ON THE POSSIBILITY OF APPLICATION OF OSCILLATING FILM SPECTROMETER IN THE STUDY OF THE DEGREE OF HOMOGENEITY OF THE DISLOCATION DENSITY DISTRIBUTION (EXAMPLE IN CASE OF ALUMINIUM)

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The feasibility of using the oscillating film method in the study of the homogeneity of the dislocation density distribution in single crystals is investigated. The components of the dislocation density tensor in aluminium single crystals are determined by means of the measurement of the half-width of interference lines and by means of the study of the Fourier transforms.

It is shown that the oscillating film method can be used for the determination of differences in local distributions of screw and edge dislocations.

1. Introduction

The purpose of the present paper was to check to what extent the oscillating film method [1, 2, 3, 4] can be used in the study of the uniformity of the density distribution of dislocations in single crystals. The photometry of interference lines registered on the film at various levels can be namely applied to the comparison of their half-widths and shapes corresponding to different points in the crystal. The calculation of the dislocation density from half-widths requires, however, the assumption of a definite model of angular dispersion of the surface elements of the single crystal which substantially influences the determination of this quantity [5, 6, 7]. In connection with these considerations the next purpose of the present paper was the performance of numerical calculations which permit the determination of the most probable model of angular dispersion of the surface elements, and the check how sensitive are the calculations of the dislocation density on the change in the parameter α, which defines the model.

Since the oscillating film method is used in principle in the determination of the dislocation density in cases when the latter is greater than 10^4-10^5 lines/cm^2, the object

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chosen for this investigation was an aluminium single crystal[1] of fairly good quality, in the form of a plate with the greatest flat surface parallel to the (100) plane. The surface of the crystal was fairly well etched to avoid the influence of mechanical treatment.

In case of ideally parallel and narrow beam and when the characteristic radiation is reflected only from the external atomic faces of the crystal, the function of the angular distribution of the surface elements of the crystal $f(y)$ can be determined from the equation

$$h(x) = \int_{-\infty}^{\infty} f(y)g(x-y)dy \tag{1}$$

in which the function $h(x)$ describes a line, which is recorded on an oscillating film during simultaneous oscillation of the crystal, while $g(\frac{1}{2}x)$ describes a line on a film at rest. The function $f(y)$ which appears in Eq. (1) describes the angular distribution of surface elements twisted by the angle y from the selected zero orientation [1–6].

In case of really performed experiments one should take into account the influence on the shape of the lines of such factors as the divergence of the beam, the width of the slit, the roughness of the crystal surface, as well as a shift of the rotation axis with respect to the reflecting surface.

In such a case the function of the angular distribution of surface elements can be determined from the following equation:

$$H^*(x) = \int_{-\infty}^{\infty} f(y)G^*(x-y)dy. \tag{2}$$

The quantities $H^*(x)$ and $G^*(x)$ appearing in this equation are connected by definition with other functions as follows

$$H^*(x) = \int_{-\infty}^{\infty} H(x-y)F_g(y)dy$$

$$G^*(x) = \int_{-\infty}^{\infty} G(x-y)F_n(y)dy$$

$$F_n(x) = \int_{-\infty}^{\infty} G'(x-y)\Phi_n(y)dy$$

$$F_g(x) = \int_{-\infty}^{\infty} G'(x-y)\Phi_g(2y)dy. \tag{2a}$$

The following conditions must be also fulfilled

$$\gamma \ll 1; \quad \beta \ll 1$$

$$\frac{\tan\varphi}{\cos^2\varphi}\Delta\varphi \ll \gamma \tag{3}$$

[1] The crystal has been prepared at the Institute of Nuclear Research by A. Modrzejewski.

where: β-is the horizontal divergence of the X-ray beam, γ-defines the maximum spread angle of the surface elements, φ-defines the divergence of the beam in vertical plane, $\Delta\varphi$-defines the divergence angle in vertical plane in the range of the photometer slit height.

The functions used in the integrals describe the shape of the interference lines obtained in the following conditions:

$H(x)$ — on an oscillating film when the crystal oscillates and is shifted,

$G(x)$ — on a film at rest with oscillating and shifted crystal,

$G'(x)$ — on a film at rest when the crystal oscillates.

