ARCHIVES OF METALLURGY

Volume 47 2002 Issue 2

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DEVELOPMENT OF MICROSTRUCTURE AND TEXTURE DURING DEFORMATION AND RECRYSTALLIZATION OF CuAI5 (110) [001] SINGLE CRYSTAL

ROZWÓJ MIKROSTRUKTURY I TEKSTURY PODCZAS ODKSZTAŁCENIA I REKRYSTALIZACJI MONOKRYSZTAŁU CUAI5 O ORIENTACJI (110) [001]

The CuAl5 single crystal with the initial orientation (110)[001] was cold rolled and subsequently compressed in a channel die in such a way that the new direction of plastic flow was parallel to the transverse direction of initial rolling and the compressed plane was parallel to the former rolling plane. The dislocation slip was the only deformation mechanism during rolling. The dislocation substructure after rolling was uniform. During the compression in a channel die, additionally, the deformation twinning as well as the shear band formation took place. The recrystallization commenced and proceded within the shear bands. After that, the recrystallization proceeded by the nucleation and growth of new grains within the areas with high density of deformation twins. The recrystallized regions of the former shear bands were filled by fine grains while the regions amongst them exhibited much bigger grains. The recrystallization texture was formed according to the theory of the oriented growth.

Monokryształ stopu CuAl5 o orientacji początkowej (110)[001] walcowano, a następnie ściskano w matrycy z kanałem, w ten sposób by nowy kierunek plastycznego płynięcia był równoległy do kierunku poprzecznego podczas walcowania, zaś ściskana płaszczyzna była równoległa do płaszczyzny walcowania. Podczas walcowania monokryształ odkształcał się tylko przez poślizg dyslokacji. Po walcowaniu monokryształ charakteryzował się stosunkowo jednorodną mikrostrukturą dyslokacyjną: Podczas ściskania w matrycy z kanałem stop odkształcał się również przez bliźniakowanie, a w mikrostrukturze tworzyły się pasma ścinania.

Rekrystalizacja rozpoczynała się w pasmach ścinania. Po zrekrystalizowaniu pasm ścinania dalsza rekrystalizacja zachodziła przez tworzenie się ziaren w obszarach o dużej gęstości bliźniaków odkształcenia i ich wzrost. Wzrost tych ziaren był znaczenie szybszy niż ziaren utworzonych w pasmach ścinania. W materiale zrekrystalizowanym w obszarach byłych pasm ścinania ziarno było drobne, natomiast pomiędzy pasmami ścinania ziarno było znacznie większe.

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Tekstury rekrystalizacji materiału odkształconego ze zmianą kierunku plastycznego płynięcia oraz materiału tylko walcowanego tworzyły się zgodnie z teorią uprzywilejowanego wzrostu. Tekstura rekrystalizacji materiału odkształcanego ze zmianą kierunku plastycznego płynięcia zawierała mniej składowych niż tekstura materiału jedynie walcowanego i rekrystalizowanego.

1. Introduction

The recognition of the microstructure and texture development during deformation with a change in the plastic flow as well as during subsequent recrystallization is particularily important for increasing of a material capacity to forming operations. Unfortunately, the formation of texture and microstructure is very complex phenomenon, especially during such treatment. The understanding of this process is easier for single crystals than polycrystals. However, a limited number of published works deal with the influence of a change in the direction of plastic flow on the developing texture and microstructure, particularly for the single crystalline metals. This issue was discussed in our earlier articles but that results pertained to the f.c.c. metals with high and medium stacking fault energy (aluminum [1] and copper [2]). The goal of the present work is to investigate this effect in the CuAl5 alloy exhibiting low stacking fault energy (about 5 mJ \cdot m⁻²).

