AN ATTEMPT OF APPLYING AN X-RAY SPECTROMETER WITH OSCILLATING FILM FOR INVESTIGATING THE REAL STRUCTURE OF Fe-\alpha SINGLE CRYSTALS PLASTICALLY DEFORMED

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The aim of this paper is to examine the extent to which an X-ray spectrometer with oscillating film (Auleytner, 1958, 1959, 1960, 1961, 1964) may be applied in defining the edge and screw components of the tensor of dislocation density in Fe- α single crystals plastically deformed. Thus far this method has not been applied in determinations of dislocation density in cases where macromosaics appeared, which is a characteristic trait of investigated Fe- α single crystals. The investigated crystals were deformed by cold rolling and tension.

In crystals with a dislocation density exceeding $10^5/\text{cm}^2$ one can determine the average angular dispersion of surface elements for the given crystallographic plane on the basis of the broadening of interference lines. The measure of the angular dispersion of surface elements is the half-width of the surface element distribution function f(x). The function f(x) defines how many surface elements in the investigated region of the crystals are rotated by an angle x from a well-defined axis lying within that plane. The surface element distribution function may be determined by means of a spectrometer with oscillating film (Auleytner 1958, 1959, 1960, 1961, 1964). A method of calculating the distribution function f(x) has been worked out by Bedyńska (Bedyńska 1960, 1963). In the present paper a simplified version is applied (Bedyńska, Chmielewska 1964). In order to calculate the function f(x) on the basis of the data obtained from the X-ray spectrometer with oscillating film the following conditions must be satisfied:

$$\alpha \ll 1$$
; $\beta \ll 1$ — condition 1
 $\frac{\operatorname{tg} \alpha}{\cos^2 \varphi} \Delta \varphi \ll \alpha$ — condition 2

with the notation: α — the dispersion angle of surface elements, β — the angle of the divergence of the beam in the horizontal plane, φ — the angle of divergence of the beam in the vertical plane, $\Delta \varphi$ — the angle of divergence of the beam in the vertical plane within the height of the photometer slit.

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In order to calculate the function f(x) we must compute the integrals

$$H_1(x) = \int_{-\infty}^{+\infty} H(y) \, \Phi_g(x - y) \, dy \tag{1}$$

$$G_1(x) = \int_{-\infty}^{+\infty} G(y) \, \Phi_h(x-y) \, dy \tag{2}$$

and then solve the integral equation

$$H_1(x) = \int_{-\infty}^{+\infty} G_1(x - y) f(y) dy.$$
 (3)

The functions H(x), G(x), $\Phi_h(x)$ and $\Phi_g(x)$ are obtained experimentally. The half-width of the surface element distribution function θ can be computed from the following formula:

$$\theta^{2} = \Delta_{H(x)}^{2} + \Delta_{\Phi_{g}(x)}^{2} - \Delta_{G(x)}^{2} - \Delta_{\Phi_{h}(x)}^{2}$$
(4)

where $\Delta_{H(x)}$, $\Delta_{G(x)}$, $\Delta_{\Phi_g(x)}$ and $\Delta_{\Phi_h(x)}$ denote the half-widths of the experimental functions H(x), G(x), $\Phi_g(x)$ and $\Phi_h(x)$. Having the half-width of the function f(x), we can calculate the dislocation density for the edge and screw components of the tensor of dislocation density. In the present paper Cottrell's model of the angular surface element distribution is assumed as the most probable. Whence to calculate the dislocation density described by the screw and edge component of the tensor of dislocation density the following formulas were used:

$$\varrho^e = \frac{\theta_e^{4/s}}{b_e^{4/s} L^{2/s}}$$
 for the edge component (5)

$$\varrho^{s} = \frac{2^{4/s} \theta_{s}^{4/s}}{b_{s}^{4/s} L^{2/s}}$$
 for the screw component (6)

where θ_e and θ_s denote the respective half-widths of the function f(x) in the case of a perpendicular and parallel shift of the crystal with respect to the axis of rotation, L is the range of this shift which in both cases amounted to 1 mm, b_e and b_s are the respective Burgers vector modules.

