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MAREK S. SZCZERBA\*

## TRANSFORMATIONS OF DOMINANT DEFORMATION SYSTEMS IN COPPER SINGLE CRYSTALS

### TRANSFORMACJE DOMINUJĄCYCH SYSTEMÓW POŚLIZGU W MONOKRYSZTAŁACH MIEDZI

A close relation between the origin of tensile instability and the onset of deformation stage IV of copper single crystals was found. The tensile instability in single glide oriented copper single crystals belongs to the end of the overshoot phenomena resulting from a change of dominant deformation system — the changeover from primary to conjugate slip dominance. The identical transformation in deformation mechanism was found to be responsible for the onset of stage IV of plastic deformation of copper single crystals tested in rolling. Structural and mechanical observations and 4.2 K electrical resistivity measurements performed on the deformed crystals show, that the change in deformation mechanism causes a significant decrease in the rate of work hardening, a sudden change in the rate of dislocation accumulation, and the redistribution of dislocation arrangements in crystal lattice. It is concluded that the transformation of dominant deformation systems may strongly control deformation properties of face centered cubic crystals at large plastic strains. Finally the low temperature slip-twin transformation is discussed in terms of the change of a dominant deformation system.

W pracy na przykładzie walcowanych i rozciąganych monokryształów miedzi wykazano, że początek IV-tego stadium deformacji oraz początek niestateczności rozciągania są zjawiskami fizycznie równoważnymi. W monokryształach miedzi o orientacji środka trójkąta podstawowego, niestateczność rozciągania należy do zjawisk kończących tzw. "overshoot" i wynika ze zmiany dominującego systemu deformacji — zamiana dominującego poślizgu pierwotnego na sprzężony. Identyczna transformacja w mechanizmie deformacji została znaleziona w walcowanych monokryształach miedzi. Przeprowadzone badania mechaniczne i strukturalne oraz pomiary oporności elektrycznej w temperaturze ciekłego helu pokazują, że transformacji dominującego systemu poślizgu towarzyszy znaczny spadek prędkości umocnienia, zmiana prędkości akumulacji dyslokacji oraz zmiana konfiguracji układów dyslokacji deformowanego kryształu. W pracy stwierdzono,

<sup>\*</sup> WYDZIAŁ METALI NIEŻELAZNYCH, AKADEMIA GÓRNICZO-HUTNICZA, 30-059 KRAKÓW, AL. MICKIEWICZA 30

że transformacja dominującego systemu poślizgu może istotnie kontrolować właściwości plastyczne kryształów regularnie ściennie centrowanych w obszarach dużych odkształceń plastycznych. W pracy podjęto również dyskusję nad niskotemperaturową transformacją typu poślizg-bliźniakowanie.

## 1. Introduction

A main aim of the present work is to show the experimental evidences for an equivalence of tensile instability and the origin of the deformation stage IV in copper single crystals. It will be shown, that the equivalence consists of a change of dominant deformation system, the transformation, which is very well known in the tensile plasticity of face centered cubic crystals and ascribed usually to the end of overshoot phenomena [1]. Since the discovery of M e c k i n g [2], there is a large number of papers dealing with physical explanation of work hardening behaviour of the deformation stage IV in face centered cubic single crystals (see for example [3]). However, still little is known about the physical reason of the origin of the deformation stage IV even in the case of pure FCC single crystals. This paper pretends to answer the question of what is the real physical reason responsible for the transition from the stage III of parabolic hardening into stage IV of linear hardening of copper single crystals tested in rolling. The affect of dominant deformation system transformation on plastic properties of copper single crystals at large strains and low temperature deformations is also discussed.

