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J. KUŚNIERZ* , J. DUTKIEWICZ*,**, P. MALCZEWSKI**, M. KUROWSKI*

NANOCRYSTALLINE MATERIALS PREPARED BY TORSION UNDER PRESSURE OF 2 GPa

NANOKRYSTALICZNE MATERIAŁY WYTWARZANE TECHNIKĄ SKRĘCANIA POD CIŚNIENIEM 2 GPa

Improved mechanical properties of materials with ultra-fine grained structure (nanostructure) are very frequently reported and severe plastic deformation is commonly proposed as a method to obtain such materials. High pressure torsion, although it cannot supply big dimension samples, generally leads to the highest degree of grain refinement The paper shows the influence of the phenomenon of intensive shear deformation during high pressure torsion (HPT) processing on the changes in structure, grain size, mechanical properties and solid solubility. The study is performed on copper brasses exemplified by CuZn30, CuZn37 and CuZn29Mn11, severely deformed up to high deformation degree (ε = 18.1 under 2 GPa pressure). The $\alpha+\beta$ brasses are particularly interesting as the deformation of two-phase $\alpha+\beta$ brasses shows the ability to superplastic deformation; additionally the better grain refinement and relation of α/β phase contents influence superplastic deformation.

Keywords: nanomaterials, high pressure torsion, $\alpha+\beta$ brasses

Polepszenie własności mechanicznych w materiałach o ultradrobnokrystalicznej strukturze jest powszechnie zauważane, a metody intensywnego odkształcenia plastycznego są wykorzystywane do wytwarzania takich materiałów. Taką metodą jest skręcanie pod wysokim ciśnieniem (metoda HPT), które chociaż nie dostarcza próbek o znacznych wymiarach jednak prowadzi do największego rozdrobnienia ziarna. W pracy przedstawiono wpływ intensywnego odkształcenia postaciowego wytwarzanego techniką HPT na zmiany mikrostruktury, wielkości ziarna, własności mechanicznych i rozpuszczalności w stanie stałym. Badania prowadzono na mosiądzach: CuZn30, CuZn37 i CuZn29Mn11 intensywnie skręcanych do ε = 18.1 pod ciśnieniem 2 GPa. W szczególności zwrócono uwagę na rozdrobnienie ziarna i wzajemny udział faz α i β, które mają wpływ na zdolność do odkształcania superplastycznego dwufazowych mosiądzów $\alpha+\beta$.

1. Introduction

The improved mechanical properties of bulk materials with ultra-fine grained (UFG) structure (nanostructure), which mostly reported, are manifested by greater strength, hardness and ductility in comparison with conventional coarse-grained materials; also the high-speed superplasticity flow is reported [1, 2]. In metal alloys with UFG structure manufactured by severe plastic deformation (SPD) the formation of metastable states or solid solutions in immiscible systems is observed [1]. Severe plastic deformation is commonly proposed as a method to obtain ultra-fine grained microstructure. Such methods of processing by means of SPD [1–3] like equal-channel angular pressing (ECAP), accumulative roll-bonding (ARB), high pressure torsion (HPT). Equal-channel angular pressing enables obtaining bulk samples of relatively great dimensions and free from porosity in opposition to the methods based on powder metallurgy, rapid cooling or crystallization from gaseous phases. The ARB method, in turn, can supply semi-final products in the form of sheets, which can be directly used for further work forming operation. High pressure torsion, although it cannot supply samples of big dimensions, leads generally to the highest degree of grain refinement.

The main objective of the paper is to present the influence of the phenomenon of intensive shear deformation during high pressure torsion (HPT) processing on the changes in structure, grain size, mechanical properties and solid solubility. The study was performed on copper brasses exemplified by CuZn30, CuZn37 and CuZn29Mn11, severely deformed up to high deformation degree. The $\alpha+\beta$ brasses are particularly interesting as the deformation of two-phase $\alpha+\beta$ brasses shows the ability of elongation up to 1000% (superplastic defor-

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* INSTITUTE OF METALLURGY AND MATERIALS SCIENCE, POLISH ACADEMY OF SCIENCE, 30-059 KRAKOW, 25 REYMONTA STR., POLAND
** PEDAGOGICAL ACADEMY, 30-059 KRAKÓW, PODCHORĄŻYCH STR., POLAND

mation) at the temperature range of 450–600°C; additionally both, better grain refinement and relation of α/β phase contents can influence the ability for superplastic deformation [4–6].

2. High pressure torsion

Among the deformation methods, where the shear deformation is directly introduced as severe plastic deformation, we find torsion under high hydrostatic pressure (method known as HPT – high pressure torsion, Fig. 1).

