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Microstructure and properties of α + β brass after ECAP processing

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ABSTRACT

Purpose: Purpose of this paper is to determine the effect of Equal Channel Angular Pressing (ECAP) processing on the microstructure and hardness of α+β brasses. The effect of deformation temperature and number of passes was investigated particularly on the shape and size of grains of both phases.

Design/methodology/approach: The specially constructed channel with 90° pressing angle, allowing heating of the tool with the sample was used for ECAP processing. The grain size was investigated using optical and transmission electron microscopy. The hardness and measurements microhardness were used to determine the effect of ECAP on the hardness of both phases.

Findings: Significant grain refinement down to 300 nm from the initial 20 μm was observed after ECAP processing at 300°C. At 400°C grain refinement occurred down to 1-3 μm. Frequent microtwins were observed within α phase. The microhardness of the β phase was higher than that of α phase, 235 HV and 173 HV respectively.

Research limitations/implications: The limitation is a size of the sample which makes difficult future applications. Another one is elevated temperature (minimum 300°C) otherwise the samples forms crack. This limits also the grain refinement which is above the range of nanomaterials.

Practical implications: Significant grain refinement allows to increase the hardness and strength of the sample preserving a good plasticity. The limitation is the size of the channel what limits the application. The material could be used in such cases when high strength of brasses is needed with sufficient plasticity and good conductivity.

Originality/value: In this paper detailed TEM studies were performed for α+β brasses showing high density of microtwins and higher density of dislocations within α phase, than in the β phase. Higher hardness of the β phase results from the ordering, which hinder deformation of this phase.

Keywords: ECAP; Severe plastic deformation; Ultra-fine grained materials; α+β brasses; CuZnPb alloys

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MATERIALS

1. Introduction

Equal Channel Angular Pressing (ECAP) was used by several authors to obtain significant grain refinement in copper and brass [1-13]. In the paper of Neishi et al., [3] authors studied superplastic deformation in two phase brass Cu-40% Zn possible at relatively low temperature due to application of severe plastic deformation (SPD). Already after one ECAP pass at 400ºC authors obtained significant grain refinement of α and β phases down to about 1 µm. Neishi and coauthors [3, 4] proved that it is possible to obtain superplastic properties after ECAP processing due to formation of large number of nuclei for phase transition and consequently small grain size. After the 1st ECAP pass it was possible to obtain superplastic deformation of 640% at the temperature of 400ºC. In the next work [4] Neishi et al., described superplastic deformation Cu-Zn-Sn alloy subjected to ECAP processing at temperature of 400ºC. Authors have shown heterogeneous microstructure of investigated alloys consisting of recrystallized and not recrystallized areas. In the recrystallized parts the grain size was estimated at 1.5 µm. However like in the case of copper [1] cavities formed during deformation tests.

2. Experimental material and procedures

The brass of composition Cu 61.2 wt% Zn37.3 wt% and Pb 1.5 wt%, rest copper (corresponding to MO60) was supplied in the form of hot pressed rods. Structure was investigated using optical microscope Leica DM-IRM, samples for metallographic examination were prepared by conventional techniques. The specimens were etched in the electrolyte 10 cm^3 HCl, 5g $FeCl_3$, 50 cm^3 C_2H_5OH and 50 cm^3 H_2O. Microstructure was investigated using transmission electron microscope (TEM) Philips CM20. Thin foils were prepared by electropolishing in the electrolyte consisting of 1/3 HNO_3 and 2/3 CH_3OH at subzero temperatures. Microhardness of α and β w phases was investigated using Micro – Hardness Tester CSM Instruments with Indentation software. The load was used in the range of 0.05 - 30 N. The ECAP processing included sequence of sample orientation changes after each pass as shown in Fig. 1. The variant B was used with 90^o rotation after each pass.

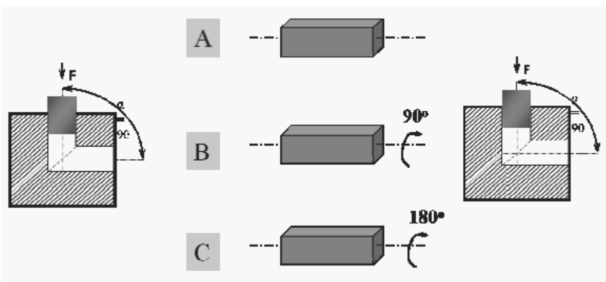

Fig. 1. Scheme of the sequence of sample rotation used between particular passes marked as A, B and C

In the frame of present investigations 3 samples were processed using ECAP: Sample 1- two passes at 400^{o}C corresponding to B scheme, Sample 2 – 1 pass at 400ºC followed by the second one at 300ºC, Sample 3: single pass at 300ºC. The technological parameters used during ECAP processing are summarized in the Table 1. The hydraulic press of maximum capacity 400 kN was used at the deformation rate between 5.6 – 6.7 mm/min.

