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Studies of Cu after severe plastic deformation

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Abstract

The aim of the present study is to examine how severe plastic deformation techniques: compression with oscillatory torsion and multi-axial compression, alter the microstructure and properties of metal, and what is the efficiency of the mentioned methods. For this reason the deformed microstructure of Cu was characterized quantitatively by use of electron backscattered diffraction and transmission electron microscopy techniques. The mechanical properties were determined using an MTS QTest/10 machine equipped with digital image correlation. The results show that severe plastic deformation through compression with oscillatory torsion and multi-axial compression leads to a refinement of the Cu to ultra-fine scale. The final structure of samples after compression with oscillatory torsion consists of cellular/subgrain structure with a low value of misorientation. The multi-axial compression technique is effective in generating high angle boundaries. The samples after compression with oscillatory torsion exhibit a higher strength compared with multi-axial compression.

Key words: severe plastic deformation, copper, fine-grained microstructure, EBSD, STEM

1. Introduction

The consequence of severe plastic deformation (SPD) is the grain fragmentation of the material to an ultrafine or even nanograined dimension. In SPD structures the refined grains can be regarded rather as ultrafine-grained where the crystallite size is in range 100 nm and 1000 nm. There is current interest in processing materials using new SPD techniques and in making use of SPD to achieve substantial grain refinement. As a consequence of this task, it has become important to characterize the microstructure-properties behaviour of materials at high strains. The use of SPD processing techniques, such as equal-channel angular pressing (ECAP) [1, 2], high-pressure torsion (HPT) [3, 4] and another [5, 6] leads to a great refinement of the microstructure and provides attractive mechanical properties.

The aim of present work is to investigate the fine grain structure formation in polycrystalline Cu during compression with an oscillatory torsion test and to compare these results with results received at multi-axis compression. The multi-axis compression is known in the literature as a technique to impose cyclic compression in two orthogonal directions [7] and is capable of refining the grain size in IF steel to about 300 nm [8]. Compression with oscillatory torsion is a deformation procedure applied to achieve large strains [9, 10] and is recognized as a method for forming ultrafine-grained materials [11, 12].

The efficiency of the compression with the oscillatory torsion method was compared with the multi-axial compression. The experimental procedures were performed using the same method of investigation, with a comparable value of deformation and on the same material. It is in accordance with suggestion in [13], that the different conclusions on the structure character may be attributed to different techniques used by different investigators.

2. Experiment

Investigations were performed using compression

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with oscillatory torsion and multi-axial compression techniques for refining polycrystalline Cu structure. The plastic deformation by compression with oscillatory torsion was performed for two effective strain levels, $\varepsilon_\mathrm{f} = 6.6$ and $\varepsilon_\mathrm{f} = 13.2$. The changes following the parameters as concerns: torsion frequency f(Hz), speed compression $v\,(\mathrm{mm\,s^{-1}})$, torsion angle α (°), and true reduction ε_h (mm) are responsible for the value of strain. In our experiment the mentioned values of deformation were obtained for:

(i) $f = 0.8$ Hz, $v = 0.04\ \mathrm{mm\,s^{-1}}$, $\alpha = 6°$, $\varepsilon_\mathrm{h} = 3$ mm; the obtained effective strain was $\varepsilon_\mathrm{f} = 6.6$,

(ii) $f = 1.6$ Hz, $v = 0.08\ \mathrm{mm\,s^{-1}}$, $\alpha = 6°$, $\varepsilon_\mathrm{h} = 3$ mm; the obtained effective strain was $\varepsilon_\mathrm{f} = 13.2$.

One can find more information about compression with oscillatory torsion in [10].

The plastic deformation by multi-axial compression was realized for two effective strain levels: $\varepsilon_\mathrm{f} = 5.4$ and $\varepsilon_\mathrm{f} = 14.9$. The average strain in a single pass was 0.2. The samples were deformed with a strain rate of $0.5\ \mathrm{s^{-1}}$. Commercial Cu (M1E) was used for these experiments. The material was homogenized at 500 ℃ for 2 h and then cooled slowly down to obtain a grain size of about 50 μm.