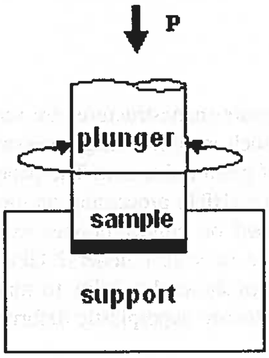

Fig. 1. Scheme of the high pressure torsion

The method of HPT [3] refers to the Bridgman torsion method [7], which was proposed in the year 1935 to study friction coefficient in dependence on pressure and rubbed materials. Admitting a cylindrical sample of 2R diameter and of height h is twisted in-between its cylindrical bases in the arrangement as presented in Fig. 1, then the maximal shear strain γ and the corresponding equivalent deformation ε according to H-v.M-H condition of plasticity [8] after N revolutions will be equal to

$$\gamma = (2\pi RN)/h; \quad \varepsilon = \gamma / \sqrt{3}. \qquad (1)$$

3. Materials and experimental technique

The chemical composition of alloys, hardness and deformation conditions are given in Table. The alloy containing Mn, after casting was homogenized at 973 K, then cut, hot rolled, quenched from the β phase region (1123 K) to room temperature and annealed at various temperatures between 723 and 873 K for 15 minutes [5, 6]. Figure 2 shows the optical microstructure of alloys 1 and 3 in the initial condition (Table). The β phase is located at the grain boundaries. The amounts of the β phase (visible in the dark contrast after etching) and the α phase, were calculated from a metallographic prepared sample according to classical quantitative phase analysis.

HPT test was performed on a sample of $2R$ = 10 mm and of height h = 1mm. X-Ray diffraction (XRD) measurements were performed using Co Kα radiation and Philips PW 1830 diffractometer. Quantitative Phase Analysis by XRD measurement was calculated using the classical method.

The structure was investigated using optical, scanning and transmission electron microscope (TEM) Philips CM20.

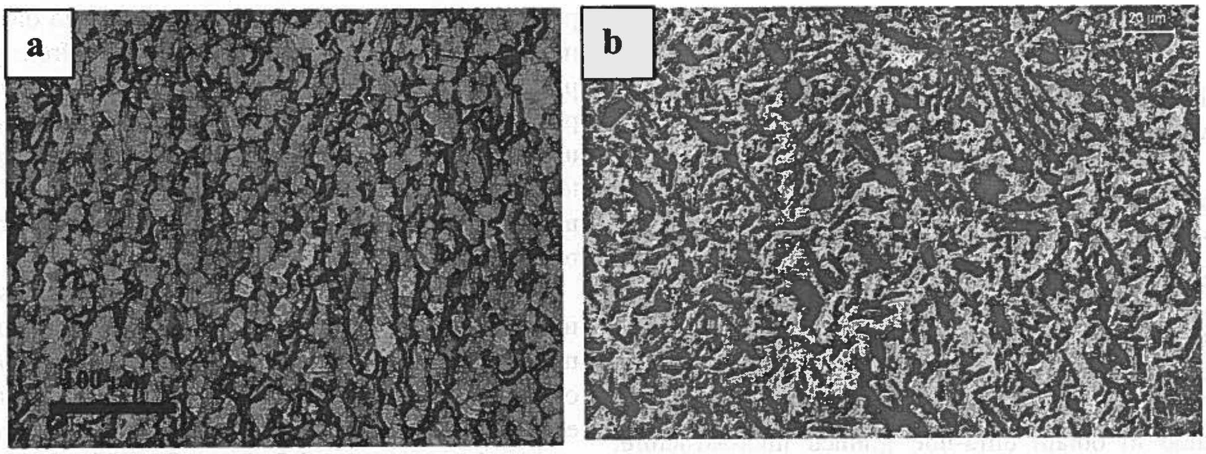

Fig. 2. Optical microstructure of alloys in the initial state: a) CuZn37 (M63 – M0) brass and b) Saturation at 1123 K and annealing at 823 K/15 min CuZn29Mn11 (MN11-N0) brass. β – phase is dark after etching

4. Results and discussion

4.1. Transmission electron microscopy

The high pressure torsion, although it cannot supply big dimension samples, it leads to the highest degree of grain refinement. In our case $N = 1$ revolution under hydrostatic pressure 2 GPa was performed and calculated according to formula (1) the maximal equivalent deformation for $N = 1$ revolution is equal to $\varepsilon = 18.1$. Figure 3 presents exemplary transmission electron micrographs of CuZn30 (M70) brass with fragmented grains of diameter less than 100 nm. In two-phase $\alpha + \beta$ CuZn29Mn11 brass, severely deformed by HPT, fragmented grains of diameter less than 50 nm are observed (Fig. 4). Additionally, in α – phase of such severely deformed alloy micro-twins are reported (Fig. 5).

