

Characterization of self-similar dislocation patterns by x-ray diffraction

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Copper single crystals deformed in uniaxial compression were investigated by x-ray line profile analysis, and the resulting statistical parameters of the developed dislocation pattern are compared to the fractal dimension of the dislocation network determined by the box-counting method on transmission electron microscope micrograph. The observed correlation between the relative fluctuation of the dislocation density and the fractal dimension of the dislocation pattern is interpreted on the basis of the generalized (fractal) composite model.

I. INTRODUCTION

A fundamental feature of the dislocation network developing during the later stages of plastic deformation of metals is that the dislocation distribution becomes inhomogeneous and different patterning processes take place. Several analytical (Kulhman-Wilsdorf,¹ Holt,² Richman and Vinals,³ Walgraef and Aifantis,⁴ Kratochvil *et al.*,^{5,6} Groma *et al.*⁷⁻⁹) and numerical (Lepinoux and Kubin,¹⁰ Gulloughlu *et al.*,^{11,12} Ghoniem and Amodeo,¹³ Cleveringa *et al.*,¹⁴ Groma *et al.*,^{15,16} Devincere and Condat,¹⁷ Fivel *et al.*¹⁸) models have been developed since the first ordered dislocation assembly was discovered. In spite of this there is not a generally accepted model of this self-organizational phenomenon.

A recent important development in this field has been attained by Hähner *et al.*,¹⁹ who interpret the dislocation patterning processes on the basis of a stochastic dislocation dynamics model. They suggest that the developing dislocation networks are self-similar fractals, and the fractal dimension can be related to the noise level of the randomly fluctuating internal stress and consequently of the deformation rate.

The aim of the investigations presented in this paper is to study whether the fractal dimension of the dislocation arrangement (determined from TEM micrograph) can be related to certain statistical parameters of the dislocation distribution (like average dislocation density and average relative fluctuation), which can be measured by x-ray line profile analysis.

In the first part of the paper the determination of the relevant characteristic parameters of the dislocation arrangement from x-ray line profile analysis is outlined. Following, the concept of the self-similar dislocation network described by the generalized composite model of Zaiser and Hähner²⁰ are briefly summarized. Then the experimental results obtained by x-ray measurements and TEM on deformed Cu single crystals oriented for ideal multiple slip are presented. Finally the results will be compared with the predictions of the generalized composite model.

II. X-RAY LINE PROFILE ANALYSIS

X-ray line broadening analysis is a frequently applied technique for the investigation of dislocation structure in crystalline materials. It is successfully applied for studying the dislocation structure in plastically deformed metals (Un-

gar *et al.*^{21,22}), recently even in ball-milled nanocrystalline metals (Ungar *et al.*²³). In the investigations reported earlier, however, the observed asymmetric x-ray line profiles were evaluated with a procedure based on the concept of restricted random dislocation distribution (Wilkins²⁴) and on the quasi-composite model proposed by Mughrabi *et al.*,²⁵ i.e., it required a specific *a priori* model for the dislocation arrangement. It has been recently shown by Groma²⁶ that from the analysis of the tail of the line profiles three important parameters of the dislocation distribution can be evaluated without any *a priori* assumption for the dislocation distribution.

This evaluation bases on the general properties of the intensity distribution $I(q)$ and its Fourier transform $A(n)$, defined as

$$A(n) = \int I(q) \exp(-2\pi qn) dq, \quad (1)$$

where $I(q)$ is the intensity distribution, and $q = 4\pi/\lambda_x \sin(\Theta)$ in which λ_x is the wavelength of the x ray, and Θ is the scattering angle.

As it is shown in Groma *et al.*²⁷ and Groma,²⁶ for small enough Fourier parameter n the natural based logarithm of the Fourier transform of the measured intensity $A(n)$ in the case of dislocations, regardless of the actual distribution of the dislocations can be given with the following form (up to fourth order in n):

