THE INFLUENCE OF EQUAL-CHANNEL ANGULAR PRESSING ON THE STRUCTURE AND PROPERTIES OF POWDER MATERIALS

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Abstract

The structure, physical and mechanical properties of samples produced from pure and titanium alloyed copper powder using equal-channel angular pressing with complex deformation route have been investigated. The samples were manufactured using the technology of pressing, sintering and deformation by equal-channel angular pressing. The structure of samples after sintering consists of copper grains, titanium particles and pores. Formation of the diffusion zone between components of powder porous sample in the course of sintering has been observed to result of surface and volume diffusion. Microstructure analysis longwise to the angular pressing axis on the lateral part of samples has shown a noticeable banding: copper grains and titanium particles are drawn longwise to the deformation direction. The maximum density was reached in the middle zone of the sample. The grain size changed in the same way. The distribution of stresses has been estimated by measuring microhardness on the lateral surface of the sample. Mechanical properties have been determined: hardness and compression tests of samples cut from the middle zone along the angular pressing axis. Decrease of non-uniformity of stress-strain state and balancing of density during angular pressing take place while the cross-section changes from circular to rectangular. High mechanical properties and noticeable banding of deformation along the angular pressing axis were observed, rather lower on the transverse cross-section.

Keywords: equal-channel angular pressing, powder samples, structure, stress, density, hardness, strength

INTRODUCTION

Technological schemes with high accumulated deformation make possible severe plastic deformations of powder billets without fracture. The presence of high value of hydrostatic compression into the deformation zone is their distinctive peculiarity [1]. Equal-channel angular pressing (ECAP) is one of those schemes utilized for obtaining materials with ultrafine-grained structure [2]. In the case of powder materials, ECAP enables the production of high-density parts. Metallic and non-metallic materials prepared by powder metallurgy gain qualitatively new mechanical and service properties, as a result of severe plastic deformation, and possess extremely high plasticity combined with high strength [3]. The main difficulty of ECAP of powder materials is obtaining high density and equidensity at limited energy-power parameters [4, 5, 6].

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The purpose of this work is experimental study of opportunities for production of high-density powder materials by equal-channel angular pressing, investigation of structure and physico-mechanical properties of finished parts.

THE MATERIAL AND EXPERIMENTAL TECHNIQUE

The prismatic-shaped billets with cross-section dimensions 15×15 mm, length 60 mm and initial porosity 20% were made of pure copper powder PMS-1 and copper powder alloyed with 0.5% of titanium powder BT1-0, They were double-action compacted on a hydraulic press (force 1600 kN).

Sintering was conducted in a synthetic-gas medium (composition: 72% H_2, 21% CO, 5.5% CO_2, 1.5% H_2O) by stepwise mode heating up to 100-120°C, 200-220°C, 300-320°C, 400-420°C, 500-520°C and 600-620°C with 30 minutes exposure to each temperature, increasing temperature up to 800°C and exposing during 1 hour.