# 2. Experimental procedure

Copper (99.999) single crystals of desired orientation were grown from seed in purified graphite moulds under vacuum of better than  $10^{-4}$  Torr. The single crystals were 4 mm  $\times$  4 mm in cross section and 80 mm long. Most of the single crystals had the main axis parallel to  $[\overline{1}45]$  crystallographic direction and the lateral surfaces parallel to  $(1\overline{1}1)$  and  $(\overline{3}\overline{2}1)$  crystallographic planes. The crystal geometry and crystal orientation (Fig. 1) were chosen in order to achieve a situation that the same slip system will predominate crystal deformation during both tension and rolling. After the [001] stereographic projection, the tensile axis [T] is parallel to  $[\overline{1}45]$  and the normal to the rolling plane, (ND), and rolling direction, [RD], are perpendicular to the  $(\overline{321})$  crystallographic plane and parallel to the  $[\overline{145}]$  crystallographic direction respectively. It is important to note that tensile deformation along the  $[\overline{1}45]$ crystallographic direction will be dominated by operation of the slip system  $B4 \equiv (111)[\overline{1}01]$  and what is again very important, the deformation during compression test along the  $\lceil \overline{321} \rceil$  will be also dominated by operation of the same slip system B4. Since the deformation of rolling test is mainly based on the tensile deformation along the rolling direction,  $RD \equiv \lceil \overline{1}45 \rceil$ , and the compression deformation perpen-

dicularly to the rolling plane ND  $\equiv (\overline{321})$ , so the slip system B4 should predominate crystal deformation both during the tensile and the rolling test. The tensile test of crystals with an expose gauge length of 50 mm was performed at room temperature and a strain rate of  $10^{-3}$  s<sup>-1</sup>. All electronically stored data of load and extension were used to obtain stress-strain curves and the rates of work hardening. Experimental observations in tension were focused on the origin of tensile plastic instability, which in this case appears as heavy strain localisation leading rapidly to sample failure (necking). X-ray measurements and slip line observations were used to check the position of crystal lattice and the changes in crystal deformation systems at the critical point of the origin of tensile plastic instability. The rolling test was also performed at room temperature and a strain rate of approximately  $10^{-3}$  s<sup>-1</sup>. The single crystal specimens were rolled gradually under good lubrication conditions in order to minimize the appearance of strain inhomogeneities, the deformation texture gradient and to achieve crystal deformation to be dominated by operation of one slip system. All of these requirements were satisfied, what was confirmed by systematic X-ray and slip line observations and the measurements of crystal shape changes in the cross section perpendicular to the rolling direction. The X-ray measurements were taken in RD-TD sections of a sample up to a cumulative rolling strain corresponding to the onset of deformation stage IV. It is to the appearance of plastic



Fig. 1. The tensile and rolling test crystal orientations and the geometry of face centered cubic single slip shown on the stereographic projection (see comments in the text)

instability again as heavy strain localisation, which now, because of the properties of the rolling test, was unable to cause the sample failure. Additional measurements of crystal shape changes and slip line and electron microscopy observations were performed to check what changes in crystal deformation system has taken place at strain levels before and after the onset of the deformation stage IV. Moreover the samples pre-deformed by rolling were tested in tension in order to construct a relationship between the  $\sigma_{proof}$  stress and the cumulative rolling strain, where each point of  $\sigma_{proof}$  stress has been obtained as the average of fifteen independent measurements. Finally, to establish how the plastic instability may affect the evolution of crystal dislocation density, 4.2 K electrical resistivity measurements of heavily deformed copper single crystals were performed, but more details of the experimental procedure will be given elsewhere [4].

