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Metallographic aspects of deformed monocrystals of CuZn30 alloys

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ABSTRACT

Purpose: The aim of the paper is to present the technique of production and the analysis of the structures of CuZn30 monocrystals compressed at a temperature of 20°C up to 400°C at a strain rate of 10^{-5} sec^{-1} to 10^{-1} sec^{-1}.

Design/methodology/approach: The range of investigations comprised the production of monocrystals for tests, the determination of their crystallographic orientation, the deformation of selected monocrystals by compressing them within the temperature range from 20°C to 400°C at a strain rate of 10^{-5} sec^{-1} to 10^{-1} sec^{-1}, as well as observations of the structures on an optical microscope.

Findings: The analysis of the results of these investigations permitted to prove a considerable influence of the temperature, the strain rate and the crystallographic orientation on the metallographic effects of the work hardening of the CuZn30 alloy with the orientation $[\bar{1}\ 3\ 9]$.

Practical implications: In the microstructures of the investigated monocrystals typical effects of plastic deformation were observed in the form of parallel cruciform lines and slip zones with locally intensified densities in various ranges of the cross-section of the sample and bands of deflection with weakly visible slip lines in the original system.

Originality/value: The analysed microstructures of compressed monocrystals prove that the main mechanism of plastic deformation is the slide. Of essential influence on the structure of plastically deformed monocrystals with the orientation $[\bar{1}\ 3\ 9]$ are both the temperature and the strain rate.

Keywords: Metals; Copper alloys; Monocrystals; Structure; Plastic deformation

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MATERIALS

1. Introduction

The development of many domains of technology is imposed by the necessity of applying non-ferrous metals and their alloys, which constitute a group of structural materials equivalent with steel. Keeping in mind the problem of raw materials copper alloys, particularly brass to plastic working are widely used in industry. They are characterized by a high plasticity and good resistance to corrosion. They are applied in electrical industry, telecommunication, in transport and machine building, particularly in the production of elements for deep drawing. Investigations concerning the fundamental mechanisms of plastic deformation of metallic materials (1-4) are mostly performed on metallurgically high-quality and chemically pure monocrystals and less frequently on polycrystalline materials. Therefore, of essential technical importance is the development of efficient techniques of producing monocrystals to be used in laboratory tests. Plastic deformation is the basic process of changing the geometrical features of solid bodies affected by external or internal forces, on which the technology of plastic working is based. The knowledge of the mechanisms of plastic deformation is, therefore, of essential importance both cognitively and with respect to its application. The development of crystallography and the methods of investigations, and mainly the techniques of utilizing the diffraction of X-rays, electrons and neutrons, contributed considerably to the development of materials engineering, particularly the knowledge concerning plastic deformation of metals and commercial alloys [1-6].

Due to the rather high interest in non-homogeneous deformation a considerable amount of investigations has been started (7-11), dealing with the influence of those factors which are responsible for this phenomenon. These are both "internal" factors, connected with the microstructure of the material, such as the grain size, precipitations and texture, as well as "external" factors connected with the conditions of deformations, viz. temperature, strain rate, the environment of deformation and the state of structure of the material. Most experiments were carried out on polycrystalline alloys. There are only few cases of investigations, in which the heterogeneity of plastic deformation concerned monocrystalline alloys. More recent methods of describing and recording the achieved results provide new information about the nature of this effect. The presently suggested theories describing this phenomenon are, however, not explicit enough, and none of them permits to explain sufficiently and to predict the occurrence of heterogeneities of plastic deformation in the given material. Every plastic deformation is a complex process, consisting in the not homogeneous and often also in a specific cooperation and organization of elementary sliding of twinning. The continuous development of the theory of dislocations, supported by modern techniques of investigations allow to understand the fundamental features of this process [9-14].

2. Experimental procedure

The charge material for the production of monocrystals was single-phase copper of the type CuZn30, assayed as CW506L according to the standard PN-EN 12163-2002 [15], from an ingot with a mass of 50 kg, smelted in the laboratory making use of the inductive method, the chemical composition of which is to be seen in Table 1.

The ingot was preliminarily forges at a temperature of 850° C to rods with a diameter of 30 mm, and then after hot forging the rods were supersaturated in water and pull-broached to the dimension ∅ 3.8 mm. The process of pull-broaching was accomplished by means of inter-operative recrystallizing annealing for 2 hours at 700°C. The obtained rods, cut into sections about 230 mm long, constituted the charge material for the production of monocrystals.

The alloy CuZn30 was monocrystallized in the laboratory of the Department of Metallic Materials and Nanoengineering at the Academy of Mining and Metallurgy in Cracow. The monocrystal of the investigated alloy was achieved by crystallization controlled by the gradient of temperature (modified Bridgman's method without a strictly assumed orientation of the respective nuclei), carried out in a vertical electrical laboratory furnace (Fig. 1), displacing the zone of the temperature gradient of the furnace versus the crucible with the charge. Due to the low temperature of vaporization of zinc (about 906^{o}C) the smelted mass of the alloy is placed in a quartz crucible with a round cross-section, an inside diameter of 4 mm and a length of about 250 mm. The crucible was characterized by a standard smoothness of the walls and a specifically defined conical shape in the zone of the nucleolus of crystallization. The tubes of the crucible were closed by hydrogen burner, forming at the front of crystallization a cone with an apex angle of about 40°.

