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## DIFFERENCES IN MICROSTRUCTURE AND TEXTURE DEVELOPMENT DURING DEFORMATION AND RECRYSTALLIZATION OF COPPER AND ALUMINIUM (110)[001] SINGLE CRYSTALS

### RÓŻNICE W MIKROSTRUKTURZE I TEKSTURZE ODKSZTAŁCONYCH ORAZ REKRYSTALIZOWANYCH MONOKRYSTAŁÓW MIEDZI I ALUMINIUM O ORIENTACJI (110)[001]

Cu and Al single crystals with the orientation (110)[001] were cold rolled and subsequently channel die compressed (CDC). The new direction of plastic flow during CDC was parallel to the former transverse rolling direction and the compressed surface was parallel to the rolling plane. The microstructure after rolling was uniform in both materials. Shear bands were formed in Cu during the CDC, while any forms of localized structural inhomogeneities were not observed in Al. In case of Al the CDC brought about only a rotation of the initial orientation around the normal direction (ND) up to about 14°.

In the case of Cu, recrystallization commenced within shear bands and proceeded by a growth of selected grains into the deformed matrix. The local crystallographic orientation measurements revealed that the oriented growth of grains played an important role in the formation of recrystallization textures. Orientations of the fastest growing grains could be obtained from the orientations found in the deformed material by their rotation around the common  $\langle 111 \rangle$  pole by about 30° or 50°.

Distribution of earliest recrystallized grains in Al was essentially random and after completion of recrystallization the grains were much larger than in Cu. The measurements of the {111} pole figures indicated that recrystallization textures of both materials were formed according to the oriented growth theory.

*Keywords:* microstructure, texture, deformation, recrystallization

Monokrystały Cu i Al o orientacji (110)[001] walcowano, a następnie ściskano w matrycy z kanałem. Podczas ściskania nowy kierunek plastycznego płynięcia był równoległy do kierunku poprzecznego podczas walcowania, zaś ściskana płaszczyzna była równoległa do płaszczyzny walcowania. Po przewalcowaniu monokrystały charakteryzowały się stosunkowo jednorodną mikrostrukturą. Podczas odkształcenia nadawanego po zmianie kierunku plastycznego płynięcia w Cu tworzyły się pasma ścinania, zaś nie tworzyły się w analogicznie odkształcanych monokrystałach Al. W przypadku Al ściskanie w matrycy z kanałem powodowało jedynie obrót orientacji wokół KN (kąąt obrotu orientacji zwiększał się z odkształceniem do około 14°).

Podczas rekrystalizacji Cu pierwsze ziarna tworzyły się w pasmach ścinania i one decydowały o postępie rekrystalizacji. Pomiar lokalnych orientacji ziaren utworzonych podczas rekrystalizacji wykazały, że tworzenie tekstury rekrystalizacji zachodzi zgodnie z teorią uprzywilejowanego wzrostu. Orientacje krystalograficzne najszybciej rosnących ziaren można było otrzymać przez obrót orientacji odkształconej osnowy wokół wspólnego bieguna  $\langle 111 \rangle$  o kąty ok. 30° i 50°. Rozmieszczenie pierwszych zrekrystalizowanych ziaren w Al było względnie losowe, a po zakończeniu rekrystalizacji ziarna były znacznie większe niż w Cu. Pomiar figur biegunowych {111} wykazały, że tekstury rekrystalizacji w Cu i Al tworzone były zgodnie z teorią uprzywilejowanego wzrostu.

### 1. Introduction

The microstructure and texture development during deformation with a change in the direction of plastic flow plays an important role in an improvement of material properties [1, 2]. The change of direction of plastic flow can have a significant influence on the deformation behavior, recrystallization, grain size after recrystallization and crystallographic texture. The investigation of these processes in single crystals is much easier than in polycrystals [3]. Therefore, it is important to properly

analyze the deformed microstructure and texture in single crystals in order to understand their effect upon the recrystallization microstructure and texture.

The stacking fault energy (SFE) in f.c.c. metals has an important influence on their deformation behaviour. So, the recrystallization behaviour during annealing of materials with different SFE may be significantly different because the nucleation of recrystallized grains is supposed to be dependent on the deformation microstructure.

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The goal of the present study is to characterise the influence of SFE and strain path on the microstructure and texture development during deformation and recrystallization of f.c.c. single crystals with the orientation  $(110)[001]$ . Two single crystals were investigated: one Cu (SFE about  $40 \text{ mJ}\cdot\text{m}^{-2}$ ) and the other one Al (SFE about  $240 \text{ mJ}\cdot\text{m}^{-2}$ ). Both single crystals were deformed by the same way, and their microstructure and texture evolution during deformation and recrystallization have been investigated in parallel.

## 2. Material and experimental procedure

The investigation was performed on the Cu and Al single crystals with the  $(110)[001]$  orientation. Specimens were uniformly rolled with the strain 0.5 at room temperature and subsequently CDC. Samples for CDC were cut off from the rolled bars in such a way that the new direction of plastic flow was parallel to the former transverse rolling direction and the compressed surface was parallel to the rolling plane (Fig. 1). Like during rolling, for the CDC the direction of plastic flow was designated as RD2, the direction of compression as ND and the direction perpendicular to the channel die side walls as TD2. Friction between the specimen and the channel walls was reduced by wrapping the specimens with Teflon tape. The rectangular-wall specimens fitted the channel die size. Before compression, surfaces of each specimen were carefully polished. This let further investigation of deformation traces by means of a light microscope (LM). Microstructure of specimens after selected amounts of deformation was also investigated by JEM-100C transmission electron microscope (TEM).