### 3. Experimental results and discussion

The tensile test of copper single crystals with the initial orientation located in the center of the stereographic triangle (see for example T and M points on the Fig. 1) usually proceeds in such a way, that one slip system (B4) predominates crystal deformation. Very precise measurements of a crystal lattice shear occurring during the tensile deformation of single glide oriented copper single crystals were performed by Basinski and Basinski [1]. The measurements show that about ninety percent of total crystal deformation — up to the end of overshoot — is produced by one slip system B4, which is accompanied by secondary slip systems responsible for the rest of crystal deformation. Then, the tensile axis T(M) rotates mainly towards the operating slip direction 4 and overshoots the symmetry boundary B4|C1 until the C1 system will take over (see [1] for full crystallographic description of FCC slip systems). The changeover from B4 to C1 dominance is called the transformation of dominant deformation system. Since both of the dominant systems are slip systems, the transformation is of slip-slip type. The choice of dominant deformation system seems to be controlled by the Schmid criterion for yielding. It is commonly observed that the transformation of dominant deformation system ends the overshoot position of crystal lattice — in the case of copper single crystals tested at room temperature the tensile axis is finally located about  $2^{\circ}$  beyond the B4|C1 boundary [1]. A very characteristic feature of the transformation is the replacement of fine slip operating on the primary plane B into heavy "coarse" slip operating on the conjugate plane C, and this conjugate slip quickly evolves into heavy strain localisation [5]. The B4|C1 transformation phenomenon is very well seen on the mechanical characteristics (Figs. 2, 3), where the tensile stresses ( $\sigma$ ) and the rates of work hardening ( $d\sigma/d\varepsilon$ ) versus true tensile strain ( $\varepsilon = \log l/l_0$ ) for M(m = 0.50) and T(m = 0.47) oriented copper single crystals are shown. Here, the Schmid factor  $m = \tau/\sigma$  reflects the ratio between the shear stress operating in the slip system and the applied tensile stress. When  $\sigma$  and  $\varepsilon$  take critical values of about 175 MPa and 0.45 respectively, a large



Fig. 2. The tensile stress-strain and the rate of work hardening curves of maximum Schmid factor in copper single crystals



Fig. 3. The tensile stress-strain and the rate of work hardening curves of soft oriented copper single crystals

drop in the rate of work hardening of the initially M oriented crystals is observed (Fig. 1, note the distance of  $10^{\circ}$  from the symmetry line). The tensile deformation becomes suddenly unstable and proceeds as an L ü d e r s front and finally evolves into premature strain localisation of necking type. The critical  $\sigma$  and  $\varepsilon$  parameters

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depend on crystal orientation and they take the higher values for the orientations located large distances away from the B4|C1 boundary. The plastic instability in copper single crystals oriented initially along [145] crystallographic direction (Fig. 1, note the distance of 20° from the symmetry line) appears when  $\sigma = 220$  MPa and  $\varepsilon \approx 0.60$ . This time the instability is so strong that without any intermediate stage. it gives rise to extremely heavy premature strain localisation of necking type. It is very important to note that the tensile instability appears when the ratio between the rate of work hardening and the current flow stress,  $R = (d\sigma/d\varepsilon)/\sigma$ , is appreciably larger than unity ( $R^M = 1.65$ ,  $R^T = 1.30$ ); so the observed tensile plastic instability is not due to the exhaustion of material work hardening capacity, and occurs well before the Considére criterion is met. This type of plastic instability and its dependence on chemical composition, deformation temperature and crystal orientation has been carefully studied in [6]. The X-ray measurements confirmed that the crystal lattice takes at the critical point the overshoot position (Fig. 1, see the position of the point  $T^{F}$ , which is shifted from an ideal position by about 2<sup>0</sup> towards the center of the stereographic projection. The deviation of the rotation path of crystal lattice is fully understandable and was already mentioned in the literature. This is caused by the activity of secondary slip systems, which usually accompany the operation of dominant deformation system. According to the results obtained by B a s i n s k i and B a s i n s k i on copper single crystals of similar crystal orientation [1], B5 and C1 are the most pronounced secondary slip systems, which accompany the dominant slip system B4.

