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Among the X-ray techniques belonging to the family of Kossel methods the divergent-beam diffraction method gives the best contrast for semiconductor specimens.

The technique has been accomplished in the scanning electron microscope (SEM) in a back reflection configuration. Epitaxial layers of GaAsSb and GaAsSbP on GaAs [100] oriented substrates were investigated. The diffraction lines from lattice planes giving only high Bragg angles were used. For the purpose of the layer strain analysis, the diffraction experiments were carried out for specimens placed horizontally (the lines \{711\} type recorded) and for tilted 45° (the lines \{551\} type recorded). A Cu foil was used as a target material.

KEY WORDS: X-ray diffraction, Divergent-beam diffraction, Crystallography, Scanning electron microscope, Epitaxial layers, Gallium arsenide, Lattice strain, Lattice mismatch, Cu target.

Abstract

The Kossel technique of X-ray microdiffraction is useful method when applied to the scanning electron microscope (SEM) (Dingley, 1975, 1978a), especially to solve metallurgical problems (Dingley, 1978b). Unfortunately it gives very weak contrast in the case of semiconductor specimens (Dingley and Razavizadeh, 1981) because of the high crystal perfection of semiconductors. Further reasons for the weak contrast are the small specimen-film distance in the SEM and blackening of the film by X-rays generated in the specimen which are emitted from it without the contribution of the diffraction phenomena. The first disadvantage does not exist in the special Kossel camera with long specimen-film distance. In such equipment satisfactory results were obtained for semiconductor epitaxial layers (Geist et al., 1982). The second disadvantage can be removed by shielding the film from the X-ray source (the source must lie outside the specimen in this case). This technique is known as a divergent-beam X-ray diffraction and is usually realized in a special apparatus with a capillary X-ray tube (Rigaku Denki "Microflex" unit). The method is restricted to the study of single crystals with a surface area not smaller than several square millimeters. Divergent-beam X-ray diffraction was also accomplished in an X-ray microanalyser with the X-ray source lying in the film plane (Bert et al., 1980) but the large source-specimen distance in this case implies a very large diffraction area. The method of divergent-beam X-ray diffraction is especially useful for the detection of differences between lattice spacings of an epitaxial layer and an underlying substrate (Bert et al., 1980) and for the strain analysis of epitaxial layers (Schutz et al., 1981).

The author of the presented paper has installed a capillary X-ray source in the SEM. The source is placed in the...
vacuum and in this case there are no requirements for its vacuum tightness and a high mechanical rigidity. Consequently the capillary source can be made very small, which facilitates placing it very close to the specimen and obtaining the small diffraction area.

Method

The X-ray source (Fig. 1) was made by an electrolytic deposition of copper on a glass cone with rounded tip. Before the copper deposition the cone was covered with thin layers of carbon and copper by vacuum evaporation. The carbon layer was to facilitate removal of the copper cone from the glass former. The process of the electrolytic deposition was interrupted when the thickness of about 10 µm was obtained, then the tip was protected with a varnish and the electrolytic deposition was continued to make the source wall thicker. The transition zone between the tip and the cone wall was made in the form of steps in order to minimize the specimen area intercepted by the source. The diameter of the tip was about 0.8 mm. To protect the photographic film from direct exposure to X-rays and backscattered electrons created inside the capillary, the cone was closed by an aperture 200 µm in diameter.

The electron beam of the microscope, after passing through this aperture, generates X-rays in the copper foil at the bottom of the capillary. The X-rays are transmitted through the target and they interact with the lattice planes of the investigated sample. The resulting diffraction pattern is registered on the photographic film placed above the specimen. The film has a central hole for the electron beam. This film position enables pseudo-Kossel lines from reflections with high Bragg angles to be registered, and in this way a high precision of the lattice parameter difference measurements is achieved.

A simple multilayer camera has been used for transport of the films. It enabled eight films in aluminium cassettes to be loaded. The dimensions of the film were 50 mm x 50 mm. The specimen to film distance was about 40 mm. Kodak Industrex C, AX, MX and ORWO TF 10 films were used. For a beam current of 5 µA and a beam voltage of 25 kV the exposure time was 5 min. for Industrex C and TF 10, 10 min. for Industrex AX and 20 min. for Industrex MX.

