

Deformation Behaviour of Copper Single Crystals with $[100]$ and $[110]$ Tensile Axis

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I Introduction

Among the hardness testing methods for material, some kinds of indentation hardness testing methods have been widely used because of the simple operation of tester and the easy preparation of specimen. Investigating the physical meaning of indentation hardness value, the deformation behaviours of indentation on (001) of Cu have been studied by observing the distribution of dislocations with etch pits method and transmission electron microscopy, and the deformation modes and the barriers to dislocation motions are considered^{(1), (2)}. Consequently, it is found that $[110]$ deformed regions of the indentation on (001) plane are wider than $[100]$ one. Because the stress around the axis of indenter distributes uniformly, it is found that the deformation in $[100]$ region of indentation is more difficult than the $[110]$ one. The results of Knoop hardness test on (001) of Cu, which is more appropriate for studying the dependence of hardness values upon the crystallographic orientation, show that the value for $[100]$ direction is higher than $[110]$, and the conclusion mentioned above is also seemed to be reasonable. For explaining these phenomena, the dislocation reactions based on the mechanism of indentation and the difference of strength between the sessile dislocations in $[100]$ deformed region and $[110]$ one are considered, however the quantitative explanation are not obtained.

It is well known that the flow stresses are represented by a linear function of the inverse of dislocation cell size^{(3), (4), (5)}. Therefore, it seems to be reasonable that the dependence of deformation behaviours of indentation and of Knoop hardness value upon the crystallographic orientation is explained by the cell size and the cell structures.

The distribution of dislocations in the indentation made by conical and Knoop indenter have been investigated by dislocation etching and transmission electron microscopy, however the sufficient results about the cell size and the cell structure have not been obtained.

In the present work, Cu single crystal plates, which have [100] and [110] tensile axis are deformed in tension and the distributions of dislocations, particularly dislocation cell structures are observed with transmission electron microscope. The results seem to be useful to explained the dependence of the deformation of indentation and Knoop hardness values on (001) plane upon the crystallographic orientation because Knoop hardness values are determined by the resistance to deformation of material along the direction of short diagonal of indenter⁽⁶⁾.

II Experimental Procedures

OFHC copper as material for specimen was selected because of its low stacking fault energy and its primitive behaviours of deformation. A single crystal was grown in graphic boat in vacuum of 2×10^{-4} mmHg by Bridgman method. Its size was $12 \times 200 \times 1$ mm³ and a set of surface (12×200 mm²) was (001) plane. The initial dislocation density of the crystal was about 5×10^4 /mm². The specimens for tensile test were cut from the crystals in $5 \times 30 \times 1$ mm³ in strain free condition and were electrolytically polished in phosphoric acid and ethyl alcohol solution. The one of them had [100] tensile axis and the other [110]. The former was called [100]-specimen and the latter [110]-one. Tensile deformation of the specimen was carried out at room temperature with strain rate $\dot{\epsilon} \cong 10^{-2}$ /sec in Shimadzu autograph IS-2000.

After the deformation, the distribution of the slip line on a (001) plane was observed by optical and electron microscope with replica method and the specimen was electrolytically polished for transmission electron microscopy.

III Results and Considerations

3-1 Stress-Strain Curves

Fig. 1 shows the shear stress-shear strain curves of [100]-specimen deformed in tension and Fig. 1 (c) shows one deformed to fracture. From the early stage of the deformation the remarkable work hardening appears, and from about 5% strain more remarkable one does in straight line. From about 20% strain the work hardening rate decreases gradually to fracture. Corresponding to the typical stress-strain curves obtained in FCC metal, stage I is not observed and the transition one is done first; subsequently the stage II and III are observed. It is already reported that when Al and Cu single crystals having [100] tensile axis are deformed in tension

with strain rate $\dot{\epsilon} = 10^{-5}$ /sec, for Al stage IV is observed after stage III and for Cu it is not done, however the serrations appear^{(7), (8)}. In the present work, both the stage IV and serrations are not observed and the strain rate $\dot{\epsilon} \cong 10^{-2}$ /sec seems to be responsible for the latter phenomenon.