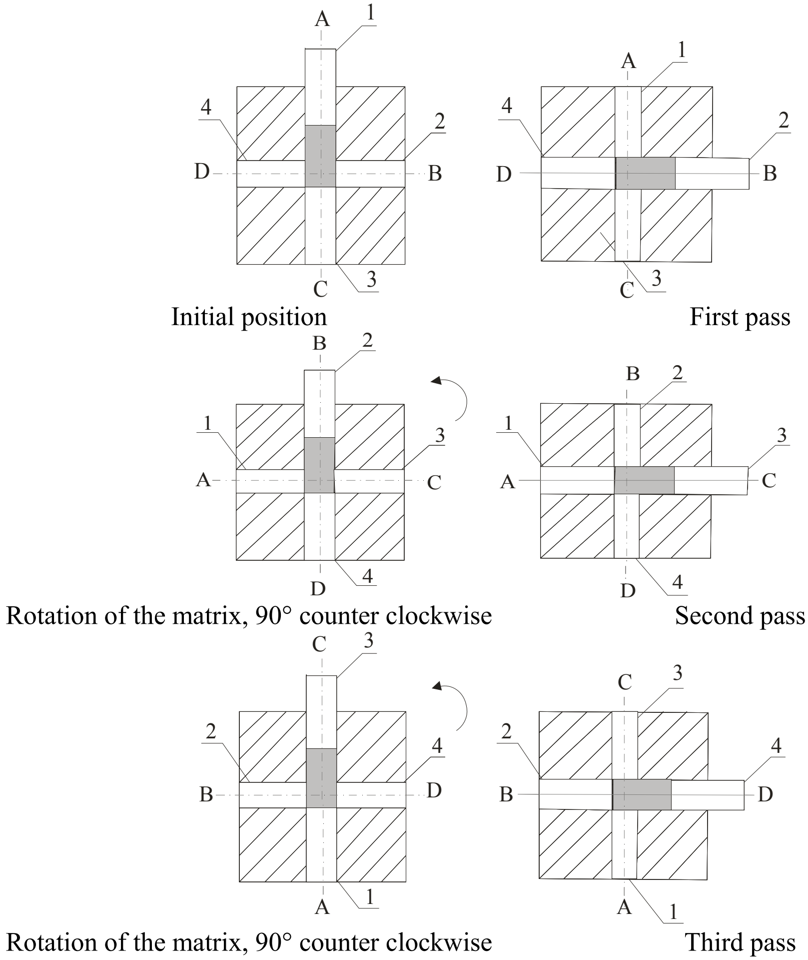
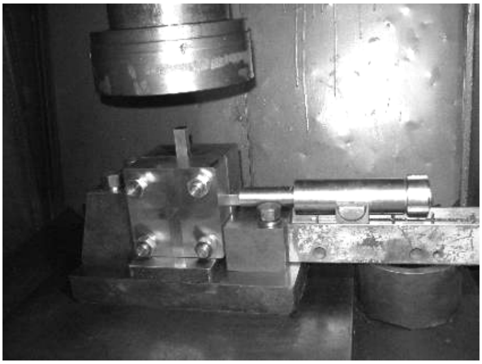


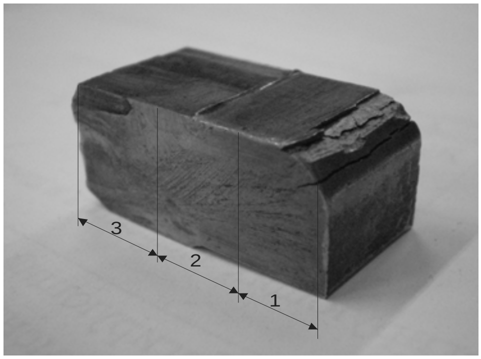
Fig.1. Scheme of equal-channel angular pressing.

ECAP was performed by three passes on the same press in the special die without extracting the sample and changing its orientation (Fig.1). Molybdenum disulphide was used as a lubricant. The length of sample after pressing became 30 mm.

The die consists of cartridge 1 and matrix 2 fixed in it with four equal channels of the same section crossed at 90°. The punches 3, 4, 5, 6 were placed in the matrix. The inner radius of matrix is 1.5 mm, outer radius zero. The backpressure was ensured by flowing lead through a contracting hole in the matrix placed before horizontal channel. Preliminary calibration of the lead billet was performed by force measurement and calculation of pressure at flow of lead through a hole. The matrix 2 was turned 90º counterclockwise after each pass without extraction of sample, to ensure continuous deformation. The photo of the special die is presented in Fig.2,a. The variations of pressure and backpressure values are presented in Table 1. The backpressure has been chosen on the assumption of mechanical properties of material, taking into account deformation hardening [7].



a)



b)

Fig.2. Photos of the die – a and the sample – b after the first pass: 1 – is the part from the backpressure side; 2 – is the middle part; 3 – is the part under the punch.