The monocrystal nucleates and grows in this case from the liquid state in the course of the gradual decrease of the temperature in the furnace. When the conical end of the crucible has cooled down, a nucleus is formed, starting from which the whole mass of the alloy in the crucible is being monocrystallized. The large gradient of temperature is necessary for the formation the nuclei of crystallization of the alloy, whereas the lower gradient increases the crystal. Thus, only a small number of crystalline nuclei is formed. In the case of a further solidification, the faster increasing nucleus of the nucleus outstrips the others, so that a monocrystal may be formed in result of the so-called natural selection occurring in the zone of the tip of the cone. A monocrystal obtained in such a way has the shape of a cylinder with a diameter of about 4 mm and a length of about 200 mm (Fig. 2).

Compression tests of selected monocrystals with some defined orientations were carried out within the temperature range of 20°C -400°C, at a strain rate of 10^{-5} sec^{-1} to 10^{-1} sec^{-1}, applying the testing machine INSTRON 3382; the tests were accomplished

Table 1.
Chemical composition of the alloy applied for the production of monocrystals

No.	Designation of the alloy and kind of the analysis	Components of the alloy, % by weight		Admissible concentration of contaminations, % by weight					
		Zn	Cu	Fe	Al	Ni	Sn	Pb	Rest.
1	ingot CuZn30 in compliance with the ladle analysis	30.3	rest.	0.024	0.039	0.024	0.003	0.01	0.3
2	CuZn30 with the PN-EN 12163:2002	28.3-30.3	rest.	0.05	0.02	0.3	0.1	0.05	0.1

in the Accredited Laboratory of the Strength of Materials (AB120) of the Polish Academy of Sciences in Cracow. The values of strength were recorded with an accuracy of up to 0.5. The final deformation of the sample during the compression test amounted to about 50%.

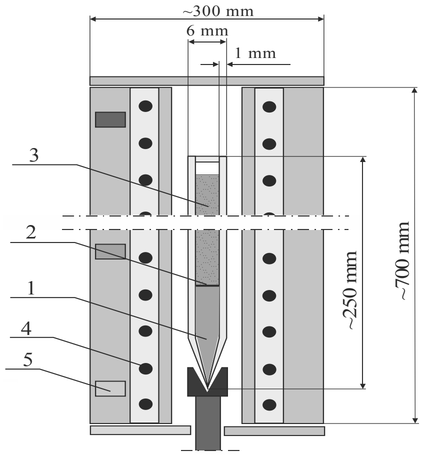


Fig. 1. Diameter of a heating stove with a quartz crucible: 1 – monocrystal, 2 – boundary surface of the solid and liquid phase, 3 – liquid, 4 – heating coil, 5 – temperature-sensitive element

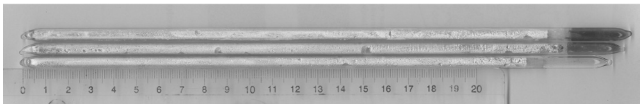


Fig. 2. Monocrystals of the alloy CuZn30 obtained in the laboratory by means of the modified Bridgman method

Metallographic observations accomplished by means of a transmission optical microscope concerned selected monocrystals of the alloy CuZn30; they were carried out immediately after the crystallization on longitudinal and transverse micro sections of the obtained samples after their free compression at a temperature within the range from 100°C to 400°C. The preparation of the microsections included classical procedures of grinding and mechanical polishing. Samples whose microsections were to be observed were etched at room temperature in a reagent containing 2 g potassin dichromate $K_2Cr_2O_2$, 100 cm^3 distilled water, 4 cm^3. Saturated aqueous solution of sodium chloride NaCl and 8 cm^2 sulphuric acid H_2SO_4 The respective experiments lasted from 20 to 120 seconds. After their etching the microsections were scanned on the transmission electron microscope Leica MEF 4A, magnified by 50 to1000 times.

3. Results and discussion

The applied modified Bridgman's method permitted to achieve monocrystals of the CuZn30 alloy adequate for plastic deformation investigations. Monocrystals are characterized by selected crystallographic orientations from various areas of the basic triangle (Fig. 3).

