ANALYSIS OF THE BROADENING X-RAY LINES BY CRYSTALS CONTAINING SUBGRAINS AND BLOCKS

K.P. RYABOSHAPKA, E.I. BOGDANOV, S.E. BOGDANOV

G.V. Kurdyumov Institute for Metal Physics, Nat. Acad. Sci. of Ukraine
(36, Academician Vernadsky Blvd., Kyiv 03142, Ukraine)

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The theoretical analysis of the intensity distribution for the X-ray scattering by crystals containing subgrains and blocks is carried out. The presence of chaotically arranged dislocation walls consisting of equidistant edge dislocations of one sign causes the broadening of X-ray lines on a Debye photograph. In the general case, the integral width can depend on the orientation of walls and the direction of the diffraction vector. The measurement of the integral width of X-ray lines allows one to determine the size of blocks and, under some conditions, the size and disorientation of blocks.

1. Introduction

A crystal can consist of fragments (subgrains) which are divided into smaller blocks. The dislocation walls can be in the form of boundaries consisting of equidistantly arranged dislocations [1—3]. The simple wall of such a type leads to a turn of blocks relatively one another, and it is a system of edge dislocations with the same Burgers vector. The edge dislocations are arranged in one plane. If such dislocation walls are distributed along the normal to a plane (e.g., the plane of type {110} for the fcc lattice and the plane of type {111} for the bcc lattice), then, at a given concentration of such walls p, the crystal is divided into blocks, whose sizes are distributed by the normal distribution law in the neighborhood of some average size $D = a/p$, where a is the size of a fragment (subgrain). The theory of the X-ray scattering by crystals divided into such blocks is developed in [4].

In the present work, the analysis of the intensity distribution for the X-ray scattering by crystals containing subgrains and blocks which have different sizes and disorientations is carried out in the kinematic approximation.

2. X-ray Scattering by Crystals Having the Dislocation Walls

In the kinematic theory of the X-ray scattering by a crystal divided into blocks by the dislocation walls, the expression for the intensity was obtained in [4] in the general case. If the dislocations in a wall are arranged at the same distance and these walls are distributed randomly along the normal to a plane for the given crystalline structure, then the integral intensity distribution may be written as [4]

$$J(\mathbf{q}_1) = \frac{J_i\sqrt{\sigma_{\xi\xi}\sigma_{\eta\eta}}}{2\pi}\frac{\sigma_\kappa}{\sigma_\kappa^2+\sigma_\kappa^2}\exp\left[-\frac{1}{4}\sum_{\xi=1,2}\frac{q_\xi^2}{\sigma_{\xi\xi}^2}\right], \quad (1)$$

where

$$\sigma_{\xi\xi}^2 = 2\sum_i \frac{R_i v_{i\xi}^2 (q_1 b_i)^2}{D_i h_i^2}, \quad (2)$$

$$\sigma_\kappa = 2\sum_i \frac{|(\mathbf{e}_i, \boldsymbol{\kappa})|}{D_i}, \quad (3)$$

$$v_{i\xi} = ([\boldsymbol{\kappa}, \boldsymbol{\tau}_i], \mathbf{e}). \quad (4)$$

Here, J_i is the integral intensity distribution; $\mathbf{q} = \mathbf{q}_1 - 2\pi\mathbf{K}_n$, where $\mathbf{q}$ is the difference of the wave vectors of the scattered wave and the incident one; $\mathbf{K}_n$ is the reciprocal lattice vector nearest to the end of the vector $\frac{\mathbf{q}_1}{2\pi}$; $\boldsymbol{\kappa} = \frac{\mathbf{q}_1}{|\mathbf{q}_1|}$ is the unit vector in the direction $\mathbf{q}_1$; the index characterizes the specified system of walls, i.e. the direction of the Burgers vectors of dislocations into a wall ($\mathbf{e}_i = \mathbf{b}_i/|\mathbf{b}_i|$ is the unit vector in the direction $\mathbf{b}_i$); σ is the stress in the crystal in the neighborhood of boundaries [5]; R_i is the stress sphere radius from the i-th dislocation wall, $D_i = a/p_i$ is the average distance between walls of the i-th type (p_i is the concentration of these walls); $h_i$is the distance between dislocations in the i-th wall; and τ_i is the unit vector along the direction of the dislocation line in the i-th wall.

