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Orientation correlation in tensile deformed [0 1 1] Cu single crystals

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Abstract

Local crystallographic orientation of tensile deformed copper single crystals was investigated by the electron backscattering technique. Statistical evaluation of the data reveals the presence of an increased crystallographic correlation at the transition point between stages II and III of work-hardening. The transition state has the lowest probability of finding geometrically necessary dislocations in circular regions of radius smaller than 8 μm. According to the present results and other data showing that the relative fluctuation of the dislocation density has a maximum at the transition point, we conclude that the transition from stages II to III of work-hardening is similar to a second-order phase transformation of the statistical dislocation system.

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Keywords: Copper; Work-hardening; Dislocations; Electron backscattering diffraction

1. Introduction

Plastic deformation of f.c.c. single crystals results generally in a heterogeneous arrangement of dislocations, which evolves gradually into a dislocation-cell structure consisting of regions of locally low and high dislocation densities. The connection between microscopic deformation mechanisms and macroscopic behavior of the single crystal is qualitatively well understood, however a generally accepted theory does not exist [1]. In order to get a better understanding of the deformation process, novel investigation techniques have to be applied, which enable a statistical evaluation of the microstructure indispensable to understand the collective behavior of dislocation ensembles. While single dislocations and the geometry of the resulting structure can be well characterized by transmission electron microscopy (TEM) [2,3], the statistical evaluation of such structures was possible only after the development of automatic orientation indexing techniques, available now in the scanning as well in the transmission electron microscope [4]. Beside electron microscopy the general theory of dislocation induced X-ray peak broadening [5] also enables a model-independent determination of the statistical properties of dislocation ensembles. It was shown by Székely et al. [6,7] that during compression of copper single crystals, a characteristic change in the relative fluctuation of the dislocation density takes place (see also Groma et al. [8]), which has a maximum at the transition point between stages II and III of work-hardening. This effect is definitely related to the collective behavior of dislocations, which may lead to a change in the deformation mechanism in stage III. Beside the determination of the total dislocation density and its fluctuation, X-ray diffraction peak broadening enables the evaluation of the polarization of the dislocation arrangement, too, which is obtained from the peak asymmetry. Dislocation polarization is mainly determined by the excess dislocations with a given sign of their Burgers vector, however its physical interpretation from X-ray data is rather difficult [9]. Similar information on the excess dislocations can be obtained by the electron backscattering diffraction (EBSD) technique available in automatic mode. EBSD has the advantage over the laboratory X-ray setups that it can be performed locally on a very fine grid (step size ≈ 0.1 μm) and the data can be directly analyzed to reveal spatial correlations [10,11]. In a common

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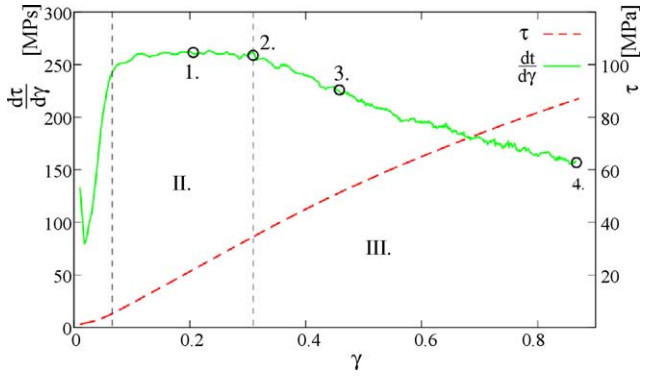

Fig. 1. Stress–strain and work-hardening rate curves of tensile deformed copper single crystal, having an initial orientation of (−1, 8, 11). The states marked by numbers from 1 to 4 were investigated by EBSD.

laboratory X-ray peak-profile measurement, a relatively large area is illuminated (≈1 mm^2) and the statistical averaging is done by the experiment itself. EBSD like local X-ray micro-diffraction studies can be also performed, however, at a synchrotron source [12], but the measurement is not automatic and the number of affordable measurement points does not allow a statistical evaluation. In this paper, we present EBSD results, complementary to X-ray and nano-indentation results [8], about the statistical properties of dislocation arrangements, which are formed during tensile straining of copper single crystals.