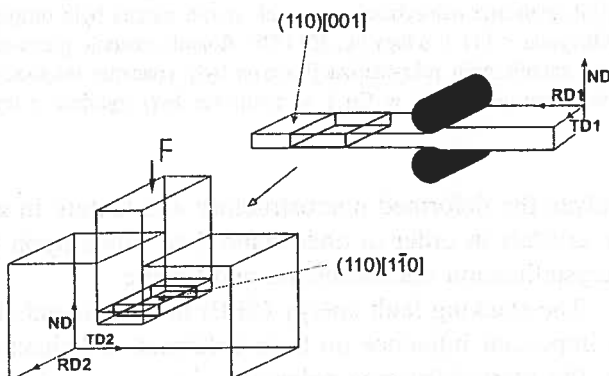


Fig. 1. The deformation scheme

Isochronal (1 hour) recrystallization annealing was carried out on specimens with a total true strain 1.0 (of 0.5 by rolling and 0.5 by CDC). The temperature stabilization was kept with an accuracy of at least  $\pm 2^\circ\text{C}$ .

The volume fraction of the recrystallized material for a given temperature was calculated by the point method.

The crystallographic orientations of recovered matrix and some grains after recrystallization were measured using Philips CM-20 electron microscope by means of a convergent beam electron diffraction (CBED) method.

The  $\{111\}$  pole figures were measured from the half thickness of the selected deformed samples as well as from the fully recrystallized ones. The measurements were performed on planes perpendicular to the ND by means of the Schulz back reflection technique using a Philips X'Pert diffractometer furnished with a textural goniometer ATC-3.

## 3. Results and discussion

### 3.1. Microstructure and texture after rolling

The microstructure of Cu and Al single crystals after rolling was relatively homogenous and very similar to each other. LM observation of free surfaces perpendicular to the TD1 revealed uniformly distributed lines which were angled to the rolling direction (RD1) at  $35^\circ$  or  $-35^\circ$ . In the case of the  $(110)[001]$  orientation, such angles are between the RD and the traces of  $(111)$  and  $(1\bar{1}\bar{1})$  planes.

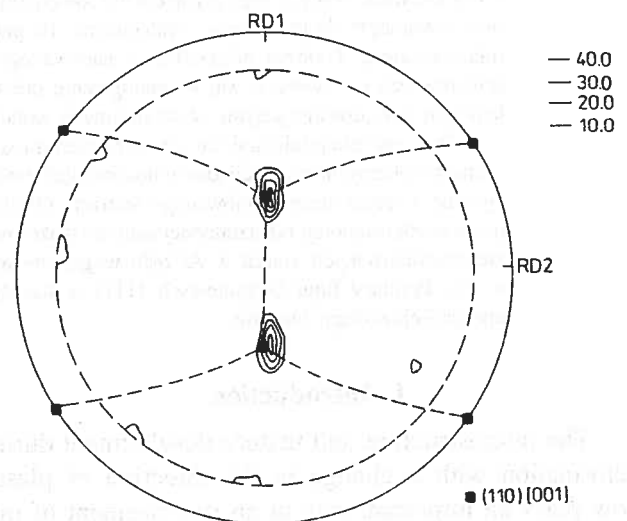


Fig. 2.  $\{111\}$  pole figure of the Al single crystal with strain 0.5

The main texture component of the rolled specimen did not show any significant difference from the initial orientation. Typical  $\{111\}$  pole figure of the rolled Al specimen is shown in Fig. 2. During homogeneous rolling of single crystals with the

(110)[001] orientation, the deformation proceeded by slip in four systems:  $(111)[01\bar{1}]$ ,  $(111)[10\bar{1}]$ ,  $(1\bar{1}\bar{1})[101]$  and  $(1\bar{1}\bar{1})[011]$ . Equal shears in these systems offset rotations of the crystallographic lattice associated with deformation in the particular system.

### 3.2. Changes in microstructure and texture during CDC

#### Cu

The  $\{111\}$  pole figure for the sample deformed to the strain of 0.5 by rolling followed by the strain of 0.5 applied by the CDC showed a strong  $(110)[5\bar{5}1]$  component<sup>1)</sup> (Fig. 3). The orientation could be transformed to the  $(110)[1\bar{1}0]$  orientation (the initial orientation for compressed samples) by its rotation around the ND by  $8^\circ$ .

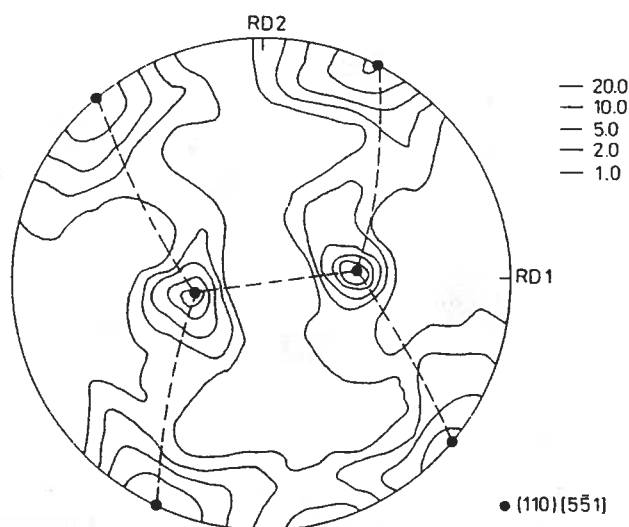


Fig. 3.  $\{111\}$  pole figure of the Cu single crystal with the initial strain of 0.5 applied by rolling followed by the strain of 0.5 applied by the CDC

The change in the direction of plastic flow brought about a significant change in microstructure. Slip lines produced on specimens' side walls perpendicular to the TD2 were composed of short segments perpendicular or parallel to the RD2 (Fig. 4a). This indicates that the segments of lines were parallel to the traces of  $(111)$  and  $(1\bar{1}\bar{1})$  or  $(\bar{1}\bar{1}1)$  and  $(1\bar{1}1)$  planes, respectively. On surfaces perpendicular to the ND, slip lines angled at  $55^\circ$  and  $-55^\circ$  to the RD2 as well as lines parallel to the RD2 were exposed (Fig. 4b). This might suggest that the inclined lines were parallel to the traces of  $(\bar{1}\bar{1}1)$  and  $(1\bar{1}1)$  planes while those parallel to the RD2 were parallel to the traces of  $(111)$  and  $(1\bar{1}\bar{1})$  planes. The above results

comply with the Taylor-Bishop-Hill theory according to which during the CDC of the  $(110)[1\bar{1}0]$  single crystal, the deformation may be accomplished by slip in eight systems:  $(111)[0\bar{1}1]$ ,  $(111)[\bar{1}01]$ ,  $(\bar{1}\bar{1}\bar{1})[011]$ ,  $(\bar{1}\bar{1}\bar{1})[101]$ ,  $(\bar{1}\bar{1}\bar{1})[\bar{1}0\bar{1}]$ ,  $(\bar{1}\bar{1}\bar{1})[01\bar{1}]$ ,  $(1\bar{1}\bar{1})[0\bar{1}\bar{1}]$  and  $(1\bar{1}\bar{1})[10\bar{1}]$ .