The local system of coordinates in the neighborhood of a node of the (hkl) type is chosen so that one coordinate axis (Q_k) lies along the direction $\boldsymbol{\kappa}$, and two other axes Q_1 and Q_2 are in the plane which is perpendicular to $\mathbf{K}_n$. These two axes coincide with the directions of the principal axes of the quadratic form in the exponent in (1).

Hence, the disorientation of the separate sections of the crystal caused by dislocation walls leads to an asymmetry of the intensity distribution in the reciprocal lattice space. The considerable difference of the sizes of broadening along the radius $\mathbf{q}_1$ of the sphere S in the reciprocal lattice space with its center in the zero node of the lattice (the Bragg's direction) and along the surface of this sphere (the anti-Bragg's direction) is observed. It follows from formula (1) that the intensity distribution along the sphere S has a Gaussian form. Iso-intensity lines in the neighborhood of the reciprocal lattice node present the ellipses with semiaxes [6] which are proportional to σ_{11} and σ_{12} (2). The iso-intensity lines depend on: 1) the sizes of the irradiating section of the crystal in the direction $\mathbf{e}_i$; 2) the parameters D_i and h_i; 3) the reciprocal lattice node; 4) the arrangement of the planes of dislocation walls. In the general case of many systems of dislocation walls, the ellipse axes are turned relatively to the crystallographic directions of the lattice by some angle which depends on the angle under consideration and the parameters of dislocation walls.

In our case, the presence of the chaotically arranged dislocation walls consisting of equidistant edge dislocations of one sign causes the broadening of the intensity lines on a Debye photograph in accordance with (1). The curve of the intensity distribution of scattered X-rays has the Lorentz form, and the integral width of the distribution takes the form

$$2\delta\theta_i = \lambda \sec\theta \sum_i \frac{|(\mathbf{e}_i, \mathbf{k})|}{D_i}, \tag{5}$$

where 2θ is the scattering angle, and λ is the X-ray wavelength.

It follows from formula (5) that the integral width can depend, in the general case, on the orientation of the system of walls in a crystal and the direction of the diffraction vector. Moreover, in the commonly used phenomenological model of blocks, $2\delta\theta_i$ is proportional to $\sec\theta$ and, for blocks of the same size ($D_i = D$) is inversely proportional to D.

It should be noted that the broadening $2\delta\theta_i$ is substantial only for small blocks $D \leq 10^{-8}$m. If a crystal is divided into blocks and subgrains and they differ strongly in sizes and the disorientation, then the intensity distributions in the direction $\mathbf{k}$ and along the sphere S will be determined by different parameters. In this case, the inequality $\sigma_k \ll \sigma_{11} \sim \sigma_{12}$ is satisfied.

3. Intensity Distribution of Scattering by Fragments and Blocks

Let two systems of dislocation walls be given in the crystal, one of which confines the blocks of the average size D_i, and other confines the subgrains (fragments), whose average size is d_i. The disorientation of blocks and subgrains is different and is characterized by the turn angles of the lattice on the boundary $\theta_i = b/H_i$ and $\theta_i' = b/h_i$, respectively, where H_i and h_i are the distances between dislocations in the i-th wall of a block and a subgrain, respectively. The size of blocks is significantly less than that of subgrains, and $\theta_i << \theta_i'$. That is, the criteria

$$\varepsilon_i = \frac{d_i}{D_i} \ll 1, \quad \Delta_i = \frac{h_i}{H_i} \ll 1 \tag{6}$$

