Vol. 91 (1997) ACTA PHYSICA POLONICA A No. 5

Proceedings of the 3rd ISSSRNS Jaszowiec '96

# INTEGRAL STRUCTURE PERFECTION DIAGNOSTICS OF SINGLE CRYSTALS USING TWO WAVELENGTHS OF X-RAY SPECTRUM

L. DATSENKO, S. KRASULYA, V. MACHULIN

Institute of Semiconductor Physics, National Academy of Sciences of Ukraine
Kiev, Ukraine

J. AULEYTNER

Institute of Physics, Polish Academy of Sciences
Al. Lotników 32/46, 02-668 Warsaw, Poland

AND V. KHRUPA*

Department of Physics, Science Laboratories, University of Durham
Durham, South Road, DH1 3LE, U.K.

A new approach to determination of microdefect structure parameters by means of single crystal diffractometer is proposed. The approach is based on the measurements of the integral reflectivity of a sample for two selected X-ray wavelengths providing with the approximations of thin and thick crystal, respectively.

PACS numbers: 81.40.–z, 61.10.–i

## 1. Introduction

Integral structure parameters of microdefects i.e. a concentration $n$ and characteristic radius $r$, entering the corresponding expressions for the Debye–Waller factor $L$ [1–3] and the coefficient of $\mu_d$, additional energy losses of X-rays due to diffuse scattering on defects, determine finally the value of the integral reflectivity $R_i$ of a dislocation-free real crystal. There are several experimental methods basing on the $R_i$ measurements which permit to obtain the integral characteristics of structure perfection of a crystal ($L$, $\mu_d$) [3–7]. Also several combinations of these methods exist [8, 9]. However, each of them is either relatively difficult in performing or may be used only by providing special conditions.

Therefore, the purpose of this work is the developing and experimental testing of a new simple approach which makes it possible to determine the integral characteristics of microdefects in a nearly perfect crystal by means of the single crystal diffractometer using two wavelengths of the X-ray spectrum.

*Permanent address: Institute of Semiconductor Physics, NAS, Kiev, Ukraine.

## 2. Theoretical background of the method

After determination of the $L$ and $\mu_d$ from the $R_i$ measurements the integral characteristics of defects i.e. $n$ and $r$ can be calculated from the following expressions:

$$L = 8nr^{9/2}(\beta H)^{3/2}, \tag{1}$$

$$\mu_d = 8\pi^2 rL\Lambda^{-2}\cos^2\theta_B, \tag{2}$$

where $H$, $\Lambda$, $\theta_B$ and $\beta$ stand for the reciprocal lattice vector, extinction length, the Bragg angle and the constant close to 0.01 for dislocation loops or large clusters.

For the case of low level of the X-ray absorption (the approximation of thin crystal, $\mu_0 t < 1$, here $\mu_0$ and $t$ are the linear absorption coefficient and the thickness of a sample) the $R_i$ of slightly distorted crystal consisting of the coherent $R_B$ and the diffuse $R_D$ components may be written in the following way [9]:

$$R_i = R_B + R_D = R_i^P e^{-L} + 2LQte^{-h}, \tag{3}$$

where $R_i^P$ is reflectivity for a perfect crystal and Q is that for a ideal mosaic sample, $R_i^P = C\pi|\chi_{rH}|I_0(C\varepsilon h)e^{-h}/2\sin 2\theta_B$, $Q = C^2\pi^2\chi_{rH}^2/\lambda\sin 2\theta_B$; $c$, $\chi_{rH}$, $\lambda$, $t$ stand for the polarization factor, real part of the Fourier coefficient of susceptibility $\chi$; X-ray wavelength and the crystal thickness respectively. $h = \mu_0 t/\gamma$, $\gamma = \cos\theta_B$, $I_0(C\varepsilon h)$ is the Bessel function of an imaginary argument. $\varepsilon = \chi_{iH}/\chi_{i0}$ is the ratio of the $\chi$ for the H-th and 0-th reflections.

When $L \ll 1$ and the hard radiation are used in the experiment (thin crystal) the integral reflectivity at the Laue diffraction has low sensitivity to the $\mu_d$ parameter [3, 9] similar to that one in the Bragg case of diffraction [10]. Therefore $R_i$ depends in this case on the static Debye–Waller parameter only.

Actually, the fit of the curve calculated by the formula (3) to the experimental data, when the $\mu_d$ is omitted, describes properly the dependences of reflectivity on a thickness [3, 11]. Such approximation fulfils even more exactly in the case of the gamma-radiation [6]. In the case of a thick crystal the approximation of the $R_i$ value depends on the both integral parameters of structure perfection. The simple expression for the $R_i$ was obtained in [12] for the case of $\mu t > 6$

$$R_i = R_i^P e^{-(L+Z)}(1 + \alpha Z/\varepsilon h\sin 2\theta_B), \tag{4}$$

where $Z = \mu_d t/\gamma$ and $\alpha = 1.5[1 - \exp Z\exp(\varepsilon h)]/(1 - Z/\varepsilon h)$.