The monocrystals were investigated by means of an X-ray spectrometer with oscillating film in cooperation with an X-ray tube with point focus (Auleytner 1958, 1959, 1960, 1961, 1964). The width of the spectrometer slit amounted to about 50 μ . The characteristic $K_{\beta}(Cu)$ radiation was utilized for producing the interference lines on the films. The (110) plane was the reflecting plane inclined to the surface of the crystal at an angle of 12°. During the investigation the crystal oscillated round the axis within a range of 2°. As in the present work we tried to seperate the edge and screw components of the tensor of dislocation density, during the exposition the crystal was also shifted perpendicularly or parallelly with respect to the axis of rotation. In both cases the crystal was shifted within the range of 1 mm. Fig. 1 shows photographs of interference lines in the case of the Fe- α single crystal deformed by rolling, and Fig. 2 shows photographs of inteference lines in the case of the Fe- α monocrystal deformed by tension.

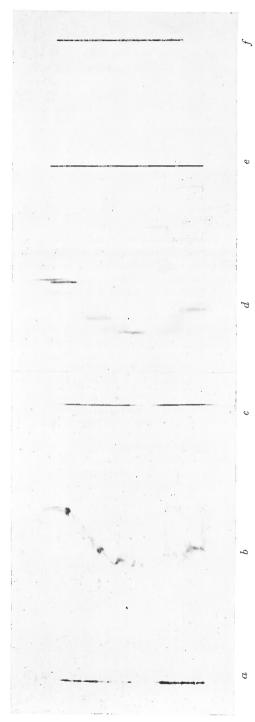


Fig. 1. Photographs of the single crystal deformed by rolling (magnification ×5). a-oscillating crystal shifted perpendicularly with respect to the axis of rotation — film fixed, b-the same as a), but the film is oscillated together with the crystal, c-oscillating crystal shifted parallelly to the axis of rotation — film fixed, d-the same as c), but the film is oscillated together with the crystal, e and f-photograps of the beam falling on flat film placed respectively midway between the slit and the crystal and in the place where the beam falls upon the crystal



Fig. 2. Photographs of single crystals deformed by tension (magnification $\times 5$). Notes a), b), c), d), e), f)—the same as in Fig. 1

Photographs b) and d) in Fig. 1 clearly show the bending of the central part of interference lines. It proves the formation of a layer with a strongly directed dispersion of macromosaic blocks.

The mutual disorientation of those blocks amounts up to 1.5° . The widening and diffusion of the lines as compared with photographs a) and c) prove the existance of a micromosaic structure. The dislocation density was calculated for the individual blocks. With the shifting of the crystal along the spectrometer axis there is revealed the presence of larger mutually screwed areas in the marked direction (it can be deduced from the splitting of the diffraction lines shown on the photographs d) Fig. 1 and Fig. 2). Deformation by tension (Fig. 2b, d) also causes the formation of macromosaic blocks, but the dislocation density of these blocks is higher in the case of single crystals deformed by rolling. This is proved by the more pronounced diffusion of interference lines than in photographs b) and d) in Fig. 1. The interference lines on the photographs presented in Figures 1 and 2 were photometred and, on the basis of the respective film characteristics, the distribution of the intensity of lines was calculated. On the basis of the shape of the obtained curves, their half-widths were calculated and then from the formula (4) the half-width of the surface element distribution