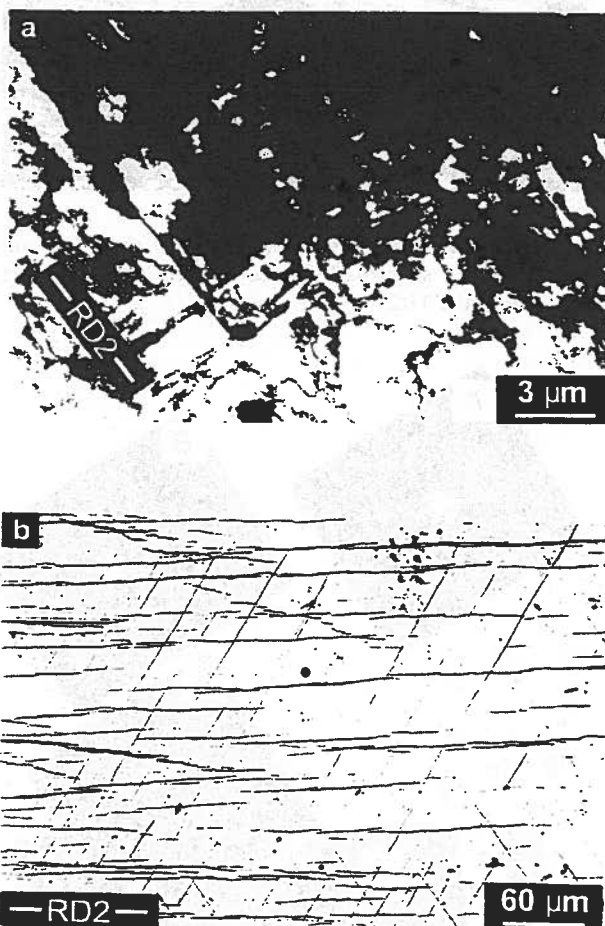


Fig. 4. Deformation markings in the Cu single crystal deformed by rolling with strain 0.5 followed by CDC: a) strain by CDC 0.12, TEM, foil perpendicular to the TD2; b) strain by CDC 0.01, LM, surface perpendicular to the ND

First shear bands were observed by the naked eye after the 0.04 strain applied by CDC. They ran across the whole thickness of the deformed sample. The shear bands formed two families mutually symmetrical with regard to the direction of plastic flow (Fig. 5). The density of shear bands increased with the increase of the amount of deformation. The first shear bands found on the surface perpendicular to the TD2 were inclined to the RD2 at angles near to  $30^\circ$  and  $-30^\circ$  and were almost parallel to the traces of  $\{100\}$  planes.

The TEM investigation of thin foils revealed the occurrence of narrow and broad shear bands. The narrow shear bands had a typical microstructure built

<sup>1)</sup> The orientations were always determined in relation to the instant direction of plastic flow.



Fig. 5. Shear bands in the Cu with true strain 0.5 applied by rolling and with additional strain 0.25 applied by the CDC, LM. Surface perpendicular to the TD2

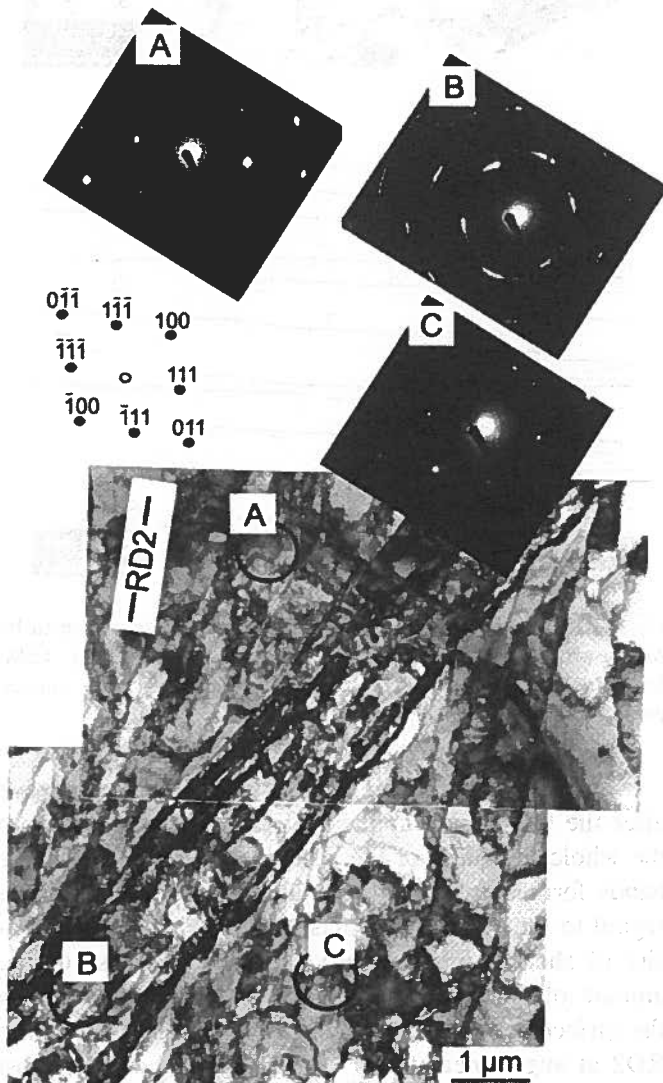


Fig. 6. Narrow shear band in the Cu with strain 0.5 applied by rolling and with additional strain 0.5 applied by the CDC, TEM

of microbands or highly elongated cells/subgrains (copper-type shear bands) (Fig. 6). The broad shear

bands were built of recovered, elongated subgrains/cells (alloy type shear bands). Such microstructure differed from the typical microstructure of the shear bands found in copper single crystals deformed without any change in the direction of plastic flow. Usually, shear bands in copper constitute clusters of microbands [4]. The similar microstructure of shear bands was also observed previously in the deformed copper single crystals with the  $(\bar{1}13)[741]$  initial orientation [5].