On the other hand Φ_n and Φ_g describe:

$\Phi_n(x)$ the shape of the beam leaving the slit very near to the latter,

$\Phi_g(x)$ the shape ot the beam leaving the slit at a distance equal to that between the crstal and the slit.

According to Bedyńska [4–6] the angular distribution function $f(y)$ of the surface elements occurring in Eq. (2) depends among others on two parameters α and N. The parameter α which defines the model of the dislocation distribution is connected with the probability p of finding a dislocation of a given sign by means of the following equation

$$\alpha = 2p-1$$

In case of a completely random distribution (Cottrell model) the probability of finding a positive dislocation is equal to the probability of finding a negative dislocation, and thus $p = \frac{1}{2}$, or $\alpha = 0$.

In case of ideal orderly arrangement (the crystal uniformly bended) all dislocations are of the same sign, and thus $p = 1$ or $\alpha = 1$.

The second parameter N denotes the number of dislocations appearing in the investigated region. It thus substantially influences the half-widths of the function $f(y)$.

The function $f(y)$ is defined according to [5] by means of the formula

$$f(y) = \frac{1}{N}\int_0^N \frac{\exp\left[-\frac{1}{2}\frac{\left(\frac{y}{\varepsilon}-n\alpha\right)^2}{n(1-\alpha^2)}\right]}{\sqrt{2\pi}\,\varepsilon\sqrt{n(1-\alpha^2)}}\,dn \tag{4}$$

where ε is the rotation angle of the lattice produced by one dislocation and depends on the Burgers vector.

In case of the Cottrell model where $\alpha = 0$, it follows according to Bedyńska and Chmielewska [5] that the function $f(y)$ is symmetric and has a maximum for $y = 0$. For $N \to \infty$ this function tends to Gaussian.

In case of ideal bending, *i.e.*, $\alpha = 1$ the function $f(y)$ is rectangular, starting from $y = 0$ with the widths equal to $\varepsilon \cdot N$, *i.e.*

$$f(y) = \frac{1}{\varepsilon N} \quad \text{for} \quad 0 \leqslant y \leqslant \varepsilon N$$

$$f(y) = 0 \quad \text{for} \quad y < 0 \quad \text{and} \quad y > \varepsilon N.$$

In intermediate cases the function $f(y)$ goes over from symmetric to a more asymmetric and rectangular form.

According to the papers [5, 6, 8] it is possible to estimate the dislocation density in the investigated crystal by approximate determination of the half-width of the function f (Eq. (2)). This is justified under the assumption that all experimental functions appearing in Eqs (2a) can be approximated by means of Gaussian functions. This is only possible when these functions are at least approximately symmetric. From these assumptions, from the formulae (2) and (2a) and from the properties of the Gaussian function (the convolution of two Gaussian functions is also a Gaussian function) it follows that also the function $f(y)$ should be on approximated Gaussian function which is possible only in the case of the Cottrell model $\alpha = 0$ and large N-values.

Given the experimental functions it is possible to calculate the half-width of the function of the distribution of the surface elements $f(y)$ by means of the formula

$$f^2 = H^2 - G^2 + \tfrac{3}{4} G'^2 + \tfrac{1}{4} \Phi_g^2 - \Phi_n^2 \tag{5}$$

where H, G, G', Φ_g and Φ_n denote the half-widths of the above-mentioned functions while f is the half-width of the function $f(y)$.

In case of undeformed crystal the best model of the dislocation distribution seems to be the Cottrell model. The dislocation density described by the edge and screw components of the dislocation density tensor is then calculated on the basis of Eqs (4)–(6):

$$\varrho^k = \frac{f^{4/3}}{2^{4/3} b_k^{4/3} L_1^{2/3}} \tag{6}$$

$$\varrho^s = \frac{f^{4/3}}{b_s^{4/3} L_2^{2/3}} \tag{7}$$

where f denotes the half-width of the surface element distribution function, b_k, b_s — the Burgers vectors of edge and screw dislocations and L_1, L_2 are the values of normal and perpendicular slipping with respect to the rotation axis.

In accordance with the references quoted above it is also possible to determine more accurately the values of the parameters α and N describing the shape of the function $f(y)$ by making use of Eq. (2) and the definition (2a). No specific assumptions about the shape of experimental function are here required, except one, *i.e.*, that the functions $H(x)$ and $G(x)$ cannot have many maxima which would indicate polygonization in the investigated region.