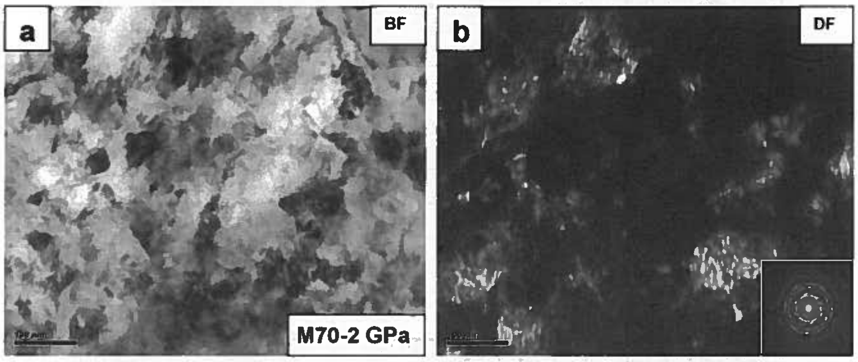

Fig. 3. Microstructure in a sheet plane of HPT deformed CuZn30 (M70) brass up to $\varepsilon = 18.1$ under 2 GPa pressure: a) M70 – BF, b) M70 – DF. Magnification mark designates 100 nm

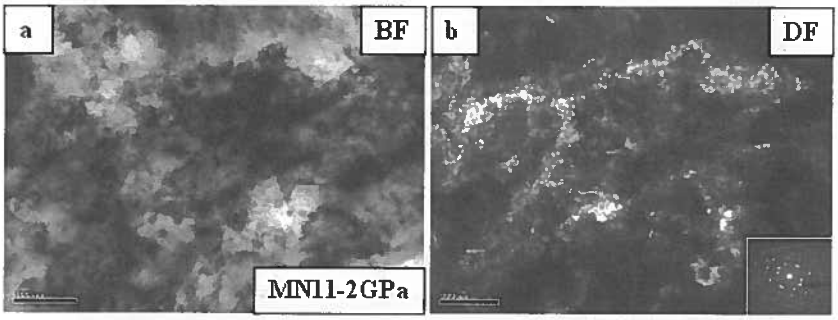

Fig. 4. Microstructure in a sheet plane of HPT deformed CuZn29Mm11 (MN11 – NB) brass up to $\varepsilon = 18.1$ under 2 GPa pressure: a) NB brass – BF, b) NB brass – DF. Magnification mark indicates 100 nm

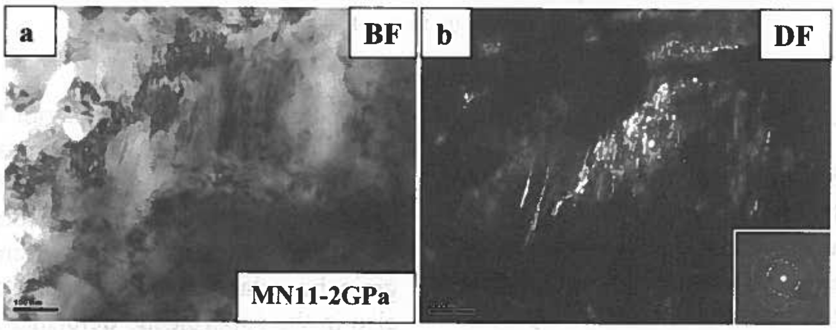

Fig. 5. Micro-twins in a sheet plane of HPT deformed CuZn29Mm11 (MN11 – NB) brass up to $\varepsilon = 18.1$ under 2 GPa pressure: a) NB brass – BF, b) NB brass – DF. Magnification mark indicates 100 nm

4.2. X-ray diffraction measurement

In the case of metal alloys, besides the grain refinement, the HPT deformation influences the solubility of the particular metals. The increase of Fe solubility up to 1.1 at% (2.2 wt%) in a solid solution, practically insoluble Al-Fe system, where the equilibrium Fe solubility in Al lattice at room temperature is equal to 0.025 at.%, is reported [1]. Taking it into consideration we had examined two samples of $\alpha+\beta$ brasses, i.e. CuZn37 and CuZn29Mn11. Figures 6a and 6b present XRD diffractograms of CuZn37 and CuZn29Mn11 in the initial state and after of HPT deformation up to ε = 18.1 under 2 GPa pressure.

