X-RAY TOPOGRAPHY IN THE STUDY OF SEMICONDUCTORS*

D. Korytár [1]
Institute of Electrical Engineering, SAS, Vrbovská cesta 102, 921 01 Piešťany, Slovakia

C. Ferrari[2]
CNR Maspec Institute, Parco Area delle Scienze 37/A Fontanini, 43010 Parma, Italy

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X-ray topography as a completely nondestructive technique is frequently used to study crystal defects, deformation fields and compositional changes in semiconductors because of its high sensitivity to strains induced by defects in crystals. Principles of surface sensitive reflection Lang topography and reflection double crystal topography and their contrast mechanisms are shortly presented and three examples of their application given. The techniques have been used to characterize crystal defects such as threading dislocations arranged into cellular structure and slip bands at surfaces of semi-insulating GaAs substrate wafers, to characterize transformation of threading dislocations into misfit dislocations in a InGaAs/GaAs strained quantum well and to characterize misorientation domains in highly mismatched InGaAs/GaAs heterostructures. In the last case tilt regions have been evidenced by synchrotron double crystal topography.

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1 Introduction

The electrophysical properties of bulk semiconductors, of their surfaces, interfaces, and heterostructures are given or affected by the crystallographic defects as composition inhomogeneities, strains and deformation fields, dislocation or precipitate density and distribution. X-ray topography as one of X-ray diffraction techniques is often used for nondestructive structural characterization of semiconductor crystals [1]. The image which is obtained in a topograph is based on the mechanisms of contrast formation; the most common are the extinction and the orientation contrasts. Through these contrast formation mechanisms the X-ray topography is suitable for visualization of crystal defects, deformation fields and compositional changes both in macro- and micro-areas of the samples. Surface related techniques of X-ray topography are mainly : a) the reflection Lang technique b) the reflection double crystal topography.

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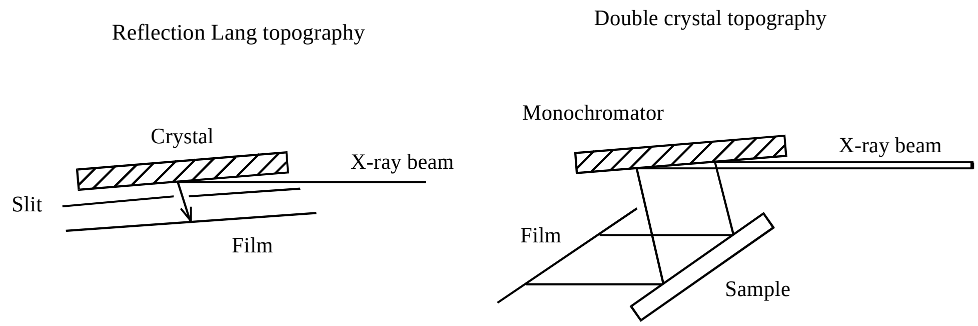
[1]E-mail address: korytar@svspn.sk
[2]E-mail address: ferrari@maspec.bo.cnr.it



(a) (b)

Fig. 1. Principles of the reflection Lang X-ray topographic techniques (a) and of reflection double crystal one (b).

In this contribution, principles and parameters of these methods and the information provided will be outlined. Namely, our results concerning slip bands, threading and misfit dislocations, cellular structures, and misorientation domains in bulk semiconductors and heterostructures will be presented.

2 Experimental techniques

The principle of the reflection Lang topography is sketched in Fig. 1a). The incident X-ray beam divergence is about 10^{-3} rad and the slit width is chosen to separate $K_{\alpha 1}$ and $K_{\alpha 2}$ lines by the sample crystal. Symmetrical (400) or asymmetrical (511), (531), and (533) $CuK_{\alpha 1}$ or $CoK_{\alpha 1}$ diffractions with penetration depths below 5 μm have been used. The simultaneous translation of the crystal and film along the wafer front plane leads to the formation of an image on the film with a contrast generated by defects in the samples.

Fig. 1b) shows the principle of the reflection double crystal topography in non-dispersive (n,-n) setting where the diffracting planes of the monochromator and sample crystal are antiparallel. As a monochromator, either 3 inch diameter Si R(511) asymmetric diffractor expanding the beam about 20 times in one direction, or 40 mm diameter Ge R(511) asymmetric diffractor expanding the beam about 14 times, both for $CoK_{\alpha 1}$ radiation, or a curvable Ge (620) diffractor for $CuK_{\alpha 1}$ radiation, have been used. According to the dynamical theory of diffraction the horizontal beam divergence after these asymmetric diffractions may be more than one order of magnitude lower than those of the incident beam and thus locating the angular position of the sample crystal on the steep flank of its rocking curve gives a very high sensitivity of the technique to local changes of lattice parameter or lattice tilts in the range of (10^{-4} – 10^{-7}). The sample crystal diffractions were chosen to have more or less non-dispersive setting, that is nearly the same Bragg angle for monochromator and sample crystal were chosen.

