RAPID STRUCTURE PERFECTION DIAGNOSTICS OF GaAs SINGLE CRYSTAL BY DIFFRACTION OF WHITE X-RAY RADIATION

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Sensitivity of X-ray integral reflectivity of GaAs single crystal to a degree of structure distortions was established to grow considerably in the Bragg diffraction case when the characteristic Ag K_{α_1} line is changed for more hard white radiation. In effect, the absorption length essentially exceeds the extinction length what results in enhancement of incoherent scattering. Measurements of X-ray integral reflectivity coordinate dependence by single crystal spectrometer permitted to determine the mean level of crystal lattice distortion as well as the degree of structure homogeneity of a sample with dislocations. The Debye–Waller static factor value was estimated from X-ray integral reflectivity magnitudes for the 800 reflection of white radiation.

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1. Introduction

Several methods of diffractometric diagnostics of single crystal structure perfection from X-ray integral reflectivity (XIR) of periodic media are well known [1–3]. They use diffraction of either characteristic or white X-ray radiation. However, most of them are not efficient in the case of a heavily absorbing crystal, such as GaAs.

The condition of weak X-ray absorption ($\mu t < 1$, where μ and t stand for a linear photoelectric absorption coefficient and thickness of a sample, respectively) for relatively thick (with thickness about 1 mm) GaAs crystals has been shown to occur at Laue diffraction of hard components of the X-ray continuous spectrum [4]. This circumstance stimulated development of nondestructive procedure for rapid

structure perfection diagnostics of GaAs single crystal plates [5]. However, such an approach did not solve a problem of bulk GaAs crystal analysis.

In our opinion the Bragg diffraction geometry is more suitable to study the structure perfection of a thick crystal, because it is possible to avoid a scattering parameters dependence on a sample thickness. Unfortunately, capabilities of X-ray structural diagnostics using the Bragg case have been much less studied as compared with the transmission geometry case. The Bragg diffraction maxima are formed at the near-surface sample areas, close to the extinction length Λ (≈ 5–50 μm). This explains why XIR of imperfect periodic media is less dependent on crystal distortion degree in the Bragg case than in the Laue one. In the last case the scattered X-ray beams are strongly affected by defects in a whole crystal.

The original results obtained by using soft X-rays [6, 7] do not contradict this idea. But the data of later theoretical [1, 8–10] and experimental [11–17] investigations have shown that sensitivity of the XIR (R_i) to structure distortion level can be considerably enhanced at the expense of a diffuse (incoherent) scattered component by using the short wavelength radiation ($\mu^{-1} \gg \Lambda$) and the high order reflections. This component of reflectivity is formed at the absorption length, μ^{-1}, of a crystal, while a range of formation of the coherent part of reflected beams is close to the extinction length Λ.

XIR sensitivity of GaAs to a degree of structure distortion is shown in this paper to be considerably increased by a transition from the relatively short-wave characteristic (Ag K_{α_1}) radiation to hard components ($\lambda \leq 0.3$ Å) of the X-ray continuous spectrum. In the latter case one can estimate the average level of GaAs crystal lattice distortions (the static Debye–Waller factor, e^{-L}) as well as the degree of structure homogeneity of a sample from the character of the XIR coordinate dependence, i.e. $R_i(x)$.

2. Peculiarities of the experimental method

The {100} oriented Te doped GaAs crystals were investigated. According to [5] these samples were characterized by inhomogeneous distribution of the dislocations. The value of dislocation density N_d was several times higher at the center and at the edges of a plate than in the intermediate areas (Fig. 1). The N_d values (according to the metallographic etch pits counting procedure) reached approximately 1×10^5 cm^{-2} and $\approx 3 \times 10^4$ cm^{-2} at the plate center and at the above-mentioned intermediate areas, respectively.

Measurements of the XIR value were carried out for the 800 reflections by means of the single crystal method [18]. The dislocation-free Ge crystal was used as a perfect control sample. Both characteristic Ag K_{α_1} and white ($\lambda = 0.2987$ Å) X-ray radiation were used. In the latter case the absorption length ($\mu^{-1} \approx 360$ μm) was more than three times larger in comparison with the extinction length ($\Lambda_{880} \approx 110$ μm).

To calculate the Debye–Waller factor, e^{-L}, the experimental data on R_i and expression (1) were applied

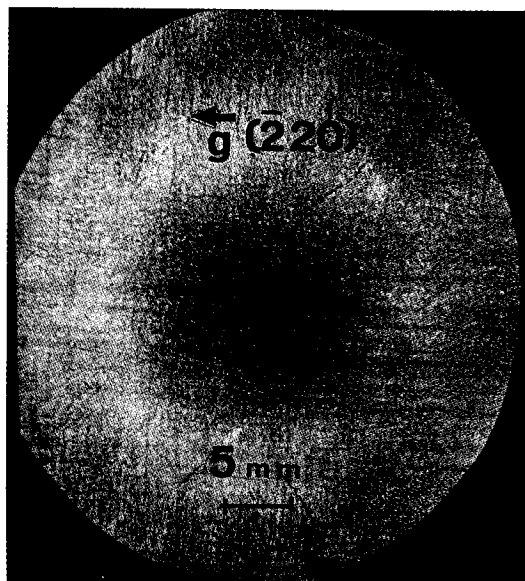$$R_i = R_i^p e^{-L} + R_i^k (1 - e^{-2L}). \tag{1}$$

Fig. 1. X-ray topograph of the GaAs specimen under investigation (Cu K_α, 220-reflection).