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

where

$$\langle \rho \rangle = \frac{1}{V} \int \rho(\vec{r}) d^3r \quad (3)$$

is the average dislocation density,

$$\langle \rho^2 \rangle = \frac{1}{V} \int \rho(\vec{r})^2 d^3r \quad (4)$$

is the average of the square of the dislocation density, and

$$\langle s^2 \rangle = \frac{2}{V} \Omega \pi \frac{g_l g_m}{|\vec{g}|} \int \rho(\vec{r}) \beta_{l,m}^{\text{tot}}(\vec{r}) d^3 r, \quad (5)$$

in which V is the volume of the crystal, \vec{g} is the diffraction vector, Ω is a parameter depending on the Burgers vector and \vec{g} (see Groma²⁶), and $\beta_{i,j}^{\text{tot}}(\vec{r})$ is the total distortion created by the dislocation system at point \vec{r} . So $\langle s^2 \rangle$ measures the correlation between the internal strain and the dislocation density. The parameters R_1, R_2, R_3 and R_4 are characteristic length parameters of the dislocation assembly, but their actual values are difficult to determine.

To investigate if there is any relation between the fractal dimension of the dislocation patterns and the parameters one can obtain from x-ray line profile a dimensionless quantity has to be introduced. An obvious choice for this is the relative fluctuation:

$$\sigma^2 = \frac{\langle \Delta \rho \rangle^2}{\langle \rho \rangle^2} = \frac{\langle \rho^2 \rangle - \langle \rho \rangle^2}{\langle \rho \rangle^2} = \delta - 1, \quad (6)$$

where

$$\delta = \frac{\langle \rho^2 \rangle}{\langle \rho \rangle^2}. \quad (7)$$

In earlier works the evaluation of the dislocation density and of the other parameters mentioned above was based on fitting form (2) to the Fourier transform of the measured intensity profile.^{27,28} A major disadvantage of this procedure comes from the experimental finding that only about the first ten Fourier coefficients are satisfactorily described by Eq. (2), and this may cause a relatively large scatter in the obtained parameters. Furthermore, there is no well established criterion for the determination of the fitting interval.

For the determination of the characteristic parameters of a distribution function usually the different order moments m_k are considered. They can be obtained from the Fourier-transform of the intensity distribution in the following way:

$$m_k = \frac{\int_{-\infty}^{\infty} q^k I(q) dq}{\int_{-\infty}^{\infty} I(q) dq} = (i)^k \frac{1}{A(0)} \left. \frac{d^k}{dn^k} A(n) \right|_{n=0}. \quad (8)$$

In the case of line broadening caused by the dislocations, however, according to Eq. (2) the second and higher order momentums are infinite, since the corresponding derivatives of $A(n)$ are infinite. So they cannot be used directly to obtain the characteristic parameters of the dislocation structure. By analyzing the integration limit dependence of the momentums, i.e., the behavior of the function

$$v_k(q') = \frac{\int_{-q'}^{q'} q^k I(q) dq}{\int_{-\infty}^{\infty} I(q) dq} \quad (9)$$

(referred to thereafter as k th order restricted moment) at large enough q value the parameters $\langle \rho \rangle$, $\langle \rho^2 \rangle$, and $\langle s^2 \rangle$ can be determined (Groma²⁶).

From Eqs. (2) and (9) the asymptotic form of the second order restricted moment and the intensity distribution are obtained as

$$v_2(q') = 2\Omega \langle \rho \rangle \ln \left(\frac{q'}{q_0} \right), \quad (10)$$

$$I(q) = \Omega \langle \rho \rangle \frac{1}{|q|^3} + \dots, \quad (11)$$

allowing the determination of the average dislocation density by fitting a straight line to the asymptotic part of the measured $v_2(q)$ versus $\ln(q)$ relation. The method is illustrated on the line profile of a Cu single crystal deformed in uniaxial compression (Fig. 1).

The line profile is usually not symmetric, this property is visible in the third order restricted moment and the consequent asymptotic form of the intensity distribution up to fourth order:

$$v_3(q') = -6 \langle s^2 \rangle \ln \left(\frac{q'}{q_1} \right), \quad (12)$$

$$I(q) = \Omega \langle \rho \rangle \frac{1}{|q|^3} - 3 \langle s^2 \rangle \frac{q}{|q|^5} + \dots, \quad (13)$$

allowing us to obtain $\langle s^2 \rangle$ from the slope of the $v_3(q)$ versus $\ln(q)$ relation in the large q regime [Fig. 1(c)].