Semiconductors as Si, GaAs, InP substrate wafers, strained $In_{0.3}Ga_{0.7}As/GaAs$ qauntum wells and highly mismatched MBE InGaAs/GaAs heterostructures have been studied by the two

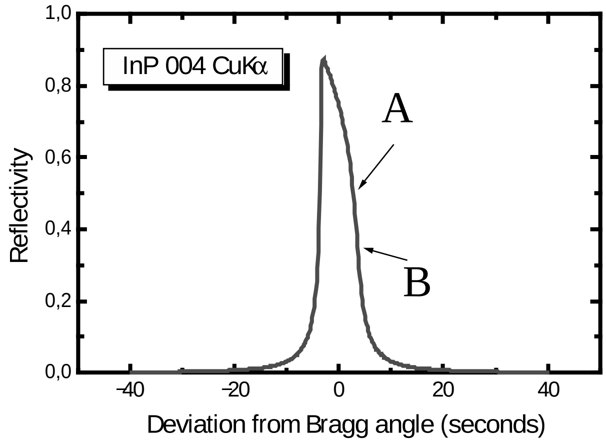


Fig. 2. The (004) rocking curve of a perfect InP sample crystal taken by a high resolution X-ray diffractometer and $CuK_{\alpha 1}$ radiation illustrating orientation contrast formation mechanism .

techniques. For epitaxial layers, separate substrate and layer peaks and images can be obtained for sufficiently large mismatches.

The contrast in X-ray topography is mainly based on two different mechanisms:

1) near the core of the defects where the crystal is so distorted to give a local Bragg condition outside the perfect crystal range predicted by the dynamical theory the kinematical theory is applied. Due to the local increase of the diffrated intensity the lattice defects appear dark and have the size of the extinction distance, that is a few μm for wavelengths in the range 1–2 Å.

2) when parts of the crystal of size larger than the extinction distance are misoriented by a quantity larger than the total reflectivity range predicted by the dynamical theory the local diffracted intensity can decrease or even vanish giving rise to the orientation contrast. The orientation contrast is well visible at a distance larger than the extinction distance near dislocations and grain boundaries.

Based on the fact that when we adjust a crystal into Bragg position relative to the incident beam, the crystal diffracts the beam with the intensity dependent on the crystal perfection and orientation. In Fig. 2 the $CuK_{\alpha 1}$ (004) rocking curve of a perfect InP samples is reported. Setting the crystal in the position A of the rocking curve in the case of a local strain or lattice tilt moves the local Bragg condition from the point A to the point B and this gives rise to a visible contrast in the topograph. This intensity contrast can be described in the first approximation by differentiating the Bragg equation

$$2d \sin \theta_{\mathrm{B}} = \lambda \tag{1}$$

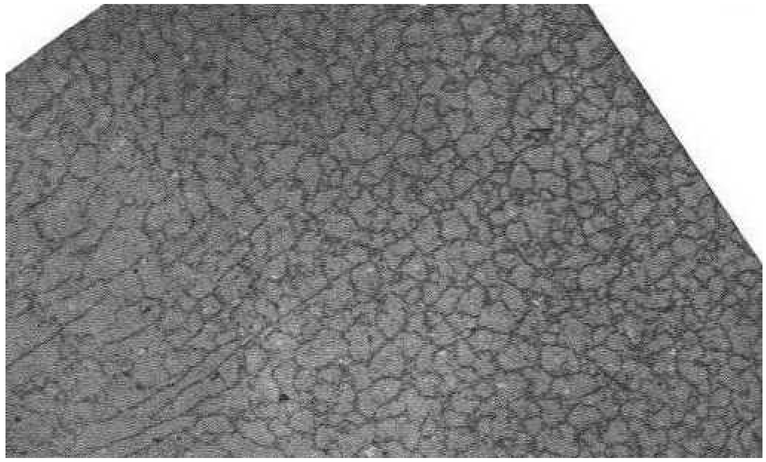


Fig. 3. Reflection Lang topograph (Cu$K_{\alpha 1}$ radiation, (531) asymmetric diffraction) of a 3 inch diameter SI GaAs substrate with relatively large threading dislocation density (10^{-4} – 10^{-5} cm^{-2}). Dislocations are mainly distributed in cellular structure and lineages. Film to sample distance decreased to 8 mm in order to increase lateral resolution. Vertical edge of the picture represents 11 mm.

