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MODES OF PLASTIC FLOW OF Zn-Cu SINGLE CRYSTALS COMPRESSED ALONG THE $[11\bar{2}0]$ DIRECTION

SPOSOBY PLASTYCZNEGO PŁYNIĘCIA KRYSZTAŁÓW Zn-Cu ŚCISKANYCH W KIERUNKU $[11\bar{2}0]$

Zinc single crystals with copper addition up to 1.5at% and orientation close to $[11\bar{2}0]$ were subjected to the compression test in the temperature range 293-673K and the initial strain rate $9\times10^{-4}\mathrm{s}^{-1}$. It has been found that — like in the case of pure zinc crystals — the deformation temperature determines the activity of the every of the four deformation mechanisms: slip in the 1st order pyramidal systems $\{10\bar{1}1\} < \bar{1}\bar{1}23 >$ (Py-1) and in the 2nd order pyramidal systems $\{11\bar{2}2\} < \bar{1}\bar{1}23 >$ (Py-2), slip in the Py-2 systems only, kink band (K.B.) formation and quasi-viscous plastic flow. It was also found that the addition of copper: i) strongly increases the flow stress; ii) activates the Py-1 systems; iii) drastically reduces the tendency toward macroscopically inhomogeneous deformation in K.B., and iv) favors quasi-viscous behavior of crystals at high temperatures. Instead, the alloying does not influence the mechanisms of kink band formation and quasi-viscous flow of tested crystals.

Kryształy cynku z dodatkiem miedzi do 1.5at% i orientacji bliskiej $[11\bar{2}0]$ poddano testowi ściskania w zakresie temperatur 293-673K z początkową prędkością odkształcenia $9\mathrm{x}10^{-4}\mathrm{s}^{-1}$. Stwierdzono, że - podobnie jak w przypadku kryształów czystego cynku — temperatura deformacji determinuje aktywność jednego z czterech mechanizmów deformacji, tzn. poślizgu w systemie piramidy 1-go rzędu $\{10\bar{1}1\} < \bar{1}\bar{1}23 >$ (Py-1) i w systemie piramidy 2-go rzędu $\{11\bar{2}2\} < \bar{1}\bar{1}23 >$ (Py-2); poślizgu jedynie w systemach Py-2; formowanie pasm ugięcia (K.B.) oraz zachowania quasi-lepkie. Wykazano ponadto, że dodatek miedzi: i) silnie podwyższa naprężenie płynięcia; ii) aktywizuje systemy Py-1; iii) drastycznie zmniejsza tendencję do makroskopowo niejednorodnej deformacji w pasmach ugięcia oraz iv) faworyzuje quasi-lepkie zachowanie kryształów w wysokich temperaturach. Stopowanie, natomiast, nie zmienia mechanizmów formowania pasm ugięcia ani quasi-lepkiego płynięcia badanych kryształów.

1. Introduction

Experimental results have shown that alloying leads to tendency toward inhomogeneous modes of plastic deformation (Portevin-LeChatelier phenomenon, Lüders deformation), irrespective of the type of the crystallographic lattice and the kind of the alloy addition. A similar tendency can be observed in a certain range of deformation temperature in alloyed zinc single crystals deformed in the basal system („jerky" flow pattern of stress — strain curve) [1-5].

Some information about the influence of alloying on the non-basal slip systems activity in zinc crystals tested in tension can be found in [6,7]. Small addition of cadmium, aluminum and copper causes increase of the flow stress in non-basal systems only and does not influence the change of deformation mechanism, i.e. twinning / quasi-viscous behavior of zinc crystals.