Both the fluctuation and the average dislocation density can be obtained from the fourth order restricted moment:²⁶

$$v_4(q') = \Omega \langle \rho \rangle q'^2 + 12 \Omega^2 \langle \rho^2 \rangle \ln \left(\frac{q'}{q_2} \right) \ln \left(\frac{q'}{q_3} \right). \quad (14)$$

(According to our experience q_2 and q_3 are nearly equal so they can be approximated with $q_0 = \sqrt{q_2 q_3}$.) It can be seen from Eq. (14) that it is useful to introduce the quantity $f(q) = v_4(q)/q^2$ since its asymptotic value is $\Omega \langle \rho \rangle$ and it has a maximum at $q_m = e^* q_0$ with a value of $12 \Omega^2 \langle \rho^2 \rangle / q_m^2$. So the difference between the maximum and the asymptotic value can be related to the relative fluctuation:

$$\frac{f_{\text{max}} - f_{\text{as}}}{f_{\text{as}}^2} = 12 \frac{\langle \rho^2 \rangle}{\langle \rho \rangle^2} \frac{1}{q_m^2}. \quad (15)$$

On Fig. 1(d) the quantity $f(q) = v_4(q)/q^2$ is plotted as a function of q together with the fitted theoretical function obtained from Eq. (14). The three free parameter fit of $f(q)$ was performed between the limits indicated by the two arrows on Fig. 1(d). It is a remarkable fact that the obtained curve follows the measured data points extremely well for q values larger than the upper fitting limit. It can be stated that from about 10^{-2} relative intensity the theoretical expression (14) describes well the tail part of the line profiles. It has to be mentioned, however, that in order to determine the parameters with reasonable accuracy the profiles need to be measured up to a scattering angle corresponding about to $2q_m$.

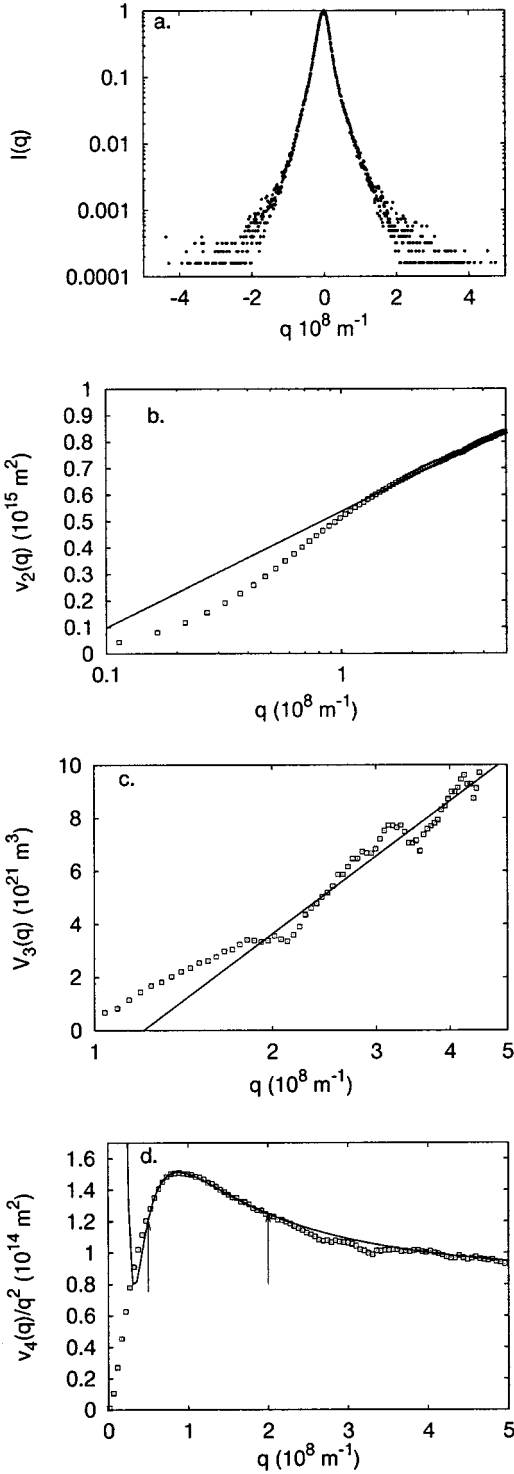


FIG. 1. (a) The measured x-ray line profile of the (200) reflection of a copper single crystal deformed in compression up to 40 MPa resolved shear stress. (b) The second order restricted moment of the measured profile with the fitted function of Eq. (10). (c) The third order restricted moment of the measured profile with the fitted function of Eq. (12). (d) The $v_4(q)/q^2$ quantity of the measured profile with the fitted function of Eq. (14).