The problem of rigorous selection of parameters α and N can be solved by means of numerical calculations with the use of a computer made according to a method described by Bedyńska *et al.* [9]. It consists in the selection of the parameters α and N for the function $f(y)$ appearing in Eq. (2) and obtained from experimental data. These parameters should be chosen to obtain the best fit of $f(y)$ to the experimental angular distribution if the surface elements for a given crystal.

The integral equation (2) is solved according to Bedyńska *et al.* [4, 5, 6, 8, 9] by means of Fourier transform method. The result of such a solution is an "experimental" Fourier

transform of the angular distribution of surface elements obtained by suitable numerical operations performed on the Fourier transforms of the experimental functions H, G, Φ_n, Φ_g. The "experimental" points of the Fourier transform f obtained in this way are fitted by the "theoretical" transform of the function f by means by the "theoretical" transform of the function f by means of the least square method by varying both α and N.

According to Refs [4, 5, 6, 8, 9] the theoretical transform depends on the parameters α and N, and is given by means of the formula

$$F(t) = \frac{|F'(t)|^2}{|F'(0)|^2} = \frac{4}{s^2t^2\varphi^2(4a^2+s^2t^2)}\left\{1+\exp(-s^2t^2\varphi)-\right.$$
$$\left.-2\exp\left(-\frac{1}{2}s^2t^2\varphi\right)\cos(st\varphi a)\right\} \tag{8}$$

where t is the subsequent number of the transform, D is the width of the region of the coordinate y in which $f(y)$ is developed into Fourier series

$$s = \frac{2\pi}{D}; \quad \varphi = \varepsilon^2(1-\alpha^2)N$$

$$a = \frac{1}{\varepsilon}\frac{\alpha}{1-\alpha^2}$$

2. Experiment

The dislocation density has been determined by means of the oscillating film method using a quasi-point X-ray source with the diameter of about 20 μ and the characteristic radiation $K\beta$ Cu. The width of the spectrometer slit was 20 μ. The crystal was adjusted to Bragg reflections from the (001) plane. During the experiment the sample oscillated about the axis within 2° and was shifted in the direction parallel to the spectrometer axis to determine the screw component, and in the direction perpendicular to the spectrometer axis to determine the edge component of the dislocation density tensor. The shift in both cases was 0.5 mm.

The photographs of the interference lines obtained are shown in Fig. 1. The levels at which the photometric measurements were made are marked in the figure.

In the photographs there are also interference lines recorded on the film at rest for a shifted, oscillating crystal (II), and for an oscillating and non-shifted crystal (III). The density of these lines has been determined by means of the photometric method. From the curves obtained in this way on the basis of Eq. (5) the half-width of the function $f(y)$ has been determined.

Fig. 2 shows examples of the shapes of interference lines obtained for one of the photometric levels.

The aluminium crystal has a face-centred regular lattice. The smallest possible Burgers vector for this lattice can be written in the form $\bar{b} = \frac{a}{2}\langle 110\rangle$. It amounts in this case to