2. Material and experimental procedure

The two single-crystalline samples of the CuAl5 alloy were subjected to homogeneous rolling at room temperature to the final true strains of 0.51 and 0.92. The specimens for the further channel die compression were cut off from the rolled bars in such a way that the new direction of plastic flow was parallel to the transverse direction of initial rolling and the compressed plane was parallel to the former rolling plane. By analogy to rolling, the direction of plastic flow during channel die compression is termed the rolling direction and assigned as RD2, the compression direction is the normal direction (ND) and the direction perpendicular to the channel side walls is the transverse direction (TD2). Friction between the sample and the walls of the channel die was reduced by wrapping samples with Teflon tape which was replaced after each deformation step of about 0.05. The specimens had rectangular walls with dimensions of 5 mm×7 mm×12 mm or 20 mm and fitted strictly the channel die. The lateral surfaces of all samples were carefully polished before compression. This let to examine, by a light microscope, deformation markings forming on these surfaces.

Recrystallization annealing was carried out in a salt bath for 1 hour. The annealing temperature was kept with an accuracy of at least $\pm 2^{\circ}$ C. The investigation of the recrystallization progress was carried out on specimens' sections perpendicular to the TD2.

The metallographic investigations were carried out by means of a light microscope and transmission electron microscopes Jeol JEM-100C, and JEM-100B.

The crystallographic orientations of grains formed during recrystallization were determined on the Philips CM20 electron microscope. The orientations were found out from Kikuchi lines formed on convergent beam diffraction patterns. The diffraction patterns were taken from an area of about 20 nm in diameter. The accuracy of the orientation determination is about 0.1°, however, an error in setting the correct RD (marked on each thin foil) in the specimen holder of the microscope may approach 10°. The Kikuchi diffractograms were solved by the computer software "Euclid's Phantasies" [3].

The X-ray pole figures for the {111} planes were measured by the Schulz back reflection technique. The measurements were performed by means of a Philips X'Pert diffractometer furnished with a textural goniometer ATC-3.

3. Results and discussion

The texture of the rolled single crystal with the orientation (110)[001] did not reveal any considerable changes comparing to the initial orientation. It results from equal strains in the four slip systems $(111)[01\overline{1}]$, $(111)[10\overline{1}]$, $(11\overline{1})[101]$ and $(11\overline{1})[011]$ operating in each element of volume of the deformed crystal. The microstructure of the deformed material was relatively uniform. The dislocation rich layers (dislocation bands) were parallel to the traces of {111} planes, on which the dislocations glide occurred. The deformation behavior as well as microstructure were identical as in analogically rolled austenitic stainless steel [4], CuAl5 [5], copper [6–8] and aluminum [9] single crystals with the same orientation.

3.1. Development of microstructure and texture during channel die compression

The {111} pole figure determined for the material subjected to channel die compression with the strain of 0.50 preceded by rolling with the 0.51 strain was relatively sharp. The main texture component was identified as $(110)[\overline{4}41]$ which was rotated around the ND by about 10° with regard to the initial orientation of the compressed samples, i.e. $(110)[1\overline{1}0]$. The component $(110)[\overline{1}19]$ was also present in texture. This component is in the twin relation to the main component $(110)[\overline{4}41]$ (Fig. 1).

Deformation twins were formed in the compressed samples. The twins were identified by electron microscopy examination (selected area diffraction patterns, dark field imaging) carried out on thin foils cut out from specimens' sections perpendicular to either the TD2 or ND (Fig. 2, 3). In the microstructure dominated twins for which traces of twin planes on the foils perpendicular to TD2 were mainly perpendicular to the RD2 (Fig. 2) while on the foils perpendicular to the ND the traces were angled to the RD2 at



Fig. 1. The {111} pole figure, material deformed by rolling (rolling direction RD1) with true strain of 0.51 followed by channel die compression with true strain 0.5 (direction of plastic flow RD2), orientations:

- (110)[441]
- (110)[T19], componet twinned to (110)[441]

55° and -55° (Fig. 3). This proves that the twins are parallel to the traces of $(\overline{1}11)$ and $(1\overline{1}1)$ planes respectively. This result confirmed the T a y l o r - B i s h o p - H i l l theory which predicted that during compression in a channel die the dominant twinning systems are: $(\overline{1}11)[\overline{1}1\overline{2}]$ and $(1\overline{1}1)[\overline{1}1\overline{2}]$. The pole figure also shows the texture component in the twin relation to the initial orientation $(110)[1\overline{1}0]$. For such relationship the $(\overline{1}11)$ plane is the twinning plane (Fig. 1). The fact that the just one twinned component was identified in the texture results from the ample dominance of a single family of twins occurring in the specimen region from which the texture was measured.