Tab.1. The modes of equal-channel angular pressing.

Pass number	Compacting pressure [MPa]	Backpressure for copper powder [MPa]	Backpressure for Cu+0.5%Ti [MPa]
1	1110	90	100
2	1220	120	130
3	1750	150	150

The copper sample obtained after the first pass of ECAP is presented in Fig.2,b. Investigation of microhardness and mechanical properties were conducted after each pass by extracting the sample from the matrix and cutting it in three parts: 1 – is the backpressure side; 2 – is the middle part; 3 – is the part under the punch. The sample was cut into six parts for determination of density of each part by hydrostatic weighing. The microhardness of cross-section of three parts of sample along the central line was measured on the PMT-3 microhardness tester by indentation of a diamond pyramid into the polished surface by a load of 25 g. The structure of each part of the sample on the lateral surface was studied on a scanning electron microscope REMMA-102.

Investigation of grain size was done by X-ray structure analysis with a diffractometer DRON 4-13, radiation CuK_{α} using diffraction line profile analysis and precise measuring of lattice parameter by centres of gravity of lines (111), (220) and (311) locations.

Mechanical properties were determined after each pass by compression testing of samples with dimensions ∅5×10 mm cut from the middle part along the pressing direction. The hardening curve was plotted with accounting of porosity using the technique [5] in coordinates 'true stress σ – relative degree of deformation ε'. The yield stress was determined by the grapho-analytical method.

RESULTS AND DISCUSSION

Visual investigation of samples (Fig.2,b) after the first and second passes has shown the presence of looseness and cracks on the butt end from the backpressure side and this part was not compacted. It possesses the same density with the initial sample, which leads to the formation of cracks [7]. The backpressure growth could not eliminate this appeared looseness. These results are well in agreement with studies presented in papers [5, 6, 8]. The above mentioned part was not found after the third pass due to growing of interparticle bonds and backpressure.

The non-uniform distribution of relative density observed after the third pass is connected with non-uniformity of stress-strain state during compaction of sample and influence of friction forces (Fig.3) [9]. The relative density varies over a wide range with a maximum value reached in the middle part of sample at the formation of a compaction zone of negligible volume. The compaction zone increases, the density grows inside it and, after the third pass, the relative density is almost equal to 1 for both materials. Growth of accumulated deformation leads to an increase of the density and dimensions of compaction zone with the density value close to pore-free material. The relative density of Cu+0.5%Ti powder material changed in the same way after ECAP passes, but its value was lower, due to the influence of hardening phase – titanium particles.

The microstructure of materials obtained after each pass is characterized by elongation of grains and titanium particles in three sections of the samples. The banding of microstructure has formed (Fig.3,a,c,e). Distortion of grain boundaries indicates a high level of internal stresses and elastic microdistortions of crystal lattice verified by the X-ray structure analysis [9].

The remaining pores located on grain boundaries were observed after the first pass with increased number of pores on butt ends [5, 10]. A coarse granularity was observed in the first part of the sample from the backpressure side. There are some grains with the diameter up to 1000-1500 nm, while most of grains are of 240-260 nm. The average grain size in this part was 280 nm. An accumulated deformation is growing into the compaction zone while grain size decreases monotonously. The average grain size in the second part was 220 nm and in the third part under the punch average grain size was 230 nm. The grain size of powder material Cu+0.5%Ti changed in the same way. The grain size in this case is larger and became 250 nm in the middle part of the sample. The uniformity of forming structure in the most intensive metal flow direction, grains' elongation and fraction of high angle boundaries are determined not only by a degree of deformation, but also, to a considerable extent, by pressing modes.