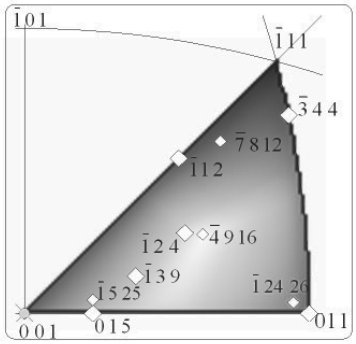


Fig. 3. Reverse polar figure with the denoted crystallographic orientations of the investigated monocrystals of the alloy CuZn30

Metallographic observations of the monocrystalline samples of the alloy CuZn30 after the hot compression test have made it possible to determine the effect of the temperature of the strain rate and crystallographic orientation of the investigated monocrystals on changes in the shape of the longitudinal section of the compressed samples and the distribution of micropores revealing after the crystallization of the alloy. The results of microscopic observations have been presented in Figs. 4-7 and those of microscopic observations in Figs. 8-11.

a) b)

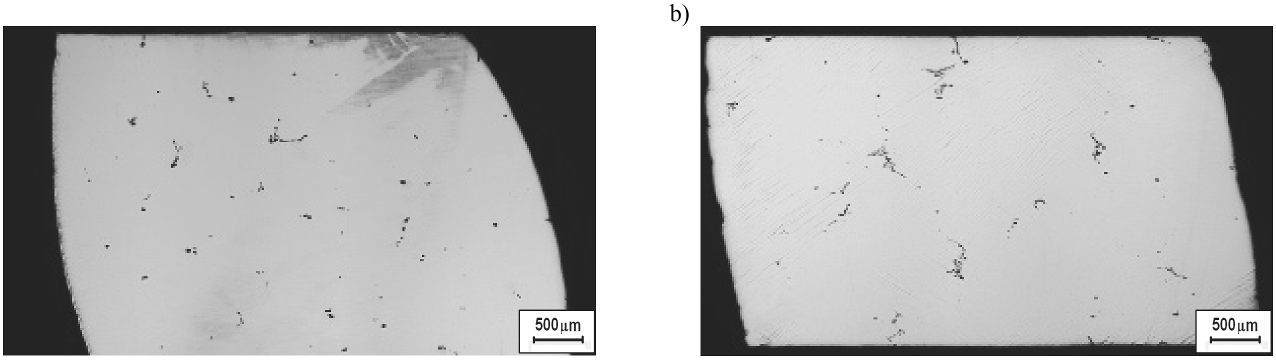


Fig. 4. Shape of the cross-section of monocrystalline samples of CuZn30 after the compression test at a strain rate of $10^{-4}s^{-1}$ and a temperature of 300°C: a) orientation [$\bar{7}$ 8 12] with a draft of 30 %; b) orientation [$\bar{1}$ 3 9] with a draft of 50%, not etched., magnified 50x

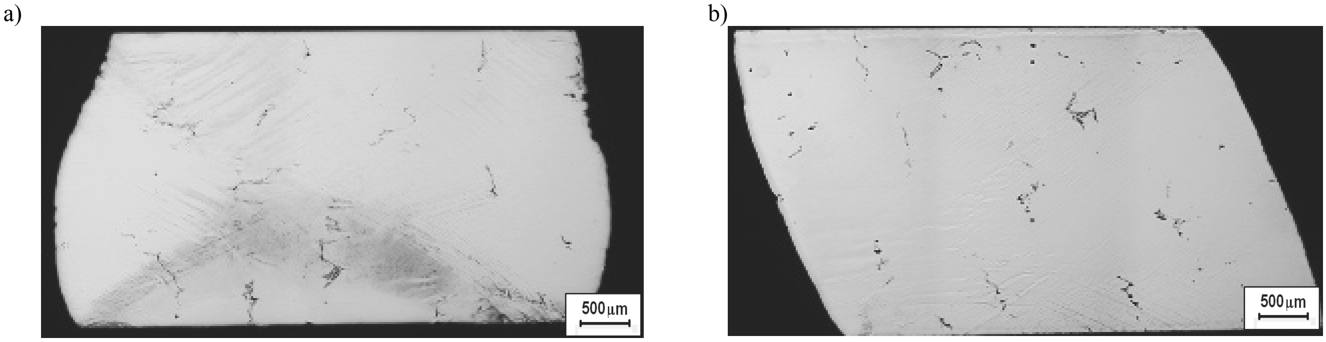


Fig. 5. The influence of the rate of plastic deformation on the shape of the longitudinal section of monocrystalline samples of the alloy CuZn30 with the crystallographic orientation $[\bar{1}\ 3\ 9]$, compressed with a draft of 50 % at 400°C: a) $\dot{\varepsilon} = 10^{-4}s^{-1}$, b) $\dot{\varepsilon} = 10^{-1}s^{-1}$ not etched, magnified 50x