are satisfied, if, for any i-th system, the inequality

$$\frac{\Delta_i^2}{\varepsilon_i} \ll 1 \tag{7}$$

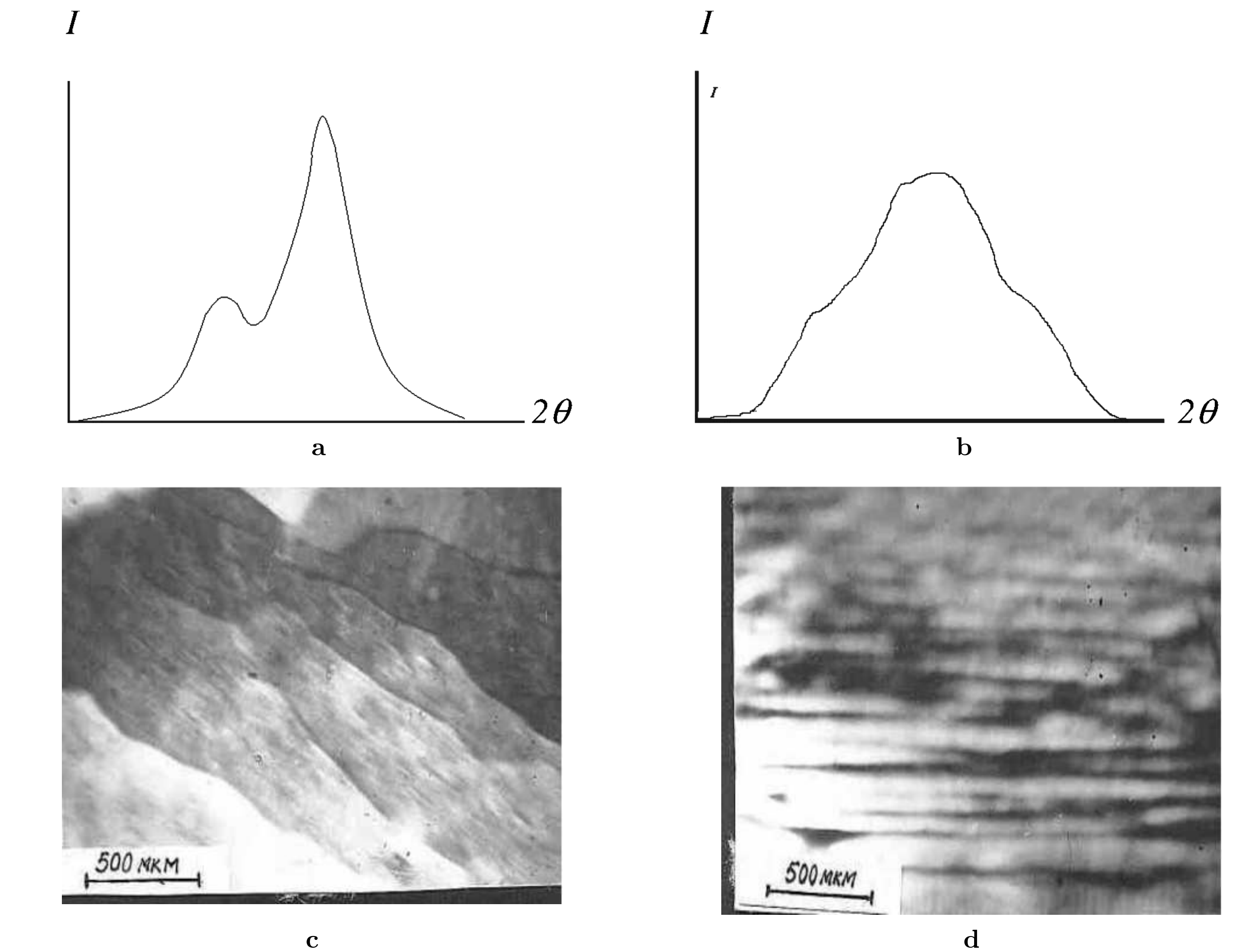
holds. Then the integral width of the intensity distribution on a Debye photograph (which is proportional to σ_k) will be inversely proportional to the size of blocks D_i, and the broadening in the azimuthal direction is determined by the value

$$\sigma_\xi \approx \frac{1}{\sqrt{D_i}} \frac{1}{h_i}. \tag{8}$$

That is, it depends on the sizes and the disorientation of subgrains. Criterion (7) will be satisfied, if, for instance, $d_i \sim 10^{-6}$m, $D_i \sim 10^{-5}$m, $h_i \sim 10^{-9}$m, $H_i \sim 10^{-8}$m.