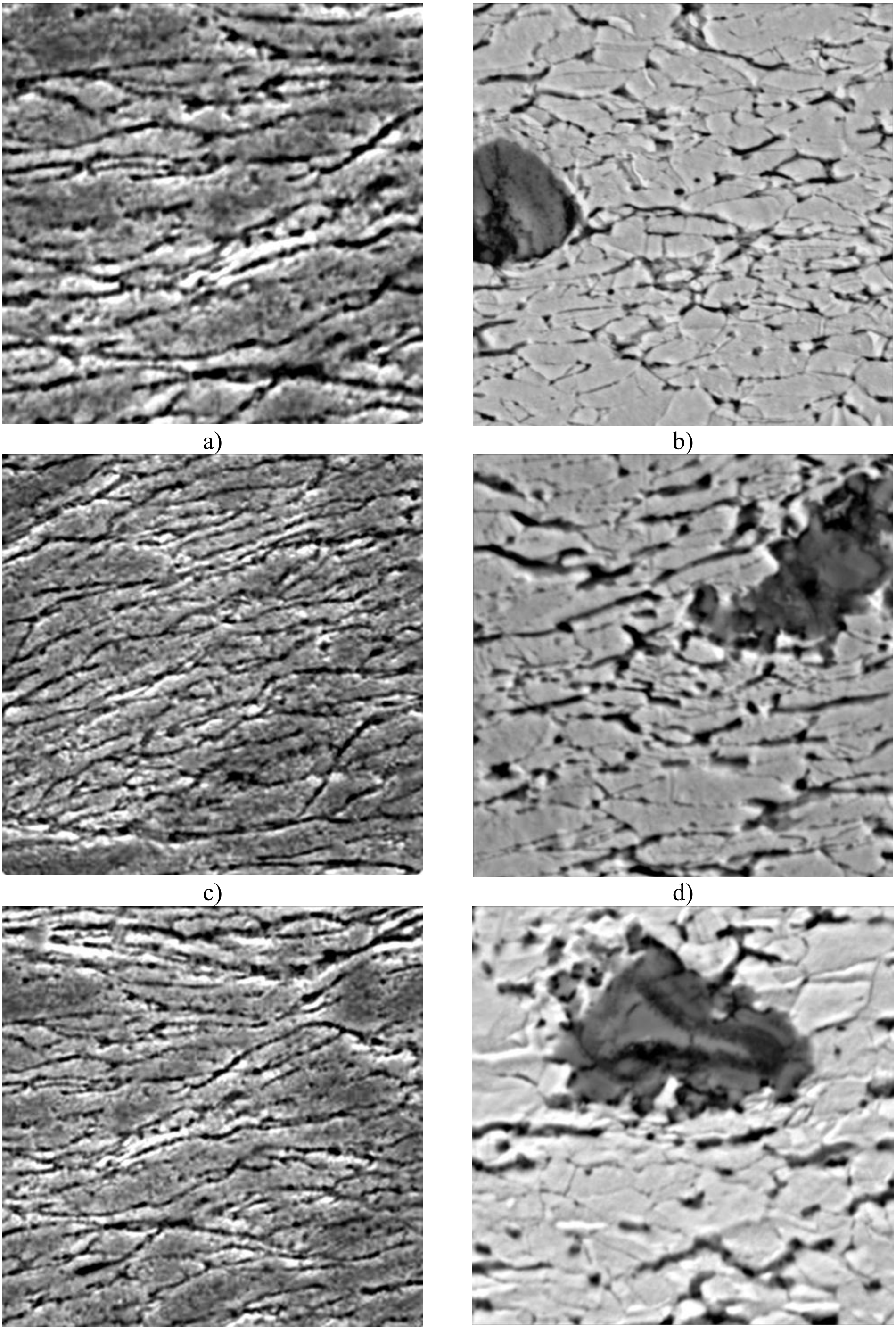


Fig.3. The microstructure of samples after the third pass, x3000: a, c, e – Cu; b, d, f – Cu+0.5%Ti; a, b – is the first part, c, d – is the second part, e, f – is the third part.