Application to semiconductor epitaxial layers

Epitaxial layers of GaAsSb and GaAsSbP on a Cr doped GaAs substrate were investigated. The substrate was [100] oriented and the layers were grown by the LPE process. In earlier papers reported, dealing with the divergent-beam method, the specimen was placed perpendicularly to the direction of the electron beam. In this case, four diffraction lines from reflections with high Bragg angles, i.e. from (711), (711) and (711) planes are recorded when a Cu target is used. For the Cu-Kα radiation the Bragg angle is 76°41'. The planes are inclined 11°25' with respect to the (100) plane. For the purpose of the layer strain analysis one must also record diffraction lines from other planes, inclined differently to the surface. For this purpose the specimen was tilted 45° and during the rotation lines from planes of the (551) type, i.e. (551), (551), (515), (515), (551), (515), and (515) were photographed in sequence. The Bragg angle for this group of planes is identical to that of the (711) type planes, i.e. 76°41'. The planes are inclined 45°34' with respect to the (100) plane.

A set of diffraction patterns from the GaAsSb/GaAs sample is shown in Fig. 2. Fig. 3a shows an enlarged part of Fig. 2e, and Fig. 3b shows schematically the diffraction lines and the distances to be measured on the film. The measurements should be made along the major axes of the pseudoellipses (Schutz et al., 1981) or in any place if the diffraction lines are approximately circles.

The technique is similar to that of Schutz et al. (1981) and their analysis can be used for determining the strain matrix and rotation matrix (and consequently the strain tensor). For this purpose, one should measure the ratio \( \eta = \frac{A_{pd}}{A_{pg}} \) for as many lines as possible and perform rather complicated calculations of matrix components. \( A_{pd} \) is the

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**Fig. 1** The capillary X-ray source. 1-target foil, 2-source wall, 3-aperture, 4-wire for mounting the source.
Divergent-beam X-ray diffraction in the SEM

distance between the lines from the layer and the substrate produced by the same wavelength, and $\Delta \rho$, is the distance between the lines from the layer or the substrate produced by the $K_\alpha$ doublet.

However, if the heteroepitaxial layer has been grown on a high symmetry plane of the substrate, one can expect that no tilt of the layer exists, and the strain is isotropic within the plane parallel to the surface. In such a case, the $R$ values are identical for reflections from symmetrically equivalent (hkl) planes. In the case of the [100] orientation the tetragonal distortion of the layer exists (Ishida et al., 1975). For the investigated sample, the differences of the $R$ values of the four (551) type planes ((551), (515), (551) and (515), Fig. 2a, c, g and i) were smaller than measurement error. The mean value was $R(551) = 1.394 \pm 0.015$. For the (711) type planes it was $R(711) = 1.670 \pm 0.013$. From the above results, the distortion of the layer lattice was assumed to be tetragonal. Having obtained the values of ratios $R(551)$ and $R(711)$, one can calculate the differences between lattice

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Fig. 2 Diffraction patterns from the GaAs$_{33}$/GaAs sample. Specimen-target distance 1.5 mm. (a), (b), (c), (d), (f), (g), (h) and (i) - specimen tilted 45°. (e) - specimen placed horizontally.
Fig. 3 (a) An enlarged part of the diffraction pattern from Fig. 2e with (711) lines shown. (b) Schematic representation of the diffraction lines. 1- doublet from the substrate, 2- doublet from the layer, \( \Delta p_{x} \) - distance on the film between the lines of \( K_{a} \) doublet from the substrate or from the layer, \( p_{d} \) - distance between the lines from the substrate and the layer produced by the same wavelength.

Spacings in the layer and the substrate, \( d \) (Bert et al., 1980). For the investigated sample they are:

\[
\frac{d_{551}}{d_{711}} = \frac{(37.82-0.76) \times 10^{-4}}{(43.24-0.76) \times 10^{-4}}
\]

where \( d \) is the lattice spacing in the substrate. Next, one can calculate the quantities of the layer strain in directions perpendicular and parallel to the surface (Ishida et al., 1975) and the relaxed lattice parameter of the layer in an unstrained state (Hornstra and Bartels, 1978). They are:

\[
\epsilon_{L} = 5.6 \times 10^{-4} \quad \epsilon_{P} = -6.2 \times 10^{-4}
\]

\[
\frac{\Delta a}{a_{\text{relax.}}} = 38.1 \times 10^{-4}
\]

where \( \epsilon_{L} \) is the strain in the direction perpendicular to the specimen surface, \( \epsilon_{P} \) the strain in the directions parallel to the surface, \( a \) is the lattice parameter of the substrate and \( a_{\text{relax.}} \) the difference between the lattice parameters of the layer and the substrate.