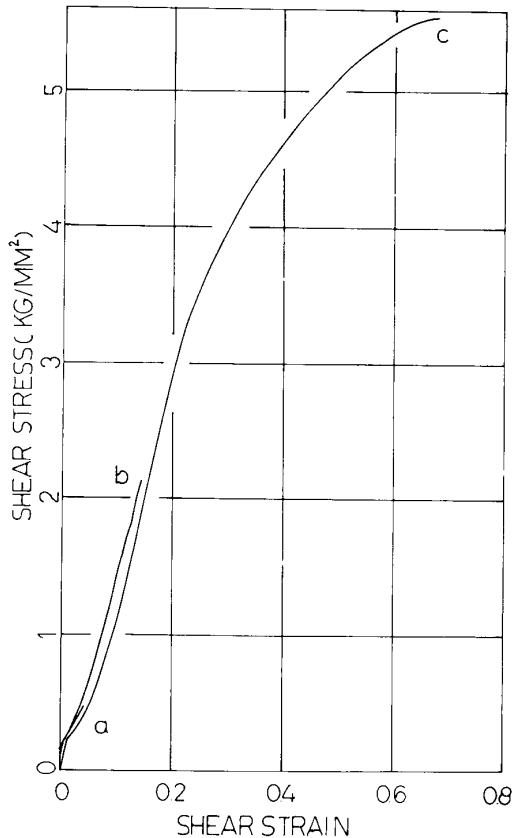


Fig. 1 Shear stress-shear strain curves of $[100]$ -specimen deformed in tension to (a) 4%, (b) 14% and (c) 68% strain.

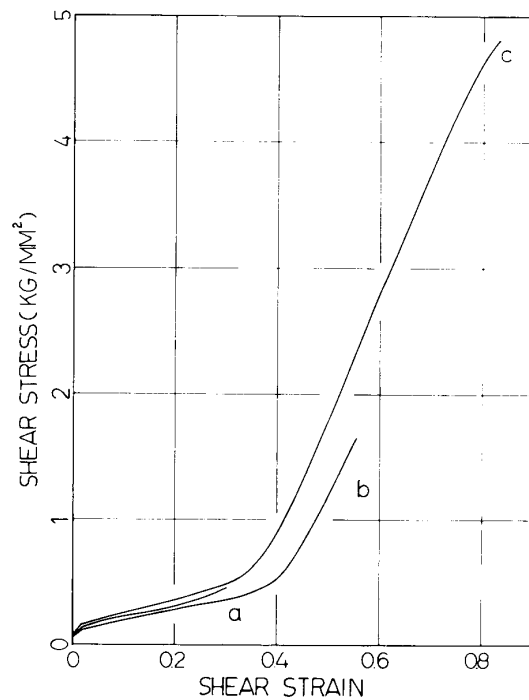


Fig. 2 Shear stress-shear strain curves of $[110]$ -specimen deformed in tension to (a) 30%, (b) 55% and (c) 80% strain.

Fig. 2 shows the shear stress-shear strain curves of $[110]$ -specimen deformed in tension. Fig. 2 (c) shows one deformed to fracture and stage I, II and III are observed. The strain to fracture is about 80% in three times.

3-2 Distributions of slip lines

Photo. 1 (a) shows the distribution of slip lines on (001) plate of $[100]$ -specimen deformed to 4% strain. The slip lines are almost in fine and parallel with each other, however in a few regions some of them are crossed one another. Also, some of the slip lines seem to be the feature in early stage of clustered band. Photo. 1 (b) shows slip lines deformed to 14% strain. The width of clustered slip band grows with increase of strain and in a few regions, fine slip lines cross one another

in perpendicular (see arrow). Photo. 1 (c) shows slip lines deformed to 37% strain. Clustered slip bands grow furthermore and fine multiple slip line take place in a few regions. At least, the operation of 4 slip systems is observed with replica method. Photo. 1 (d) shows slip lines deformed to fracture and new and sharp slip lines are observed over the whole region. A prominent cross slip^{(7), (8), (9)}, which is observed in Al and Cu single crystal deformed in [100] tensile direction is not observed. This seems to be responsible for the difficulty of cross slipping because of strain rate $\dot{\epsilon} \approx 10^{-2}$ / sec.