III. SELF-SIMILAR DISLOCATION PATTERNS

Hähner *et al.*¹⁹ investigated the fractal properties of the dislocation structure developing in $\langle 100 \rangle$ oriented copper single crystals deformed in tension. They found that the dis-

location pattern is a self-similar hole fractal, i.e., the probability of the cell size (λ) being larger than Λ is the hyperbolic:

$$P(\lambda > \Lambda) \propto \Lambda^{-D} \quad (16)$$

between the limits $\lambda_{\min} < \lambda < \lambda_{\max}$, where D is the fractal dimension. From this, the probability density of the size distribution is

$$p(\lambda)d\lambda = [D\lambda_{\min}^D]\lambda^{-D-1}d\lambda, \text{ if } \lambda \in [\lambda_{\min}, \lambda_{\max}]. \quad (17)$$

Based on the stochastic dislocation dynamics theory of Hähner²⁹ the fractal character of the dislocation assembly can be connected to the statistical properties of the evolution of the dislocation structure.

Following the argument of Zaiser *et al.*²⁰ 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 ρ_w dislocation density. To calculate spatial averages of the quantities Zaiser *et al.*²⁰ introduced a spatially weighted cell size distribution $[p_v(\lambda)]$ in which every microstructural element is weighted with its volume in the embedding E dimensional space: $p_v(\lambda) \propto p(\lambda) \times \lambda^E$:

$$p_v(\lambda) = [(2-D)\lambda_{\max}^{D-2}]\lambda^{-D+1}. \quad (18)$$

The composite cell structure is considered in such a way that the hyperbolic size distribution of the cells is valid from $\lambda=0$ to $\lambda=\lambda_{\max}$ but the cells smaller than λ_{\min} are considered to be the cell walls. According to this model the cell wall volume fraction can be given as

$$f_w = \int_0^{\lambda_{\min}} p_v(\lambda)d\lambda = \left[\frac{\lambda_{\min}}{\lambda_{\max}} \right]^{2-D}. \quad (19)$$

Assuming that dislocations can be found only in the cell walls with dislocation density ρ_w the average dislocation density can be given as

$$\langle \rho \rangle = f_w \rho_w. \quad (20)$$

Similarly, the average of the square of the dislocation density is

$$\langle \rho^2 \rangle = f_w \rho_w^2. \quad (21)$$

The external stress is the average of the local stresses, i.e., the stress in the cell walls, which can be expressed with the local dislocation density (ρ_w), and the stress exerted by the cells, which can be expressed by the size of the cells:

$$\tau_{\text{ext}} = \langle \tau_f \rangle = \int_0^{\lambda_{\max}} p_v(\lambda) \tau_f(\lambda) d\lambda, \quad (22)$$

where $\tau_f(\lambda)$ is the following:

$$\tau_f = \begin{cases} \alpha_f^{\text{eff}} G b \sqrt{\rho_w}, & \lambda \leq \lambda_{\min}, \\ \zeta \frac{G b}{\lambda}, & \lambda \geq \lambda_{\min}. \end{cases} \quad (23)$$

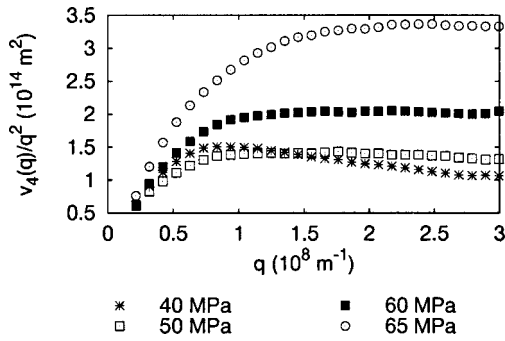


FIG. 2. The $v_4(q)/q^2$ quantities of copper samples deformed in the compression up to the resolved shear stresses indicated.