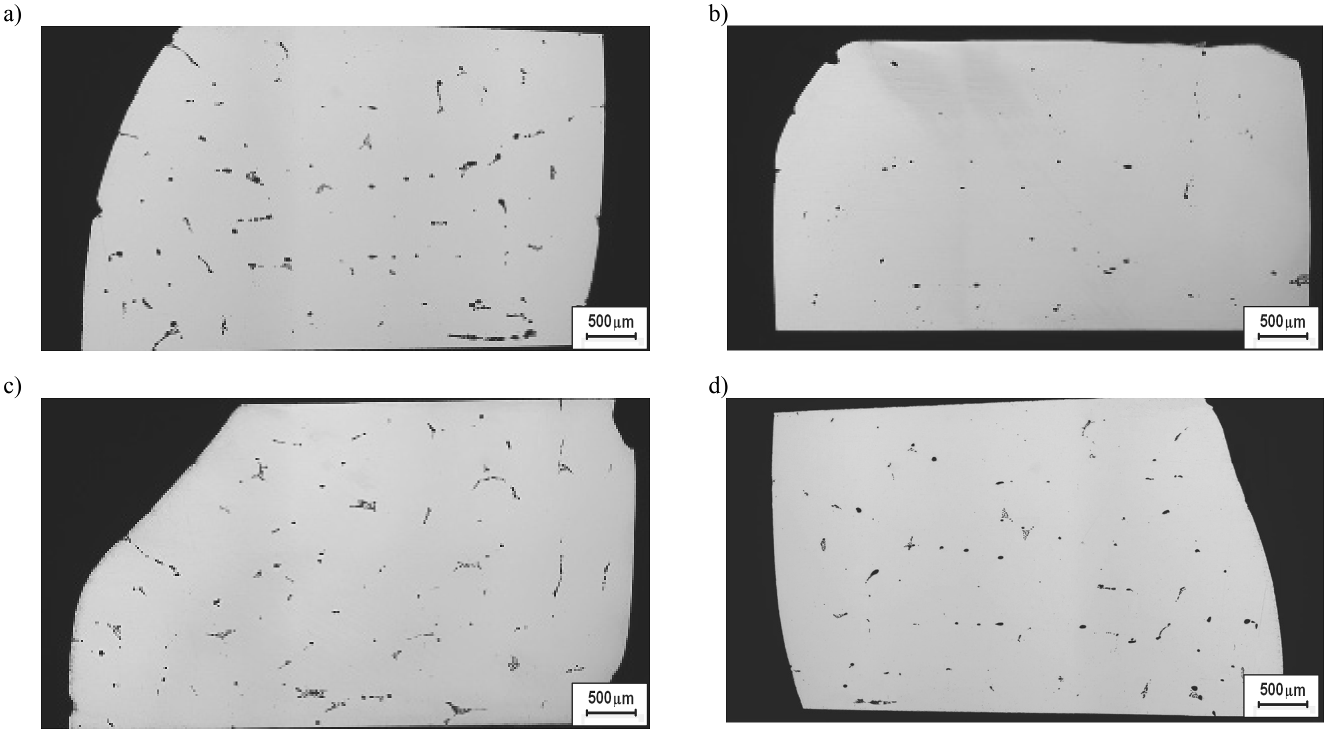


Fig. 6. The shape of the cross-section of monocrystalline samples of the alloy CuZn30 after the compression test with a draft of 30% at 300°C, about $10^{-3}s^{-1}$, depending on the crystallographic orientation: a) $[\bar{3}\ 4\ 4]$, b) $[\bar{4}\ 9\ 16]$, c) $[\bar{1}\ 24\ 26]$, d) $[\bar{1}\ 5\ 25]$, not etched, magnified 50x

Samples of monocrystals with the crystallographic orientation $[\bar{7}\ 8\ 12]$ compressed with a draft of 30% at a strain rate of about $10^{-3}sec^{-1}$ in the temperature range from 300°C to 400°C have a nearly orthorhombic cross-section deflected at an angle of about 11°-15° from the direction of the exerted compressive force (Fig. 4a). A similar shape of the cross-section was displayed by samples with the orientation $[\bar{1}\ 3\ 9]$ after their deformation by 50% at 300°C and 350° C with about $10^{-4}sec^{-1}$. The deviation of the axis of the samples from the direction of the exerted compressive force is similar, amounting to 12°-15° (Fig. 4b).

Figs. 5a-b illustrates monographic ally the influence of the strain rate in the range of $10^{-4}s^{-1}$ to $10^{-1}s^{-1}$ on the shape of the longitudinal section of monocrystals with the crystallographic orientation $[\bar{1}\ 3\ 9]$, compressed with a draft of 50 %. After deformation in the range $\dot{\varepsilon} = 10^{-4}s^{-1}$-$10^{-3}s^{-1}$ the cross-section of the samples is characterized by rounded edges and an increased volume of the bottom part and also by a cylindrical shape in the upper part of the cross-section. In the case of the investigated parameters of deformation the shape of the cross-sections of the samples is nearly orthorhombic. The deviation of the sample axes from the direction of the exerted compressive force amount to 5°-15°. After the deformation at 300°C and a strain rate of about $10^{-3}s^{-1}$ monocrystals with the crystallographic orientation $[\bar{3}\ 4\ 4]$, $[\bar{1}\ 24\ 26]$ and $[\bar{1}\ 5\ 25]$ are characterized by a diversified shape of the cross-section (Fig. 6). The deviation of the samples from the direction of the exerted compressing force amounts to about 5° to 7°.

