# LETTER TO THE EDITOR

# MECHANICAL TWINNING IN POLYCRYSTALLINE COPPER AT 20 K SYED M. RAZA, NAEEM FAROOQUI, SYED. B. H. ABIDI

and

### ABDUL. K. KHAN

Department of Physics, University of Baluchistan, Quetta, Pakistan

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Recent observations of mechanical twins in the deformation process of polycrystalline copper at 20 K suggest that twinning is a characteristic of low-temperature deformation and occurs by the movement of partial dislocations. At low strains, cross-slip is partially expected due to widening of stacking fault ribbon. Cross-slip is inhibited at larger strains due to strain enhancement effect. The latent hardening properties of copper are extended for the prediction of general characteristics of plastic flow. Plastic accomodation in polycrystals will then occur by contiguous single or double slip domains within the grains. It is concluded that the twin surfaces are formed due to strain-enhancement and stress-raising effects.

Mechanical twinning has been studied in f. c. c. metals by Blewitt et al. 1.2) since 1954. The exact mechanism of twinning is not known, except that it should occur by the propagation of a half dislocation and its associated stacking fault across each plane of a set of parallel (111) planes. However, the stacking faults are wider than the equilibrium separation of the partials, so that the twin process should involve one of the half dislocation being anchored while the other half for the twin to thicken, the half dislocation should also climb onto successive twin planes. Nevertheless, mechanical twinning is thought to take place by a dislocation mechanism for the same reason as slip but the dislocations that cause twinning are partials and not unnit dislocations.

Further studies on f. c. c. metals<sup>3-8)</sup> and copper-base alloys<sup>9-11)</sup> revealed the fact that mechanical twinning in most cases occurred at low temperatures. In some cases, twinning can be artificially produced at 78 K and room temperature<sup>3,4)</sup>. The apparent restriction of twinning to certain orientations, at low temperatures, may be ascribed to high shear stress attained on crystals with these orientations, since the stress necessary to produce twinning is high.

It has been confirmed that twinning in copper-base alloys  $^{9-11}$  increases with increasing stacking fault energy. Possibly, the solid solution hardening may inhibit widening of stacking fault ribbon due to pinning of stacking faults. Therefore, cross-slip may be comparatively easier in solid solutions than in  $f.\ c.\ c.$  metals. The role of mechanical twinning in the deformation process of polycrystalline copper at 20 K is critically discussed.

Copper (99.999%) rods of 5 mm diameter were annealed for 50 minutes at 800 °C in  $1.3 \times 10^4$  Pa of hydrogen to permit recrystallization, and to extract oxygen<sup>12)</sup>. They were then allowed to cool to 650 °C in the hydrogen gas for about 15 minutes, and were subsequently held at this temperature without hydrogen gas for 30 minutes in a dynamic vacuum of  $1.3 \times 10^{-2}$  Pa. All annealed copper specimens were chemically polished for 4 minutes at 40 °C in a solution containing  $H_3PO_4$  (25 vol %), acetic acid (25 vol%) and nitric acid (50 vol %), to remove about 15  $\mu$ m of the surface. To facilitate the measurement of the grain size, surfaces of few sample specimens were etched for 2 seconds in a solution of ferric chloride (0.016 kg Fe Cl<sub>3</sub> in 50 ml of concentrated HCl and 200 ml of water). The average grain diameter of copper specimen was 140  $\mu$ m. The chemi-

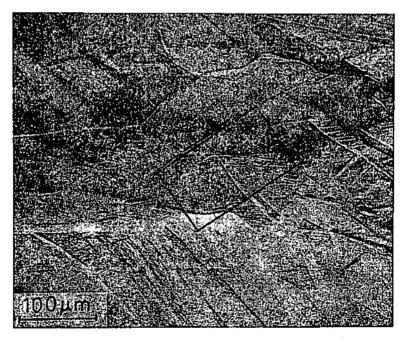


Fig. 1. Copper (grain size = 140  $\mu$ m); T = 20 K; Stress level = 98 MN/m<sup>2</sup>; Strain (%) = 9.8;  $\times$  200.

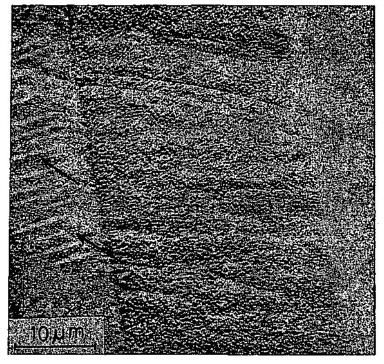


Fig. 2. Copper (grain size = 140  $\mu$ m); T = 20 K; Stress level = 98 MN/m<sup>2</sup>; Strain (%) = 9.8; × 2000.

cally polished specimens of copper (99.999%) were deformed in compression at a strain rate of  $2.3 \times 10^{-5} \, \mathrm{s^{-1}}$ . The continuous flow cryostat (Oxford Instruments) was used in the experiment. The deformed specimens at 20 K were then examined by the scanning electron microscope. In order to render any oxide film on the surface of the specimen (if present) conducting, the copper specimens were coated with gold in a bell jar at a vacuum of  $1.3 \times 10^{-4} \, \mathrm{Pa}$  for about 5 minutes, a layer of approximately 20 nm of gold was thus deposited.

