials Processing Technology 130-131 (2002) 181-188.
[18] C.J. Luis Pérez, P. Gonzáles, Y. Garcés, Equel channel angular extrusion in a commercial Al-Mn alloy, Journal of Materials Processing Technology 143-144 (2003) 506-511.
[19] C.J. Luis Pérez, R. Luri-Irigoyen, D. Gaston-Ochoa, Finite element modelling of an Al-Mn alloy by equal-channel angular extrusion, Journal of Materials Processing Technology 153-154 (2004) 846-852.
[20] J. Mao, S.B. Kang, J.O. Park, Grain refinement, thermal stability and tensile properties of 2024 aluminium alloy after equal-channel angular pressing, Journal of Materials Processing Technology 159 (2005) 314-320.
[21] J.P. Mathieu, S. Suwas, A. Eberhardt, L.S. Toth, P. Moll, A new design for equal channel angular extrusion, Journal of Materials Processing Technology 173 (2006) 29-33.
[22] G. Niewielski, D. Kuc, K. Rodak, F. Grosman, J. Pawlicki, Influence of strain on the copper structure under controlled deformation path conditions, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 109-112.
[23] G. Purcek, S.A. Burhanettin, I. Miskogli, H.O. Pey, Processing of eutectic Zn-5%Al alloy by equel-channel angular pressing, Journal of Materials Processing Technology 148 (2004) 279-287.
[24] M. Richert, A. Korbel, The effect of strain localization on mechanical properties of A199,992 in the range of large deformation, Journal of Materials Processing Technology 53 (1995) 331-340.
[25] S. Rusz, H. Dyja, The influence of technological parameters on the rotary pressing process, Journal of Materials Processing Technology 157-158 (2004) 604-608.
[26] G. Salishchev, R. Zaripova, R. Galeev, O. Valiakhmetov, Nanocrystalline structure formation during severe plastic deformation in metals and their deformation behaviour, Nanostructured Materials 6 (1995) 913-916.
[27] V.A. Teplov, V.P. Pilugin, V.S. Gaviko, E.G. Cheryshov, Nanacrystalline structure of non-equilibrium Fe-Cu alloys obteined by severe plastic deformation under pressure, Nanostructured Materials 6 (1995) 437-440.
[28] R Valiev, A. Korznikov, V. Alexander, Nanomaterials produced by severe plastic deformation, Annales de Chimie Science des Materiaux 27 (2002) 1-2.
[29] R.Z. Valiev, R.K. Islamgaliev, Bulk nanostructured materials from severe plastic deformation, Progress in Materials Science 45 (2000) 106-107.
[30] Z.F. Zhang, S.D. Wu, Y.J. Li, S.M. Liu, Z.G. Wang, Cyclic deformation and fatique properties of Al-0.6wt.%Cu alloy produced by equal channel angular pressing, Materials Science and Engineering A 412 (2005) 279-286.
[31] L.J. Zheng, H.X. Li, M.F. Hashmi, C.Q. Chen, Y. Zhang, M.G. Zeng, Evolution of microstructure and strengthening of 7050 Al alloy by ECAP combined with heat-treatment, Journal of Materials Processing Technology 171 (2006) 100-107.
[32] A.P. Zhilyaev, Experimental parameters influencing grain refinement and microstructural evolutin during high - pressure torsion, Acta Materialia 51 (2003) 753-765.
[33] G. Niewielski, D. Kuc, K. Rodak, F. Grosman, J. Pawlicki, Influence of strain on the copper structure under controlled deformation path conditions, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 109-112.