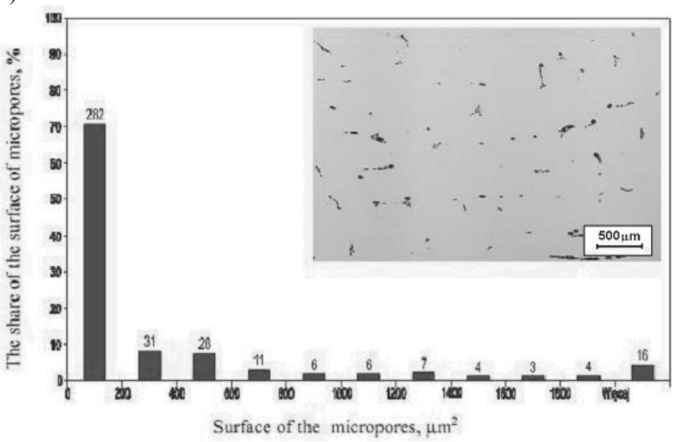
The micropores on the cross-sections of the tested monocrystals differed in their shape and size, mostly in the shape of numerous clusters or situated linearly. Basing on the analysis of the parameters of stereological micropores (Fig. 7a) detected in the investigated monocrystals of the alloy CuZn30 is has been found that the crystallographic orientation of the monocrystals does not influence the number and size of micropores (from about 249 to 304) with a surface up to 200 μm^2. It has also been found that the larger the surface of the micropores, the smaller is their amount number; (the number of micropores with a surface exceeding 1800 μm^2, for instance, amounts to about 4-16. The share of the surface of micropores with a maximum size oscillates from 0.15% to 0.45% whereas the share of minimum surface sizing up to 200 μm^2 amounts to about 0.02 %-0.12%. A microanalysis revealed that the chemical composition of the internal surface of the micropore includes contaminations of oxygen and sulphur with a mass fraction of about 5.50 and 1.68 - respectively (Fig. 7b). The structure of plastically deformed monocrystals with the orientation $[\bar{1}\ 3\ 9]$, is essentially affected by the temperature and strain rate. The results of the temperature of deformation within the range of 300 to 400°C when $\dot{\varepsilon} = 10^{-4}sec^{-1}$ and the effect of the strain rate between $10^{-5}sec^{-1}$ and $10^{-1}sec^{-1}$ on the structure of a CuZn30 monocrystals with an orientation $[\bar{1}\ 3\ 9]$ were determined concerning a constant temperature of compression amounting to 400°C, as shown in Figs. 8 -11.

After the compression test at a temperature of 300°C in the structure of the tested monocrystal besides micropores parallel as well as crisscrossing gliding bands were to be seen with a locally intensified density in various areas of the cross-section of the sample. The gliding bands occurring in the structure of the deformed monocrystal have a width of about 10-20 μm. Similar bands of plastic deformation have also been revealed after the compression test at a temperature of 350°C (Fig. 8), as well as in areas with clusters of micropores, whereas in a monocrystal deformed at a temperature of 400°C micro-areas of deformation were detected characterizing localized macro-twins or bands of deformation.

Moreover, in the zone of maximum macroscopic deformations of the sample there occurred microareas of fine grains, probably arising due to dynamic and static recrystallization in the course of the compression of the sample (Fig. 9). In the case of a monocrystal compressed at a strain rate of $10^{-5}sec^{-1}$ (the time of sampling amounting to about 3 hours) distinct smooth zones have been observed with irregular boundaries similar to dynamically recrystallized grains obtained in the process of hot deformation. An increase of the strain rate from $10^{-3}sec^{-1}$-$10^{-1}sec^{-1}$ involves besides the typical effects of plastic deformation densely arranged broad bands of deflections, probably occurring already during the initial stage of deformation and hampering the development the slip bands. Considerable local stresses occurring in the path of shear due to the retardation of dislocations in primary system cause that the increasing deformation may lead to a gliding in other planes of the system (Figs. 10 and 11). After a deformation at a strain rate of about $10^{-3}sec^{-1}$ in the structure of the monocrystal mainly parallelly arranged slip of multiple bands were to be observed. Probably there are also bands of deflection with only weakly visible slip lines in the primary system (Fig. 10a). In the monocrystal subjected to a compression test at a strain rate of about 10^{-2} sec^{-1} (for about 36 seconds) both straight and curved bands of multiple slip were to be observed (Fig. 10b). After compression at a strain rate of about $10^{-1}sec^{-1}$ (time of testing about 6 seconds) parallel and criss-crossing bands of multiple slip, as well as probably broad bands of deflection were detected (Fig. 11). The fragmentation of the bands of gliding prove the actuation and development of a secondary gliding in the band, which prevents a further deflection (Fig. 11a).

Basing on structural investigations it has been found that the temperature and strain rate influence considerably the metallographic effects of the cold work of CuZn30 monocrystals with the orientation $[\bar{1}\ 3\ 9]$. The analysed microstructure of

a)



b)