Figs. 1 and 2 represent the scanning electron micrographs of the deformed surfaces of polycrystalline copper at 20 K. It is interesting to note that the mechanical twins are formed near the boundary of annealing twins.

Slip bands in polycrystalline metals at low temperatures were found to be of lengths commensurable with diameter of the grains in the pre-yield band formation  $^{13}$ ). This suggests that the intergranular stress field is significantly enhanced near grain boundaries, at low temperatures. Fig. 1 shows that the length of slip bands in copper at 20 K is almost equal to the size of the grains, i. e., 140  $\mu$ m which is indicative to support our view that the backward stresses do not seem to be important due to long slip band formation and glide accommodation on discrete slip systems. Possibly, the strain incompatibilities in the favourably oriented grain of a polycrystal due to latent hardening may become the cause of stress state changes near grain boundaries, at low temperatures. Evidence of forward

stresses resulting in the form of serrated cracks  $^{13}$ ) near grain boundaries according to Hall-Petch mechanism suggest that there should be a stress-raising effect due to pile-up of dislocations, Since, copper exibits higher latent hardening behaviour than aluminium  $^{14}$ , the glide ability of any slip system do not only need Schmid factor  $F_s$ , but  $F_s|LHR$  (where LHR is the latent hardening ratio). Strong latent hardening makes less probable the simultaneous homogeneous activation of many slip systems in the grains of a polycrystal, as predicted, for instance, by Taylor's theory, and more likely the fragmentation of grains into separated areas with only one or two active slip system  $^{15}$ . Plastic accomodation in polycrystals will then occur according to an energetically more favourable process  $^{15}$ , namely the formation of contiguous single or double slip domains within the grains which is an observes case  $^{13}$ . The same observation can also be verified from Figs. 1 and 2.

The isolated screw dipoles in copper single crystals at 20 K, in the process of deformation, seem to be retained by virtue of pinning 16). This indicates that there is the least possibility of cross-slip at low temperatures. Again, the inhomogenieties of deformation, as observed on the surface of copper single crystals by Buck and Essmann<sup>17)</sup> suggest that cross-slipping is restricted even in the initial stages of hardening. Since, we know that copper has medium stacking fault energy, cross-slip should be relatively easier than for metals having low stacking fault energies. There are at least two slip systems operative in single crystals and necessarily in polycrystals in plastic deformation. Occurence of secondary slips correspond to latent hardening behaviour which is reminiscent of the long hands in easy glide. This may reduce the occurrence of cross-slip for interacting slip systems at low temperatures due to freezing in of secondary slips on the primary ones. Thus, the only alternate for cross-slip in f. c. c. metals is by Friedel-Escaig model 18) in which the driving force for cross-slipping is the widening of the stacking fault ribbon. Thus, partial cross-slip is expected in the initial stages of hardening. The nucleation of twinning stress due to widening of stacking fault ribbons is also possible. At high strains, cross-slip is restricted due to more and more widening of stacking fault ribbons. With further increasing strain; sessiles, Cottrell-Lomer barriers and other sources are formed at dislocation pile--ups which leads to stress-raising effffect causing restriction on cross-slip. This would ultimately result in the enhancement of strains in the contiguous single or double slip domains of the grains, thereby producing twin surfaces due to unlocking of partial dislocations.

The pinning of stacking faults through solute atoms becomes a source for attracting barriers, sessiles and Cottrell-Lomer locks, thereby reducing the width of the faulted ribbon and making the cross-slip relatively easier. Therefore, work hardening mechanism in solid solutions, at low temperatures, is different than for normal pure metals.

## Hence, we conclude that:

- (i) Nucleation of twinning stresses may be due to widening of stacking fault ribbons.
- (ii) Latent hardening behaviour may contribute significant effects on the formation of twinning surfaces, at low temperatures.
- (iii) Twinning in f. c. c. metals occurs due to strain enhancement and stress-raising effects.

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(iv) The mechanism for the formation of twinning surfaces in case of f. c. c. metals, at low temperatures, is quite different than in the case of solid solutions.

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## PROUČAVANJE MEHANIČKIH DVOJNIKA U POLIKRISTALNOM BAKRU PRI 20 K

SYED M. RAZA, NAEEM FAROOQUI, SYED B. H. ABIDI

## ABDUL K. KHAN

Department of Physics, University of Baluchistan, Quetta, Pakistan

## UDK 538.951

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Proučavanje mehaničkih kristala dvojnika u procesu deformacije polikristalnog bakra pri 20 K navodi na pretpostavku da je dvojničenje karakteristika niskotemperaturne deformacije i da se zbiva gibanjem parcijalnih dislokacija. Pri malim deformacijama očekuje se djelomično poprečno klizanje zbog širenja vrpci pogrešaka u slaganju. Pri većim deformacijama efekt pojačanog naprezanja spriječava poprečno klizanje. Autori zaključuju da se površine dvojnika formiraju upravo zbog efekata pojačane deformacije i popratnog rastućeg naprezanja.