The examinations of specimen surfaces revealed also deformation markings almost parallel to the RD2 and to the traces of (111) and $(11\overline{1})$ planes. These markings were specially well recognized after low deformations (Fig. 4). Though the examination of thin foils revealed deformation twins parallel to the RD2 (Fig. 5), the number of such twins was not enough to shown their presence on the pole figure (Fig. 1).



Fig. 2. Deformation twins, material deformed by rolling (rolling direction RD1) with true strain of 0.92 followed by channel die compression with true strain 0.46, TEM, section perpendicular to RD1

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Fig. 3. Deformation twins, material deformed by rolling with true strain of 0.92 followed by channel die compression with true strain 0.12, TEM, section parallel to the rolling plane



Fig. 4. Microstructure of the material deformed by rolling with true strain of 0.51 followed by channel die compression with true strain 0.01, light microscope, compressed surface



Fig. 5. Microstructure of the material deformed by rolling with true strain of 0.92 followed by channel die compression with true strain 0.46, TEM, section perpendicular to the RD1

Almost simultaneously with the formation of areas with high density of deformation twins, the shear bands developed in the compressed samples. In the sample rolled to the 0.92strain first shear bands appeared after subsequent strain of 0.06. In the sample rolled to the 0.51 strain first distinct shear bands appeared after additional strain of 0.15. One may draw a conclusion that the easier the shear bands develop, during the compression after the change in the direction of plastic flow, the more strengthened the material is, i.e. the higher deformation was applied before the change in the strain path. The work [8] showed evidences that the formation of shear bands is associated with the passage of newly generated dislocations through barriers which impede their motion. In the case of the material investigated in the present work, either the dislocation bands formed during unidirectional rolling or deformation twins formed during the compression in a channel die might constitute the barriers for the dislocation motion. The dislocation structure in the samples deformed to 0.51 and 0.92 strains was similar. The differences resulted from the different dislocation density and different density of deformation twins in the microstructure of both samples. These differences are also the reason that during the channel die compression of the sample rolled to the 0.92 strain the first shear bands were formed at much higher stress than in the sample rolled to the 0.51 strain (920 MPa and 750 MPa respectively).

The shear bands revealed on sections perpendicular to the TD2 were inclined to the RD2 at the angles from a range of $\pm 22^{\circ} \div 32^{\circ}$. The position of shear bands is thus

consistent with the literature data [10, 11]. The shear bands revealed on sections perpendicular to the ND were sloped to the RD2 at angles from the range of $\pm 50^{\circ} \div 85^{\circ}$. On sections perpendicular to the RD2 they were almost parallel to the <113> and <133> directions in the matrix. The directions, to which the shear bands are parallel, do not lie in the {111} planes, consequently the shear bands are not parallel to the {111} planes in the matrix. This suggest that shear bands could have been formed as a result of the deformation in several systems with the {111} planes operating at the same time or, alternatively, as a result of deformation in nonoctahedral slip systems. The study of Cu single crystals with the same initial orientation, deformed in the same way, in which shear bands were also non-parallel to the traces of {111} planes, showed that the shear bands formation as a result of multisystem slip on {111} planes is more likely [12].

The shear bands thickness usually was not more than several μ m. Thickness and density of the shear bands were much more lower then in the (110)[001] copper single crystals deformed in the same way [2].

4. Changes in texture and microstructure during annealing of deformed crystal

4.1. Texture of the rolled and recrystallized crystal

Fig. 6 shows the {111} pole figure measured at the half of thickness of the crystal which was rolled to the strain of 0.92 and subsequently annealed at 415°C for one hour. The following components were identified in this texture: $\{211\}<\overline{328}>$, $\{531\}<\overline{592}>$ and



Fig. 6. The {111} pole figure, material rolled to 0.92 true strain and annealed at 415°C for 1 h; position of maxima for orientations:



 $\{531\}<4\overline{97}>$. The highest intensity of reflections exhibited the following components: $(121)[\overline{238}], (211)[\overline{328}], (\overline{211})[328]$ and $(\overline{121})[238]$. These components can be obtained from the orientation (110)[001], which predominates in the texture of the deformed material, by its rotation around common $\{111\}$ poles, lying on the rolling plane, by an angle of about 30°. The components: $(513)[52\overline{9}], (153)[2\overline{59}], (15\overline{3})[\overline{259}]$ and $(51\overline{3})[\overline{529}]$ could have been gotten from the (110)[001] component by its rotation around the common <111> poles lying on the rolling plane in the vicinity of the main deformation texture components. In this case the angle of rotation was about 45°. Weaker components of the recrystallization texture: $(153)[74\overline{9}], (513)[47\overline{9}], (51\overline{3})[479]$ and $(15\overline{3})[749]$ exhibit twin relationship with the $\{531\}<592>$ components. The recrystallization texture is similar to the texture in analogically deformed CuAl5 single crystal with the same initial orientation, rolled to the 1.61 strain and annealed at 385°C [17]. However, in Ref. [17], the texture was measured in a material layer located at one forth of the thickness of the rolled crystal.

Because the microstructure in the rolled crystals with the orientation (110)[001] was relatively uniform, one can argue, that the new grains reached the orientations that stimulated their fast growth into the deformed matrix as a result of the formation of recrystallization twins. Since the quadruple twinning of the (110)[001] orientation does not lead to the components of the recrystallization texture [17], so to get these components, at least quintuple twinning of this orientation is required. It seems more likely that formation of the fast growth orientation is a result of the lower-fold twinning of the deformation texture orientations close to orientation (110)[001].

4.2. Microstructure and texture of the recrystallized crystal deformed by a channel die compression after preceding rolling

The recrystallization was investigated on samples subjected to compression in a channel die to the strain of 0.50 after the preceded 0.51 strain applied by rolling.

4.2.1. Light microscopy

First grains were formed within shear bands (Fig. 7a). The shear bands are favorable nucleation sites because of large local misorientations in the crystal structure and high stored energy of deformation [18, 19]. In sample annealed at 320°C the shear bands were fully recrystallized (Fig. 7b). Much faster recrystallization of shear bands than of the matrix was also observed in a polycrystalline CuAl5 alloy [16]. The recrystallized shear bands had not any appreciable influence on the course of recrystallization at higher temperatures. Only a few grains from the shear bands grew into the deformed matrix. Usually, the growth of grains formed within shear bands was confined to a shear band. Further recrystallization proceeded mainly by the formation of new grains in the regions

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containing high density of deformation twins. Their growth determined the progress of recrystallization at temperatures above 300°C (Fig. 7b-c). The size of grains after recrystallization was differentiated. In the regions of former shear bands the grains were fine, with a diameter of about a few μ m, while in other regions the grains were much bigger, about 30 μ m (Fig. 7d).



Fig. 7. Microstructures of recrystallized materials. Deformation applied by rolling with true strain of 0.51 followed by channel die compression with true strain of 0.5; micrographs a, b and d – section perpendicular to RD1, micrograph c – compressed surface

4.2.2. Crystallographic orientations of selected grains (TEM)

The crystallographic orientations of selected grains as well as subgrains in the recovered matrix were determined for samples annealed for one hour at temperatures 295°C and 310°C. The poor quality of diffractograms (Kikuchi patterns) obtained from the recovered regions of shear bands made their solution impossible. The local crystallographic orientations within the matrix were close to the main components of deformation texture determined by the X-ray method (Fig. 8). Thus, it is believed that they are representative for the entire matrix.



Fig. 10, 11a,b	43	44	45	46	47	48	49	50	52	53	57	58	59	60	61	62	65	68	69	70
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Fig. 8. The {111} pole figure. Positions of orientation poles determined by TEM are superimposed on the deformation texture from Fig. 1; sample deformed by rolling (rolling direction RD1) with true strain of 0.51 followed by channel die compression with true strain of 0.5 (direction of plastic flow RD2) and subsequently annealed at 310°C for 1 h