The state of stress operating in a crystal during rolling is more complex in comparison with that of the tensile deformation. However, a detailed analysis performed by M e c k i n g [7] suggests that for the case of crystal orientation and geometry chosen in this experiment the stress state generated by rolling can be approximated by a deviatoric part of stress tensor corresponding to the state of plane strain (as experimentally observed in [8], rolling deformation of copper single crystals of the orientation  $(ND)[RD] = (\overline{321})[\overline{145}]$  proceeds up to the onset of deformation stage IV with negligible longitudinal strain tensor component along the transverse direction TD). It is not unreasonable to assume, that the effective state of stress in a crystal during rolling will be dominated by the compression stress component acting normal to the rolling plane (ND) and the tensile stress component along the rolling direction RD, and  $\sigma_{RD} = -\sigma_{RP} = \sigma$ . Now, using the law of second order tensor transformation one can easy predict the most stressed slip system for a given crystal orientation. At the very beginning of rolling B4 system predominates crystal deformation. The ratio m between the resolved shear stress  $\tau$  and the magnitude of the compression stress component  $\sigma$  takes the highest value for B4 system and is by about factor of two and more higher than that of any other slip system (Table). Such the "orientation factor capacity" of B4 slip system could mean, that even if a real stress state of the rolling test will somehow differ from the assumed one, the B4 slip system will be still the most stressed slip system first satisfying the criterion for yielding. Keeping in mind that the dominant B4 slip system is accompanied by operation of secondary slip systems let as assume for simplicity, that all crystal deformation is produced by one system. Then the expected rotation path of rolling direction RD and normal to the rolling plane ND will be like it is shown on the Fig. 1. Rolling direction rotates toward the operating slip direction (RD  $\rightarrow$  4) and may overshoot the B4|C1 boundary and the rolling plane toward the normal to the slip plane (ND  $\rightarrow$  B) overshooting eventually the B4|C1 symmetry line. Using again the law of tensor transformation, one can easy check that such a situation will favor the C1 system. So, there is a critical amount of strain which must be imposed to a crystal in order to change the dominant slip system, and the most probable changeover will be identical with that occurring during the tensile deformation B4|C1. Later it will be shown that the real lattice rotation differs not very much from the ideal single slip situation and the deviations will be caused by the operation of secondary slip systems.

TABLE

The ratio  $m = \tau/\sigma$  for all of the slip systems of copper single crystals with the initial (m<sub>1</sub>) and firnal (m<sub>F</sub>) rolling orientation shown on the Fig. 7.

$A2 m_I = .078 m_F = .293$	$B2m_I^{}=.428\ m_F^{}=.467$	$C1 m_I = .350 m_F = .826$	$D1 m_I = .000 m_F = .066$
$A3 m_1 = .505 m_F = .251$	$B4 m_1 = .933 m_F = .784$	$C3 m_I = .428 m_F = .591$	$D4 m_I = .000 m_F = .057$
$A6 m_I = .583 m_F = .418$	$B5 m_I = .505 m_F = .317$	$C5 m_1 = .078 m_F = .236$	$D6 m_I = .000 m_F = .123$

Figure 4 shows the work hardening behaviour of copper single crystals rolled in the orientation  $(\overline{321})$  at room temperature. The most interesting feature is a transition from the deformation stage III of parabolic hardening into stage IV of linear hardening with the strain hardening coefficient  $\Delta\sigma/\Delta\varepsilon$  of approximately  $10^{-3}$  of the shear elastic modulus  $\mu$ . It is extremely interesting that the critical parameters needed to generate the plastic instability in tension fit perfectly well to the observed values of stress (220 MPa) and strain (0.60) at which the transition from the deformation stage III into the deformation stage IV does occur. The slip lines patterns (Fig. 5) show then the activation of heavy 'coarse' slip occurring on the slip plane C (the identical crystallography of slip line patterns was observed in tension). Also the electron microscopy observations taken before and right after the point of plastic instability (note the position of a and b points on the Fig. 4) show the transformation of the dislocation substructure from the cell like (Fig. 6a) into well-defined microband structure elongated along the expected traces of the conjugate plane C (Fig. 6 b). Very important information comes from the systematic X-ray measurements of the crystal lattice rotation (Fig. 7). Here, the measurements of crystal lattice position are presented for six steps of rolling deformation up to the value of  $\varepsilon = 0.65$ . The analysis of the experimental data show clearly that the critical point of the onset of deformation stage IV is fully equivalent to the point of the tensile instability and does belong to the end of overshoot phenomena. Table shows the new m ratio for each slip system when the crystal lattice takes its final position at the onset of deformation stage IV. Now the C1 system takes over and the proportion  $m_{C1}/m_{B4}$  more than 1.05 seems enough to favor



