Statistical properties of dislocation structures investigated by X-ray diffraction

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Abstract

The method of X-ray line profile analysis was applied to obtain statistical parameters (average dislocation density, net dislocation polarization and average dislocation density fluctuation) of the dislocation structure developed in copper single crystals deformed in uniaxial compression. It is found that during the plastic deformation, while the dislocation density increases monotonously, the average fluctuation has a maximum at the transition from stage II to stage III work hardening. The fractal properties of the dislocation structure are also investigated. Strong correlation was found between the fractal dimension and the relative dislocation density fluctuation. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Static properties; Dislocation structures; Fractal properties

1. Introduction

The X-ray line profile analysis of deformed crystalline materials has proved to be a powerful tool to investigate the properties of the dislocation network [1–5]. Recent theoretical developments have made possible to determine certain parameters of the dislocation structure, like the average dislocation density, net dislocation polarization, and the average dislocation density fluctuation with high accuracy [4]. These parameters are important, since they provide a possibility for the experimental verification of the recently proposed stochastic dislocation dynamical theories which are focusing on the distribution functions of dislocations, and the internal stress [6–10].

In the present work, X-ray line profile analysis is applied for the investigation of copper single crystals deformed in uniaxial compression. The variations of the statistical parameters of the dislocation assembly during the deformation process are presented. Furthermore, the parameters measured by X-ray diffraction, will be correlated to the fractal dimensions determined from TEM-micrographs. The results are compared to the predictions of stochastic dislocation theories.

2. X-ray line profile analysis

The evaluation method of the X-ray diffraction line profile analysis used in this paper is based on the analytical results of Groma et al. [2–4]. According to this in the case of dislocation caused line broadening the asymptotic form of the Fourier-transform of the intensity distribution can be given with the following form up to fourth order in the Fourier parameter $n$ (see [2,4]).

$$\ln(A(n)) = A(\langle \rho \rangle) n^2 \ln \left( \frac{n}{R_1} \right) + i \langle s^2 \rangle n^3 \ln \left( \frac{n}{R_2} \right) + \frac{1}{2} A^2 (\langle \rho^2 \rangle - \langle \rho \rangle^2) \times n^4 \ln \left( \frac{n}{R_3} \right) \ln \left( \frac{n}{R_4} \right)$$

(1)

where $\langle \rho \rangle$ is the average dislocation density, $\langle \rho^2 \rangle$ the average of the square of the dislocation density, and $\langle s^2 \rangle$ a parameter measuring the correlation between the internal strain and the dislocation density [4]. The coefficient $A$ appearing in Eq. (1) is commonly referred to as the dislocation contrast factor (for its actual value see [4]). The quantities $R_1$, $R_2$, $R_3$ and $R_4$ are characteristic length parameters of the dislocation assembly, their actual values, however, are difficult to determine.

The evaluation method used in the present work is based on the analysis of the integration limit dependence of the moments of the measured intensity distribution, i.e. the behavior of the function [4]

$$v_k(q') = \frac{\int_{-q'}^{q'} q^k I(q) dq}{\int_{-\infty}^{\infty} I(q) dq}$$

(2)

(referred to hereafter as $k$th order restricted moment) from which at large enough $q'$ value, the parameters $\langle \rho \rangle$, $\langle \rho^2 \rangle$, $\langle s^2 \rangle$...
and \( \langle s^2 \rangle \) can be determined [4]. On Fig. 1, an example of the evaluation method is presented on a line profile of the \( \langle 200 \rangle \) reflection of a copper single crystal deformed in compression. The measured profile is plotted on Fig. 1a, while the different order restricted moments are shown in Fig. 1b. The line profiles are usually not symmetric resulting in a nonzero third order restricted moment which has the asymptotic form

\[
v_3(q') = -6 \langle s^2 \rangle \ln \left( \frac{q'}{q_1} \right) \tag{4}
\]

allowing to obtain \( \langle s^2 \rangle \) from the slope of the \( v_3(q) \) versus \( \ln(q) \) relation in the large \( q \) regime. (In Fig. 1b, the second order restricted moment of the profile of Fig. 1a is plotted with the fitted curve given by the expression (4)).

Both the fluctuation and the average dislocation density can be obtained from the asymptotic form of the fourth order restricted moment [4]

\[
v_4(q') = \Lambda(\rho)q'^2 + 12 \Lambda^2(q^2) \ln \left( \frac{q'}{q_0} \right) \ln \left( \frac{q'}{q_0} \right) \tag{5}
\]

In Fig. 1b, the \( f(q') = v_4(q')/q'^2 \) quantity corresponding to the profile of Fig. 1a is plotted. As it can be seen from expression (5), the asymptotic value of \( f(q') \) is \( \Lambda(\rho) = \rho^6 \), the formal dislocation density and it has a maximum determined by the ratio \( \langle \rho^2 \rangle/\langle \rho \rangle^2 \). The fitted function \( f(q') \) obtained from expression (5) is also plotted on the figure.

It is important to note that determining \( \langle \rho \rangle \) both from the expressions (3) and (5) gives a possibility for the internal checking of the consistency of the evaluation method.