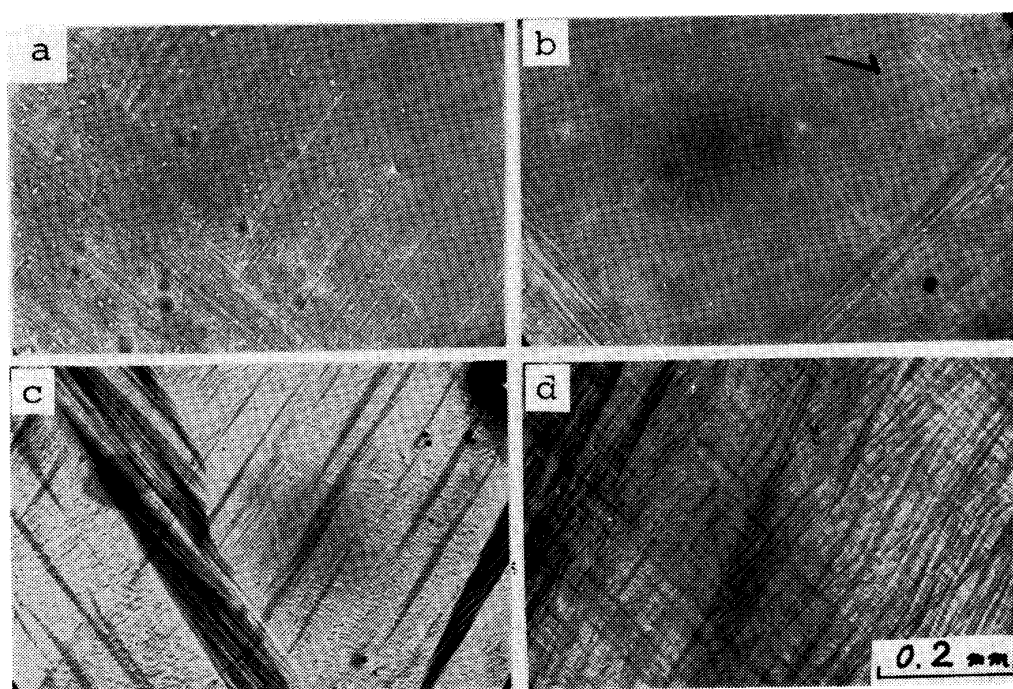


Photo. 1 Slip lines on (001) surface of [100]-specimen ;
 (a) deformed at 4%, (b) 14%, (c) 37% and (d) 67% strain.

Photo. 2 (a) shows a replica micrograph of the distribution of slip lines on (001) plane of [110]-specimen deformed to 30% strain. The slip lines are in fine and straight, and the operative slip system seems to be one. Photo. 2 (b) shows the one deformed to 55% strain. Slip lines are in fine and straight same as Photo. 2 (a), however slip lines with relatively high step are observed in the spacing of $1.5 \mu - 2.0 \mu$. The operative slip system seems to be two or more. Photo. 2 (c) shows the one deformed to fracture, and almost slip lines are coarse and in spacing of $0.5 \mu - 1.0 \mu$.

3-3 Distributions of dislocations

Photo. 3 shows the transmission electron micrographs of [100]-specimen deformed to 4% strain. It is found that dislocation bundles parallel to [110] direction shown in Photo. 3 (a) are observed in about 60% of all observed micrograph