For microstructure investigation an FEI INSPECT F SEM microscope with a field emission type gun, equipped with EBSD facility, and a Hitachy HD-2300A STEM microscope were employed. The evolution of misorientation distribution and crystallite size in deformed samples was observed using the electron backscattered diffraction (EBSD) technique, which is the only possible tool for analysis of relatively large areas [14]. Compared to the scanning transmission electron microscopy (STEM) based method, the field emission gun scanning electron microscope (FEG-SEM/EBSD) technique has the important advantage that large amounts of quantitative data over far larger areas are required to fully characterize the grain refinement processes. Orientation maps from EBSD analysis were acquired with a step size of 50 nm. For the microstructure characterization individual areas of 14 μm × 36 μm were studied.

A substantial strain accumulation during multi--axis compression occurs in the centre of the sample causing a significant grain refinement. For this reason, the multi-axially compressed specimens were prepared from the central part of the sample's deformed volume. For this purpose, the samples for SEM and STEM structure analyses were cut perpendicularly to the long axis direction. The structure studies using STEM and SEM were conducted on samples extracted from the 0.8 radius in the longitudinal plane section.

For the STEM examinations, foils were prepared by electropolishing using D2 electrolyte. For the SEM observations, specimens were polished by using the ion thinning device (PECS) manufactured by Gatan Inc. The mechanical properties: yield stress (YS), ultimate tensile strength (UTS), and uniform elongation (EL) were determined on an MTS QTest/10 machine equipped with digital image correlation (DIC) [15].

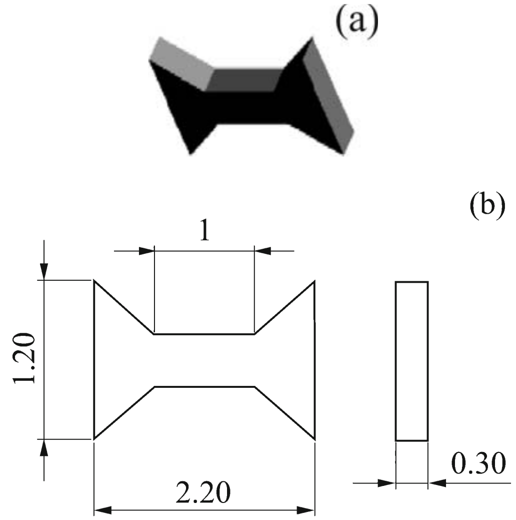

Fig. 1. Mini tensile test sample: (a) view of sample, (b) sample geometry.

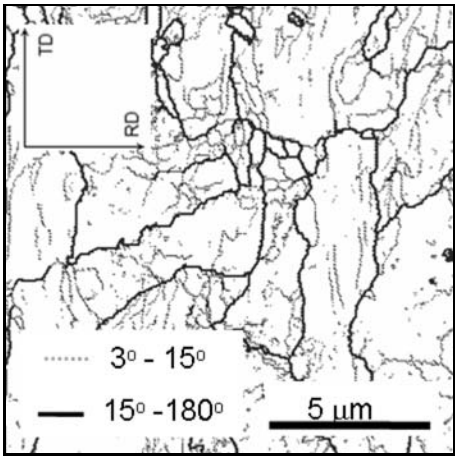

Fig. 2. EBSD map of Cu after compression with oscillatory torsion at $\varepsilon_\mathrm{f} = 6.6$.

Due to the small volume of materials available for testing, one way of measuring mechanical properties was then to use mini tensile test samples. In this work small size samples with a total length of 2.2 mm were used (Fig. 1).