In the case of compressed pure zinc single crystals with orientations lying on the $[11\bar{2}0]$ – $[10\bar{1}0]$ line of stereographic triangle four mechanisms contribute to plastic deformation. They are as follow: i) slip in the 1st order pyramidal systems $\{10\bar{1}1\} < \bar{1}\bar{1}23 >$ (Py-1) and the 2nd order pyramidal systems $\{11\bar{2}2\} < \bar{1}\bar{1}23 >$ (Py-2), ii) homogeneous slip in the Py-2 systems only, iii) mechanically unstable, localized deformation in kink band (K.B.) and iv) double slip in prismatic and (coarse) basal systems $\{10\bar{1}0\} < 11\bar{2}0 >$ (Pr) and $(0001) < 11\bar{2}0 >$ (Ba), respectively [8-10]. It has been found that the latter correlates with the quasi-viscous mode of plastic flow at high temperatures whereas mechanical behavior of crystals tested at lower temperatures — in particular the

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occurrence of the steady state flow, work softening or drastic mechanical instability — is strongly influenced by the K.B. dimension. Moreover, a mechanism of K.B. nucleation, based on coordinated movement of atoms along the planes close to the prismatic ones (of the 1st or the 2nd order) was also proposed [9].

The question which appears still open is how the alloying influences these mechanisms. To form a comprehensive view of the problem, systematic studies of the influence of copper addition on the deformation mechanisms of zinc single crystals were undertaken.

2. Experimental

Cylindrical samples (∅6mm × 9mm) of zinc crystals (99.995%) and zinc crystals with copper addition in the amount of (in at. %): 0.1, 0.2, 0.5, 0.9 and 1.5 were used in the experiment. Crystals of the initial orientation close to $[11\bar{2}0]$ ($\chi_0 = 90°$, $\lambda_0 = 3°$; where χ_0 , λ_0 — the angle between the crystal axis and the directions [0001] and $< 11\bar{2}0 >$, respectively) were obtained by Bridgman method at the growth rate of 0.85cm/h. The crystals were compressed up to 10% of the height reduction at the initial strain rate of $9 \times 10^{-4}s^{-1}$, within the temperature range from 293K to 673K. The flaked graphite was used as a lubricant and at least three specimens were tested at each deformation conditions. The analysis of deformation behavior of ZnCu crystals was performed on the basis of mechanical characteristics and slip marking observations on crystal faces parallel to the (0001) and close to $\{10\bar{1}0\}$ crystallographic planes.

3. Results and discussion

3.1. Mechanical characteristics and macrostructure

Basing upon the analysis of the compression curves and observations of macrostructure of crystals, one may conclude that the copper addition increases the flow stress and, especially in the temperature range 523-623K, strongly influences the plastic behavior of crystals (Figs. 1, 2).

At low temperature 293-473K, the mechanical characteristics of whole crystals has a two-fold course. The initial work hardening of crystals (ranging up to 5% of deformation at 293K and 1% at 473K) follows the flow stress stability or, at higher temperatures, the work softening of crystals (Fig. 1). It is worthy of note that the crystals with higher copper content begin to work soften already at 373K and the work softening of pure zinc crystals compressed at the 473K is associated with a „smooth" formation of macroscopic K.B.. Copper addition is responsible for the different shape instability, i.e. buckling of pure zinc crystals and obliquity of alloyed crystals (Fig. 2).