TABLE I

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|--|--|--|--|--|--|--|---|--|
| Burgers | $\vec{b}_{\pmb{k}}$: $\langle 100 \rangle$ | | \vec{b}_k : $\langle 100 \rangle$ | | $\vec{b}_{\pmb{k}} \colon \frac{1}{6}\langle 112 \rangle$ | | $\vec{b}_{\pmb{k}} \colon \frac{1}{6} \langle 112 \rangle$ | |
| Investigated erystal | \vec{b}_s : $\langle 100 \rangle$ | | \vec{b}_{ε} : $\frac{1}{2}\langle 111 \rangle$ | | \vec{b}_s : $\langle 100 \rangle$ | | \vec{b}_s : $\frac{1}{2}\langle 111 \rangle$ | |
| Iron deformed by tension | $\varrho^e [\mathrm{cm}^{-2}]$ | $\varrho^s \ [{ m cm}^{-2}]$ | $\varrho^e [\mathrm{cm}^{-2}]$ | <i>ρ</i> ^s [cm ⁻²] | <i>ρe</i> [cm ⁻²] | <i>ρ</i> ^s [cm ⁻²] | <i>ρ^e</i> [cm ⁻²] | <i>ρ</i> ^s [cm ⁻²] |
| | 5.89×10 ⁷ | 4.28×10 ⁷ | 5.89×10 ⁷ | 5.18×10 ⁷ | 1.94×10 ⁷ | 4.28×107 | 1.94×108 | 5.18×10 ⁷ |
| Iron deformed by rolling | $\begin{vmatrix} 2.73 \times 10^7 \\ 1.14 \times 10^7 \\ 1.90 \times 10^7 \end{vmatrix}$ | $\begin{vmatrix} 3.10 \times 10^7 \\ 2.92 \times 10^7 \\ 8.57 \times 10^7 \end{vmatrix}$ | $\begin{vmatrix} 2.73 \times 10^7 \\ 1.14 \times 10^7 \\ 1.90 \times 10^7 \end{vmatrix}$ | $\begin{vmatrix} 3.76 \times 10^{7} \\ 3.09 \times 10^{7} \\ 1.04 \times 10^{8} \end{vmatrix}$ | $\begin{vmatrix} 9.02 \times 10^7 \\ 3.75 \times 10^7 \\ 6.29 \times 10^7 \end{vmatrix}$ | $\begin{vmatrix} 3.10 \times 10^7 \\ 2.92 \times 10^7 \\ 8.57 \times 10^7 \end{vmatrix}$ | $\begin{array}{c} 9.02 \times 10^{7} \\ 3.75 \times 10^{7} \\ 6.29 \times 10^{7} \end{array}$ | 3.76×10^{7} 3.09×10^{7} 1.04×10^{7} |

function f(x) was found. As in the investigated crystal there occur dislocations with various Burgers vectors possible for this structure and as it is difficult to determine which of those dislocations have the biggest influence upon the respective components of the dislocation density tensor three possible Burgers vectors $\langle 100 \rangle$, $\frac{1}{2}\langle 111 \rangle$ and $\langle \frac{1}{6}\langle 112 \rangle$ were taken into consideration in the calculations of those densities (Van Bueren 1960). Putting together those vectors respectively in pairs we obtained for each of the investigated cases four pairs of values for ϱ^e and ϱ^s calculated on the basis of formulas (5) and (6). The results are shown in Table I.

From the data in Table I we can conclude that in the case of iron deformed by tension the dislocation densities are comprised within the limits:

$$\begin{split} 5.89 \times 10^7 / \mathrm{cm^2} & \leq \varrho^e \leq 1.94 \times 10^8 / \mathrm{cm^2} \\ 4.28 \times 10^7 / \mathrm{cm^2} & \leq \varrho^s \leq 5.18 \times 10^7 / \mathrm{cm^2} \end{split}$$

and as far iron deformed by rolling:

$$1.14 \times 10^7/\text{cm}^2 \le \varrho^e \le 9.02 \times 10^7/\text{cm}^2$$

 $2.92 \times 10^7/\text{cm}^2 \le \varrho^s \le 1.04 \times 10^8/\text{cm}^2$.

On the basis of these data one can conclude that deformation by tension (4 per cent of deformation) causes the formation of larger edge dislocation densities than in the case of the 4 per cent deformation by rolling. One can also state that in the majority of the investigated cases the dislocation density for the rolled crystal described by the screw component of the tensor dislocation density is bigger than the dislocation density described by the edge component of this tensor. The obtained results characterize the real structure of the investigated Fe monocrystals and they point to the difficulties which crop up during this kind of investigation.

A number of problems have not been explained yet. Crystals intended for the investigation should be cut along precisely defined crystallographic planes. Also, the thickness of the layer deformed by mechanical treatment accopanying cutting and polishing should be determined. This layer should be removed by galvanochemical means.

Investigations should be carried out for various crystallographic directions. A more precise method of calculating the dislocation density should be applied (Bedyńska 1963), and the obtained results ought to be compared with the results obtained by other methods (Bedyńska 1963, Precht 1966). Because of all this, the dislocation densities given here should be looked upon as merely approximate data. However, the oscillating film method makes it possible to characterize the mosaic structure of Fe monocrystals sufficiently and quickly.

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