Table 1.
Technological parameters used In ECAP processing

	Temperature [^{o}C]	Strain rate [mm/min]	Load [t]
Sample 1	400	5.6	4-4.5
	400	6.8	3.6
Sample 2	400	6.3	3.8-4.5
	300	6.7	4
Sample 3	300	6.8	4-4.2

3. Results

3.1. Optical microscopy

Fig. 2 shows optical microstructure from the investigated α + β brass in the initial stage. One can see 3 types of phases: dominating α phase, which contribute to about 70% of the sample area, β phase visible as slightly darker, of estimated fraction near 30% and the smallest particles of lead of less than 1%. The largest grains are that of the α phase of the average size near 20 µm and that of the β phase slightly below 10 µm. From the shape of grains one can see that the initial material was in the as annealed condition. Fig. 3 shows optical microstructure taken from the sample 2 after 1st pass at 400^{o}C and the second at 300^{o}C. One can see that the shape of grains of both phases α and β is more elongated, and some dark details can be seen within α phase. However, not basic differences in the grain size can be seen, what is astonishing in comparison to works of Neishi [3,4].

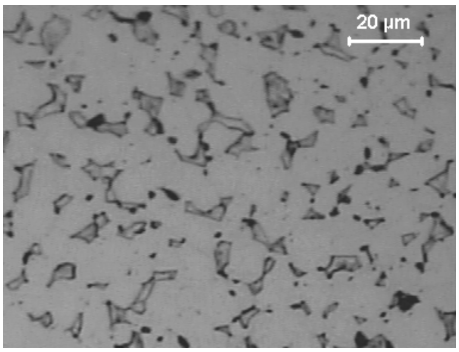

Fig. 2. Optical microstructure of investigated α+β brass in the initial annealed stage

3.2. Transmission electron microscopy

Twice ECAP pressing allowed to obtain a fine grain size structure, however not nanocrystalline, as results from TEM shown, what is in agreement with observations of Neishi et al. [3, 4]. The grains are elongated along the pressing direction, however in optical micrographs it was not possible to determine the existence of a low angle grain boundaries. The optical microscopy allowed to state that the highest degree of deformation was in the central part of the sample, while in the starting part and at the end of the sample deformation manifested by grain shape change was much smaller. Microstructures obtained using transmission electron microscopy from the ECAP pressed α+β brass allowed to determine much more structural details than using optical microscopy. Fig. 4 shows a TEM micrograph taken from the sample 2 after two passes. One can see dark grain of size slightly larger than 1 μm in the reflecting condition. Selected area diffraction pattern (SADP) from this grain indicates that it is β phase at [001] zone axis orientation.

Diffraction pattern (Fig. 4b) indicates that it is B2 ordered phase. It is surrounded by slightly smaller grains of the α phase on the right side as results from the frequent microtwins existing within grains. Small changes in the contrast indicate most probably small differences in orientation.

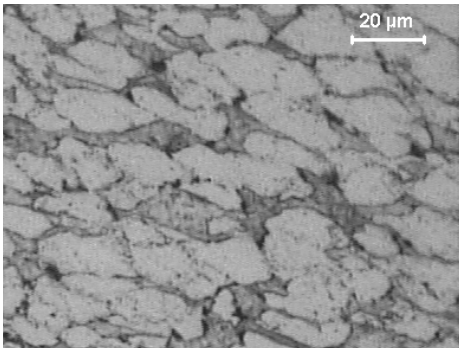

Fig. 3. Optical microstructure of Sample 2 (ECAP at 400°C, then 300°C) – picture from the middle part of the sample

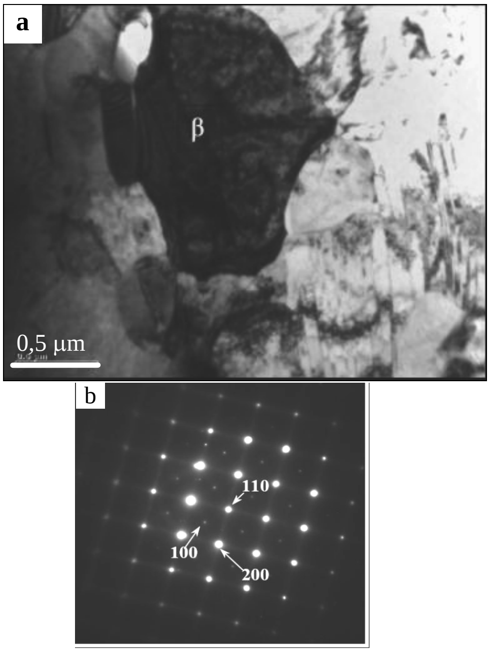

Fig. 4. Sample 2 after two ECAP passes (a) TEM micrograph (b) electron diffraction pattern

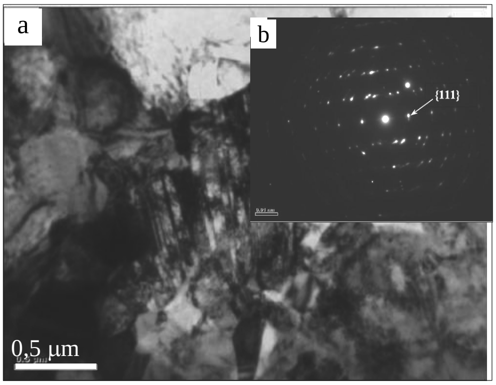

Fig. 5. Sample 2 after two ECAP passes (a) TEM micrograph (b) electron diffraction pattern

Both phases show rather high dislocation density, however that in the α phase seems to be higher. Fig. 5 shows a microstructure of the sample 2 where in the central part high density of twins in the α phase can be seen SADP from this area is presented in Fig. 5b. One can see a twin orientation at [110] zone axis orientation. The twinning plane (111) is marked in the SADP and it can be seen that it is perpendicular to the lines representing twin boundaries.