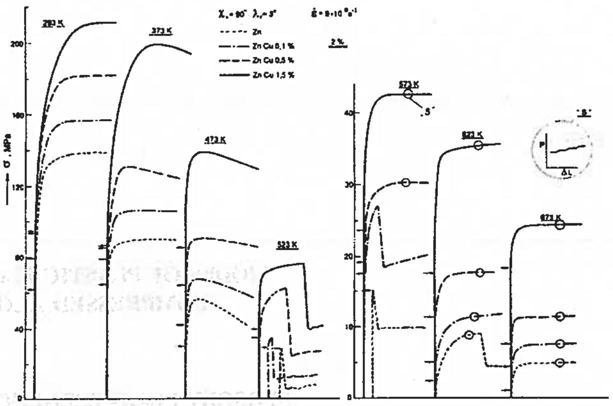

Fig. 1. Compression curves of Zn-Cu single crystals

Beginning with the temperature 523K, formation of macroscopic K.B. and localized basal slip within them make significant contribution to plastic flow of crystals. However, a more important observation is that the K.B. nucleation is followed by restored strain hardening of crystals (as in the case of ZnCu0.5-1.5% at 523K and of pure Zn at 623K) or it takes place in the „elastic" range (as in Zn crystals at 523-573K) (Figs. 1, 2). The formation of first macroscopic K.B. is accompanied by large load drop (up to 70% reduction of load) and deformation is then continued at low stresses by the formation of „new" K.B. (propagation of the front of K.B.), and basal slip in „old" K.B. (Figs. 1, 2, 3f). In consequence, compressed crystals assume finally a „mushroom" shape (Fig. 2).

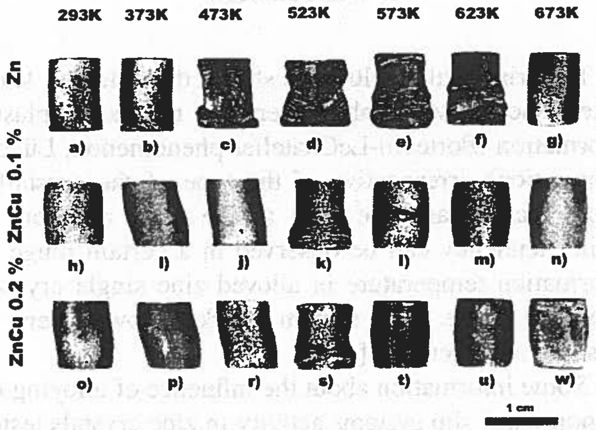

Fig. 2. Macrostructure of compressed Zn-Cu single crystals

Alloying causes drastic suppression of the tendency for K.B. formation. In crystals with higher copper concentration (0.2% and above) macro-localization of deformation in K.B. takes place only at 523K, whereas in pure zinc — in a wide range of temperature up to 623K (Fig. 2). In the first ones the K.B. are small (see for example Fig. 2l), whereas the thickness of plate-like K.B. in pure zinc attains 1mm [9].

High-temperature deformation of the highly-alloyed crystals (above 523K) and pure zinc crystals (above 623K) occurs almost without strain hardening (except of initial strain ranging up to about 1-2% of strain) and is accompanied by high strain rate sensitivity (the value of $m = d\ln\sigma/d\ln\dot{\varepsilon}$ exceeds 0.2). No sample shape instability is observed during this quasi-viscous deformation (only slight tendency toward „barrelling"), although the compression curves show a „step-like" character suggesting inhomogeneity of plastic deformation (Figs. 1, 2).

Such an experimental patterns of the behavior of crystals during plastic straining drive to the question about the mechanisms responsible for work hardening, the steady state flow and / or the work softening of crystals at lower temperature, especially if these effects are apparent (caused by shape instability) or are a „secondary" ones resulting from the activity of an additional („new") deformation mechanism, leading in turn to the shape instability. Moreover, it is also important to show whether the mechanisms of K.B. formation and of quasi-viscous flow are influenced by copper addition. The attempt to get the answer to these questions was a subject of microscopic observations. The results of them was summarized below.

3.2. Pyramidal slip systems

Slip marking observations of crystals compressed at temperatures up to the 473K lead to the four main conclusions:

- all alloyed crystals — similarly as pure zinc crystals — become deformed plastically already at the early pre-yielding stage, well below the „macroscopic yield stress" (MYS, marked by arrows in Fig. 1), i. e. the stress value at which the load / strain curve loses the linearity;
- the deformation may result from slip in three systems: Py-1, Py-2 and Ba, and formation of small K. B. (Figs. 3a-e, 4);
- the pre-yielding and the further plastic stage of compressed crystals proceed by essentially different mechanisms, especially the activity of the Py-1 systems is strongly influenced by deformation temperature and by copper content, and
- the instability of crystals shape originates in heterogeneous distribution of Py-2 slip, which in pure and low alloyed crystals is accompanied by formation of small K.B., nucleated at the areas of contact with the anvils.