The parameters α_f^{eff} and ζ have a weak ρ_w dependence but in the investigated deformation regime they can be approximated by constants. From Eqs. (19), (22), and (23) one can arrive at the relation

$$\tau_{\text{ext}} = \alpha_f^{\text{eff}} \zeta G b \frac{f_w}{D-1} \sqrt{\rho_w}. \quad (24)$$

IV. EXPERIMENTAL RESULTS

Copper single crystals were deformed by uniaxial compression in the $\langle 100 \rangle$ direction at $2 \times 10^{-4} \text{ s}^{-1}$ constant strain-rate up to resolved shear stresses of 40, 50, 60, and 65 MPa.

The x-ray line profiles of the $\langle 200 \rangle$ reflection were obtained by a double crystal diffractometer using Cu $K\alpha$ radiation. The measured profile and the different order restricted moments for the sample deformed up to 40 MPa are plotted in Fig. 1. The three parameters characterizing the dislocation arrangement (the dislocation density, the fluctuation and the $\langle s^2 \rangle$) are obtained by fitting the theoretical functions (10), (12), and (14) on the corresponding restricted moments calculated from the measured profiles. The fitted curves are also shown in Fig. 1.

Figure 2 shows the $v_4(q)/q^2$ quantities for the different samples as a function of q . It is apparent that with increasing deformation, the asymptotic value of $v_4(q)/q^2$, i.e., the dislocation density is increasing (Fig. 3), while the δ parameter (and consequently the σ^2 relative fluctuation) is decreasing (Fig. 4). The fact that the fluctuation is decreasing with deformation indicates that the dislocation network tends toward homogeneity.

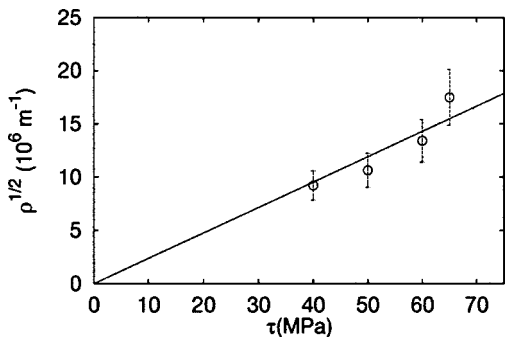


FIG. 3. The square root of the measured dislocation densities vs the resolved shear stress with the fitted $\tau = a\sqrt{\rho}$ function.

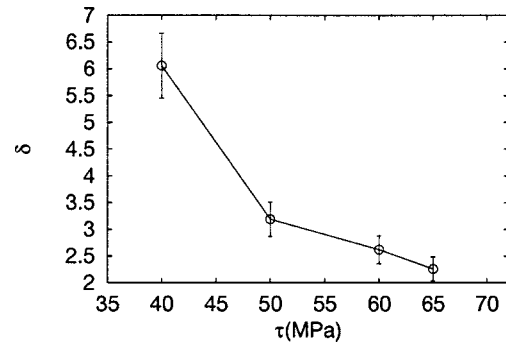


FIG. 4. The obtained $\delta = \langle \rho^2 \rangle / \langle \rho \rangle^2$ quantity related to the fluctuation vs the resolved shear stress.

In order to see if there is any connection between the fractal scaling properties of the dislocation network determined from TEM images and the statistical parameters obtained by x-ray line profile analysis TEM micrographs of the samples investigated by the x-ray method were analyzed.

The TEM investigations were performed on thin foils of $\langle 100 \rangle$ orientation. The fractal dimensions of the dislocation structures were determined by the box-counting method (Fig. 5). Since the measurements of the fractal dimensions were carried out on (quasi) two dimensional sections of the dislocation structure, the fractal dimension of the three dimensional dislocation network can be obtained by adding one to the measured dimensions. According to Fig. 5(b) the linear scaling range extends to approximately 2 orders of magnitude, which is sufficiently large to claim that the investigated structure is a fractal. The measured dimensions as a function of the resolved shear stress are plotted in Fig. 6. Within the