Fig. 4 shows the diffraction patterns from the GaAsSbP/GaAs sample for different distances between the target and the specimen. In the case of a small distance, equal to 0.5 mm, a part of the diffraction pattern is shadowed by the source, but one can still carry out the needed measurements. The diameters of the areas from which the full diffraction patterns originate (four reflections for the specimen placed horizontally and two reflections for the specimen tilted 45°) correspond to 1.3 mm and 0.4 mm for 1.5 mm and 0.5 mm distances respectively in the case of the horizontal specimens, and to 1.1 mm and 0.35 mm respectively for the specimens tilted 45°. For one reflection only, these diameters are substantially smaller.

Fig. 5 shows the geometry of the diffraction for the (711) and (551) lattice planes, 1- electron beam, 2- the end of the source, 3- sample, 4- X-rays.

Conclusions

The preliminary results presented in this paper demonstrate the usefulness of the X-ray divergent-beam method in
the SEM. Its accuracy of the lattice spacings determination can be estimated to be $1 \times 10^{-4}$, or 0.00005 nm. Divergent-beam diffraction is a single crystal method and is less precise than multiple crystal methods (Hart, 1981). However this technique is very convenient for the assessment of the strain distribution, whereas multiple-crystal methods are best suited for an exact determination of strain amplitudes.

The diameter of the source tip was 0.8 mm, but it can be made smaller. It seems to be possible to obtain half of this value. Such a small source will allow diffraction studies from areas 200 µm in diameter or even smaller. Moreover, it is easy to measure a lattice mismatch in different places on the specimen surface by simply moving the specimen across the source, which is rather complicated matter in the case of diffractometer methods.

A Cu target was used, but it is possible to make targets from other materials. This can be accomplished by vacuum evaporation of a thick layer of the target material (several microns) and, after protection of the tip with a varnish, electrolytic deposition of copper on the wall of the source. Very useful should be a Ti target for GaAs and related compounds. It would enable the (400) line to be recorded, thus obtaining directly the strain in the direction perpendicular to surface. However, it can be applied only for rather thin layers because of the low penetration depth of the Ti-Kα radiation compared to Cu-Kα radiation.

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References


Discussion with Reviewers

Reviewer IV: What are the corresponding measurements for the GaAsSbP/GaAs sample? Author: They are:

$R(711) = 0.924 \pm 0.015 \quad R(551) = 0.637 \pm 0.026$

$\left(\frac{\Delta d}{d}\right)_{711} = (23.43 \pm 0.62) \cdot 10^{-4}$

$\left(\frac{\Delta d}{d}\right)_{551} = (17.88 \pm 0.85) \cdot 10^{-4}$

$\varepsilon_{\perp} = 5.6 \times 10^{-4} \quad \varepsilon_{\parallel} = -6.2 \times 10^{-4}$

$\left(\frac{\Delta a}{a}\right)_{\text{relax.}} = 18.3 \times 10^{-4}$

Reviewer IV: Explain how the diffraction area changes with the reflection recorded. Author: The diffraction area depends on the angle of the Kossel cone: the tilt angle of the specimen and the angle between an axis of the Kossel cone and an electron beam. It grows with the increase of these angles. The area is smaller when only one reflection is considered (Fig. 2a, c, g, i) and larger if the diffraction pattern contains several reflections (Fig. 2e). However, one can see (Fig. 5a) that in the case of the (711) planes, the outer vertices of pseudoeiipses (where the measurements are made) originate in the proximity of the source axis. Thus, the diffraction area determining the measurements is very small.
Reviewer IV: Would you explain how you deduce there is a tetragonal distortion of the GaAsSb/GaAs sample, and how big is it?

Author: Tetragonal distortion can be assumed if the R values are identical for several symmetrically equivalent planes \( \{hkl\} \) (Schutz et al., 1981). It is better to use planes with large angles between them (as with \( \{551\} \) planes). The quantity of the distortion can be derived from the R values of two planes differently inclined to the sample surface (Ishida et al., 1975, Bert et al., 1980).

Reviewer IV: D.J. Dingley