Divergent-Beam X-Ray Diffraction in the Scanning Electron Microscopy and its Use for the Study of the Semiconductor Epitaxial Layers

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Abstract

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.

Introduction

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 re-
quirements 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 trans-
ition 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 expo-
sure to X-rays and backscattered elec-
trons 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 spec-
imen. The film has a central hole for
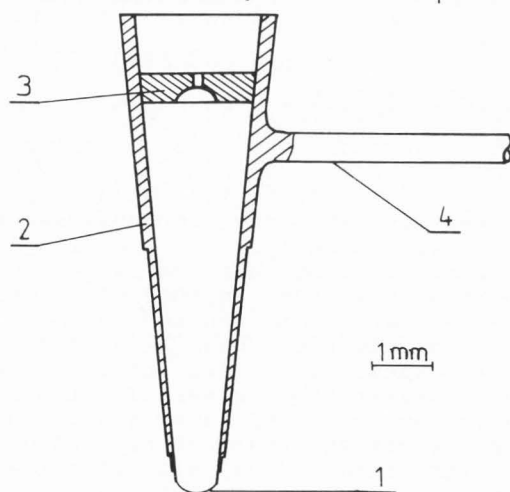the electron beam. This film position

Fig. 1 The capillary X-ray source. 1-
target foil, 2- source wall, 3- aperture,
4- wire for mounting the source.

enables pseudo-Kossel lines from reflect-
ions with high Bragg angles to be regist-
ered, and in this way a high precision
of the lattice parameter difference
measurements is achieved.

A simple multiplate 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 Indust-
rex 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 diff-
raction lines from reflections with high
Bragg angles, i.e. from (711), (7$\bar{1}$1),
(71$\bar{1}$) and (7$\bar{1}\bar{1}$) planes are recorded when
a Cu target is used. For the Cu-Kα_1 rad-
iation the Bragg angle is 76°41'. The
planes are inclined 11°25' with respect
to the (100) plane. For the purpose of
the layer strain analysis one must also
record diffraction lines from other
planes, inclined differently to the
surface. For this purpose the specimen
was tilted 45° and during the rotation
lines from planes of the {551} type, i.e.
(551), (55$\bar{1}$), (51$\bar{5}$), (5$\bar{1}$5), (5$\bar{5}\bar{1}$), (5$\bar{5}$1),
(5$\bar{1}$5), 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 diff-
raction 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 conseq-
uentely the strain tensor). For this
purpose, one should measure the ratio
$R=\Delta p_d/\Delta p_\lambda$ for as many lines as possible
and perform rather complicated calculat-
ions of matrix components. Δp_d is the

distance between the lines from the layer and the substrate produced by the same wavelength, and Δp_λ is the distance between the lines from the layer or the substrate produced by the K_α 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] orient-

ation 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 ((55$\bar{1}$), (5$\bar{1}$5), (5$\bar{5}$1) and (515), Fig. 2a, c, g and i) were smaller than measurement error. The mean value was R(551)= 1.394±0.015. For the {711} type planes it was R(711) = 1.670±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