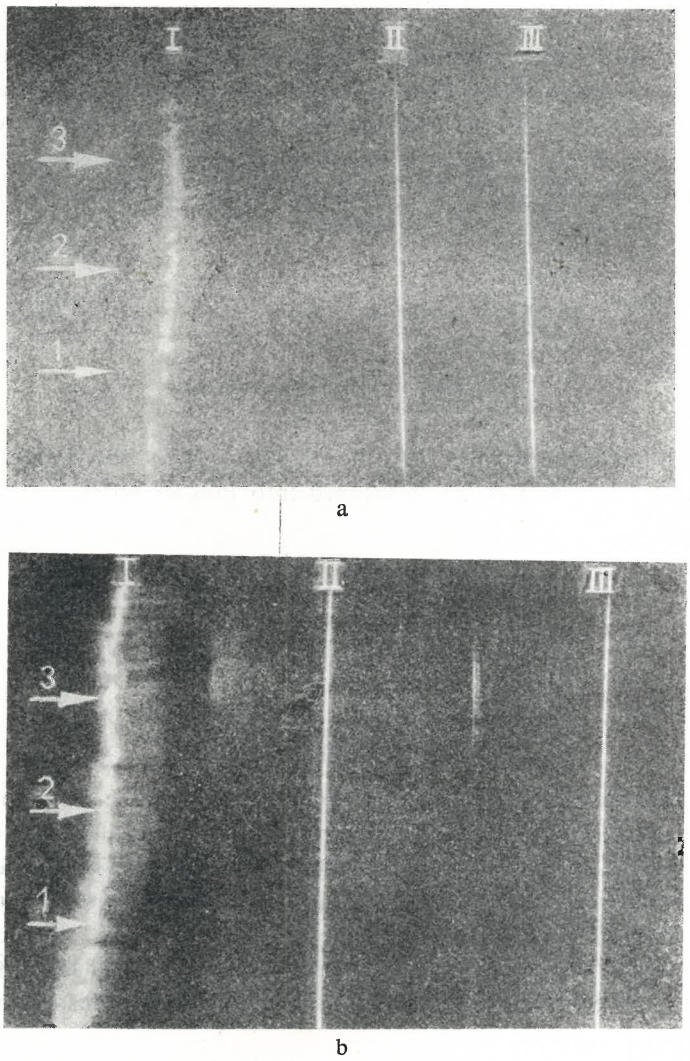

Fig. 1. Examples of photographs of interference lines for the following components a) screw and b) edge dislocations (the levels at which photometric measurements were made are indicated by arrows). *I.* Oscillating film — oscillating and shifted crystal. *II.* Film at rest — oscillating and shifted crystal. *III.* Film at rest — oscillating and non-shifted crystal

2.8635 Å. Making use of Eqs (6) and (7) we have calculated the density of screw and edge dislocation connected with this Burgers vector, assuming the Cottrell model and using the value of the half-width of the spread function of surface elements.

The results obtained are listed in Table I. The calculations have been performed for the Burgers vectors $\bar{b}_k = \frac{a}{2}\langle 110\rangle$, $\bar{b}_s = \frac{a}{2}\langle 110\rangle$, where $\bar{b}_k$ denotes the Burgers vector for edge dislocation and $\bar{b}_s$ for screw dislocation.

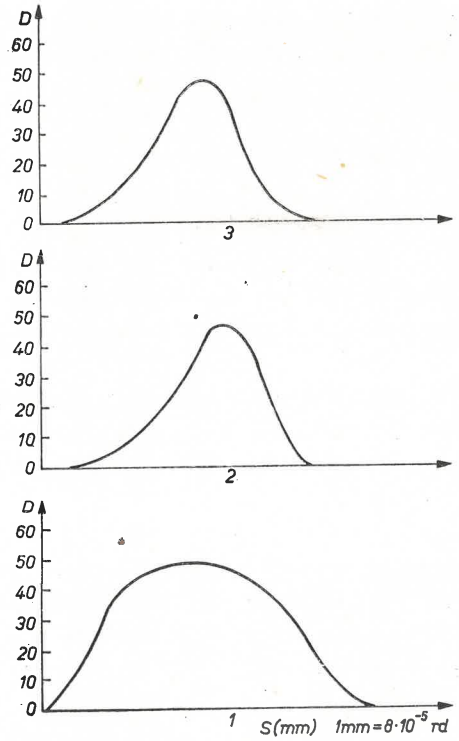

Fig. 2. Shapes of interference lines obtained at one of the indicated levels. *1.* in case of oscillating film and crystal — the latter shifted perpendicularly to the spectrometer rotation axis, *2.* in case of oscillating crystal shifted perpendicularly to the spectrometer rotation axis — film at rest, *3.* in case of oscillating and non-shifted crystal — film at rest

TABLE I

Level	ϱ_k [lines/cm^2]	ϱ_s [lines/cm^2]
1	$4.46 \cdot 10^7$	$1.22 \cdot 10^8$
2	$1.22 \cdot 10^7$	$5.21 \cdot 10^7$
3	$1.18 \cdot 10^7$	$4.15 \cdot 10^7$

Evaluation of experimental results

In accordance with Ref. [9] the parameters α and N which described the angular distribution of surface elements in the investigated crystal have been chosen in the following way: several dozens consecutive points of the "experimental" Fourier transform of the angular distribution function of surface elements have been determined strating from Eqs (2) and (2a). The behaviour of this transform should be described by Eq. (8) which gives the behaviour of the theoretical transform depending on α and N.