R_i^p, R_i^k symbols stand, respectively, for XIR of a perfect and ideally-mosaic crystal. It should be noticed that suitability of the static Debye–Waller factor for XIR describing a crystal containing dislocations has been formerly confirmed [19]. The later experimental investigations [2, 20, 21] have shown that the expression (1) may satisfactorily describe XIR of crystals containing dislocations. It should be also pointed out that similar model for describing XIR of crystals with dislocations was independently applied also in Refs. [11, 12].

To measure the $R_i(x)$ dependences, the samples were successively translated along the $\langle 110 \rangle$ direction with 1 mm step. The L value was determined for every x from $R_i(x)$ graph (Fig. 2). The $L(x)$ coordinate dependence computed according to the $L = (\mathbf{H} \cdot b)\Lambda^2 N_d/4\pi$ formula [21], (where $\mathbf{H}$ and b stand for a diffraction vector and Burgers vector, respectively) was also analyzed (Fig. 3) to check the reliability of results obtained by this calculation using Eqs. (1). The N_d value was obtained from the etched pits counting procedure.

3. Results and discussion

The coordinate dependences of the R_i/R_i^p ratio, obtained for both the characteristic and white radiation are shown in Fig. 2. One can see that the shape of both graphs reproduces qualitatively the W-like character of dislocation distribution along the plate diameter (Fig. 1). However, such a kind of XIR distribution is considerably better distinguished for more hard radiation. In this case considerable enhancement of XIR in the regions with higher N_d values is observed as compared with that for a perfect crystal. Therefore, R_i sensitivity to the lattice distortion level has been increased.
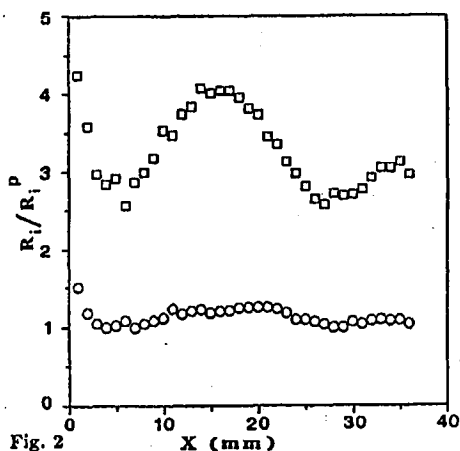
Fig. 2.   The R_i/R_i^p ratio versus coordinate x for Ag K_{α_1} radiations (o) and for $\lambda =$ 0.2987 Å(□).
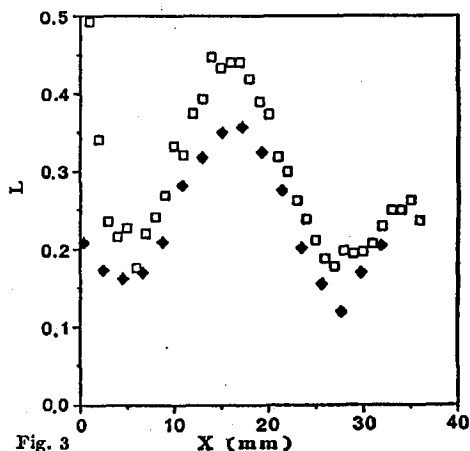
Fig. 3. $L(x)$ dependencies calculated from diffractometric (□) and metallographic (◇) data.

The results obtained can be explained as follows. The integral contribution of the diffuse component to the total XIR depends mainly on a level of photoelectric absorption of scattered radiation, contrary to the coherent part of reflectivity, which is determined completely by the extinction phenomena [1, 9, 10, 14]. It happens due to different angular intervals of coherent and diffuse component formations. The latter has more wide angular region of the primary beam acceptance so it predominates at the larger depth where the coherent part of scattering disappears due to the extinction phenomena. When the wavelength decreases, the absorption length of a medium increases according to the cubic law. As a result, an expansion of the surface layer accumulating the total incoherent component of scattering, takes place. This effect leads generally to an XIR increase. Taking into account the low level of L ($L \ll 1$), one can rewrite (1) in the following way:

$$R_i/R_i^p \approx 1 + 3L(\mu A)^{-1}\cos\theta, \qquad\qquad (2)$$

where θ is the Bragg angle. One can see that the experimentally observed increment of XIR, described by the second term in Eq. (2), depends on the $(\mu A)^{-1}$ parameter at constant L. The latter increases approximately by a factor of three in our case at a transition from characteristic to continuous spectrum what is in good agreement with the data of Fig. 2.

The comparison of the coordinate dependences, $L(x)$, obtained by both above-mentioned methods is shown in Fig. 3. It is necessary to note that the non-coincidence of the H and b vectors directions should be taken into account by using the metallographical data. One can see a good correlation between two $L(x)$ dependences. A shape of the $L(x)$ curves is also in concordance with the independent diffractometric data obtained in the Laue case of diffraction [5]. This circumstance confirms reliability of the present results. From $L(x)$ dependence one can compute

also the mean $\langle L(x)\rangle$ value as well as the normalized $\langle \Delta L(x)\rangle$ value which may characterize quantitatively the average level of crystal lattice distortion and degree of sample homogeneity, respectively ($\langle L(x)\rangle = 0.292$, $\langle \Delta L(x)\rangle = 0.092$).

4. Conclusions

In conclusion we would note that the L_{800} values for the indicated interval of dislocation density, N_d, are in satisfactory agreement with those known for various reflections [2, 5, 19–22]. Taking into account the above-mentioned results one can conclude that the new approach proposed by us is promising for a rapid preliminary structure diagnostics of bulk GaAs crystals.

Acknowledgments

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