3. Results

The changes in the microstructure after processing by compression with oscillatory torsion and multi-axial compression are shown in SEM/EBSD patterns (Figs. 2–5). The results of the investigation show that at the beginning of the deformation, a fragmenta-

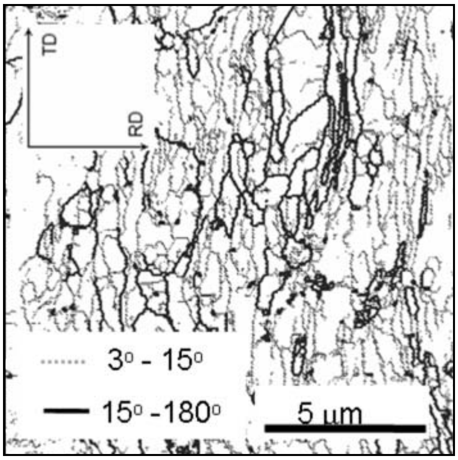

Fig. 3. EBSD map of Cu after compression with oscillatory torsion at $\varepsilon_\mathrm{f} = 13.2$.

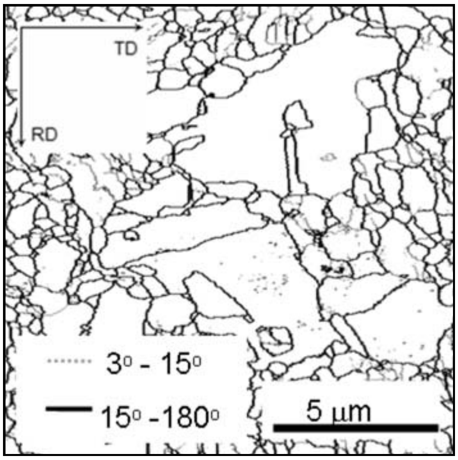

Fig. 4. EBSD map of Cu after multi-axial compression at $\varepsilon_\mathrm{f} = 5.4$.

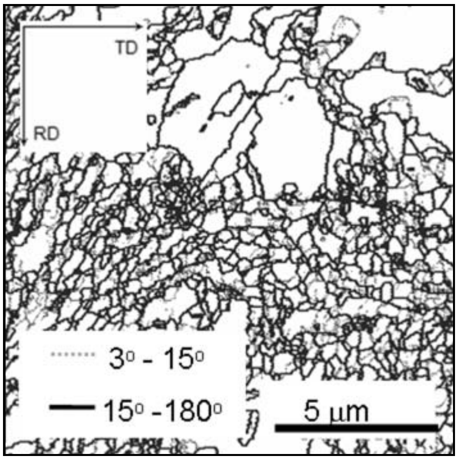

Fig. 5. EBSD map of Cu after multi-axial compression at $\varepsilon_\mathrm{f} = 14.9$.

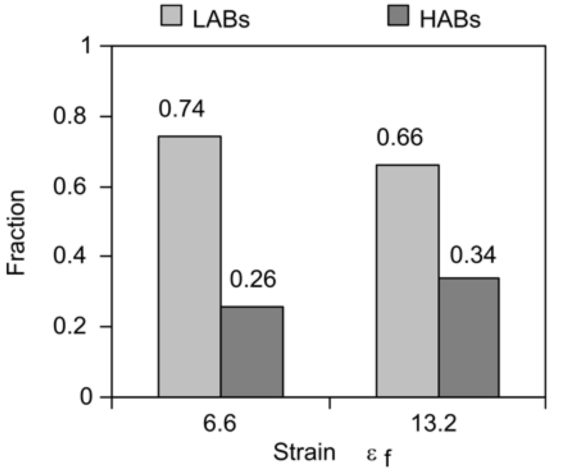

Fig. 6. Influence of deformation on misorientation value in Cu after compression with oscillatory torsion.

tion of the original grains occurs. The microstructures processed by compression with oscillatory torsion are rather inhomogeneous and during further deformation the homogeneity of the microstructure is increased (Figs. 2, 3). After initial deformation, most of the boundaries between neighbouring subareas are classified as low angle. With further strain accumulation, the misorientation angle slowly shifts towards a high value.