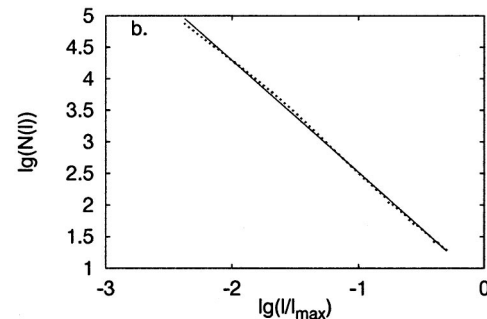
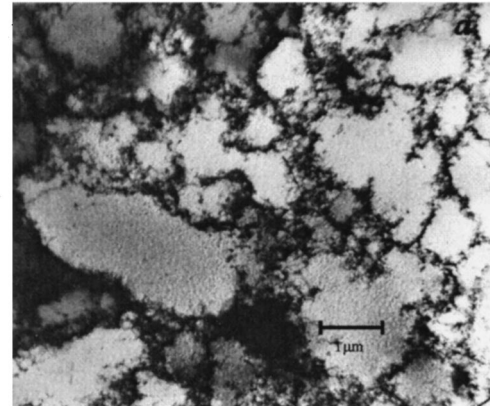


FIG. 5. (a) TEM micrograph showing the dislocation arrangement after compression. (b) The common log-log plot of the box-counting method applied on the TEM micrograph.

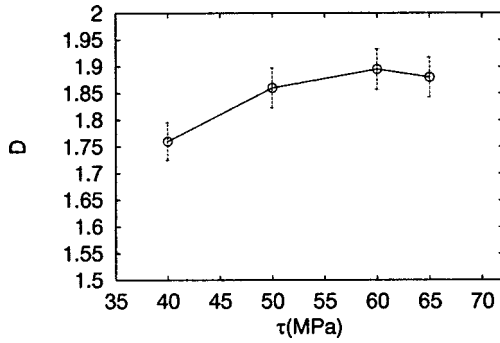


FIG. 6. The measured fractal dimensions of the dislocation patterns developing during compression.

experimental error the measured dimensions at corresponding stresses are equal with the values reported by Hähner *et al.*¹⁹

The measured dimensions are increasing with increasing stress, and tend to 2 (in three dimensions it tends to 3), i.e., with increasing stress the dislocation structure tends to fill up homogeneously the space. This finding is in agreement with the results of the x-ray line profile measurements, which showed that the relative fluctuation is decreasing during the deformation meaning also that the dislocation arrangement tends to homogeneity. Furthermore the D vs δ plot of Fig. 7 suggests a strong correlation between the relative fluctuation obtained from the x-ray measurements and the fractal dimensions measured on the TEM micrograph of the samples.

V. DISCUSSION

For the interpretation of the observed correlation between the relative fluctuation obtained from x-ray measurements and the fractal dimension determined from TEM micrograph the statistical parameters of the dislocation distribution need to be expressed in the framework of the generalized composite model of Zaiser and Hähner. From the model outlined in Sec. III the average of the dislocation density and the average of the square of the dislocation density can be determined as $\langle \rho \rangle = f_w \rho_w$ and $\langle \rho^2 \rangle = f_w \rho_w^2$. With these the quantity δ measured by the x-ray method can be expressed as

$$\delta = \frac{\langle \rho^2 \rangle}{\langle \rho \rangle^2} = \frac{1}{f_w} \tag{25}$$

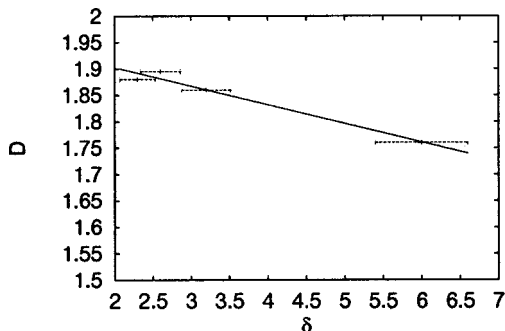


FIG. 7. The correlation between the fractal dimension and the δ parameter measuring fluctuation with the fitted linear relation $D = 1.975 - 0.0354\delta$.