3. Fractal properties of dislocation structures

In a recent work, Hähner et al. [11] by analyzing TEM micrographs, investigated the fractal properties of the dislocation structure developing in \( \langle 100 \rangle \) oriented copper single crystals deformed in tension. They interpreted the observed properties within the framework of the stochastic dislocation dynamics theory proposed by Hähner [6], and found that the dislocation pattern developing in unidirectional deformation is a self-similar hole fractal, i.e. the probability of the cell size (\( \lambda \)) being larger than a given \( L \) is hyperbolic:

\[
P(\lambda > L) \propto L^{-D} \tag{6}
\]

between the limits \( \lambda_{\min} < \lambda < \lambda_{\max} \), where \( D \) is the fractal dimension. They obtained the same value for \( D \) by box-counting and from the self-correlation function [12]. Since the fractal dimension determined from TEM micrographs corresponding to a two dimensional cross-section of the three dimensional dislocation array, the real fractal dimension of the dislocation cell structure is \( D + 1 \).

According to the suggestion of Zaiser and Hähner [13], the hole fractal can be considered as a generalized composite consisting of dislocation free regions, i.e. the cell interiors with hyperbolic size distribution, and the cell walls with \( \rho_w \) dislocation density. Using this assumption, the average dislocation density and the average of the square of the dislocation density can be given as

\[
\langle \rho \rangle = f_w \rho_w \quad \langle \rho^2 \rangle = f_w^2 \rho_w^2 \tag{7}
\]

The externally applied stress \( \tau_{\text{ext}} \) is the average of the local stresses, i.e. the stress in the cell walls, which can be expressed with the local dislocation density \( \rho_w \), and the stress exerted by the cells, being inversely proportional to the cell size. By carrying out the stress averaging, Zaiser and Hähner [13] found that

\[
\tau_{\text{ext}} = \alpha_f \xi Gb \frac{f_w}{D-1} \sqrt{\rho_w} \tag{8}
\]
4. Results and discussion

The X-ray line profile evaluation method outlined in Section 2 is applied to copper single crystals deformed in uniaxial compression from the \(\langle 100 \rangle\) direction at \(2 \times 10^{-4}\) s\(^{-1}\) strain-rate. The X-ray line profiles were measured at two different reflection (\(\langle 200 \rangle\) and \(\langle 220 \rangle\)).

The squared root of the measured formal dislocation density \(\rho^* = \Lambda(\rho)\) versus external (resolved) shear stress is shown in Fig. 2. The \(\tau \propto \sqrt{\rho}\) relation can be seen from the linear fits plotted in the figures (the slopes of the straight lines in the two reflections are different because of the different contrast factors of dislocations in the two reflections).

The ratio \(\delta = \langle \rho^2 \rangle / \langle \rho \rangle^2\) (related to the relative dislocation density fluctuation \(\sigma^2 = \langle \rho^2 \rangle - \langle \rho \rangle^2 / \langle \rho \rangle^2\) as \(\sigma^2 = \delta - 1\)) versus stress is plotted in Fig. 3. The relative fluctuations obtained from the two reflections agree within the experimental error. An important feature of the dislocation density fluctuation is that it exhibits a sharp maximum at 40 MPa resolved shear stress corresponding to the stress level at which the stage II to III transition takes place. This indicates that the observed maximum originates from the structural transition in the dislocation network. Similar observations were made by Diehl [14,15] by measuring the relative fluctuation of the macroscopic strain on a copper single crystal deformed in tension. He found that the strain fluctuation increases during stages I and II of the work hardening, but decreases during stage III.

In order to relate the above result to the properties of the dislocation structures, TEM investigations were performed on (1 0 0) oriented thin foils. The fractal dimensions of the dislocation structures were determined by the box-counting method (Fig. 4). The \(D\) versus \(\delta\) plot (Fig. 5) indicates a strong correlation (with a linear relationship in the investigated regime) between the relative fluctuations obtained from the X-ray measurements and the fractal dimensions obtained from the TEM micrographs.

From Eq. (7) of the fractal composite model outlined in Section 3, the quantity \(\delta\) measured by the X-ray method can be expressed as

\[
\delta = \frac{\langle \rho^2 \rangle}{\langle \rho \rangle^2} = \frac{1}{f_w} \quad (9)
\]

At the same time, the \(f_w\) volume fraction of walls can be expressed by the parameters introduced in the fractal composite model (\(\lambda_{\min}, \lambda_{\max}\) and \(D\)). So, the parameter \(\delta\)
Fig. 5. The observed correlation between the fractal dimension of the dislocation structure and the $\delta$ parameter describing the fluctuation with the fitted linear relation $D = 1.975 - 0.0354\delta$.

quantifying the relative fluctuation, can be related to the volume fraction of the dislocation cell walls and through Eq. (8) to the external stress and the fractal dimension. This yields a possibility for the experimental verification of the assumptions of the fractal composite model.

According to the above results X-ray line profile analysis and fractal analysis of TEM images give comparable results with respect to the statistical properties of the dislocation structure developing during plastic deformation.

5. Conclusions

The $\langle 100 \rangle$ oriented copper single crystals deformed in uniaxial compression were investigated using X-ray diffraction and fractal analysis of TEM micrographs. It has been found that:

- the evaluation method of X-ray line profile analysis applied in the present work is capable of providing important statistical parameters of the dislocation structure (like average dislocation density, net dislocation polarization, relative fluctuation of the dislocation density) characterizing the dislocation structure;
- the relative fluctuation of the dislocation density has a maximum during the plastic straining at a stress corresponding to the transition between hardening stages II and III;
- the relative fluctuation and the fractal dimension of the dislocation array are in strong correlation.

Acknowledgements

The authors are grateful to Professor T. Ungár and M. Zaiser for valuable discussions and comments on the manuscript and to Dr. L. Zsoldos for assistance in the experimental work. The financial support of the Hungarian Scientific Research Fund (OTKA) under contract numbers T 029701 and 030791 is acknowledged.

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