Fig. 4. The work hardening properties of copper single crystals pre-deformed by rolling at room temperature. Note the position of the plastic instability point taken after the tensile test measurements



Fig. 5. Slip line patterns of heavy slip in the conjugate plane C observed in the RD-ND section at the onset of the deformation stage IV of copper single crystals

new dominant deformation system. Similar proportion of m values corresponds to the room temperature overshoot of copper crystals tested in tension (see the overshoot position in the tensile test shown on the Fig. 1). Moreover, the deviations of the real crystal rotation path from the ideal single slip situation strongly suggest that the most pronounced secondary slip systems are those belonging to the slip planes C and B note that RD path deviates toward the operating slip direction 5 and the ND path deviates first toward the normal of the slip plane C and then 95toward the normal of the slip plane B.



Fig. 6 a. The dislocation substructure taken in the RD-ND section before the onset of the deformation stage IV. Note the point a position on the Figure 4



Fig. 6 b. The dislocation substructure taken in the RD-ND section after the onset of the deformation stage IV. Note the point *b* position on the Figure 4

It is to be emphasized that the plastic instability does not appear gradually — evolutional process — but instead, its activation, induced at a critical point of the deformation process, is rather discontinuous. This changes suddenly the crystal microstructure and properties — transformational process. The transformation of the dominant deformation system may strongly affect further deformation process and the evolution of crystal structures, especially those containing high densities of dislocations. Although the work on electrical resistivity of heavily deformed copper single crystals is still in progress [4], some experimental data are shown on the figure 8, where for simplicity al changes in the resistivity due to plastic work are ascribed to the changes of dislocation density. A detailed slope analysis performed in [4] shows that a sudden change in the rate of the dislocation and develops into the observed oscillations of the dislocation density, which start at a cumulative





a)

Fig. 7. The experimental data of crystal lattice rotation (a) taken after the inverse (111) pole figures X-ray measurements in the RD-TD section (b)



Fig. 8. The 4.2 K electrical resistivity versus cumulative rolling strain of copper single crystals. The specific dislocation resistivity is after Basinski [1] of the order of  $10^{-19} \ \Omega \text{cm}^3$ 

rolling strain of approximately 1.4 (Fig. 8). Further structural observations suggest that those oscillations may be connected with the conversion of plastic instabilities into well-defined shear bands, suggested already in the literature for copper single crystals of similar crystallographic orientations [8, 9].

As it was already mentioned the B4|C1 transformation is of slip-slip type, where one dominant slip system is replaced by the other dominant slip system. However, the transformation may suffer a transition from slip-slip into slip-twin type and this can be controlled by chemical composition (stacking fault energy), deformation temperature and crystal orientation. As it was shown on copper based Cu–Al single crystals [5], a change of solute concentration affect strongly the  $\gamma/b$  value and hence stresses operating in alloy crystals may satisfy a condition for twinning to occur. The same affect may have the deformation temperature. Blewitt and co-workers [10] and also recent investigations [11] have shown that for the crystal orientations used in the experiment the liquid helium temperature is enough to change the transformation from slip-slip into slip-twin type. Then, the dominant slip system is replaced by a twin system operating on the conjugate plane C in the most stressed twin shear direction. So, the deformation twinning would also belong to the phenomena of dominant deformation system transformation.

### 4. Conclusions

As it is observed in the case of copper single crystals, the transformation of the dominant deformation system generates plastic instabilities. These are responsible for the tensile instability of face centered cubic crystals, as well as, for the obset of the deformation stage IV. They do belong to the end of overshoot phenomena. The deformation twinning may also be considered as the transformation of the dominant deformation system. The transformation of the dominant deformation system appears discontinuously leading rapidly to heavy strain localisation. The critical parameters of the phenomenon depend strongly on crystal orientation and this is probably a main reason why polycrystals reveal rather smooth transition from the stage III of parabolic hardening into the stage IV of linear hardening.

All these strain-induced phenomena possess a transformational character, which must be taken into account in order to develop a proper physical theory of large plastic deformation of crystalline materials.

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