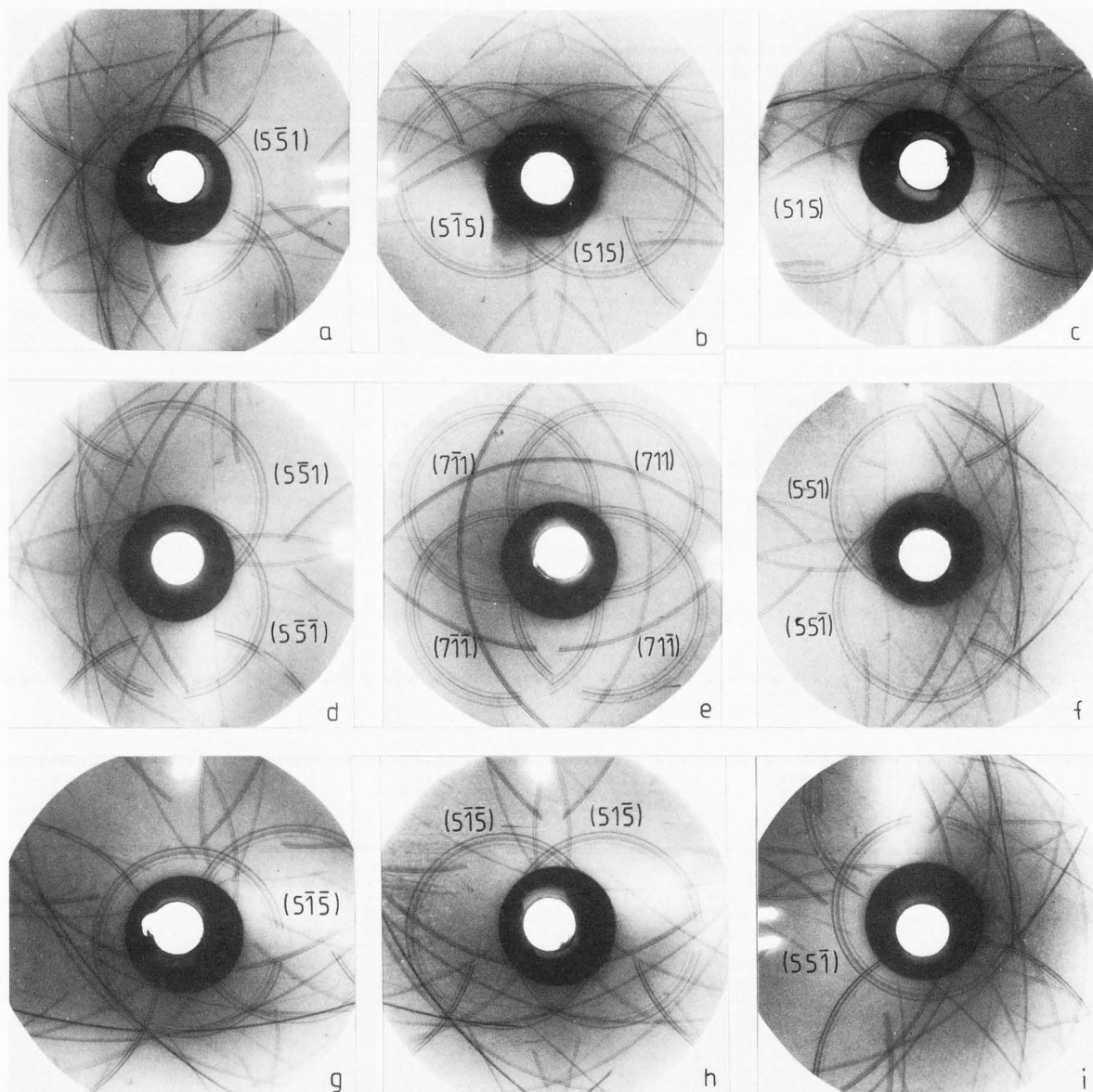Fig. 2 Diffraction patterns from the GaAsSb/GaAs sample. Specimen-target distance 1.5 mm.(a), (b), (c), (d), (f), (g), (h) and (i) - specimen tilted 45°. (e)- specimen placed horizontally.
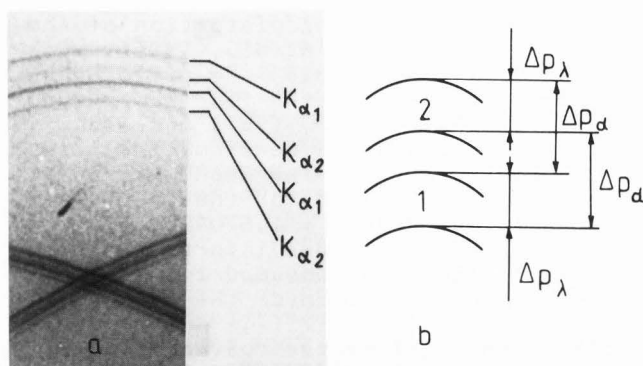
Fig. 3 (a) An enlarged part of the diff-raction pattern from Fig. 2e with $(7\bar{1}\bar{1})$ lines shown. (b) Schematic representation of the diffraction lines. 1- doublet from the substrate, 2- doublet from the layer, Δp_λ- distance on the film between the lines of K_α doublet from the substrate or from the layer, Δp_d- distance between the lines from the substrate and the layer produced by the same wavelength.