The α-interval from 0 to 1 has been divided into m equal subintervals; next for each consecutive α value starting from $\alpha = 0$ the least square method was used for obtaining

the best fit of both transforms with N as varying parameter. The difference between these two transforms can be expressed by the so-called correlation R-index

$$R^2 = \frac{\sum_{i=1}^{i_m} (F_0(i) - F_T(i))^2}{\sum_{i=1}^{i_m} F_0(i)^2} \tag{9}$$

where $F_0(i)$ is the consecutive value of the experimental and $F_T(i)$ of the theoretical transform of the angular distribution function of surface elements.

The R-index was dependent on the value of α and for a certain value α_{opt} became minimum.

This indicated that one should look for the best fit of the theoretical to the experimental transform, in the vicinity of this particular value α_{opt}. By repeating this procedure for the reduced range around α_{opt} it was possible to determine more precisely this value of α for which the best fit was obtained (R_{min}).

To the value of α found in the above-mentioned procedure there corresponds the parameter N which gives directly the dislocation density in the investigated crystal region. The results obtained for the point of the crystal corresponding to the region at half the height of the diffraction pattern are summarized in Table II. The columns 2 and 3 of this Table give the values of the R-index and the dislocation density values obtained for various

TABLE II

1	2	3	4	5
α	R_k %	ϱ_k [lines/cm^2]	R_s %	ϱ_s [lines/cm^2]
0.00	7.68	$3.48 \cdot 10^6$	9.18	$1.12 \cdot 10^7$
0.10	8.27	$2.86 \cdot 10^6$	6.73	$6.79 \cdot 10^6$
0.20	10.05	$2.02 \cdot 10^6$	8.37	$3.94 \cdot 10^6$
0.30	11.70	$1.48 \cdot 10^6$	10.14	$2.72 \cdot 10^6$
0.40	15.37	$1.10 \cdot 10^6$	11.24	$2.07 \cdot 10^6$
0.50	16.40	$8.97 \cdot 10^5$	11.97	$1.67 \cdot 10^6$
0.60	17.09	$7.55 \cdot 10^5$	12.50	$1.40 \cdot 10^6$
0.70	17.57	$6.51 \cdot 10^5$	12.89	$1.20 \cdot 10^6$
0.80	17.91	$5.73 \cdot 10^5$	13.20	$1.05 \cdot 10^6$
0.90	18.15	$5.12 \cdot 10^5$	13.44	$9.37 \cdot 10^5$
1.00	18.35	$4.63 \cdot 10^5$	13.76	$8.59 \cdot 10^5$

assumed values of the parameter α, for measurements of the edge component of the dislocation density tensor. The columns 4 and 5 give analogous values for the screw component of the dislocation density tensor. Best fit values have been obtained