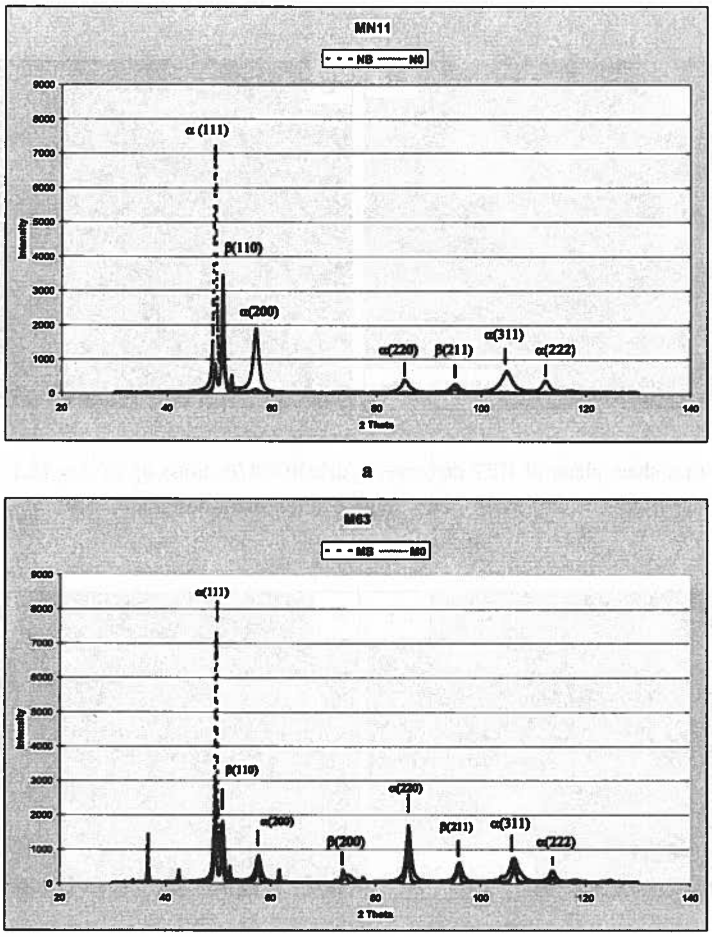

Fig. 6. XRD diffractograms of the examined brasses: a) CuZn29Mn11 (MN11) brass, where N0 indicates the initial state and NB, the state after HPT deformation up to ε = 18.1 under 2 GPa pressure and b) CuZn37 (M63) brass where M0 indicates the initial state and MB, the state after HPT deformation up to ε = 18.1 under 2 GPa pressure

4.3. Discussion

The influence of high pressure torsion on the properties of the examined alloys can be noticed from Table. First of all, as can be expected, the hardness of HPT deformed alloys increased notably due to grain refinement and possibly due to residual stresses. X-ray diffraction measurements indicated considerable changes in the fraction of α phase as related to the fraction of β phase. In deformed CuZn37 brass the fraction of β phase increased contrary to the behavior of CuZn29Mn11 brass where there appeared the decrease of the β phase fraction. The partition of manganese between α and β phases is not equal, affecting the deformation behavior in the initial texture, grains misorientation and character of grain boundaries, and that may play an important role also in the superplastic deformation [8]. The role of Mn contents and the relative relation of α to β contents remains to be explained.

TABLE

Characteristics of the examined brasses processed by HPT under 2 GPa

Alloys	Composition [wt %] /Treatment	Equiv. Deformation ε	Vickers Hardness [MPa]	Grain size [nm]	α phase (XRD) [%]	β phase (XRD) [%]
1	CuZn29Mn11 /1073 K+quench. (MN11-N0)	0	1737.4	–	32.6	67.4
2	CuZn29Mn11 /As 1 (MN11-NB)	19.1	2374.0	<50	88.5	11.5
3	CuZn29Mn11 /As 1+823 K/15 min (MN11-N3)	0	1481.3	–	50.3	49.7
4	CuZn29Mn11 /As 3 (MN11-N4)	18.1	2413.3	–	72.7	27.3
5	CuZn37 (M63-M0)	0	940.8	–	71.8	28.2
6	CuZn37 (M63-MB)	18.1	1509.0	<100	63.2	36.8
7	CuZn30 (M70)	0	1187.0	–	100	–
8	CuZn30 (M70)	18.1	2060.1	<100	100	–

5. Conclusion

Intensive shear deformation during high pressure torsion (HPT) processing, besides refinement of grain size, influences the mechanical properties (considerable increased HV) and solid solubility, which depends on the particular alloy: in the case of CuZn37 brass we notice an increase of β phase in CuZn37 brass deformed by HPT; whereas in HPT deformed CuZn29Mn11 brass, it appears the decrease of the β phase fraction.

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