#### Al

Texture of the sample deformed to the total strain of 1 (rolling to the 0.5 followed by the CDC to the 0.5 strain) was relatively sharp but the range of spreading of the main component  $(110)[\bar{3}31]$  was larger than for samples subjected to rolling alone (Fig. 7). This orientation is turned by about  $14^\circ$  around the ND in relation to the  $(110)[\bar{1}10]$  orientation.

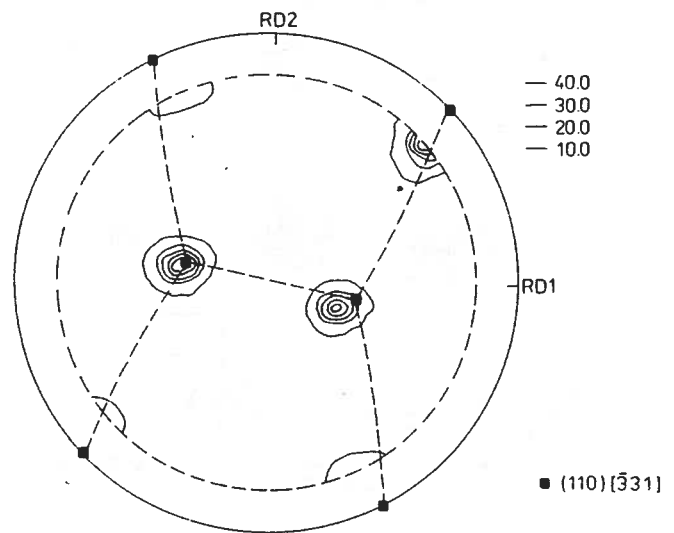


Fig. 7.  $(111)$  pole figure of the Al single crystal with the initial strain of 0.5 applied by rolling followed by the strain of 0.5 applied by the CDC

After the modest amount of deformation (0.04) by the CDC, the slip lines observed on surfaces perpendicular to TD2 were approximately parallel or perpendicular to the RD2 (Fig. 8a). This suggests that these lines are parallel to the traces of the  $(111)$ ,  $(11\bar{1})$ ,  $(\bar{1}11)$  or  $(\bar{1}\bar{1}1)$  planes. Thus, for small amounts of deformation applied by the CDC, deformation occurs by slip in eight systems, the identical ones as in Cu. After larger amounts of deformation (above 0.2) applied by the CDC, slip lines were angled to the RD2 at about  $22 \div 28^\circ$  and  $-(22 \div 28^\circ)$  (Fig. 8b). The steady inclination of slip lines on the surface perpendicular to the TD2 (strains larger than 0.2 applied in compression) may result from the

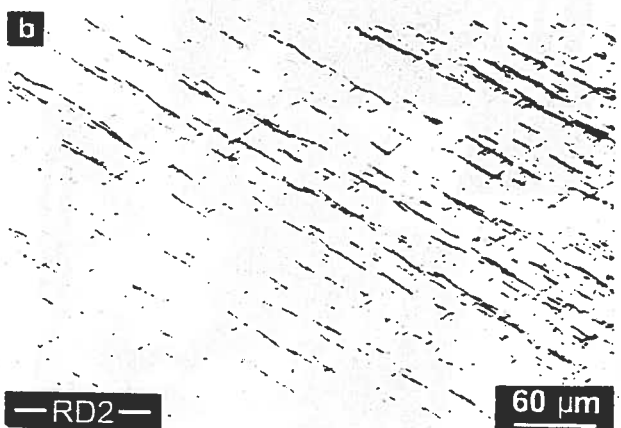
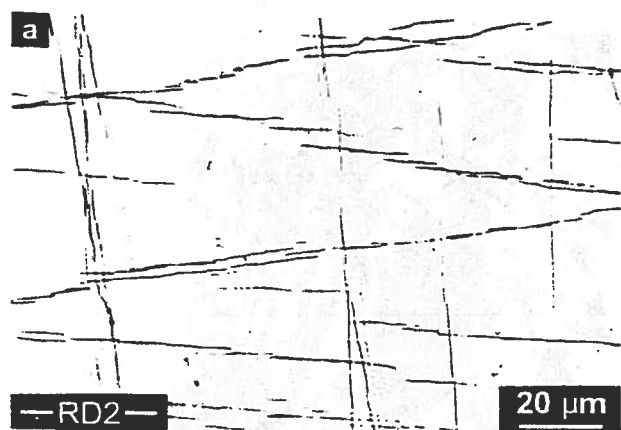


Fig. 8. Microstructure of the deformed Al single crystals, surface perpendicular to the TD2: a) strain 0.5 by rolling and 0.04 by CDC; b) strain 0.5 by rolling and 0.5 by CDC



Fig. 9. Dislocation microstructure of the deformed Al single crystal, strain 1, section perpendicular to TD2

large spreading of texture components on the pole figures from the deformed material and from an inaccuracy in measuring of this inclination (slip lines were highly

waved) alike. The change in orientation of slip lines was accompanied by increasing shear strain in the compression plane evidencing the lattice rotation around the ND. The lattice rotation was evidenced on texture (Fig. 7). Any forms of localized microstructural inhomogeneities like shear or transition bands were not observed.

The dislocation microstructure was strongly recovered. It consisted of subgrains elongated in the direction of plastic flow (Fig. 9). This is due to the high SFE of Al. In such materials cross slip occurs easily and recovery proceeds fast resulting in the formation of the subgrain microstructure [5].