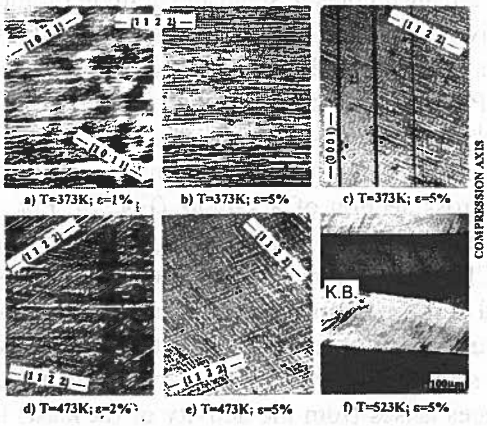

Fig. 3. Slip traces on (0001) (a, b) and on ~ {10$\bar{1}$0} (c-f) planes of ZnCu1.5% single crystals

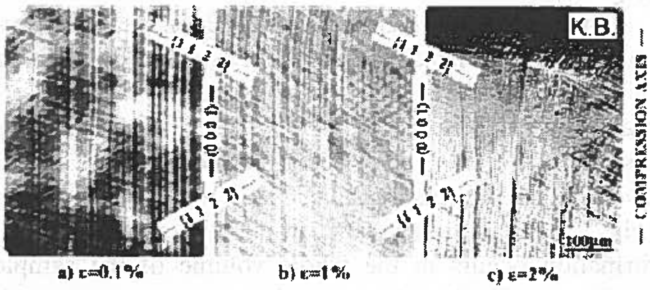

Fig. 4. Formation of small kink bands in the „dead" areas of compressed zinc single crystals

In a certain range of temperature, the plastic flow is initiated simultaneously in Py-1 and Py-2 systems. In pure zinc crystals the weak activity of Py-1 slip systems was observed as a pre-yielding behavior only in the narrow range of temperature 293-333K [8]. The copper addition considerably activates the Py-1 systems, and in ZnCu1.5% the nearly homogeneous distribution of the Py-1 slip traces can be observed after, at least, 1% of total strain of crystals at 473K (Fig. 3a). Thus the interaction of two types of the pyramidal systems is responsible for the strong work hardening, both in the pre-yielding and at the beginning of plastic stage of compressed highly-alloyed crystals (Fig. 1). The enhanced activity of Py-1 systems in alloyed crystals — as it seems — is mainly connected with the drastic increase in pyramidal dislocations density in the „as grown" crystals (for example, by two orders of magnitude in the ZnCu0.3% crystals with respect to the pure zinc crystals [11]). It can not be also excluded that the activity of the Py-1 systems in Zn-Cu crystals, especially in these with copper content exceeding the solid solubility limit (0.3% at 373K, 0.9% at 473K and 1.6% at 573K) may be associated with the formation of clusters of copper atoms, supersaturated solid solution or precipitations of the hexagonal ε-Zn_3Cu (Zn_4Cu) phase [12].

At advanced plastic stage and at higher temperatures the activity of the Py-1 systems disappears and the Py-2 systems become the dominant deformation systems (Fig. 3b-e). Plastic behavior of crystals is then controlled by the mechanisms activated near the anvils. In pure zinc crystals small K.B. are formed, which penetrate only part of the cross section of a sample (Fig. 2). Fig. 4 shows the sequence of slip events leading to the formation of such K.B. in zinc crystals with orientation $[10\bar{1}0]$, compressed at room temperature; this is a case when crystals work soften just at about 1% of strain [9]. At the early plastic stage the nearly homogeneous distribution of the slip traces arises from the activity of (at least) two Py-2 systems and the Ba system (Fig. 4a). With increasing strain, however, one of the Py-2 systems — a different one at each of anvil — becomes the dominant slip system (Fig. 4b).