In the sample annealed at 295°C the shear bands were partly recrystallized. The crystallographic orientations of grains which were formed within shear bands and grew into the matrix often exhibited relatively low values of the coincidence factor Σ^* with the orientations from the deformed matrix. They could be obtained from the orientation of the deformed matrix by a rotation around the common $\langle 111 \rangle$ poles by angles from the range of 22–48°. E.g., by about 22° (Σ 21, for the grain no. 22 in Fig. 9a), by about 32° (Σ 39, for the grain no. 30 in Fig. 9b), by about 38° (Σ 7, for the grain no. 16 in Fig. 9a) and by 48° (Σ 19, for the grain no. 9 in Fig. 9a). There were also found grains growing from a shear band into the matrix even so their orientations had no common <111> pole with the orientations of the deformed matrix, e.g., grains no. 2 and 5 in Fig. 9a.

The grains which growth was confined to shear bands alone were relatively small (e.g., the grain no 6 in Fig. 9a). Such grains usually exhibited with the matrix relatively large values of Σ .

^{*} calculated according to [20].



Fig. 9. Partly recrystallized shear bands in two areas (a) and (b) of the sample annealed at 295°C for 1 h. Deformation applied by rolling with true strain of 0.51 followed by channel die compression with true strain of 0.5; sites where crystallographic orientation were determined are numbered







The above results correspond with the results collected by Humphreys and Hatherly [21] which showed that the activation energy for migration of grain boundaries in aluminum has minimal value for grains which orientations could be obtained from a matrix orientation by its rotation around a common <111> pole by angles: about 38° and by 47°. Also, Kronberg and Wilson [22] showed that the grains with high boundry mobility in copper had orientations which could be obtained from the orientation of the matrix by the rotation around a common <111> pole by about 22° or by about 38°.

In the recrystallized grains, recrystallization twins occurred which formed with the recovered matrix flat, and thus low-mobile, boundaries. Orientations of some of those twins could be obtained by the rotation of the orientation of the recovered matrix around a common <111> pole by angles: about 24° (Σ 67, the twin no. 25 in the grain 23 in Fig. 9b) and about 28° (Σ 13, the twin no. 35 in the grain no. 30 in Fig. 9b). The other analyzed recrystallization twins had no common <111> pole with the recovered matrix. Orientations of some of them could be obtained by the rotation of the matrix orientation around a common <211> pole by angles: about 26° (Σ 53, the twin no. 20 in the grain no. 21) and by 108° (the twin no. 10 in the grain no. 9), Fig. 9a. In some recrystallized grains, twins (twins no. 11 and 12) nearly parallel to the deformation twins in the matrix (twins no. 13 and 14) were observed (Fig. 9a). The crystallographic orientations of such twins were consistent with the orientations of deformation twins.

After the completion of recrystallization within shear bands the new grains nucleated in the regions of high density of deformation twins (Fig. 10) and especially in sites where deformation twins intersected (Fig. 11a-b). The growth of these grains was so much faster than the growth of grains from shear bands. That is why only these grains controlled the progress of recrystallization in regions between shear bands. The orientations of such grains were determined in the sample which was annealed at 310°C for one hour. No preferred growth of grains with a common <111> pole with the orientations of the recovered material was observed. The biggest grains out of all ones with convex boundaries had orientations which could be obtained by the rotation of the

No of recrystallized grain	No of matrix	Angle of rotation	Common pole <211>			
67	69	140°				
67	68	144°				
67	70	144°	1			
66	69	75°	<221>			



Fig. 10. Grains formed within areas of high density of deformation twinns in sample annealed at 310°C for 1 h. Before annealing, deformation was applied by rolling with true strain of 0.51 followed by channel die compression with true strain of 0.5; sites where crystallographic orientation were determined are numbered



Fig. 11. Grains formed at intersections of deformation twins in sample annealed at 310°C for 1 h. Before annealing, deformation was applied by rolling with true strain of 0.51 followed by channel die compression with true strain of 0.5; sites where crystallographic orientations were determined are numbered

orientation of the recovered matrix around a common poles: <110> by an angle of about 55° (Σ 41, the grain no. 40 in Fig. 11a), <211> by an angle of about 132° (the grain no. 63 in Fig. 11b) and <211> by an angle of about 144° (Σ 83, the grain no. 67 in Fig. 10). The determined relationships between the deformed matrix and the grains formed within them are shown in the tables at Figures 10 and 11. It appears from that data that the value of Σ itself is not sufficient to state positively that the grain will show a tendency to the fast growth. It is believed that an important role plays also the mutual relationship between the crystallographic orientation of a grain and deformed matrix as well as the orientation of the boundary between them.