### 3.3. Recrystallization microstructure and texture of materials deformed with a change in the direction of plastic flow

The recrystallization was investigated on samples subjected to rolling to the 0.5 strain followed by the CDC to the 0.5 strain.

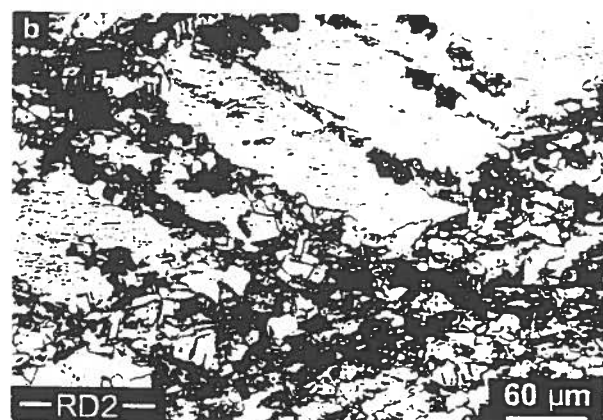
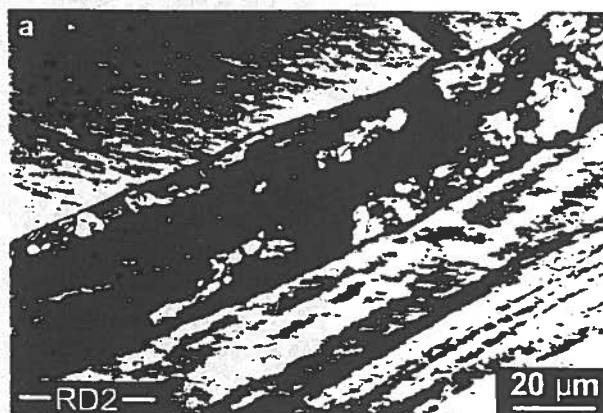
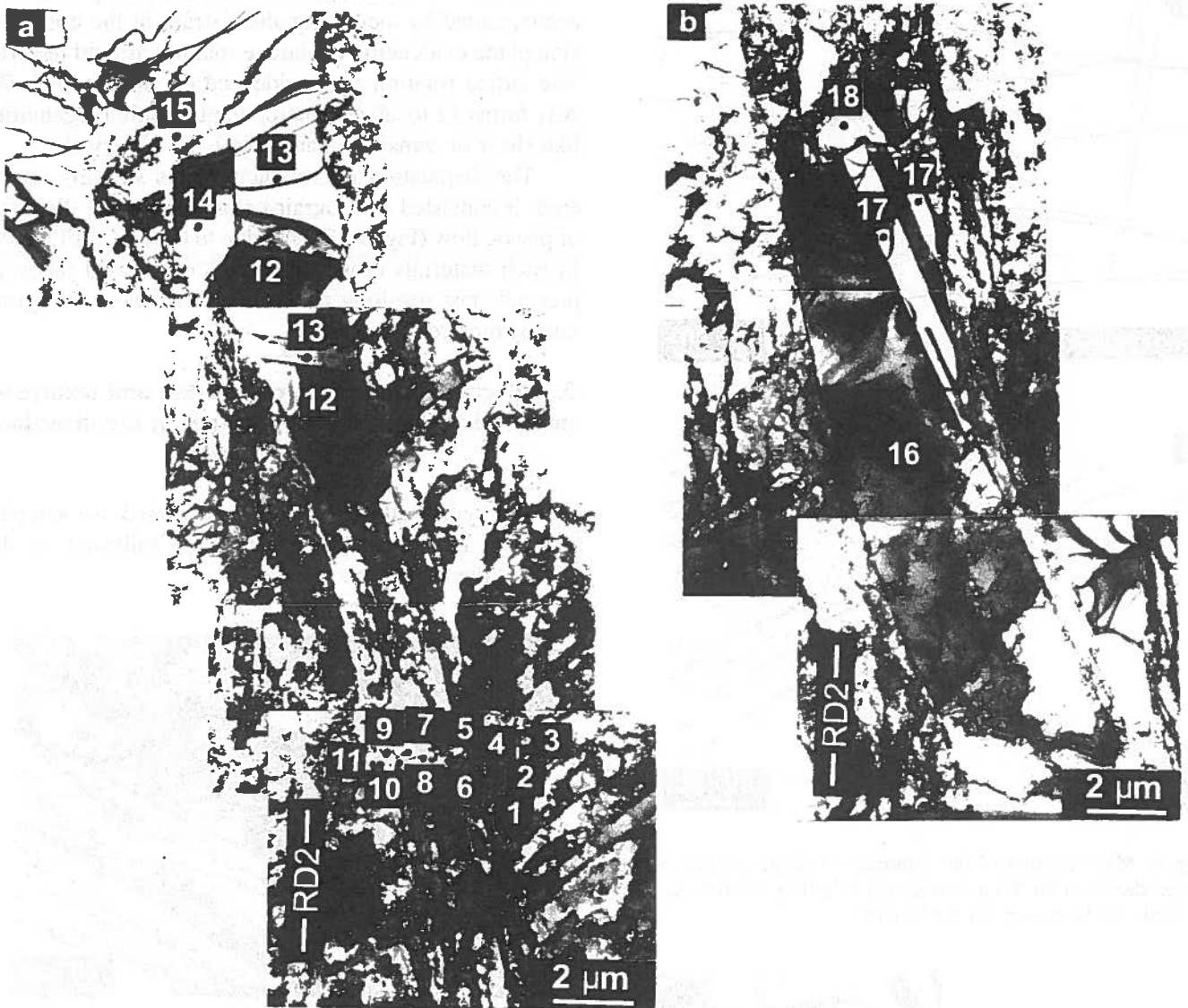


Fig. 10. Microstructure of the Cu after deformation (rolling, strain 0.5 and additional 0.5 strain by the CDC) followed by annealing for 1 h: a) at 160°C and b) at 200°C

### Cu

During annealing at 160°C recrystallization commenced within shear bands (Fig. 10a). Shear bands provide pref-