In samples processed by multi-axial compression, the fragmentation process carried out seems to be faster and more effective than the compression with oscillatory torsion (Fig. 4). However, the microstructure seems to be inhomogeneous during subsequent deformation (Fig. 5). The coarse equiaxed structures are characteristic for recrystallized grains. This means that the dynamic recrystallization process causes obvious grain growth. The dynamic recrystallization process was also confirmed by STEM examinations.

A detailed quantitative analysis using the EBSD technique reveals a clear difference between the deformed structures resulting from compression with oscillatory torsion and multi-axis compression processing. For both methods of deformation, the boundary misorientation angles were measured and the results are presented in Figs. 6 and 7. The fraction of low-angle boundaries (LABs) smaller than 5°, in samples deformed by compression with oscillatory torsion, changes from about ~ 75 % to ~ 65 % for $\varepsilon_\mathrm{f} = 6.6$ and $\varepsilon_\mathrm{f} = 13.2$, respectively.

The samples deformed by multi-axial compression show a quite different distribution of boundaries misorientation. The samples have a large fraction of the high-angle boundaries. The fractions of HABs are ~ 70 % and ~ 80 % for $\varepsilon_\mathrm{f} = 5.4$ and $\varepsilon_\mathrm{f} = 14.9$, respectively. In terms of HABs production, the multiaxial compression is far more effective than compres-

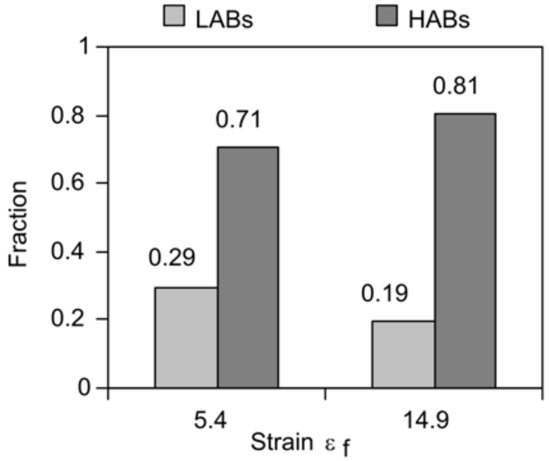

Fig. 7. Influence of deformation on misorientation value in Cu after multi-axial compression.

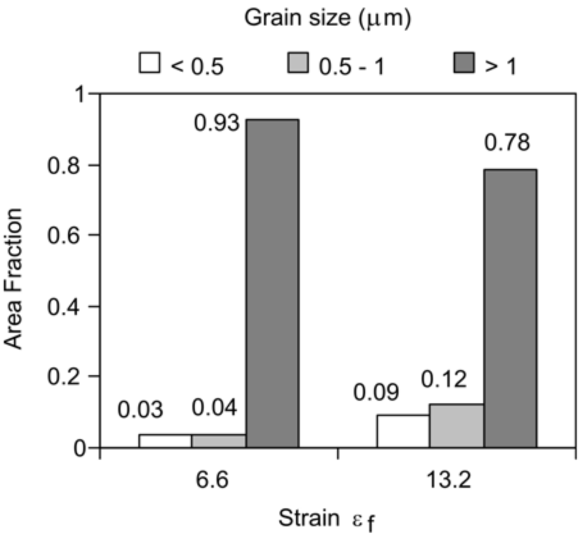

Fig. 8. Influence of deformation on area fraction of grains after compression with oscillatory torsion.