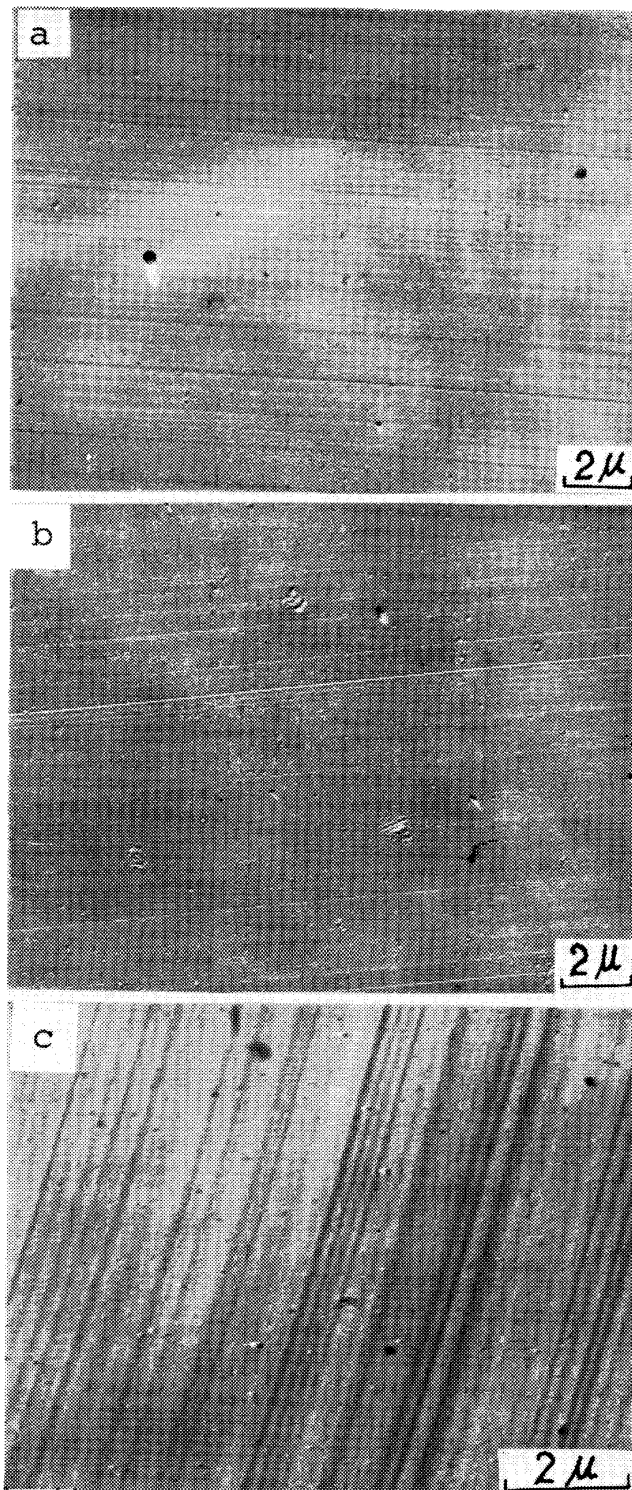


Photo. 2 Electron micrographs of slip lines on (001) surface of $[110]$ -specimen ; (a) deformed at 30%, (b) 55% and (c) 80% strain.

and dislocation cell structures shown in Photo. 3 (b) are observed in about 10%. In the rest, dislocation tangles are observed and dislocation density is low.

Photo. 4 shows the micrographs deformed to 37% strain. In Fig. 4 (a) the cell structures with distinct cell walls are observed and the dislocation density in cell walls

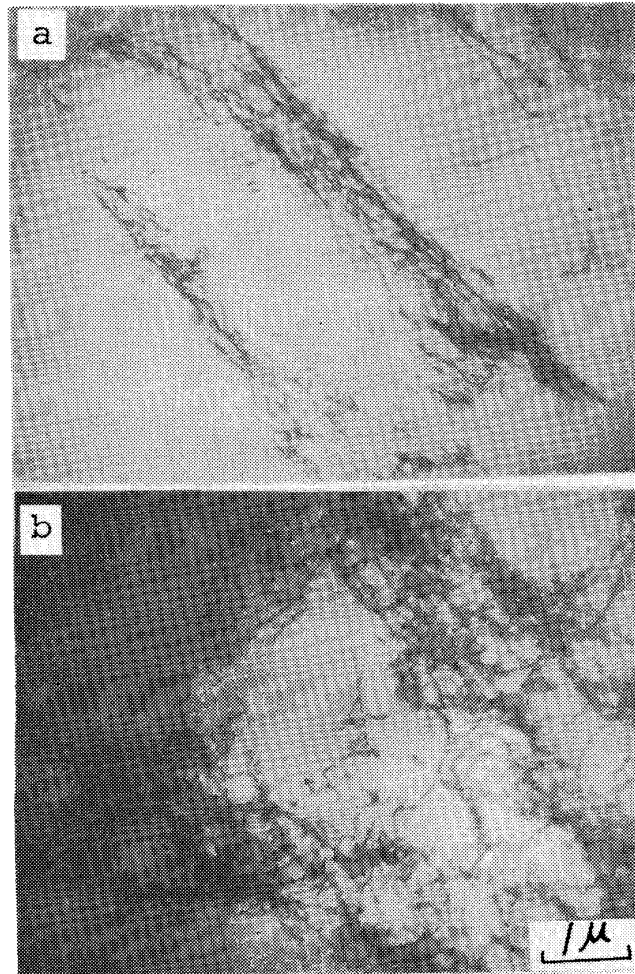


Photo. 3 Distribution of dislocations of $[100]$ -specimen deformed at 4% strain.