Disorientation	$\Sigma$	Number of orientation
$30^\circ\langle 111 \rangle$	13	12, 15, 16
$40^\circ\langle 111 \rangle$	19	16
$52^\circ\langle 111 \rangle$	61	18
$14^\circ\langle 210 \rangle$	69	15
$32^\circ\langle 211 \rangle$	87	12

Fig. 11. Partly recrystallized shear band in Cu. Material deformed (strain 0.5 by rolling and additional strain 0.5 by the CDC) and 1 h annealed at  $190^\circ\text{C}$

ential sites for recrystallization because they constitute regions of localized deformation with a high dislocation density and significant local disorientations. After

annealing at  $200^\circ\text{C}$  the shear bands were fully recrystallized while the matrix was only recovered (Fig. 10b). This demonstrates the significant difference in the poten-

tial for recrystallization of the both regions. After the full recrystallization of shear bands, further recrystallization of the sample proceeded by a growth of selected grains from a shear band into the deformed matrix. Thus, after completion of recrystallization, fine grains (about 10 μm in diameter) filled the former shear bands while coarse grains (about 100 μm in diameter) occurred between them. The similar influence of shear bands on the recrystallization behavior was also observed in other f.c.c. metals [6, 7]. The recrystallized grains contained often recrystallization twins.

The determination of local crystallographic orientations was performed by CBED in the partially recrystallized sample (annealed at 190°C). After such treatment, the shear bands were partly recrystallized (Fig. 11 a, b). The crystallographic orientation (close to the orientation {631}<493>) in the recovered regions of a shear band differed from that in the matrix, on both sides of a shear band, by maximum of 15°. The orientation differences between adjacent subgrains in the broad shear bands did not exceed 8°. Orientations of subgrains within shear bands and orientations of the deformed matrix fell into the range of the orientation spreading determined by the X-ray diffraction for the deformed material (Fig. 12).

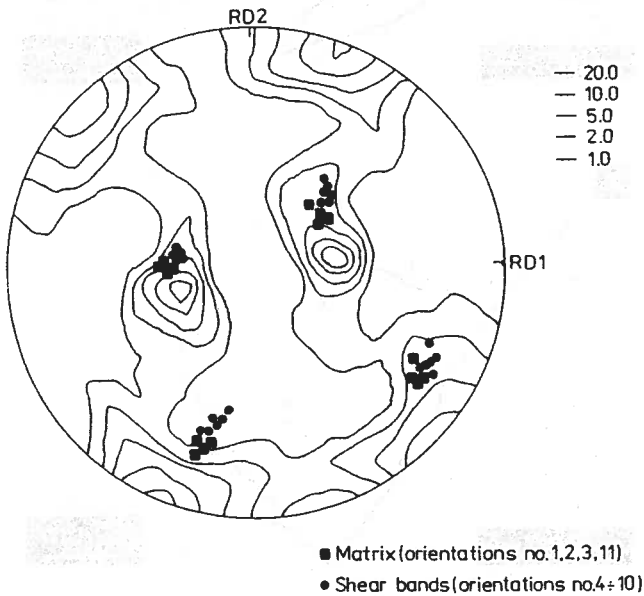


Fig. 12. Orientations determined by CBED in matrix and in the recovered areas of shear bands from Fig. 11 a, b superimposed on Fig. 3

The shape of the new grains suggested that the grains grew faster along the direction of shear bands than in any other direction (Fig. 11 a, b). Among grains which grew along a shear band, larger sizes reached those grains which orientations could be obtained from

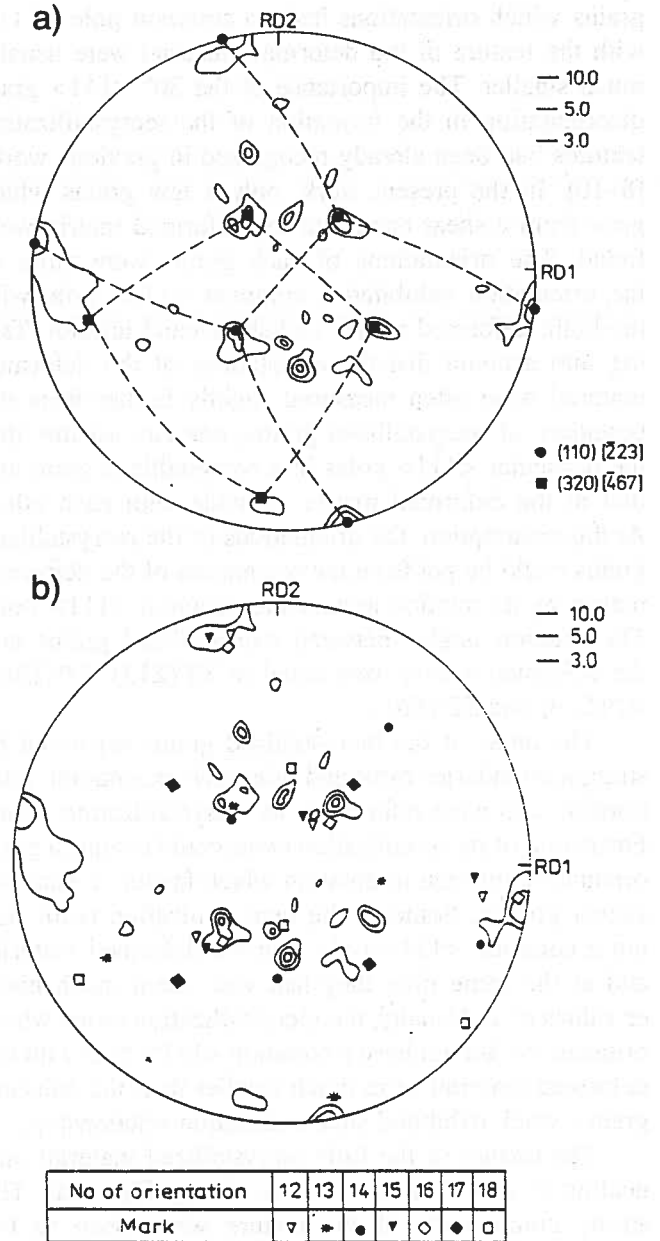


Fig. 13. a) (111) pole figure of Cu after full recrystallization (1 h annealed at 380°C) b) crystallographic orientations of the recrystallized grains from Fig. 11a, b, superimposed on Fig. 13a

orientations of the shear band by a rotation around a <111> pole by about 30°. For such a disorientation, the parameter Σ<sup>2)</sup> is equal to 13 (Σ13). Orientations of such grains could also be obtained from the orientation of the matrix surrounded the shear bands by the rotation around the following common poles: <111> by about 40° (Σ19), <210> by about 14° (Σ69) and <211> by about 32° (Σ87). The above disorientations are related to lower mobility of a grain boundary comparing to the well known disorientation 30° <111>. In addition, the