Therefore, varying the absorption level of the sample by choosing the wavelength of X-ray spectrum one can provide the needed level of absorption. In one case the sample may be considered as a thin one and then, formula (3) can be applied. In another case the thick crystal approximation takes place and the $R_i$ may be described by expression (4). Having determined parameter $L$ in the first case which is known that not depends on the wavelength one can calculate the second characteristic $\mu_d$ by the last expression (4). Then, using formulas (1) and (2) one may estimate the $r$ and $n$ values.

## 3. Experiment results and discussion

Dislocation-free silicon crystals grown by Czochralski method (Cz-Si, oxygen concentration close to $10^{18}$ at/cm$^{-3}$) in initial state (sample 1) and that after

annealing during 4 hours at 850°C were chosen for investigation by the proposed approach. All $R_i$ measurements were carried out in the Laue geometry by means of the single crystal diffractometer for the 220 reflections using the characteristic Ag $K_{\alpha_1}$ line and the wavelength of continuous spectrum corresponding to the Cu $K_{\alpha_1}$ radiation. For determination of the intensity of primary beam the perfect ($e^{-L} > 0.999$) float-zone grown silicon reference sample was used.

All results of the $R_i$ measurements and the determination of the integral characteristics of the crystal perfection $L$, $\mu_d$, $r$ and $n$ which were calculated by means of formulae (1)–(4) are given in Table.

TABLE

The values of integral reflectivity, $L$, $\mu_d$ as well as the radius $r$ and concentration $n$ of microdefects.

| $\lambda \times 10^8$ [cm] | $\mu_0 t$ | $R_i \times 10^6$ | $R_i^p \times 10^6$ | $L \times 10^3$ | $\mu_d$ [cm$^{-1}$] | $r$ [$\mu$m] | $n$ [cm$^{-3}$ |
|---|---|---|---|---|---|---|---|
| 0.5594 | 0.96 | 5.69 | 2.88 | 4 | – | | |
| 1.5405 | 18.7 | 3.38 | 4.79 | – | 1.6 | 0.1 | $2 \times 10^7$ |

Values of $r$ and $n$ obtained by independent method using X-ray diffraction in the Bragg geometry are 0.2 $\mu$m and $8 \times 10^7$ cm$^{-3}$, respectively (see our article in this issue).

It is important to note that the experimental value of $R_i$ considerably exceeds the level of this parameter for a thin perfect crystal. On the contrary, in the case of a thick crystal the corresponding experimental $R_i$ value was lower than that for the perfect crystal.

The value of the Debye–Waller factor ($L = 0.004$) in the initial state of the crystal was close to the meaning of these parameters known for Cz-Si crystals with a high oxygen concentration [4–6]. It testifies the reliability of the proposed procedure for determination of the $L$. The calculated data concerning the $n$ and $r$ parameters determined by utilizing the $\mu_d$ value are reasonable, too. They are close to these parameters for Cz-Si crystals which contained $9 \times 10^{17}$ at/cm$^{-3}$ of oxygen and were annealed at temperatures near the 800°C [13]. It means that proposed approach makes it possible to obtain reliable information about the integral structure characteristics of microdefects.

This paper was done by partial financial support of the Ukrainian Scientific and Technology Committee and the Committee for Scientific Research (Poland).

## References

[1] M.A. Krivoglaz, *Theory of X-ray and Thermal Neutron Scattering by Real Crystals*, Plenum, New York 1969.

[2] P.H. Dederichs, *Solid State Phys.* **27**, 135 (1972).

[3] L.I. Datsenko, V.B. Molodkin, M.E. Osinovskii, *Dynamical Scattering of X-rays by Real Crystals*, Naukova Dumka, Kiev 1988 (in Russian).

[4] N.M. Olekhnovich, A.L. Karpei, *Phys. Status Solidi A* **82**, 365 (1984).

[5] S.N. Voronkov, F.N. Chukhovskii, D.I. Piskunov, *Sov. Phys.-Solid State* **27**, 1151 (1985).

[6] J.R. Schneider, R. Bouchard, H.A. Graf, H. Nagasawa, *Acta Crystallogr. A* **48**, 804 (1992).

[7] I.R. Entin, V.I. Khrupa, L.I. Datsenko, *J. Appl. Cryst.* **23**, 355 (1990).

[8] J. Auleytner, *X-ray Methods in the Study of Defects in Single Crystals*, Pergamon Press and Polish Scientific Publs., Warszawa 1967, p. 86.

[9] V.F. Machulin, V.I. Khrupa, *Structure Perfection X-ray Diagnostics of Slightly Distorted Crystals*, Naukova Dumka, Kiev 1995 (in Russian).

[10] V.I. Khrupa, S.M. Krasulya, V.F. Machulin, V.B. Molodkin, L.I. Datsenko, *Metallofizika* **17**, 27 (1995).

[11] L.I. Datsenko, V.I. Khrupa, M.Ya. Skorokhod, V.V. Nikolayev, M.E. Osinivskii, S.I. Olikhovskii, *Metallofizika* **9**, 65 (1987).

[12] V.V. Kochelab, V.B. Molodkin, S.I. Osinovskii, *Phys. Met.* **11**, 802 (1992).

[13] S. Iida, H. Sugiyama, Y. Sugita, H. Kawata, *Jpn. J. Appl. Phys.* **27**, 1081 (1988).