In the „dead" areas of crystals deformation is initially accommodated by Ba slip, causing local buckling of basal planes (Fig. 4b), and subsequently also by formation of small K.B. (Fig. 4c). Then, the heterogeneous distribution of Py-2 slip, accompanied by K.B. formation and Ba slip inside them, leads to crystal buckling (Fig. 2). On the other hand, in alloyed crystals the deformation occurs in the whole volume of the sample by the two Py-2 systems (conjugate or un-conjugate), one of them distinctly prevailing over the other (Fig. 3e) and the crystals undergo gradual obliquity (Fig. 2). It seems that the lack of small K.B. in these crystals results from the strong work hardening in pyramidal systems, which „shifts" the nucleation of K.B. to higher stresses (the higher ones the lower deformation temperature and higher strain rate [9]).

3.3. Macroscopic kink bands and quasi-viscous flow

Apart from the chemical composition of the tested crystals, the dominant role of pyramidal systems ends at the 473K and at higher temperatures the one- or two-fold changes (depending on copper addition) of the dominant deformation mechanism take place. In crystals containing at least 0.5%Cu the transition from the pyramidal systems activity to the quasi-viscous flow begins just at about 523K, but still at this temperature the K.B. nucleation prevents the crystal to attain the state of steady flow (Fig. 1). On the other hand, in pure and low-alloyed (0.1%Cu) zinc crystals, formation of macroscopic K.B. dominates in the range of temperature 523-573K (K.B. nucleates in the „elastic" or at the beginning of the plastic stage) and the transition to the quasi-viscous behavior occurs at a much higher temperature (623K) (Fig. 1).

This means that alloying disturbs the sequence of change of deformation mechanisms with temperature and favors the quasi-viscous behavior of zinc crystals at the expense of K.B. formation (Figs. 1, 2). In order to explain this behavior one can recall the results of experiments described in [9]. As it was shown in pure zinc crystals with low density of dislocation, K.B. nucleate in „elastic" stage at rather low stresses (9MPa and 20-25MPa at 623K and 77K, respectively). However, when K.B. formation is preceded by plastic deformation, dislocation substructure make the nucleation of K.B. difficult and the K.B. nucleation stress is highly temperature and strain rate dependent. Then, it seems that the observed strong suppression of macro-K.B. formation in alloyed zinc crystals — similarly as in the case of small K.B. — results from the combined effect of the increased initial dislocation density and the additional accumulation of dislocations during preceding plastic deformation.

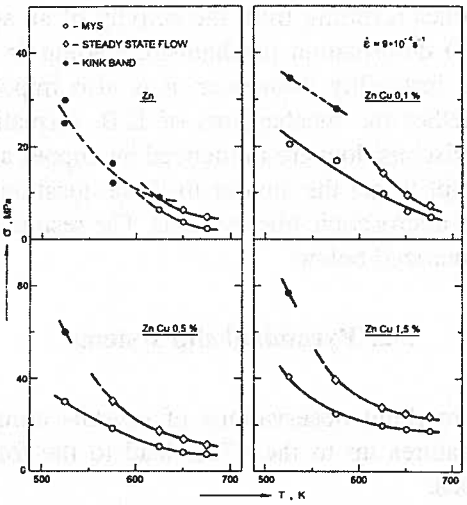

Fig. 5. Temperature dependence of kink band nucleation stress and of yield stress, and steady state flow stress of copper alloyed zinc single crystals

Moreover, the alloying also changes the mutual relations between the K.B. nucleation stress, the macroscopic yield stress, and the steady state flow stress. Fig. 5 shows that the ratio: K.B. nucleation stress / MYS equals ~ 1.3 for pure Zn at 623K and ~ 1.7 for ZnCu0.1% at 523-573K, and raises up to ~ 2 for highly alloyed crystals.