From Fig. 2 the contrast $\delta I/I$ is given by the equation

$$\frac{\delta I}{I} = K\left(\frac{\Delta d}{d}\right)\mathrm{tg}\theta_{\mathrm{B}} \pm \delta\theta \qquad (2)$$

In Eqs. (1) and (2), d is the lattice parameter, Δd the lattice parameter variation, θ_{B} the Bragg angle, $\delta\theta$ the local tilt angle, λ the wavelength, and K is the slope of the rocking curve flank. The detailed contrast of the individual defects, given by strain fields around them, can be calculated by means of elasticity theory and dynamical theory of X-ray diffraction [2] and depends on the X-ray beam parameters (divergence and spectral dispersion), material and geometrical setting parameters. As a practical result, X-ray diffraction corresponding to the reciprocal lattice vector **h** is not sensitive to lattice displacement vectors **u** perpendicular to **h**.

3 Results

The most perfect crystal material ever found or produced, as-grown floated zone (FZ) silicon, shows in X-ray topographs homogeneous contrast without any crystal defects and strains. The quality of III-V semiconductors is steadily improving, too. Fig. 3 shows reflection Lang topograph (Cu$K_{\alpha 1}$ radiation, (531) asymmetric diffraction) of a 3 inch diameter semi-insulating (SI) GaAs substrate with relatively large threading dislocation density (10^{-4} – 10^{-5} cm^{-2}) with dislocations distributed mainly in cellular structure and lineages.

Figs. 4a-c) show reflection double crystal topographs of semiinsulating GaAs for radiation detectors [3]. Because of higher strain sensitivity the width of crystal defects is higher and the spatial resolution lower than in Lang topography. In Fig. 4a) we can see dislocation-free SI

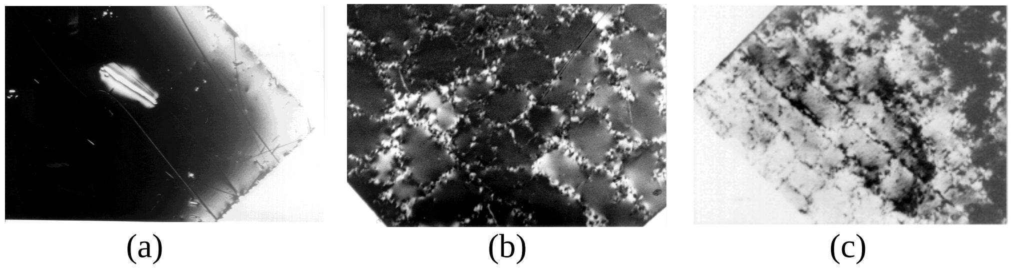


Fig. 4. Reflection CuK$_{\alpha 1}$ radiation (620) double crystal topographs of a dislocation- free SI GaAs wafer prepared by VGF technique (a), of a LEC SI GaAs wafer with dominating cellular structure (b) and another one with dominating slip dislocations (c). Vertical edges of the pictures correspond to 2.7 mm.

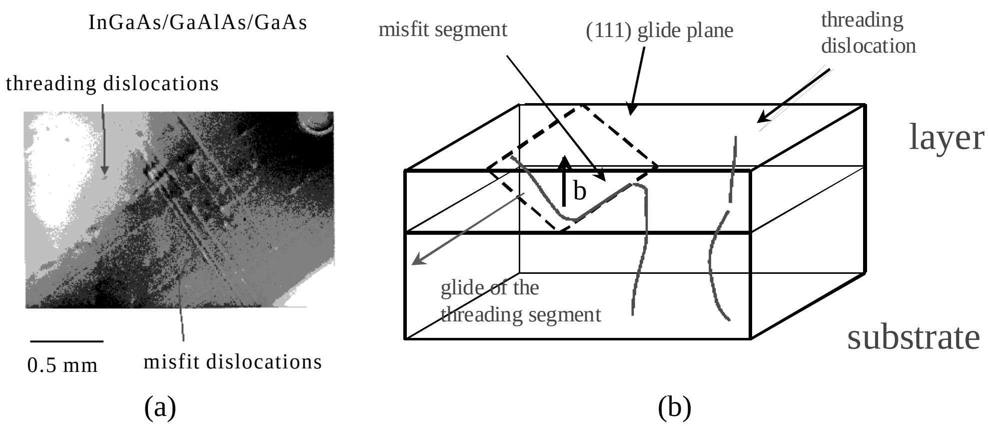


Fig. 5. Misfit dislocations generation in an InGaAs/GaAlAs/GaAs laser structure (a) according to the model proposed by Matthews and Blakeslee [4] (b).