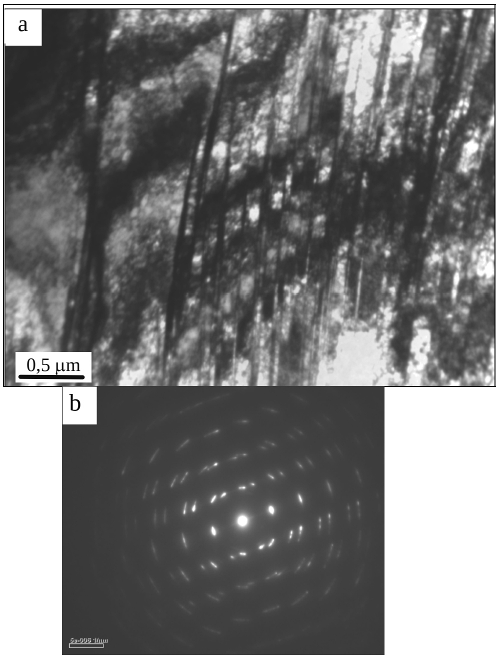

Fig. 6. Sample 2 after two ECAP passes (a) TEM micrograph (b) electron diffraction pattern

Deformation twins formed in a larger grain can be seen in Fig. 6. One can see that their thickness is very small from 50 – 200 nm and they are bent due to plastic deformation of twins during ECAP. Deformation of twins can be seen also in SADP as extension of spots along Debye-Scherrer rings. The microstructure in the central part is rather non-homogeneous and there are also places like those shown in Fig. 7, where fine grains are equiaxial of size less than 0.5 μm. SADP from this area shows rings of reflections from α and β grains, typical for ultra fine grain materials. The hardness and microhardness measurements, which were performed for the brass in the initial stage and after various stages of ECAP at 400^{o}C, showed increase of hardness after application ECAP. The increase of hardness is more pronounced in the sample 2 than in the sample 1 where the second pass was at 300^{o}C, as compared to 400^{o}C in the sample 1. The difference between average hardness of deformed and non-deformed state is about 40HV. The sample 3 where only one pass at 300^{o}C was done show a similar hardness increase like sample 2, with 2 passes at 400 and then 300^{o}C.

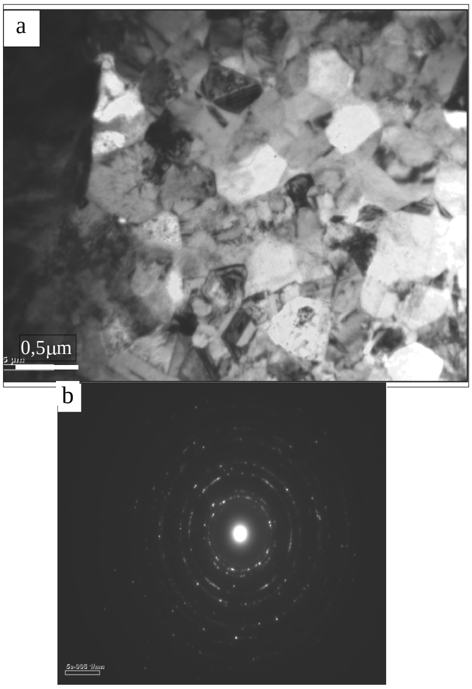

Fig. 7. Sample 2 after two ECAP passes (a) TEM micrograph (b) electron diffraction pattern

Microhardness studies of the $\alpha + \beta$ brass in the initial stage show higher hardness and Young's modulus for the β phase than for the α phase (respectively β - 235 HV and E = 162 GPa as compared to α phase - 173 HV and E = 131 GPa). It results from the ordering of the β phase below 400ºC. Results of the microhardness studies after ECAP, show that the average hardness increases of the α phase from 173 to 235 HV, and Young modulus from 143 to 203 GPa. Due to narrow elongated shape of β phase, its microhardness could not be determined.

4. Conclusions

1) ECAP hot pressing at 400^{o}C of $\alpha + \beta$ brasses causes elongation of grains of both phases and their refinement down to several μm. Additional ECAP hot pressing at 300^{o}C causes further grain refinement to the range from 0.3 to 1.5 μm.
2) In the α phase after ECAP hot pressing at 300^{o}C frequent microtwins (of thickness from 50 to 200 nm) were observed and relatively high dislocation density, higher than in the β phase.
3) In the initial stage β phase show higher hardness and Young's modulus than for the α phase ($HV\beta$ = 235 HV and $HV\alpha$ phase -173 HV). After ECAP the average hardness of the α phase increases from 173 to 235 HV.

Acknowledgements

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