a) for the screw component at

$$\alpha = 0.11 \qquad R_{min} = 6.71\,\%$$

$$\varrho_s = 6.37 \cdot 10^6 \text{ lines/cm}^2$$

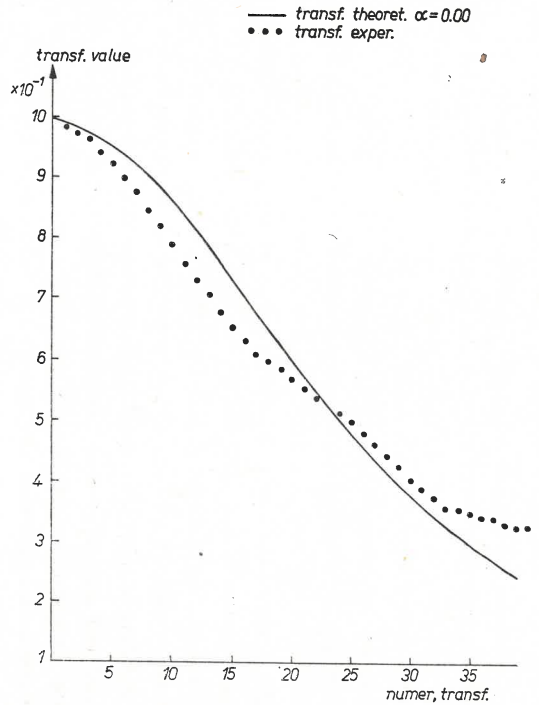

Fig. 3

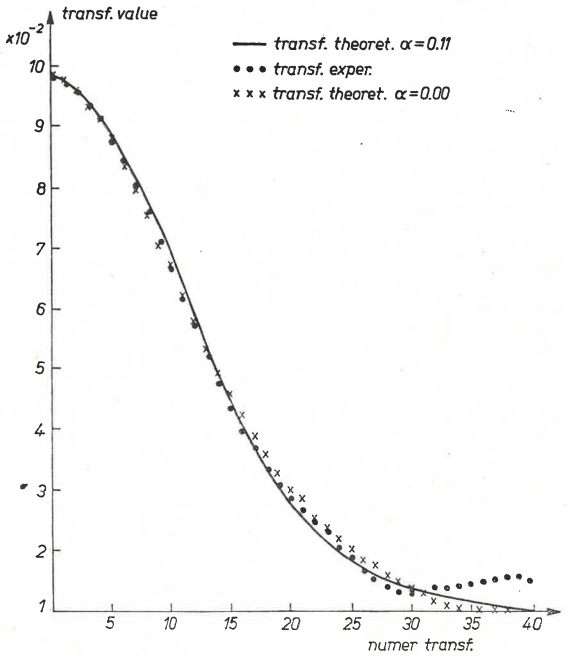

Fig. 4

Fig. 3. The dependence of the values of subsequent coefficients of the Fourier transform of the function $f(y)$ on the number of the transform in case of the edge component of the dislocation density tensor and best fit for $\alpha = 0.00$

Fig. 4. Dependence of the values of subsequent coefficients of the Fourier transform of the function $f(y)$ on the number of the transform in case of screw dislocation component for $\alpha = 0.00$ and $\alpha = 0.11$ (solid line is best fit)

b) screw component at

$$\alpha = 0 \qquad R_{\min} = 7.68\,\%$$

$$\varrho_k = 3.48 \cdot 10^6 \text{ lines/cm}^2.$$

The behaviour of the experimental and the theoretical Fourier transform for the screw and edge component of the dislocation density tensor are shown in Figs 3 and 4.

It can be seen from the figures that the theoretical curves are in good agreement with experimental points, the best agreement occurring for the screw component at $\alpha \rightleftharpoons 0.11$.

3. Conclusions

1. The dislocation density has been estimated from the half-width odf lines. At three different points in the crystal it amounts to $1.18 \cdot 10^7$, $1.22 \cdot 10^7$ and $4.46 \cdot 10^7$ lines/cm^2 for edge dislocation, respectively. For screw dislocations the respective values are $4.15 \cdot 10^7$, $5.21 \cdot 10^7$ and $1.22 \cdot 10^8$ lines/cm^2. This means that the distribution of the dislocation density in the investigated crystal was not uniform, and that at each point at which the measurement was made screw dislocations prevail. For calculations of the dislocation density we have accepted the Cottrell model.

2. In order to check the validity of this assumption we have made computer calculations of the dislocation densities for one of the previously measured points (level 2) using a specially written program. We have studied the dependence of the R-index for screw and edge dislocation on the value of the parameter α which defines the model ($\alpha = 0.00$ for the Cottrell model and 1.0 for uniform bending model). The edge dislocation density obtained for $\alpha = 0.00$ was $3.47 \cdot 10^6$ while that of the screw dislocations is $1.12 \cdot 10^7$. The best value of R has been obtaines for edge dislocations under the assumption of the Cottrell model ($R = 7.68\,\%$), whereas for screw dislocations the best value of R was for $\alpha = 0.11$, *i.e.*, for a model slightly deviating from the Cottrell model. The R-index for screw dislocation density of $6.37 \cdot 10^6$ lines/cm^2 amounted in the latter case 6.71 % This fact indicates some degree of ordering of screw dislocations in the investigated crystal.

The differences between the dislocation densities estimated on the basis of the half-width and those calculated for the best fit values, amount to about half an order of magnitude. In spite of that, the ratios of the screw to edge dislocation densities estimated by both methods are similar, and amount to 0.23 and 0.3 for the half-width and the best fit method respectively. The oscillating film method can thus be used for the determination of differences in local density distributions with simultaneous determination of the ratio of screw to edge dislocation density. The differences between the edge dislocation densities found at different points of the crystal did not exceed in our case half-an order of magnitude, while for screw dislocations these differences were slightly higher.

3. The results obtained indicate that the use of the method of dislocation density estimation on the basis of the half-width can lead to overestimated values owing to the approximate assumptions concerning the dependence of half-width on the dislocation density.

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