The mechanical and the structural evidences indicate that alloying does not change the mechanisms of K.B. formation neither the quasi-viscous flow, discussed in detail for pure zinc crystals in [9,10].

There are several features indicating that in tested crystals the K.B. results from the lattice shearing occurring along the planes nearly parallel to the 1st order prismatic planes, in particular:

- the K.B. formation is always accompanied by a distinct acoustic effect, which means that the process occurs at the rate of elastic wave;
- the initial traces of the K.B. / matrix interface, seen on the $(10\bar{1}0)$ plane, are inclined by only a few (5-8°) degrees to the [0001] crystallographic direction (Fig. 2);
- the deformation associated with K.B. formation has a plane strain character (no strain component in the direction perpendicular to the plane containing the [0001] crystallographic direction and compression axis has been noticed) (Fig. 2);
- in the areas of K.B. only the traces of basal slip are observed (Fig. 3f), and
- the load drops are rather independent of the size of formed macro-K.B. (Fig. 1).

The evident decrease in size of the first formed K.B. with the increase of copper addition (Fig. 2l) may be associated with the properties of the Ba slip occurring in the structure with increased dislocation density. As it was shown in [9], the K.B. formation causes the step-like reorientation of the crystallographic lattice (Fig. 3f), and thus leads to the increase (up to ~ 0.1) of the Schmid orientation factor for the Ba system. The activity of this slip system is then „driven" by the effect of „geometrical softening", yet the greater density of obstacles results in the shortening of the basal slip distance making difficult the basal slip in K.B., and, in consequence, as observed in alloyed crystals, the size of K.B. decreases.

The results of topological (slip traces) observations, the mode of changes of the cross-section of samples, shape stability and the „step-like" course of load-deformation characteristics of alloyed zinc crystals compressed at high temperatures prove that the mechanism of quasi-viscous flow is similar to that occurring in pure zinc crystals [9,10]. It is based on the activity of the prismatic 1st order systems $\{10\bar{1}0\} < 11\bar{2}0 >$ (and / or pyramidal 1st order systems $\{10\bar{1}1\} < 11\bar{2}0 >$) accompanied by the basal slip. Slip in non-basal systems leads to strain hardening of crystals at the initial stage of deformation (the higher the lower the deformation temperature), while the strain hardening diminishes when the deformation in the basal system begins to localized in the coarse bands (the localization in meso-scale). This behavior would be responsible for mechanical instability of compressed crystals.

4. Conclusions

Deformation temperature (in the range 293-673K) and copper addition (up to 1.5at%) influence the mechanisms of deformation of zinc single crystals compressed along the direction close to $[11\bar{2}0]$.

1. The deformation temperature determines the activity of the every of the four deformation mechanisms: slip in the 1st order pyramidal systems $\{10\bar{1}1\} < \bar{1}\bar{1}23 >$ (Py-1) and in the 2nd order pyramidal systems $\{11\bar{2}2\} < \bar{1}\bar{1}23 >$ (Py-2), slip in the Py-2 systems only, kink band (K.B.) formation and quasi-viscous behavior.
2. The copper addition: strongly activates the 1st order pyramidal systems accompanying the 2nd order pyramidal systems, drastically reduces the tendency toward formation of small kink bands at lower temperature and — at high temperature — favors tendency toward quasi-viscous flow at the expense of macroscopic kink band formation.
3. Slip in the 1st order pyramidal systems is active at the initial stage of deformation. The domination of the 2nd order pyramidal system leads to shape instability (buckling or obliquity) of specimens and results in the effect of steady state flow or „work softening" of crystals.
4. Localization of deformation (in meso-scale) in coarse basal slip bands in crystals deformed by the 1st order prismatic systems is responsible for quasi-viscous behaviour of crystals at high temperature and for the mechanical instability of crystals („step-like" course of compression curves).
5. The experimental evidences show that the mechanisms of kink band formation and quasi-viscous flow are not influenced by the copper alloying of zinc crystals.

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