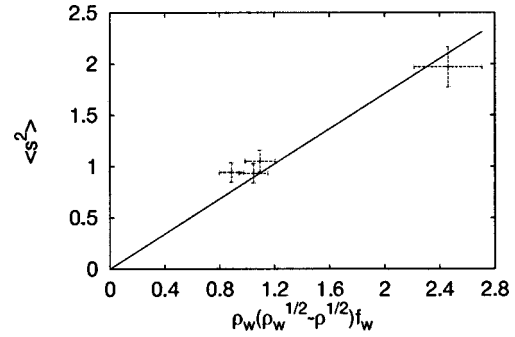


FIG. 8. The connection between dislocation polarization, dislocation density, and cell wall volume fraction according to Eq. (27).

and consequently it is related through Eq. (19) to the parameters of the fractal composite (λ_{\min} , λ_{\max} , and D). So, the δ parameter quantifying the relative fluctuation, can be related to the volume fraction of the dislocation cell walls. Since δ decreases, i.e., f_w is increases with increasing deformation, at least in the investigated strain regime the dislocation cell walls tend to fill up the space, which is in agreement with the result of the composite model predicting [Eq. (19)] that $f_w = (\lambda_{\min}/\lambda_{\max})^{2-D}$ tends to 1 as D tends to 2.

The parameter $\langle s^2 \rangle$ measuring the asymmetry of the x-ray profile can also be related to the generalized composite model since $\langle s^2 \rangle \propto \langle \rho \beta_{\text{tot}} \rangle$ and the total distortion β_{tot} can be expressed by the stress in the cell walls τ_w as follows:

$$\langle s^2 \rangle \propto f_w \rho_w (\tau_w - \tau_{\text{ext}}). \tag{26}$$

(Since the x-ray measurements were performed on unloaded samples $\langle s^2 \rangle$ is proportional to the stress difference $\tau_w - \tau_{\text{ext}}$.) Assuming the commonly accepted proportionality between the flow stress and the square root of dislocation density $\tau_w \propto \sqrt{\rho_w}$ and $\tau \propto \sqrt{\langle \rho \rangle}$ we arrive at a relation between the parameters determined from the x-ray line profiles:

$$\langle s^2 \rangle \propto f_w \rho_w (\sqrt{\rho_w} - \sqrt{\langle \rho \rangle}). \tag{27}$$

On Fig. 8 the measured $\langle s^2 \rangle$ data points are plotted as a function of the quantities given by the right-hand side of the above relation together with the fitted linear curve. It can be seen that the linear relationship is satisfied within experimental errors.

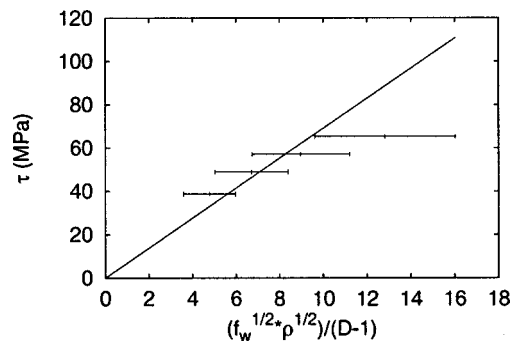


FIG. 9. The external stress vs the experimentally measured parameters of the dislocation pattern (cell wall volume fraction, dislocation density, and fractal dimension), and the fitted function of Eq. (24) obtained from the fractal composite model.

From the x-ray measurement $f_w = 1/\delta$ and $\rho_w = \rho/f_w = \rho\delta$ can be measured, while from TEM micrograph D can be obtained, so the relationship (24) can be checked experimentally. It can be seen on Fig. 9 that there is a qualitative agreement with the $\tau_{\text{ext}} \propto [f_w/(D-1)]\sqrt{\rho_w}$ relation is within experimental errors, although some systematic quantitative deviation can be observed at higher stresses.

VI. CONCLUSIONS

Dislocation structures developed in copper single crystal samples deformed in uniaxial compression were investigated both by x-ray line profile measurements and by fractal analysis of TEM micrographs. The observations were related to the (generalized) fractal composite model of Zaiser and Hähner.

The x-ray line profile analysis is able to provide statistical parameters of the evolved dislocation distribution, which can be compared with both the TEM results and the parameters of the fractal composite model.

The δ parameter connected to the relative fluctuation of

the dislocation density is decreasing with increasing deformation, while the fractal dimension obtained from the TEM micrographs is increasing, both meaning that the dislocation network tends to fill in the space homogeneously. For the investigated Cu single crystal in the accessible regime the $D = 1.975 - 0.0354\delta$ linear relation was obtained between δ and the fractal dimension D .

It can be stated that the fractal composite model describes satisfactorily the correlation between the statistical parameters obtained from x-ray and TEM measurements and the mechanical behavior of the material.

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