<sup>2)</sup> Σ is defined as the reciprocal of the ratio of the number of coincidence lattice sites to the number of overall lattice sites (calculated according to Ref. [11]).

grains which orientations had no common pole  $\langle 111 \rangle$  with the texture of the deformed material were usually much smaller. The importance of the  $30^\circ \langle 111 \rangle$  grain disorientation in the formation of the recrystallization textures had been already recognized in previous works [8–10]. In the present work, only a few grains which grew from a shear band into the deformed matrix were found. The orientations of such grains were close to the orientation exhibited a common  $\langle 111 \rangle$  pole with the both: deformed matrix and shear band interior. Taking into account that the orientations of the deformed material were often measured slightly farther from the boundary of recrystallized grains, one can assume that the particular  $\langle 111 \rangle$  poles in a recrystallized grain and that in the deformed matrix coincide with each other. At this assumption, the orientations of the recrystallized grains could be got from the orientation of the deformed matrix by its rotation around the common  $\langle 111 \rangle$  pole. The rotation angles between recrystallized grains and the deformed matrix were equal to  $30^\circ (\Sigma 13)$ ,  $33^\circ (\Sigma 39)$ ,  $40^\circ (\Sigma 19)$  and  $52^\circ (\Sigma 61)$ .

The areas of the recrystallized grains separated by straight boundaries exhibited often twin orientation relationship and were referred to as recrystallization twins. Formation of recrystallization twins could change a grain orientation into the orientation which favours a faster or slower growth. Some of the recrystallization twins had not a common  $\langle 111 \rangle$  pole with the deformed material and at the same time they had with them much higher values of  $\Sigma$ . Usually, the recrystallization twins which orientations did not have a common  $\langle 111 \rangle$  pole with the deformed material were much smaller than the adjacent grains which exhibited such orientation relationship.

The texture of the fully recrystallized material (annealing at  $380^\circ\text{C}$ ) was relatively strong (Fig. 13a). The strong components of the texture were close to the  $(110)[\bar{2}23]$  and  $(320)[\bar{4}67]$  orientations. These orientations could be transformed from orientations forming maxima on the deformation texture by a rotation around the common  $\langle 111 \rangle$  poles by the angles of  $35^\circ$  and  $50^\circ$ , respectively. The comparison of the X-ray texture with the results of the CBED measurements showed that the texture component  $(110)[\bar{2}23]$  corresponded to those grains which orientations could be transformed to the orientation of the deformed material by a rotation around the common  $\langle 111 \rangle$  pole by about  $30^\circ$ . Fig. 13b shows the recrystallization texture determined by the X-ray diffraction with the superimposed orientations of grains belonging to the analyzed shear band determined by the CBED.

#### Al

The first recrystallized grains were found in the sample annealed at  $130^\circ\text{C}$  (Fig. 14a). The arrangement of

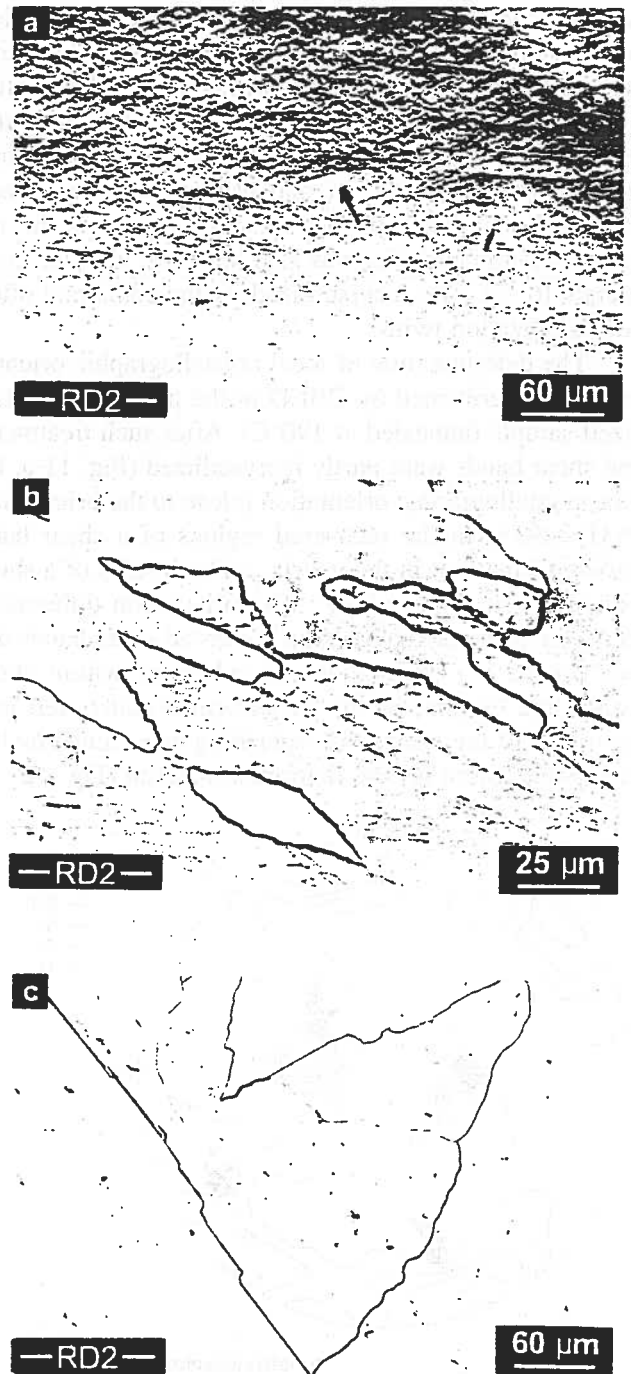


Fig. 14. Microstructure of the Al after deformation followed by annealing at: a)  $130^\circ\text{C}$ , b)  $133^\circ\text{C}$  and c)  $150^\circ\text{C}$