spacings in the layer and the substrate, Δd (Bert et al., 1980). For the invest-igated sample they are:

$$\left(\frac{\Delta d}{d}\right)_{551} = (37.82^{\pm}0.78)\cdot 10^{-4} \quad (1)$$

$$\left(\frac{\Delta d}{d}\right)_{711} = (43.24^{\pm}0.78)\cdot 10^{-4}$$

where d is the lattice spacing in the substrate. Next, one can calculate the quantities of the layer strain in direct-ions 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:

$$\varepsilon_\perp = 5.6 \times 10^{-4} \qquad \varepsilon_{||} = -6.2 \times 10^{-4}$$

$$\left(\frac{\Delta a}{a}\right)_{relax.} = 38.1 \times 10^{-4} \quad (2)$$

where $\varepsilon_\perp$ is the strain in the direction perpendicular to the specimen surface, $\varepsilon_{||}$ the strain in the directions parallel to the surface, a is the lattice para-meter of the substrate and Δa the differ-ence 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 1.3mm and 0.4 mm for 1.5 mm and 0.5 mm distan-ces respectively in the case of the ho-
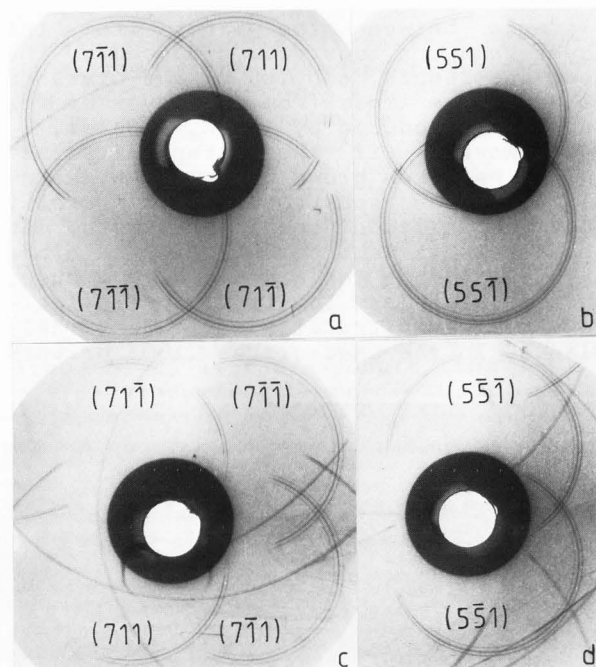

Fig. 4 Diffraction patterns from the GaAsSbP/GaAs sample for different target-specimen distances. (a) and (b) - 1.5mm, (c) and (d) - 0.5 mm.
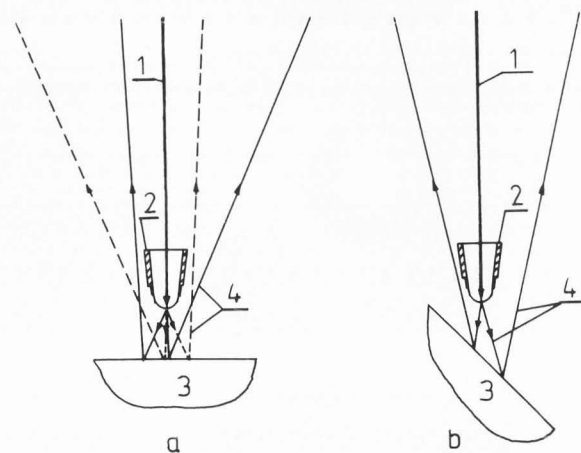

Fig. 5 Geometry of diffraction for (a)- {711} and (b) - {551} lattice planes. 1- electron beam, 2- the end of the source, 3- sample, 4- X-rays.

rizontal 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.

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 1×10^{-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-K_α radiation compared to Cu-K_α radiation.

Acknowledgement

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Discussion with Reviewers

Reviewer IV: What are the corresponding measurements for the GaAsSbP/GaAs sample?
Author: They are:
$R(711) = 0.924 \pm 0.015$ $R(551) = 0.637 \pm 0.026$

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

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

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

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

Reviewer IV: Explain how the diffracting area changes with the reflection recorded.
Author: The diffracting 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 pseudoellipses (where the measurements are made) originate in the proximity of the source axis. Thus, the diffracting 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