Granularity after the second pass is lower and the grain size decreases continuously with the growth of the degree of accumulated deformation. Grains elongated during the first pass in the pressing direction became more rounded in shape after the first pass due to changing of the metal flow direction. There are no pores on grain boundaries into the second and third parts of samples, the density of samples into the compaction zone is close to the density of compact material. The largest grain size remained in the butt end from the back pressure side and granularity had been observed. Reduction of average grain size happened as accumulated deformation grew. It became 160 nm for the copper powder material and 210 nm for the Cu+0.5%Ti powder material.

The least granular, regular, band microstructure with small-angle boundaries was obtained after the third pass. In this case grains possess gradual elastic distortion with evident deformation texture. The reason of this distortion may be long-range stresses from non-equilibrium grain boundaries [9, 11]. The smallest grain size has been obtained in the compaction zone (approximately 80 nm for the copper material and 180 nm for the Cu+0.5%Ti material), while with growth of the distortion level of all grains, contours of boundaries became fuzzy. The great variation in diameter of grains was not observed in the investigated parts of the samples, like after first and second passes.

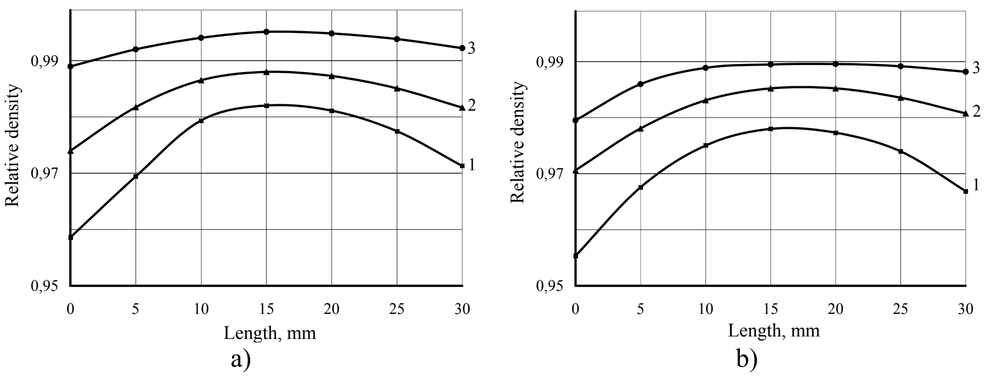


Fig.4. Distribution of relative density along the sample: Cu – a, Cu+0.5%Ti – b; after passes: 1 - the first pass; 2 - the second pass; 3 – the third pass.

The microhardness change along central line of each part of the sample is the same. The lowest microhardness values are on butt ends and maximum in the central part of the sample with increases from outer ends to the centre.

Nonmonotonic variation of microhardness decreases with the growth of accumulated deformation. The microhardness in the compaction zone of copper sample increased to 845 MPa after the third pass due to formation of fine-grained structure with average grain size 80 nm and in the Cu+0.5%Ti powder material it has grown to 865 MPa, which is stipulated by the formation of fine-grained structure and presence of hardening phase – titanium particles.

The dependence of stress on true degree of deformation for axial compression enables an estimation of the degree of strengthening of deforming powder material.