GaAs prepared by vertical gradient freeze (VGF) technique, further topographs show wafers of Liquid Encapsulated Czochralski (LEC) SI GaAs with dominating cellular structure (Fig. 4b) and another one with dominating slip dislocations (Fig. 4c) evidenced by the distribution of threading dislocations along [110] directions. Cross hatched structure corresponding to slip dislocations is discernible.

In slightly mismatched or strained quantum well based heterostructures, misfit between substrate and epitaxial layer lattice parameters can cause problems to technologists and device engineers.

The substrate/layer misfit can be relieved by misfit dislocations by several mechanisms. Fig 5a) clearly demonstrates the mechanism of formation of misfit dislocation predicted by Matthews and Blakeslee [4] according to the model depicted in Fig. 5b).

Composition graded epitaxial layers have attracted the attention of technologists and device engineers because of the possibility to control the interfacial strain profile. Step-like, linear and

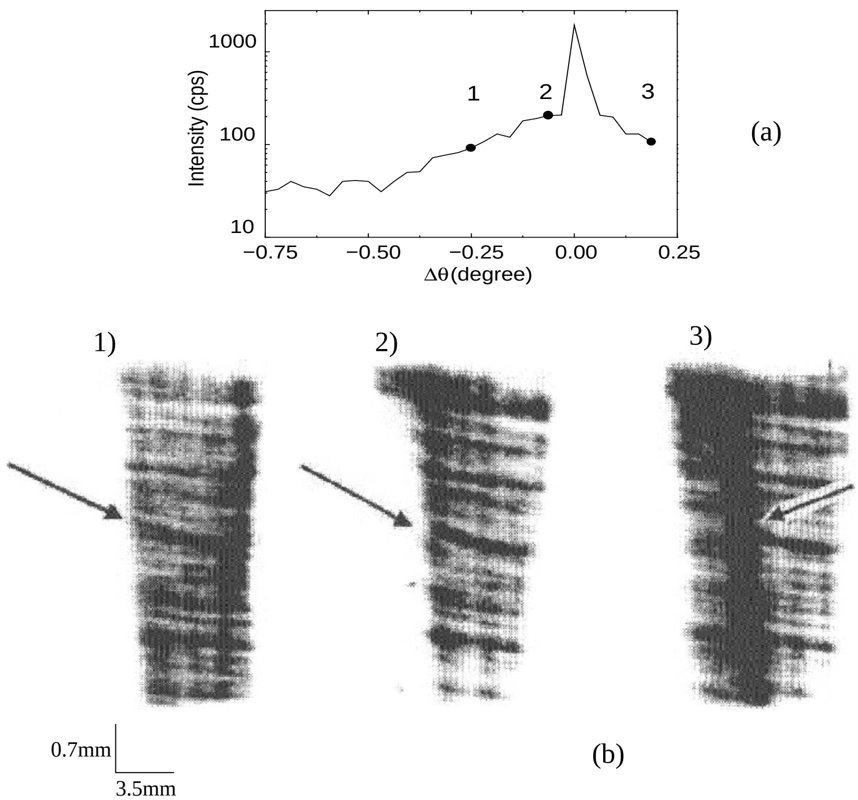


Fig. 6. Broad (004) synchrotron radiation rocking curve of a square root graded InGaAs/GaAs heterostructure (a) and X-ray double crystal topographs (b) taken at three points of the reflection curve with the exposure time about 30 secs at the beamline ID19 in ESRF Grenoble [7]. See text for the details.

square root compositional gradients are of interest.

Fig. 6a) shows a (004) rocking curve of a square root graded InGaAs/GaAs heterostructure ($\mathrm{In_xGa_{1-x}As/GaAs}$ with $x_{\min} = 0.005$, $x_{\max} = 0.350$ and the total layer thickness $t = 2330$ nm) where spontaneously generated large lattice tilts [5] can minimize the energy of the system [6]. The thick graded InGaAs heterostructure gives a very broad diffraction tail marked by the points 1, 2 and 3 in Fig. 6a). Synchrotron radiation X- ray double crystal topographs (Fig. 6b), taken at three points of the reflection curve with the exposure time about 30 secs at the beamline ID19 in ESRF Grenoble [7], show clearly that the presence of tilted regions in the epitaxial layer is an additional contribution to broadening of local rocking curves caused by the composition gradient. Large domains (see arrows in Fig. 6b) in the epilayer with different orientation diffract at different incidence angles. The image is formed by different blocks at the points 1, 2, and 3 of the rocking curve. The blurr of misfit dislocations images is caused by broad peak of the epitaxial layer due to compositional gradient and tilts.

4 Conclusion

From the results presented it was demonstrated that the X-ray topography gives valuable information on two-dimensional distribution of crystal defects, strains, and compositional inhomogeneities in a variety of semiconductor materials starting from the substrates and covering small to high mismatch heterostructures.

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