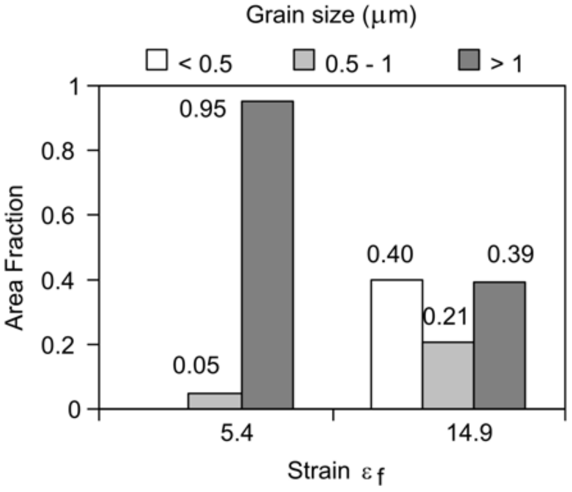

Fig. 9. Influence of deformation on area fraction of grains after multi-axial compression.

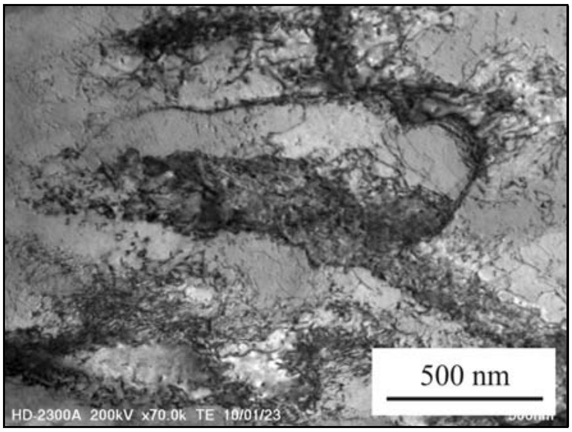

Fig. 10. Microstructure of Cu after compression with oscillatory torsion at $\varepsilon_f = 6.6$.

sion with oscillatory torsion (about 35 % HABs for compression with oscillatory torsion and about 80 % for multi-axial compression).

Histograms presented in Figs. 8 and 9 show the distribution of the area fraction of grains after compression with oscillatory torsion and multi-axial compression. Three types of grains were detected: ultrafine ~ 0.5 μm, fine ~ 1 μm, and above 1 μm. It is essential to clarify that the grain boundaries' misorientations presented in Figs. 8 and 9 are only HABs. The area fractions of ultrafine ~ 0.5 μm and fine ~ 1 μm grain detected by the EBSD technique for compression with oscillatory torsion samples are about 10 % and 20 % for $\varepsilon_f = 6.6$ and $\varepsilon_f = 13.2$, respectively. The area fraction of ~ 0.5 μm and ~ 1 μm grains in multi-axial compression evidently changes with deformation, and at $\varepsilon_f = 14.9$ it reached about 60 %.

The compression with oscillatory torsion results in the development of characteristic cellular/subgrain structure (Figs. 10, 11). Figure 10 shows the microstructure consisting of cells with a high dislocation density. Figure 11 presents a region of mixture coarser sub(grains), where some of sub(grains) showed reduced dislocation density when compared to Fig. 10. The change in the deformation parameter led to the transformation of cell boundaries into sub(grains), but a significant fraction of low-angle grain boundaries persists after reaching high effective strain of $\varepsilon_f = 13.2$.

The microstructure of multi-axially compressed samples after effective strains of $\varepsilon_f = 5.4$ and $\varepsilon_f = 14.9$ are shown in Figs. 12 and 13, respectively. The STEM

Table 1. Average values of structural parameters: grain size (D), subgrain size (d), and mechanical properties: yield stress (YS), ultimate tensile strength (UTS), uniform elongation (EL) of severely deformed Cu

Method	Effective strain ε_{f}	d (μm)	D (μm)	UTS (MPa)	YS (MPa)	EL (%)
Initial state			50	228	149	34.9
Compression with oscillatory torsion	6.6	–	0.93	326	276	15.6
	13.2	0.28	0.73	–	–	–
Multi-axial compression	5.4	0.21	0.47	258	139	45.5
	14.9	0.25	0.33	–	–	–