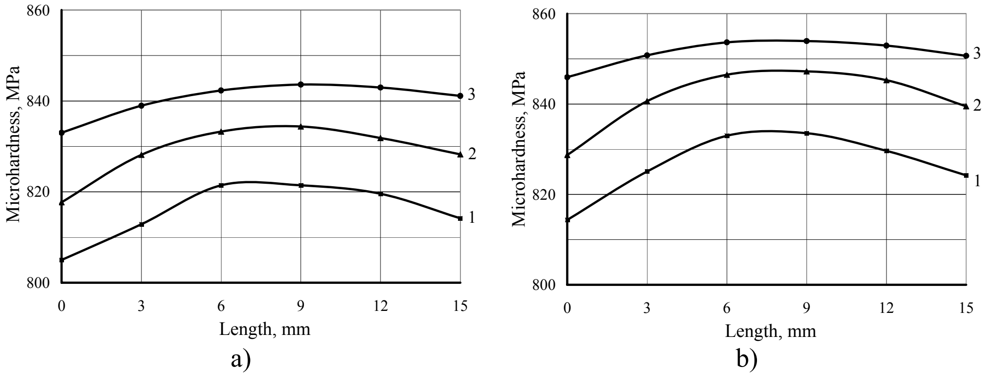


Fig.5. Changing of microhardness by central line of cross-section of sample: Cu – a, Cu+0.5%Ti – b; after passes: 1 - is the first pass; 2 - is the second pass; 3 – is the third pass.

A gradual densification of material with slight hardening of powder particles was observed after the first pass, hardening coefficient is 0.47, yield stress 225 MPa. After the second pass the value of relative density almost reached the density of pore-free material, with an increase in strength properties. A higher value of yield stress was ensured by growing density, lower grain size and presence of hardening titanium particles in samples made of Cu+0.5%Ti powder material. Samples of maximal relative density, fine-grained band structure and high mechanical properties, higher hardening coefficient and yield stress, were produced after the third pass.

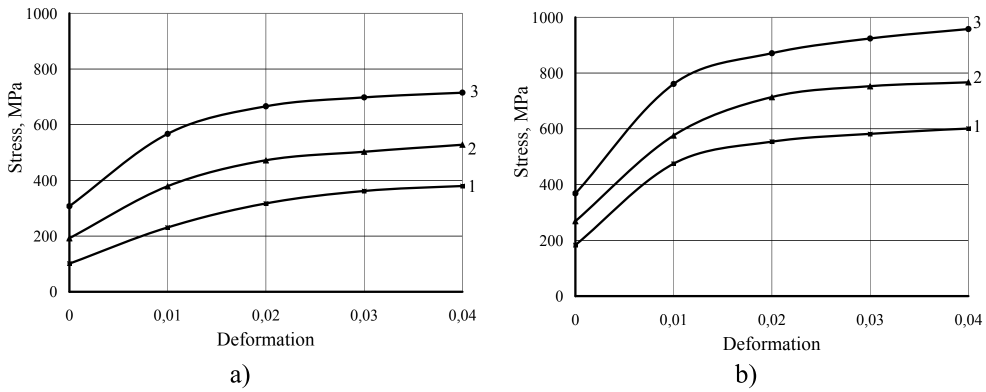


Fig.6. Hardening curves of samples Cu – a, Cu+0.5%Ti – b; after the passes: 1 - is the first pass; 2 - is the second pass; 3 – is the third pass.

Plastic properties may be characterised by the relative degree of deformation of the height of sample till fracture, which has been determined by appearance of cracks on a lateral surface of the sample. After the first pass samples fractured after deformation of 0.035-0.040, but after the second and third passes the degree of deformation approached 1.

CONCLUSIONS

The influence of equal-channel angular pressing route on the structure, physical and mechanical properties of pure and titanium alloyed copper powder materials has been studied experimentally. Significant densification of powder material and formation of compaction zone after the first ECAP pass was established, with a dramatic non-uniformity of stress state, physical and mechanical properties. The relative density after the second pass was almost equal to 1 and the volume of deformation zone became two-thirds of billet's volume. The compaction zone has grown to 80% of billet's volume after the third pass and was characterized by high density, fine-grained structure and high mechanical properties.

The microstructure of powder materials after each pass of ECAP is characterized by elongation of grains and titanium particles with an increase in plasticity, which may be approximately described by relative degree of deformation that approaches 1 after the second and third ECAP passes. The presence of titanium particles improves strength properties of material, which impedes obtaining high porosity. The average grain size was 80 nm for the pure copper and 180 nm for the Cu+0.5%Ti powder material.

It has been established experimentally that, using initially porous powder billets, powder materials with nanostructure, high mechanical properties and density can be produced by three ECAP passes.

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