is higher than one shown in Photo. 3 (b). The feature shown in Photo. 4 (a) are observed in about 50% of all observed micrograph. The rest as observed in Photo. 4 (b) shows the dislocation density in the walls is lower and the density in cell is higher than one in Photo. 4 (a). The shape of cell structures is in circular or square and the cell sizes are in $1.5 \mu\text{--}2.5 \mu$ in diameter. Photo. 5 shows the micrographs deformed to 68% strain, i.e. fracture. It is found that cell structures are observed in the whole region, and the cell sizes are almost $0.3 \mu\text{--}0.7 \mu$ in diameter and the dislocation density in cell is higher.

Photo. 6 shows the transmission electron micrograph of $[110]$ -specimen deformed to 55% strain. Dislocation bundles parallel to $[110]$ are observed in whole region and the spacing of them are about 1μ , which corresponds to the spacing of the slip lines with relatively high step observed in Photo. 2 (b). The dislocation cell structures are hardly observed.

Photo. 7 shows the micrograph deformed to 80% strain. Dislocation bundles in

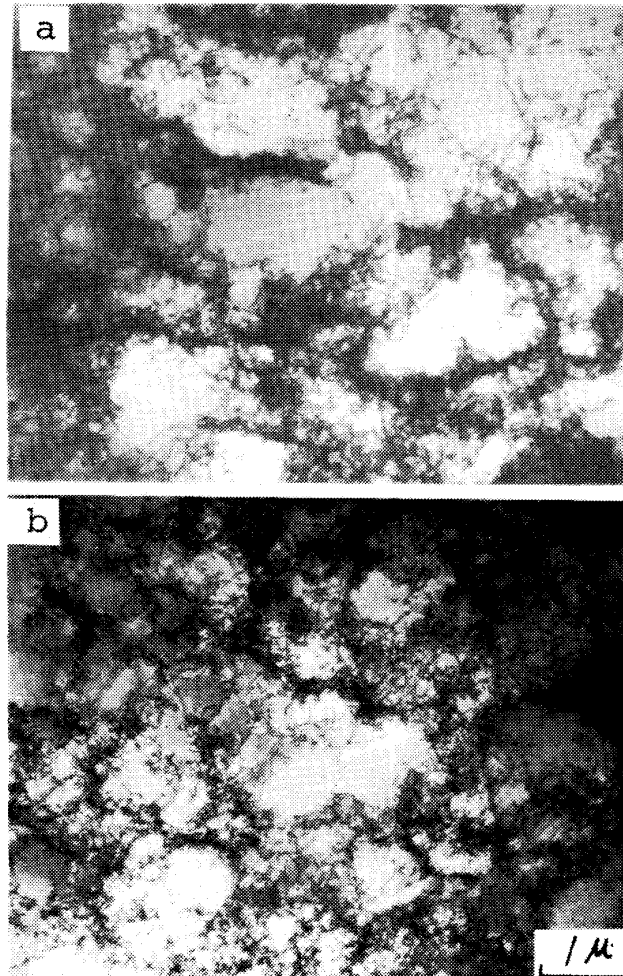


Photo. 4 Distribution of dislocations of $[100]$ -specimen deformed at 37% strain.

higher density parallel to $[110]$ with narrow spacing are observed in whole region. However, dislocation bundles perpendicular to them and the rectangular cell structures are observed too. A spacing of dislocation bundles corresponding to long side of the rectangle seems to be agreement with one of the coarse slip band observed in Photo. 2 (c). The cell sizes are about $2\mu \times 1.5\mu$.