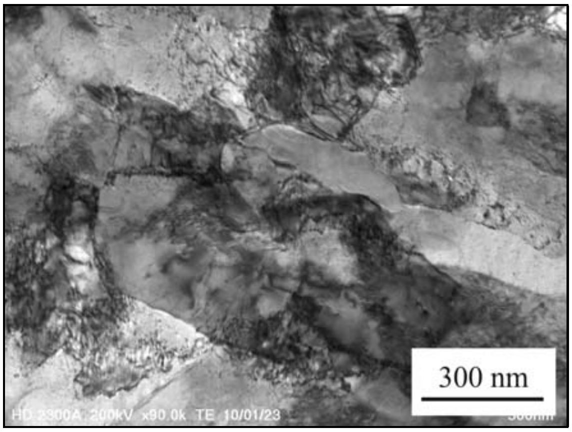

Fig. 11. Microstructure of Cu after compression with oscillatory torsion at $\varepsilon_{\mathrm{f}} = 13.2$.

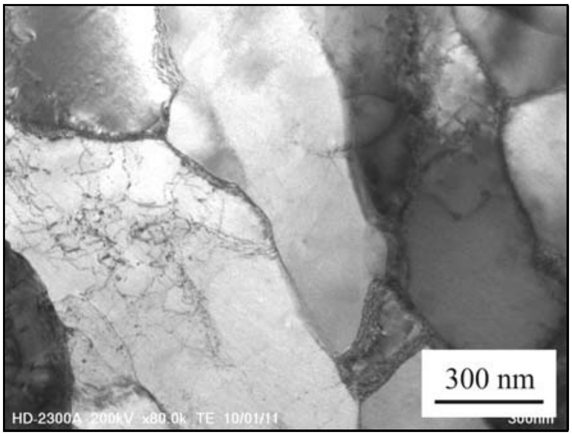

Fig. 12. Microstructure of Cu after multi-axial compression at $\varepsilon_{\mathrm{f}} = 5.4$.

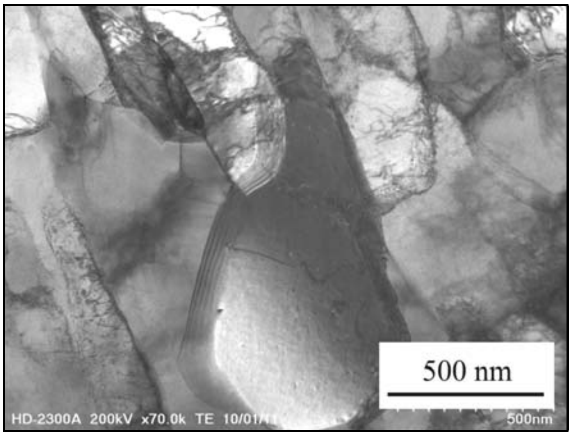

Fig. 13. Microstructure of Cu after multi-axial compression at $\varepsilon_{\mathrm{f}} = 14.9$.

microstructures of multi-axially compressed samples show an approximately equiaxed sub(grain) structure with low dislocation density and with a more random misorientation of individual subgrains and grains than does the microstructure taken from compression with oscillatory torsion samples (Fig. 12). This highly deformed structure confirms that there are new grains with well resolved angle boundaries.

There is a larger fraction of high-angle grain boundaries and a larger progress in recovery after straining than after compression with oscillatory torsion. The 'sharp' boundaries of greater grain size refer to already equilibrium grain boundaries. Recrystallized grain interiors appear to be free of dislocation (Fig. 13).

The microstructural parameters and mechanical properties for both methods of deformation are summarized in Table 1.