recrystallized grains within the sample was relatively random, no tendency for clustering of the recrystallized grains was found. This resulted from the uniform microstructure of the deformed material in which no preferential nucleation sites occurred. In such microstructure the nucleation of new grains is strongly confined. In most cases, the grains formed during recrystallization were oblong-shaped and were elongated in the  $[1\bar{1}0]$  di-



rection (RD2). For the annealing temperatures higher than 130°C, the regions adjoining to free surfaces of the compressed samples acted as preferential nucleation sites resulting in the formation of grain conglomerates. In order to find out whether the formation of new grains at specimen surfaces results from more inhomogeneous deformation of such regions, before annealing, the layers adhering to the specimen surfaces were removed by grinding down to about 0.5 mm followed by electrochemical polishing. Despite of this, the surfaces were as before favourite nucleation sites. This shows, that in the absence of structural inhomogeneities, the free surface may sufficiently lower the nucleation energy and come into effective site for new grains formation. The grains forming along slip bands were also observed (Fig. 14b). The specimen annealed at 150°C was fully recrystallized (Fig. 14c). Since the number of nuclei was low and their growth was relatively fast, after completion of recrystallization the grains were very large (grain size was about 600  $\mu\text{m}$ ).

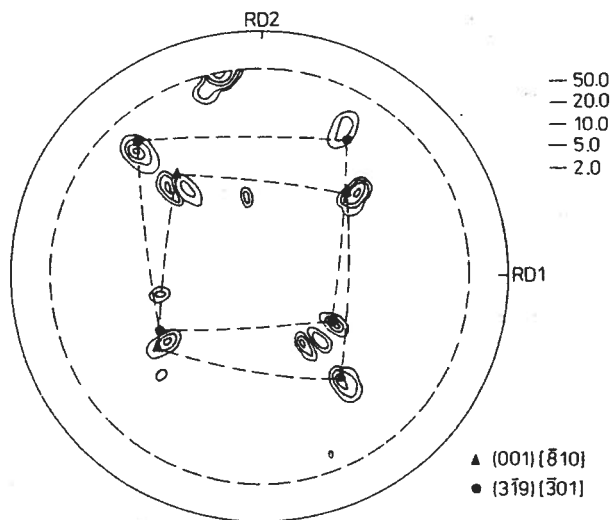


Fig. 15. [111] pole figure of the Al single crystal annealed at 150°C

The recrystallization texture was measured for fully recrystallized material. The texture components close to the (001)[ $\bar{1}00$ ] and ( $3\bar{1}9$ )[ $\bar{3}01$ ] orientations were identified (Fig. 15). The (001)[100] component often appears in recrystallization texture of single and polycrystalline aluminium [12–13]. The (001)[100] and ( $3\bar{1}9$ )[ $\bar{3}01$ ] components may be produced by the rotation of the deformation texture components around the  $\langle 111 \rangle$  poles inclined toward the rolling plane, by an angle of about 30° (Fig. 16).

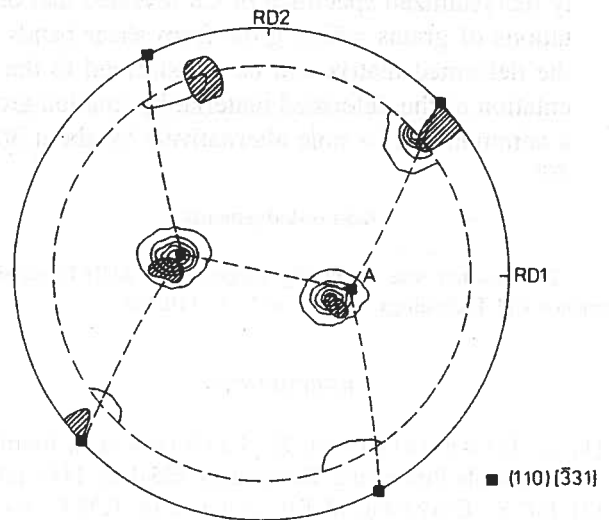


Fig. 16. Comparison of textures from Fig. 15 after rotation around  $\langle 111 \rangle$  pole marked A by 30° (hatched areas) with texture from Fig. 7

#### 4. Conclusions

1. During the homogeneous rolling, the deformation mechanism of the investigated f.c.c. single crystals with the (110)[001] orientation was recognized as the dislocation glide. The rolling texture did not show any significant changes comparing to the initial orientation.
2. The change in the direction of plastic flow brought about a significant change in microstructure. Shear bands were formed in Cu during CDC, while any forms of localized microstructural inhomogeneities were not observed in Al. In the case of Al the CDC brought only a rotation of the initial orientation around the ND by about 14°.
3. In the case of Cu, recrystallization commenced within shear bands and proceeded by a growth of selected grains from shear bands into the deformed matrix. Since the number of such grains in the population of the grains formed within the shear bands is relatively small, after recrystallization fine grains were in areas of former shear bands while the coarse ones in areas between them. The number of nuclei in Al was low hence the grain size after recrystallization was large.
4. The processes predicted by the theory of the oriented growth play an important role in the formation of recrystallization textures in both materials. The strong components of the recrystallization textures might be obtained by the rotation of the deformation texture components around the common  $\langle 111 \rangle$  poles by an angle of 30° for Al and 35° or 50° for Cu.

5. CBED measurements of local orientations in partially recrystallized specimen of Cu revealed that orientations of grains which grow from shear bands into the deformed matrix can be transformed to the orientation of the deformed material by rotation around a common  $\langle 111 \rangle$  pole alternatively by about  $30^\circ$  or  $50^\circ$ .

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