According to these results, it is found that in $[100]$ -specimen the cell structures are formed from the onset of deformation, however in $[110]$ -specimen they are not formed even in considerably deformed stage. In the former, it seems to be thought that eight slip systems on four slip planes operate simultaneously, the cell structures are formed from the early stage of the deformation, and the cell formation is advanced with increasing the deformation because the tensile direction is hardly changed by the deformation. In the latter, it seems to be thought that two slip systems operate simultaneously and dislocation bundles are mainly formed by dislocations on two slip systems. Also, the dependence of Knoop hardness values

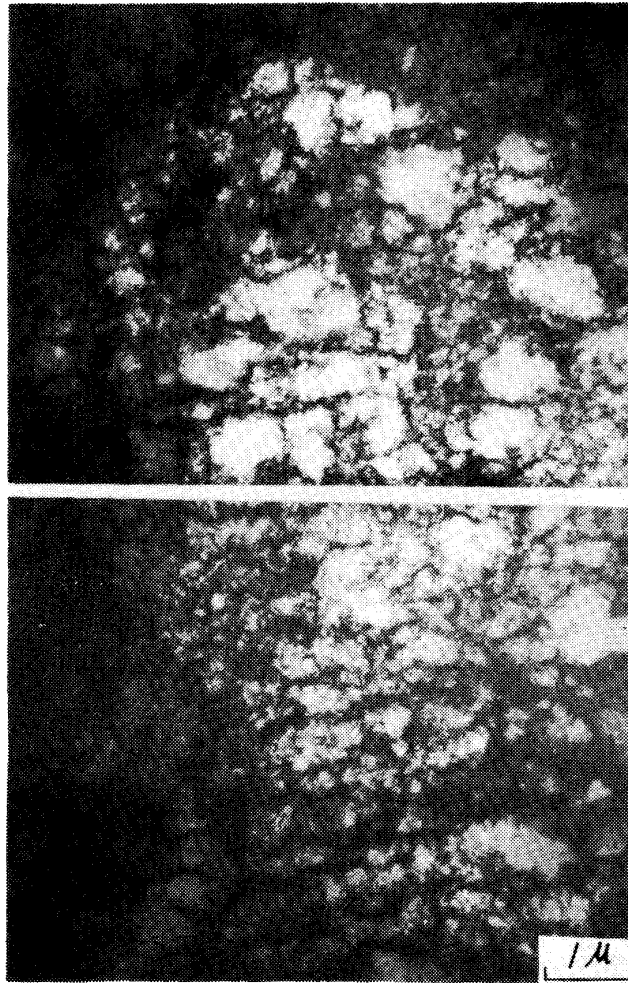


Photo. 5 Distribution of dislocations of [100]-specimen deformed at 68% strain.

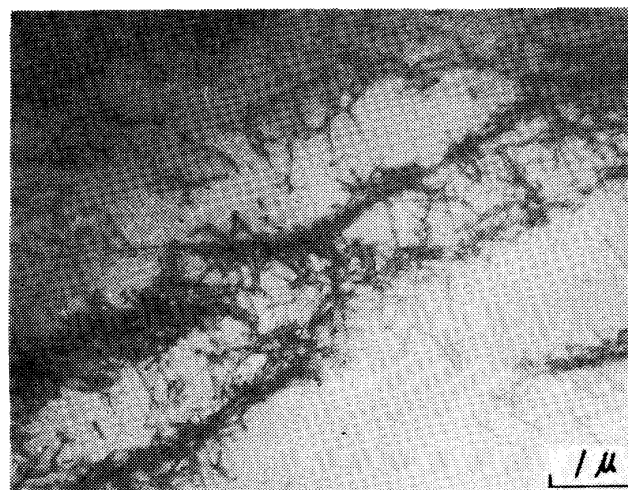


Photo. 6 Distribution of dislocations of [110]-specimen deformed at 55% strain.

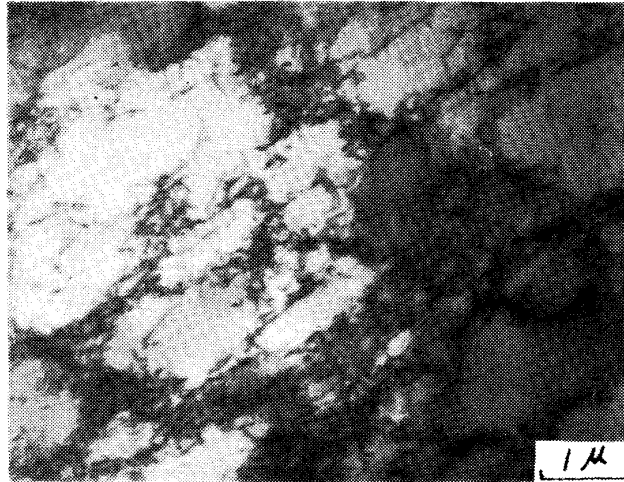


Photo. 7 Distribution of dislocations of $[110]$ -specimen deformed at 80% strain.