The samples after compression with oscillatory torsion at $\varepsilon_{\mathrm{f}} = 6.6$ exhibit a high strength (UTS: 326 MPa; YS: 276 MPa) and a moderate elongation (EL: 15.6 %). An evident decrease in strength (UTS: 258 MPa; YS: 139 MPa) and increase in elongation (EL: 45.5 %) take place in the samples after multi-axial compression. For this mode of deformation the grains can be refined effectively; the average grain size (D) and subgrain size (d) for $\varepsilon_{\mathrm{f}} = 5.4$ reach 0.47 μm and 0.21 μm, respectively. The subsequent deformation to $\varepsilon_{\mathrm{f}} = 14.9$ slowly reduced the subgrain size. Comparison of the microstructures between the mentioned methods of deformation clearly shows that dynamic recovery and recrystallization (DRX) play an important role in multi-axial compression process. The strain rate of $0.5\,\mathrm{s}^{-1}$ during multi-axial compression leads to a larger temperature increment, which contributes to DRX during deformation.

4. Discussion

The results show that multi-axial compression and compression with oscillatory torsion processing result in ultra-fine grained structure, but the deformed microstructures produced by the mentioned methods are quite different. The microstructure after compression with oscillatory torsion contains subgrains simultaneously. Meanwhile microstructures after multi-axial compression have high-angle grain boundaries mainly. The HABs fraction in multi-axially compressed samples reaches 80 %, which is much higher than that in compression with oscillatory torsion samples: 35 %. Obtaining UFG structures with high-angle grain boundaries is a rather difficult task and depends on many factors such as strain, strain rate, temperature, applied deformation techniques, etc. To support this, an interesting conclusion has been drawn in reports where different methods of SPD were used. Sun et al. [16] reported that the ECAE process was more effective than cyclic extrusion compression (CEC) in generating HABS, that was, the ECAE route Bc with a strain of ~ 8 showed similar results to the CEC process with a strain of 60.

Despite the fact that multi-axial compression is an attractive process because it leads to a greater refinement of the microstructure and to a higher presence of high-angle boundaries than compression with oscillatory torsion, is less effective in strengthening. The changes in the microstructure of Cu after the multi-axial compression process are controlled by the dynamic recrystallization (DR) process. From the present results, it is concluded that (DR) plays an important role in the process.

Our investigations are in accordance with the results depicted in [17]. Dalla Torre and co-workers mentioned that the copper hardness was not related to the true strain and the size of elements that form the structure, and the maximal hardness was reached with a relatively small deformation, which did not give rise to dynamic recrystallization. Mishra and co-workers [18] examined the structure and properties of Cu samples after the ECAP process. These authors explained that the two-pass sample showed a significant jump in strength over the initial sample, and the rise in strength with a higher number of passes was not as significant. It was found that the highest strength is observed after the first or the second pass. This is suggestive of the fact that for two passes, the fraction of boundaries that has a low angle increases significantly, while the fraction of large angle boundaries becomes small. The additional passes simply increased the fraction of grains of ~ 200 nm in size, and the low-angle grain boundaries are a minority. In [19] it is mentioned that after twist hydroextrusion (THE) of Cu, the principal changes in structure and properties are observed for $\varepsilon = 2.7$, and with increasing total deformation the properties of Cu change non-monotonously.

In conformity with data from the literature it can be presumed that for samples deformed by compression with oscillatory torsion, the strain hardening or dislocation strengthening play a main role in the strength increase, and the formation of submicron subgrains or dislocation cells also concur to the strength substantially. The strengthening in multi-axial compressed samples may be attributed to grain refinement hardening, but low strengthening is related to the fast course of dynamic recrystallization process.

5. Conclusion

1. The subgrain/grain size during multi-axial compression and compression with oscillatory torsion processing reaches an ultrafine dimension.

2. The microstructure after compression with oscillatory torsion contains dislocation cells or subgrain boundaries simultaneously. Meanwhile, in microstructure after multi-axial compression there dominate high-angle grain boundaries. It is established that the multi-axial compression method is more effective than compression with oscillatory torsion processing in generating HABs.