The recrystallization twins occurred also in grains formed in regions of high density of deformation twins, e.g., twins no. 38 and 39 in the grain no. 40 and the twin no. 42 in the grain no. 41 (Fig. 11a), the twin no. 66 in the grain no. 67 (Fig. 10). The relationship between the crystallographic orientations of recrystallization twins and the adjacent regions of the recovered matrix are shown in the tables at relevant Figures.

4.3. Texture of recrystallized material

The texture of fully recrystallized material which was formerly rolled to the 0.51 strain then compressed in a channel die with a strain of 0.69 and subsequently annealed at 360°C for one hour is shown in Fig. 12. Strong, mutually symmetrical, components of this texture $(21\overline{1})[113]$ and $(2\overline{1}1)[\overline{1}13]$ could be obtained by the rotation of orientations occurring near the $(110)[\overline{1}19]$ component of the deformation texture around a common <111> poles (lying in the rolling plane) by about 30° (Fig. 12). Also weaker com-



Fig. 12. {111} pole figures for sample annealed at 360°C for 1 h. Before annealing, deformation was applied by rolling (rolling direction RD1) with true strain of 0.51 followed by channel die compression with true strain of 0.69 (direction of plastic flow RD2); positions of maxima for orientations:

• (110)[T19], component of deformation texture

▲ near {211}<113>, this orientation can be obtained by rotation of orientation ● around a common <111> pole by about 30°

ponents: {110}<119>, {131}<411>, {131}<101> and {153}<301> were found in the recrystallization texture. The {131}<411> components could be obtained by a rotation of the orientations belonging to the vicinity of the (110)[$\overline{119}$] component of the deformation texture around common <111> poles (lying in the rolling plane) by about 30°. On the other hand, the components: {110}<119>, {131}<101> and {153}<301> fell into the range of spreading of the main components of the deformation texture.

No matter that the formation of the global recrystallization texture can be justified by the theory of the oriented growth (assuming the rotation of the deformation texture around the common $\langle 111 \rangle$ pole by an angle of about 30°), the determination of local orientations showed that the grains which exhibit relationships with the deformed matrix other than the typical one for this theory also have some influence on the formation of the recrystallization texture.

5. Conclusions

1. The microstructure of the single crystal deformed only by rolling was relatively uniform. Texture of such deformed material was relatively sharp with the (110)[001] component alone. The single crystal deformed with the change in the direction of plastic flow developed heterogenous structure and more complicated deformation texture. The change in the direction of plastic flow from [001] during rolling onto [110] during channel die compression induced the change of the deformation mechanism from slip onto slip and twinning.

2. The easier the shear bands were formed the higher strain was applied before the change in the direction of plastic flow; the density of shear bands rose with the increase of the applied strain. Shear bands were composed of elongated subgrains and were not parallel to the traces of $\{111\}$ planes in the matrix.

3. Recrystallization commenced in shear bands. After completion of recrystallization in shear bands, further recrystallization proceeded owing to nucleation and growth of new grains in the regions of high density of deformation twins. The sizes of grains after recrystallization was differentiated – fine grains occur in regions of former shear bands while bigger ones in the other regions.

4. The material deformed with the change in the direction of plastic flow showed more simple recrystallization texture than the material deformed with plastic flow along the one direction. The strongest components of recrystallization textures could be obtained from the deformation texture by its rotation around common <111> poles by about 45° or about 30° respectively for the material rolled in one direction and in the material rolled and further compressed in a channel die with a change in the direction of plastic flow.

5. For selection of the grains, growing faster than others, is not sufficient neither low value of coincidence factor nor misorientation typical for that justified by the theory of the oriented growth.

Acknowledgment

The financial support from the Polish Committee for Scientific Research, grant no. 7 T08A 02016 is greatly appreciated.

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REVIEWED BY: JAN POSPIECH

Received: 10 January 2002.