2. Experimental

Copper single crystals of 4N purity having initial orientation near the [0 1 1] pole of the stereographic triangle have been deformed in tension at room temperature at a strain rate of $10^{-3}\,s^{-1}$. The stress-strain curve (resolved shear stress as a function of shear strain) as well as the work-hardening rate are shown in Fig. 1. The deformation was stopped at characteristic points, marked by numbers from 1 to 4, and the corresponding deformation states were investigated by EBSD as well as by nano-indentation [8].

The sample numbers and the corresponding strain and stress values are given in Table 1. Sample 1 represents the middle of stage II, sample 2 the transition state between stages II and III, while samples 3 and 4 are representative for stage III. The EBSD measurements were performed on a Philips XL-30 SEM equipped with an EDAX-TSL system for automatic recording and indexing. Scans of 150 μm × 120 μm

Table 1
Strain and stress values of the selected deformation states for EBSD investigation

Sample number	Strain (γ)	Stress (τ) (MPa)
1	0.21	24
2	0.31	33
3	0.46	52
4	0.87	87

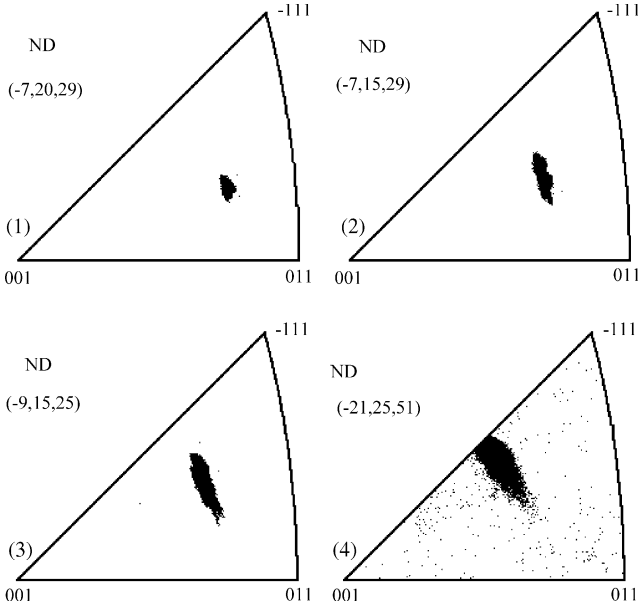

Fig. 2. Inverse-pole figures of the sample tensile axis for the investigated states (1–4).

were measured on each sample using a hexagonal grid with a step size of 0.5 μm. Indexing of Kikuchi patterns could be done on 99% of the measured points on the first three samples, while for the largest deformation state 4, the indexing rate was only 96%.

The measurements were carried out on sections with surfaces perpendicular and parallel to the tensile deformation axis. Since the two results are quite similar, in the following, only the results for the perpendicular sections will be shown.

Fig. 2 shows the distribution of the tensile axis (normal direction, ND) in the crystal local coordinate system. With increasing deformation, the spot of orientation becomes more elongated and moves to the line connecting the 0 0 1 and −1 1 1 poles. The average orientations of the tensile axes are also given in figure.

It is known that from a continuum point of view the geometrically necessary dislocations (GNDs) are the ones, which are required to support a given curvature of the crystalline lattice [13]. So, by measuring the local curvature of the crystal, we can conclude on the density of the GNDs. However, due to the electron opacity in crystalline materials, related to the small interaction depth of backscattered electrons with the surface (of about 20 nm) [14], not all the components of the rotation gradient tensor can be retrieved from EBSD. This makes rather difficult an exact quantitative evaluation of the components of the dislocation density tensor [15]. For this reason, we present mainly qualitative results for the GND density, which are based on total lattice curvature data.

Fig. 3 shows disorientation maps characterizing the four investigated states. The disorientation was calculated in each case as the smallest misorientation angle from the average orientation of the scans. A visual inspection of the pictures reveals that the structures have different length-scales, the