3. Deformation of Cu by the compression with oscillatory torsion method leads to an important improvement in strength properties. The increase of the flow stress is due to strain hardening.

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References

[1] PRANGNELL, P. B.—BOWEN, J. R.—APPS, P. J.: Mat. Sci. Eng., *A 375–377*, 2004, p. 178. doi:10.1016/j.msea.2003.10.170

[2] SHIH, M. H.—YU, C. Y.—CAO, P. W.—CHANG, C. P.: Scripta Mater., *45*, 2001, p. 793. doi:10.1016/S1359-6462(01)01098-3

[3] RENTENBERGER, C.—MANGLER, C.—KARNTHALER, H. P.: Mat. Sci. Eng., *A 387–387*, 2004, p. 795.

[4] SABIROV, I.—PIPPAN, R.: Scripta Mater., *52*, 2005, p. 1293. doi:10.1016/j.scriptamat.2005.02.017

[5] RICHERT, M.: Mat. Sci. Eng., *A 129*, 1990, p. 1.

[6] TSUJI, N.—ITO, Y.—SAITO, Y.—MINAMINO, Y.: Scripta Mater., *47*, 2002, p. 893. doi:10.1016/S1359-6462(02)00282-8

[7] KUZIAK, R.—ZALECKI, W.—WĘGLARCZYK, S.—PIETRZYK, M.: Solid Phenom., *101–102*, 2005, p. 43. doi:10.4028/www.scientific.net/SSP.101-102.43

[8] PETRYK, H.—STUPKIEWICZ, S.—KUZIAK, R.: J. Mater. Process. Technol., *204*, 2008, p. 255. doi:10.1016/j.jmatprotec.2007.11.068

[9] PAWLICKI, J.—GROSMAN, F.: Arch. Civil Mech. Eng., *4*, 2004, p. 45.

[10] PAWLICKI, J.—GROSMAN, F.: Mechanic, *201*, 2003, p. 139.

[11] RODAK, K.: Arch. Mater. Science, *1*, 2006, p. 29.

[12] RODAK, K.—GORYCZKA, T.: Solid State Phenom., *130*, 2007, p. 111. doi:10.4028/www.scientific.net/SSP.130.111

[13] DEGTYAREV, A. M. V.—CHASHCHUKINA, T. I.—VORONOVA, L. M.—PATSELOV, A. M.—PILYUGIN, V. P.: Acta Materialia, *55*, 2007, p. 6039. doi:10.1016/j.actamat.2007.04.017

[14] PRANGNELL, P. B.—BOWEN, J. R.—GHOLINIA, A.: In: Proceedings 22nd Riso International Symposium on Materials Science. Eds.: Dinesen, A. R., Eldrup, M. D., Jensen, J. Riso National Laboratory, Denmark 2001, p. 105.

[15] CHEVALIER, L.—CALLOCH, S.—HILD, F.—MARCO, Y.: Eur. J. Mech. – A/Solids, *20*, 2001, p. 169. doi:10.1016/S0997-7538(00)01135-9

[16] SUN, P. L.—YU, C. Y.—KAO, P. W.—CHANG, C. P.: Scripta Mater., *457*, 2002, p. 377. doi:10.1016/S1359-6462(02)00117-3

[17] DALLA TORRE, F.—LAPOVOK, R.—SANDLIN, J.—THOMSON, P. F.—DAVIES, C. H. J.—PERELOMA, E. V.: Acta Mater., *52*, 2004, p. 4819.

[18] MISHRA, A.—KAD, B. K.—GREGORI, F.—MEYERS, M. A.: Acta Mater., *55*, 2007, p. 13. doi:10.1016/j.actamat.2006.07.008

[19] VARYUKHIN, V. N.: Mater. Science Forum, *503–504*, 2006, p. 591. doi:10.4028/www.scientific.net/MSF.503-504.591