X-Ray Diagnostics of Nanoscale Defects in Single-Crystals by Deformation Dependencies Method for Total Integrated Intensity of Dynamical Diffraction

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One of the necessary conditions for creating functional materials with new properties is the presence of an appropriate diagnostic base for control of structural characteristics. X-ray diffraction methods play an important role in solving this problem. These non-destructive and at the same time highly informative methods make it possible to detect defects of small sizes and concentrations, in particular micro- and nano-sized ones. To increase the speed of diagnosis, it is advisable to measure certain integral parameters. Along with other integral methods, the method of deformation dependencies of total integrated intensity of dynamical diffraction (TIIDD) has become widespread. The purpose of this work is to determine the interconnection between the parameters of the deformation dependences of total integrated intensity of dynamical diffraction and the parameters of imperfect structure in single-crystalline materials.

The following model is proposed and used in the work :

$$R = R_{\mathrm{ib}}^{\mathrm{coh}} + R_{\mathrm{i}}^{\mathrm{dif}}\,(1+1,65(\alpha'/\alpha)BT + 1,6(\beta'/\beta)B^2T^2)\exp(-6,97\cdot10^{12}(\gamma'/\gamma)\left|1/r\right|^2\sqrt{M_0} + \mu_0 l\Lambda\delta/r),$$

where $R_{\mathrm{ib}}^{\mathrm{coh}}$ is the coherent component of the total integrated intensity of dynamical diffraction for the curved crystal with microdefects, $R_{\mathrm{i}}^{\mathrm{dif}}$ is the diffuse component of the total integrated intensity of dynamical diffraction for the crystal with defect structure, *BT* is the effective deformation, α, β, γ, δ and α', β', γ' are parameters of the model for coherent and diffuse components, respectively.

To establish the connection between the parameters of the deformation dependences of the diffuse component of TIIDD and the parameters of defects, Si samples annealed at different temperatures for different times were selected. According to the theory of the formation of SiO2 precipitates during annealing of CZ Si single crystals, the concentration and average size of these precipitates are determined by the temperature and time of annealing. It is also known that, in addition to precipitates, prismatic dislocation loops and small dislocation loops formed in the annealed samples. The concentrations of small dislocation loops are proportional to the volume fraction of precipitate clusters.

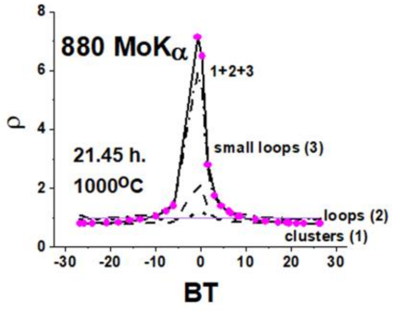

Fig. 1. The deformation dependencies of coherent and diffuse components of TIIDD, normalized to the deformation dependencies of IIDD for perfect crystal: dashed lines are the deformation dependencies of the coherent component of TIIDD, solid lines are the deformation dependencies of the diffuse component of TIIDD

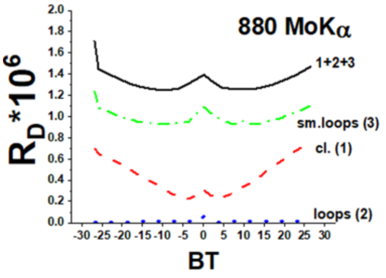

Fig. 2. The deformation dependencies of diffuse component of TIIDD R_D: *1* — presence in single-crystal clusters-precipitates SiO_2 only, *2* — presence in single-crystal ‘large’ dislocation loops only, *3* — presence in single-crystal ‘small’ dislocation loops only, $1+2+3$ — total deformation dependency of TIIDD taking into account all mentioned defect types in single-crystal (single-crystal Si, reflex (880) radiation MoK_α).

Figure 1 shows a typical dependence of TIIDD on the effective strain for a high-order reflex (880). By analyzing such deformation dependences of TIIDD, phenomenological relations between the deformation dependences of parameters of the diffuse component of TIIDD α', β', γ' and defect characteristics were found:

$$\alpha'/\alpha = 0,$$

$$\beta'/\beta = 0,73(R_{\max}/\Lambda)/L,$$

$$\gamma'/\gamma = 0,0779/L.$$

Here $R_{\max}$ is the the largest of the values of the average radii of clusters, loops and small loops present in the studied sample, Λ is the extinction length, L is the Krivoglaz factor (static Debye-Waller factor).

Figure 2 demonstrates the growth of the diffuse component of TIIDD with increasing effective strain for defects of different types with their simultaneous presence in the crystal when using the high-order reflex (880).

In Figure 2, the curve 1 describes the TIIDD in the presence of only precipitate clusters in the single crystal SiO_2, the curve *2* describes the TIIDD in the presence of only "large" dislocation loops, the curve *3* describes the TIIDD in the presence of only "small" dislocation loops, $1+2+3$ is the complete deformation dependence of the TIIDD taking into account the presence of all specified types of defects in the single crystal: clusters, "large" and "small" dislocation loops.

It can be seen from Figure 2 that the value of the normalized diffuse component of the deformation dependences of the TIIDD increases steepest for clusters, increases equally weakly for small dislocation loops and in the presence of all three types of defects in the sample, and sharply decreases with decreasing deformation for dislocation loops.

As a result, with a relatively weak degree of effective with a significant contribution of the diffuse component the experimental deformation dependence at separate intervals will coincide with deformation dependences of TIIDD calculated for small dislocation loops. The deformation dependences of the TIIDD, calculated for clusters and for dislocation loops, for which the contribution of the diffuse component is small, will coincide with separate intervals of the experimental deformation dependences of the TIIDD at much higher degrees of effective deformation. This makes it possible to carry out multi-parameter diagnostics of the defective structure of single crystals using only one deformation dependences of the TIIDD.

The interconnection between parameters of deformation dependencies of diffuse component of total integrated intensity of dynamical diffraction (TIIDD) and parameters of imperfect structure of single-crystalline materials is determined empirically. It is shown that deformation dependencies of diffuse component of total integrated intensity of dynamical diffraction are qualitatively different from each other for various types of micro- and nano-defects. As a result, the deformation dependencies of total integrated intensity of dynamical diffraction are selectively sensitive to each defect type for single-crystal with a several types of defects at different values of the effective deformation..