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Double- and triple-crystal X-ray diffractometry of microdefects in silicon

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Abstract. The generalized dynamical theory of X-ray scattering by real single crystals allows to self-consistently describe intensities of coherent and diffuse scattering measured by double- and triple-crystal diffractometers (DCD and TCD) from single crystals with defects in crystal bulk and with strained subsurface layers. Being based on this theory, we offer the combined DCD+TCD method that exhibits the higher sensitivity to defect structures with wide size distributions as compared with any of these methods alone. In the investigated Czochralski-grown silicon crystals, the sizes and concentrations of small oxygen precipitates as well as small and large dislocation loops have been determined using this method.

Keywords: dynamical scattering, triple-crystal diffractometer, double-crystal diffractometer, microdefects.

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1. Introduction

Silicon single crystals contain microdefects (MDs) of different types and sizes ranged from nano- to micrometers [1]. Such complicated defect structures arise both during crystal growth and in consequence of various technological treatments. Besides, disturbed or distorted surface layers are present in these crystals with strains caused by intentional modification or due to natural surface relaxation, including strains due to "mirror image forces" from point defects [2] and MDs [3]. The consecutive and self-consistent description of X-ray diffraction patterns from such crystals, which are measured by high-resolution double- (DCD) or triple-crystal (TCD) X-ray diffractometers, should take into account the influence of all the above mentioned factors on the reflectivities of investigated samples.

The TCD measurements provide the most complete diffractometric characterization of both defects in the crystal bulk [4-6] and strains in disturbed surface layer [7]. However, when analyzing diffraction patterns, the consideration is usually limited to the investigation of the diffuse scattering (DS) intensity distributions only in those reciprocal space regions where the coherent component can be neglected [8-9]. Such approach can cause systematical errors when determining defect characteristics. There exist also restrictions connected with the low sensitivity of reciprocal space maps measured by TCD in the cases when MDs have very small radii and corresponding DS intensity distributions are very smooth, or MDs have very large radii and corresponding DS peaks are located within the total reflection range.

The DCD measurements provide more sensitive characterization of MDs with very small radii due to the additional integration of DS intensity over the horizontal divergence. However, the determination of MD characteristics and, especially, parameters of disturbed surface layers by this method can be ambiguous [10, 11]. The circumstances stated above lead to the idea of joint using both diffractometric methods with the aim to compensate disadvantages of each of them by advantages of the another one [12].

In this work, the analytical expressions for the description of X-ray diffraction profiles measured by DCD [13-16] and TCD [17-19] from single crystals with homogeneously distributed defects of several types and disturbed surface layers, which take into account also the DS intensity from defects in monochromator and analyzer, have been used to develop the combined method for the characterization of defect structures in single crystals with joint treatment of DCD and TCD profiles. It should be emphasized that the used approach [13-19] allows to consider the X-ray scattering in crystals containing MDs of arbitrary sizes due to account for multiple DS effects in coherent and diffuse components of the diffracted intensity.

2. Experimental

The investigated Czochralski-grown silicon sample has been cut from the central part of 10 cm wafer with (111) surface plane perpendicular to the growth direction. The wafer was of *p*-type conductivity with the resistance of 10.5 Ohm×cm. The thickness of sample after chemical etching was 480 μm. The sample contained $1.1\cdot10^{18}$ cm^{-3} and less than 10^{17} cm^{-3} of oxygen and carbon atoms, respectively, and was annealed at 750 °C for 50 h in argon atmosphere under 150 kPa pressure.

Measurements of diffraction profiles in the symmetric Bragg reflection geometry have been carried out near the reciprocal lattice point (111) in dispersionless scheme (*n*, –*n*, *n*) by automated TCD with widely open detector window. The RCs have been measured when the investigated sample was mounted on analyzer place and two monochromators in dispersion position were mounted on first two axes. Thus, the scheme of DCD measurements was (*m*, *m*, –*n*). At the treatment of DCD profiles, vertical and horizontal divergences as well as dispersion of X-ray beam were taken into account.

3. Treatment and analysis of measurement results

The TCD method has a low sensitivity to small nm-sized MDs (and to large MDs with sizes of the order of the extinction length) because DS intensity from these MDs is very weak in measured reciprocal space regions. On the other hand, DCD method has high sensitivity to both small and large MDs due to the additional integration of DS intensity over the horizontal divergence, but has a disadvantage of the wide uncertainty range for characteristics of MDs with intermediate sizes, what is a consequence of the same integration. The joint treatment of TCD and DCD diffraction profiles (Fig. 1) allows to increase the sensitivity to MD characteristics in the whole size range from nano- to micrometers.

Such joint treatment of measured diffraction profiles was carried out in the iterative way, when MD characteristics determined from TCD profiles were used as initial ones at the treatment of DCD profiles, and vice versa. At the treatment of the measured diffraction profiles, we used the model of a defect structure in the investigated silicon sample, which supposed the presence of randomly distributed disk-shaped oxygen precipitates and chaotically oriented circular dislocation loops with Burgers vectors $\boldsymbol{b} = \langle 110\rangle/2$ [1, 15, 18].