4. Determination of Sizes of Blocks and Subgrains by the Broadening of X-ray Lines

In [7], the broadening of the scattering intensity distribution in the azimuthal direction (along the sphere S) obtained by the oscillating crystal method was considered. The pattern on the X-ray photograph oscillates by a small angle, which exceeds the disorientation angle δ, in the neighborhood of the reflection position relative to the axis normal to the directions of the incident ray $2\pi\mathbf{K}_0$ and the diffraction vector $2\pi\mathbf{K}_n$ of a radiating node of the reciprocal lattice. In this case, the different sections of the region with increased intensity in the neighborhood of the reciprocal lattice node are successively placed in the reflecting position. Hence, the X-ray pattern fixes the integral intensity from all points lying along the direction



X-ray intensity distribution: *a* — original (as-received) molybdenum monocrystal, *b* — after diffusion of silicon into molybdenum, *c* — X-ray pattern before diffusion, *d* — after diffusion

$\mathbf{t} = [\boldsymbol{\kappa}, \mathbf{L}]$, where $\mathbf{L} = [\mathbf{K}_0, \mathbf{K}_n]$ is the vector along the oscillation axis. By integrating the total intensity as in [7], we obtain

$$I\left(Q_k\right) = \frac{f^2\sqrt{\pi}}{Sv}\frac{1}{\sigma_k}\exp\left[-\frac{1}{4}\frac{Q_k^2}{\sigma_k^2}\right], \quad (9)$$

where $\sigma_k^2 = 2\sum_i \frac{R_i}{D_i}\frac{(q_1 b_i)^2}{h_i^2}\left([\boldsymbol{\kappa}, \boldsymbol{\tau}_i], \mathbf{L}\right)^2$, S is the constant ~ 1, and $\nu \sim R^3$ is the crystal volume.

The experimental quantity δ is proportional to σ_k. By performing the measurements for different nodes, we can determine (if $\frac{\Delta_i^2}{\varepsilon_i} \ll 1$) the quantities $R_i b_i^2 / D_i h_i^2$ for different values of i (i.e. for different systems). In the case of the investigation of the structure of molybdenum monocrystals (see, e.g., [8]), we can determine the size of blocks before and after the diffusion treatment. Using (8), we determined the average size of blocks to be $\sim 5 \cdot 10^{-4}$m (Figure, *c*) and $\sim 2 \cdot 10^{-5}$m (Figure, *d*).

5. Conclusion

Thus, if criterion (7) is satisfied, the measurement of the integral width on Debye photographs allows us to determine the size of blocks. Under the condition that $D_i = D$, $h_i = h$, we can obtain the values of Dh^2 which correspond to the sizes of grains. If $\left(\frac{h_i}{H_i}\right)^2 \frac{D_i}{d_i} \gg 1$ is satisfied, then the measurements of the quantity δ on Debye photographs allow us to determine the average size of subgrains d_i and the disorientation of blocks θ.

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АНАЛІЗ РОЗШИРЕННЯ РЕНТГЕНІВСЬКИХ ЛІНІЙ КРИСТАЛАМИ, ЩО МІСТЯТЬ СУБМЕЖІ ТА БЛОКИ

К.П. Рябошапка, Є.І. Богданов, С.Є. Богданов

Р е з ю м е

Проведено теоретичний аналіз розподілу інтенсивності розсіяння рентгенівських променів кристалами із вмістом субзерен та блоків. Показано, що наявність хаотично розміщених дислокаційних стінок, які складаються із еквідистантних краєвих дислокацій одного знака, приводить до розширення рентгенівських ліній на дебаєграмі. Інтегральна ширина в загальному випадку може залежати від орієнтації стінок та напрямку дифракційного вектора. Вимірювання інтегральної ширини рентгенівських ліній дозволяє визначити розмір блоків, а за деяких умов розмір і розупорядкування блоків.