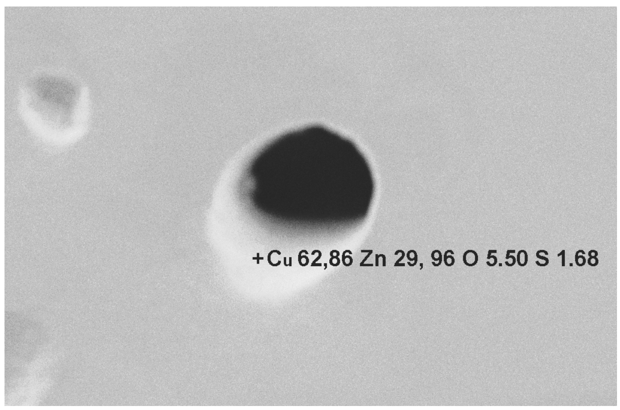


Fig. 7. Micropores occurring in a monocrystal of the alloy CuZn30 with the crystallographic orientation $[\bar{3}\ 4\ 4]$: a) numerical fraction of micropores, b) results of the local microanalysis of the chemical composition of the internal surface of a micropore

compressed monocrystals (Figs. 8-11) prove that the main mechanism of plastic deformation is the sliding, which consists in the shearing translation of the layers of the crystal in definite directions due to dislocations without any changes of the volume

a) b)

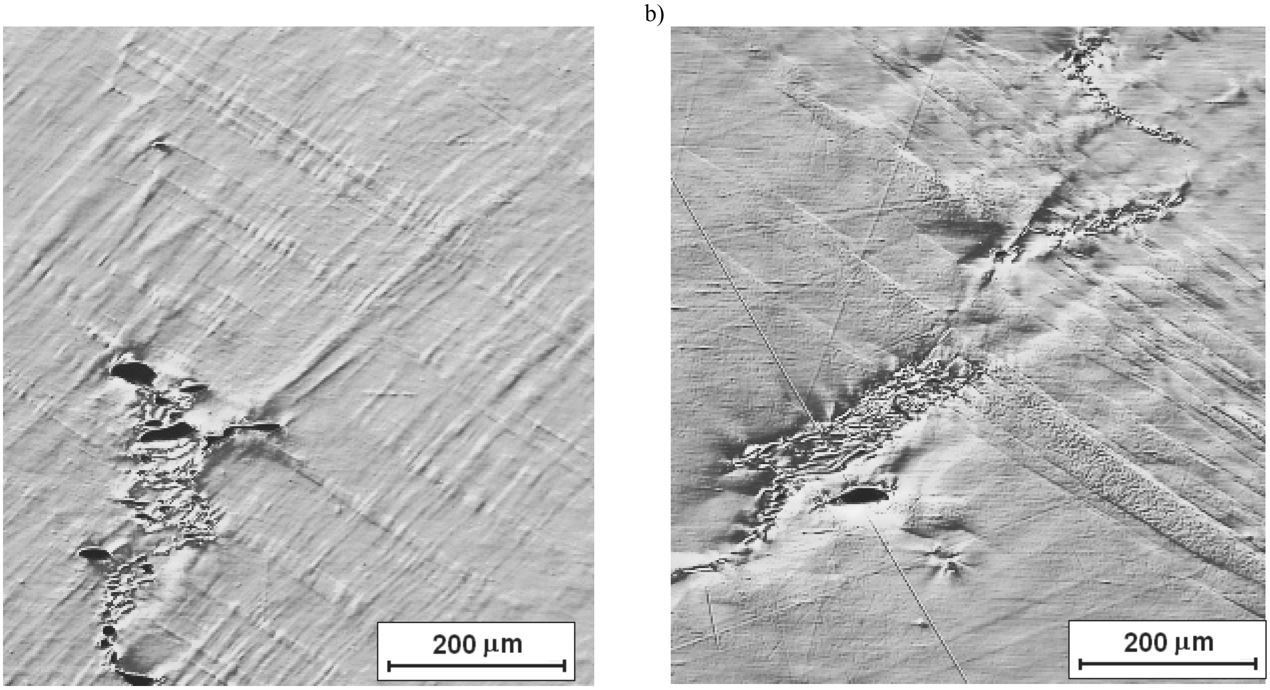


Fig. 8. Micropores and both parallel and criss-crossing bands of multiple slip on the longitudinal section of a CuZn30 monocrystal with the crystallographic orientation $[\bar{1}\ 3\ 9]$ after free compression with a draft of 50%: a) at 350°C and $\dot{\varepsilon} = 10^{-4}s^{-1}$, magnified 50x, b) at 400°C and $\dot{\varepsilon} = 10^{-4}s^{-1}$, magnified 200x

a) b)