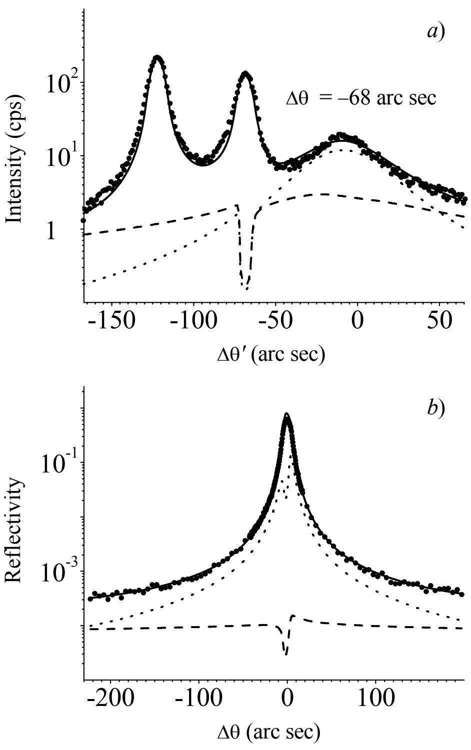

Fig. 1. Fitted diffraction profiles (solid lines) measured by TCD (*a*) and DCD (*b*) for Si (111) reflection of CuK$_{\alpha_1}$ radiation from Czochralski-grown silicon sample annealed at 750 °C for 50 h. Dashed and dotted lines describe DS intensities from oxygen precipitates and dislocation loops, respectively.

The proposed method has allowed to simultaneously determine characteristics of small and large MDs in the crystal bulk, namely, the characteristics of small oxygen precipitates of the radius $R_P = (7.7 \pm 0.2)$ nm, thickness $h_P = (2.2 \pm 0.05)$ nm, and numeric density $n_P = (2.8 \pm 0.3)\times10^{13}$ cm^{-3}, as well as characteristics of large and small dislocation loops (see Fig. 2).

Additionally, the proposed approach has allowed for the quantitative investigation of the strain in subsurface layer, which is caused by “mirror image forces” from point defects and MDs. This strain was described by the exponential law $\varepsilon_\perp = \varepsilon_{0\perp}\exp(-z/t_0)$, where z is a depth, and the parameters determined by fitting the experimental diffraction profiles are $t_0 = (7.0 \pm 0.7)$ nm and $\varepsilon_{0\perp} = (1.0 \pm 0.1)\times10^{-4}$ (see Fig. 3).

Besides, the quantitative description of the asymmetrical behaviour of pseudo-peak heights on TCD profiles measured at opposite deviation angles of the investigated sample has allowed to determine the characteristics of dislocation loops in TCD monochromator, which has been made of Czochralski-grown silicon crystal, namely, $R_L = (1.0 \pm 0.03)$ μm and $n_L = (1.1 \pm 0.1)\times10^8$ cm^{-3}.

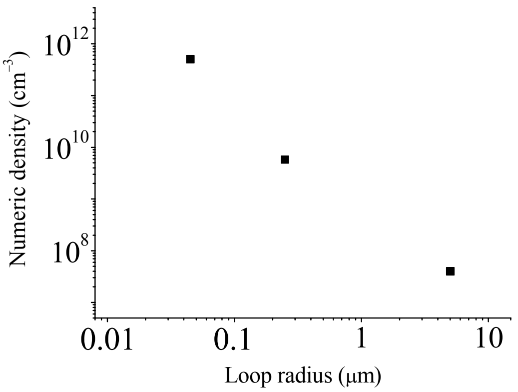

Fig. 2. Determined numeric density of dislocation loops (sizes of markers correspond to error bars).

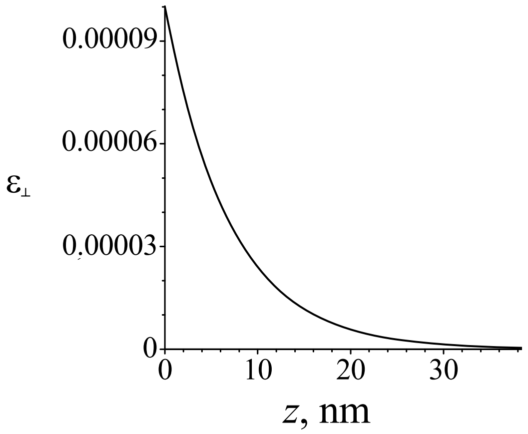

Fig. 3. The strain profile caused in a subsurface layer by "mirror image forces" from point defects and MDs in the investigated silicon single crystal.

In the whole, the proposed complex diffractometric method with using the analytical formulas of the generalized statistical dynamical theory for X-ray scattering by real single crystals offers the new possibilities for the extended quantitative characterization of the defect structures in imperfect single crystals.

4. Conclusion

Thus, the joint treatment of DCD and TCD diffraction profiles can substantially improve the completeness and uniqueness of characterization of complicated defect structures in real single crystals. Of course, the key role in the adequate treatment of measurement results is played by applying the formulas of the generalized statistical dynamical theory for X-ray scattering by real single crystals, which allows to self-consistently describe the coherent and diffuse scattering intensities. The exclusive importance of account for the simultaneous presence of various-type MDs for the correct interpretation of diffraction patterns from silicon crystals (see, e.g., also [6, 20, 15]) should be emphasized as well.

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