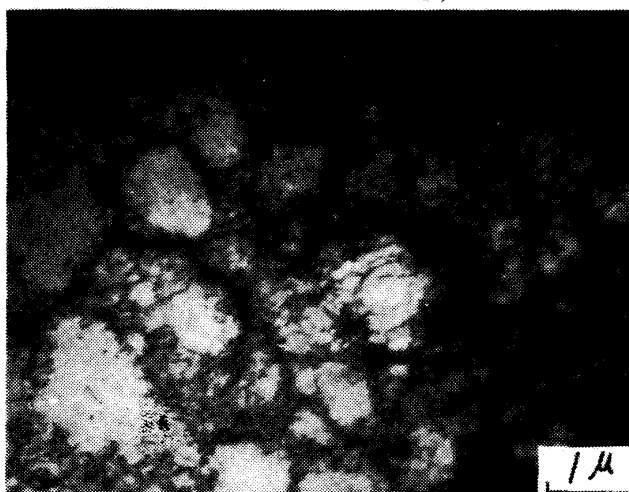
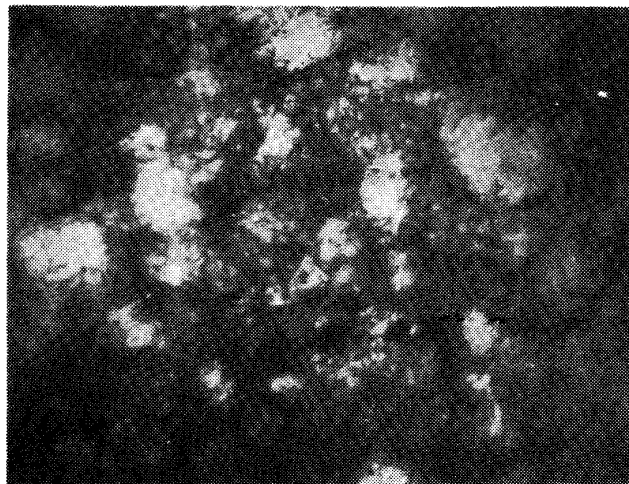


Photo. 8 Distribution of dislocations in the region beneath indentation center on Cu (001) plane.

upon the crystallographic orientation seems to correspond to the dependence of cell formation upon it.

Photo. 8 shows the transmission electron micrographs in the region beneath indentation center, which is made on (001) plane of Cu single crystal under the following conditions: a conical indenter with apex angle 144° , a load of 200 g, a loading time for 60 sec and room temperature. Over the whole region, distinct cell structures are observed, the shapes of them are almost in circular and the cell size is smaller than 1μ in diameter. These features seem to correspond to one observed in the crystal with [100] axis deformed in tension. If it is assumed that the distribution of dislocation in the specimen deformed in compression corresponds to one in tension, from the results of [100]-specimen, the state of deformation beneath indentation center seems to correspond to one deformed nearly to fracture.

VI Summary

Investigating the dependence of indentation and Knoop hardness values on Cu (001) plane upon the crystallographic orientation with dislocation cell structures, Cu single crystal plates which have [100] and [110] tensile axis and (001) plane are deformed in tension and the distribution of dislocations at some stages of deformation is observed by transmission electron microscope. The results obtained are as follows;

(1) In [100]-specimens, the dislocation cell structures are observed in early stage of deformation. Increasing the strain, the cell size decreases and the dislocation density in the cell walls increases.

(2) In [110]-specimens, the cell structures are hardly observed nearly to the fracture and the dislocation bundles parallel to [110] direction are mainly observed.

(3) Comparing the cell structures, the deformation beneath the indentation center seems to correspond to one in [100]-specimen deformed in tension nearly to fracture.

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