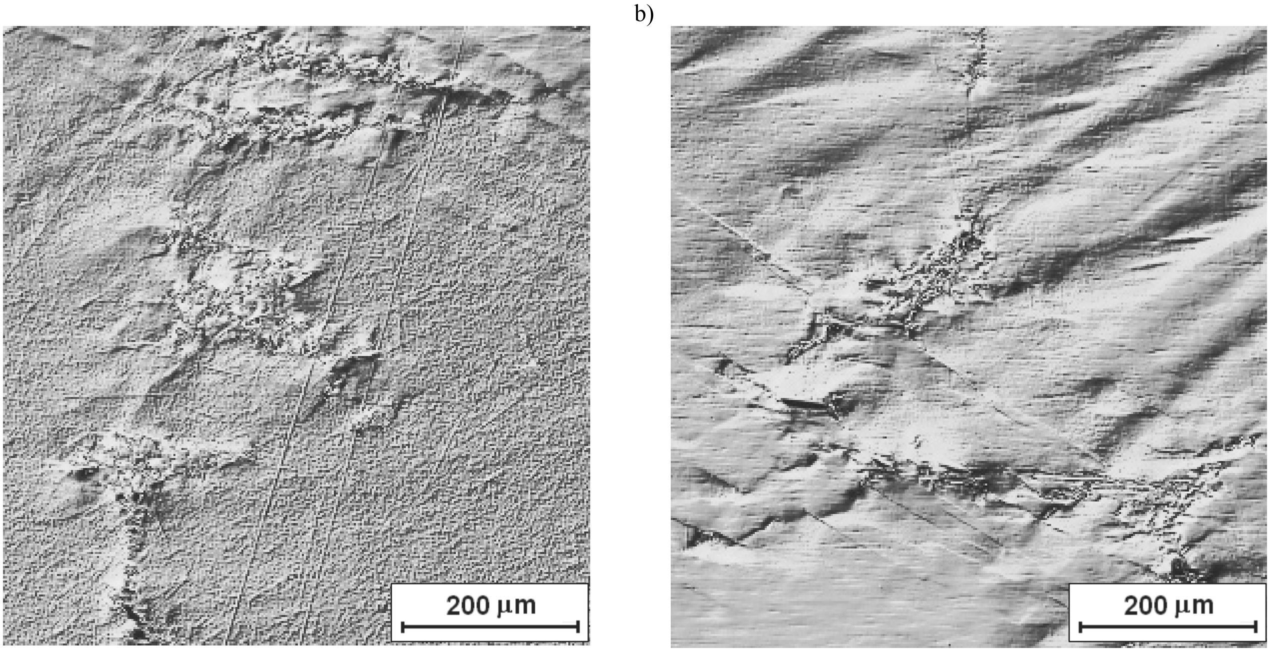


Fig. 9. Structure of a CuZn30 monocrystal with the crystallographic orientation $[\bar{1}\ 3\ 9]$ after deformation with a draft of 50 % at 400°C and $\dot{\varepsilon} = 10^{-4}s^{-1}$: a) recrystallized grains, magnified 200x, b) slip of multiple lines of deformation and microtwins, magnified 200x

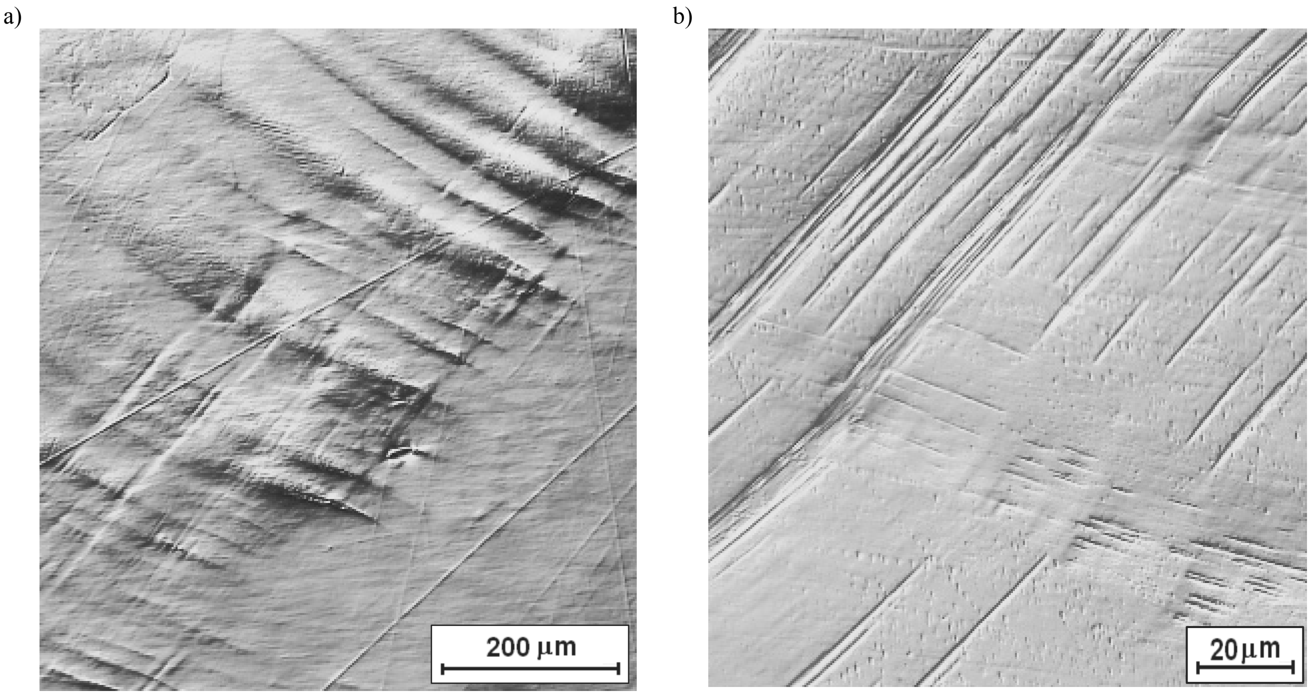


Fig. 10. Bands of deflection and criss-crossing slip of multiple lines in the microstructure of a CuZn30 monocrystal with the crystallographic orientation $[\bar{1}\ 3\ 9]$ after deformation with a draft of 50% at 400°C: a) when $\dot{\varepsilon}$ = $10^{-3}sec^{-1}$, magnified 200x, b) when $\dot{\varepsilon}$ = $10^{-2}sec^{-1}$ magnified 1000x

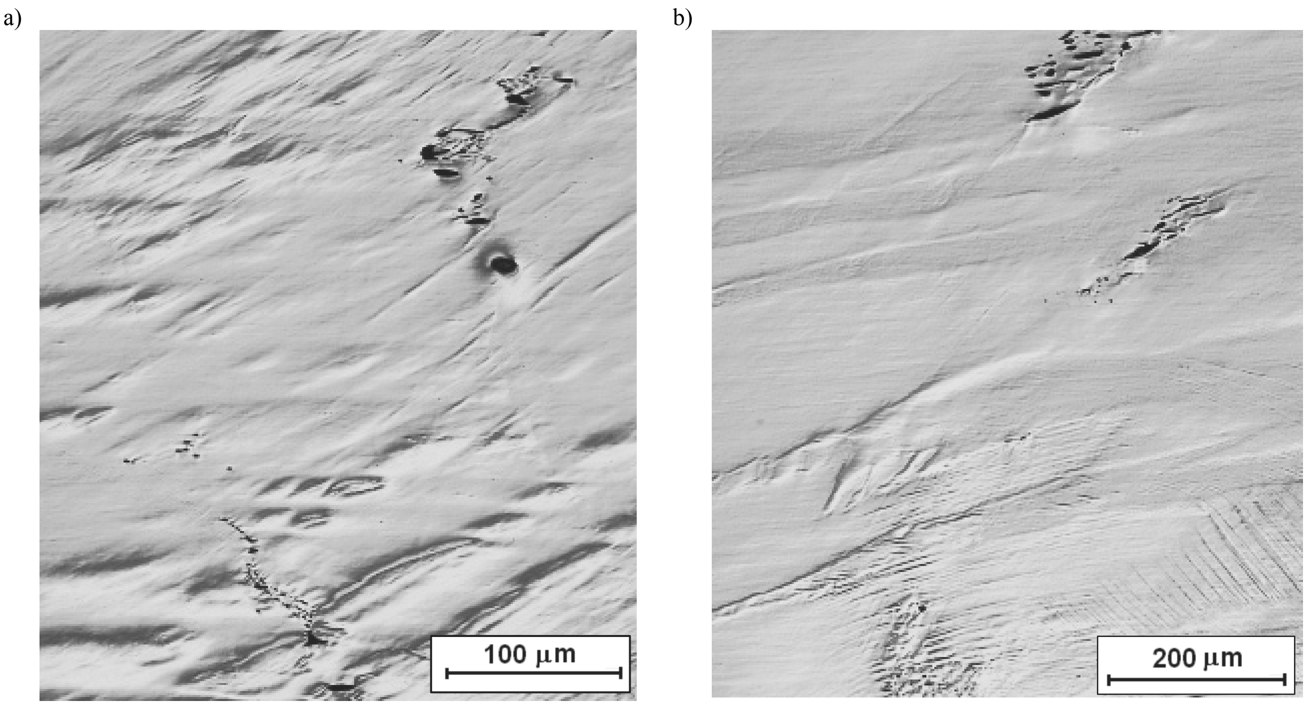


Fig. 11. Structure of a CuZn30 monocrystal with the crystallographic orientation $[\bar{1}\ 3\ 9]$ after deformation with a draft of 50 % at 400°C and $\dot{\varepsilon}$ = $10^{-1}s^{-1}$: a) bands of deflection and micropores, magnified 400x, b) local paths of shear and clusters of micropores, magnified 200x

and in the crystallographic orientation of the mutually displaced parts of the crystal. At elevated temperature the respective slip lines integrate forming clusters – slip bands, between which irregular effects of strain do occur. These phenomena indicate that sliding is a heterogenous process, because the plastic deformation takes place in the region of the paths of shear where dislocations may be extended to considerable distances.

4. Conclusions

Basing on these investigations the following conclusions may be drawn:

1. Bridgman's modified method permits to achieve CuZn30 monocrystals with selected crystallographic orientations, adequate for laboratory investigations concerning plastic deformation;
2. The revealed irregularities in the structure of monocrystals in the form of micropores result most probably from reactions of contaminations in the chemical composition of the alloy with oxygen from the crucible - the quartz capsule;
3. Monocrystals of the alloy CuZn30 with the orientations $[\bar{1}\ 3\ 9]$ display after their compression at a temperature of 400°C and a strain rate of $10^{-4} sec^{-1}$ a typical single-phase structure of the deformed α-solution with many slip lines and criss-crossed slip bands or microtwins;
4. In the zone of maximum microscopic strains of the sample occurs a structure of fine grains, probably resulting from the dynamic and static recrystallization in the course of hot deformation and cooling.

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