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ABSTRACT

Convergent beam electron diffraction (CBED) using either the (200) systematic row and/or the (011) zone axis was successfully applied to distinguish between As and Ga planes in (011) GaAs samples in order to identify the respective planes. The information existing in the 200 disc is different, depending on whether a particular disc refers to the As or the Ga plane. Therefore, this method can be generally applied in transmission electron microscopy for in-situ samples and without any chemical etching.
The increasing interest in III-V and II-VI semiconductor compounds has brought attention to the polarity of these crystals especially with the current interest in growing polar crystals on non-polar substrates. Differences in crystal growth conditions of homo- or heterostructure layers have been observed when the GaAs substrate crystal is facing different polar sides; (111) Ga or As and (100) Ga or As. This clear difference was observed for Au deposited on the As and Ga sides, respectively, and annealed at 350°C. Triangular patches aligned along <110> in GaAs are formed on the gallium face, whereas circular patches are formed on the arsenic face. Other important properties connected with the polarity of the crystal are α- and β-type dislocations in sphalerite crystals, depending on whether their extra half-plane terminates on the Ga or As face. These dislocations show very different mobility. Therefore, it is very important to determine the Ga and As planes in the crystal. The only method used up to now to distinguish between these two different crystal planes is a selective chemical etch using HF:H2O2:H2O (1:1:2). Specific etching figures make it possible to differentiate between As and Ga planes for polar crystal faces. However, the chemical etching cannot be applied for nonpolar (011) crystal faces.

In this paper, we describe a particular application of the convergent beam electron diffraction (CBED) method to identify the As and Ga planes in (011) GaAs. It has already been shown that the specific features in convergent diffraction discs are rich in information about crystal structure, point groups, structure factors, and polarity of the crystal.
Two sets of GaAs (111) surfaces were prepared: with the As and Ga faces up, respectively. These two faces were verified by etching methods (Fig. 1a and 1b). Cross-sections were prepared from these samples so that the electron beam was parallel to <011> and either Ga or As planes were glued together (Fig. 2a and 2b).

Two methods were explored; one described by Tafto\textsuperscript{7} using the (200) systematic row in the CBED pattern, and one utilizing the full CBED pattern of the (011) pole.\textsuperscript{5} In the first method, the systematic (200) reflection was excited with two other reflections on the Ewald sphere that fulfill the Bragg condition (Fig. 3a). For sample I in Fig. 2a [As (111) planes glued together], the constructive interaction of the (755) and (955) beams with the (200) beam resulted in the appearance of a white cross in the (200) CBED disc correlated with the As plane (Fig. 3b). The interaction of the (755) and (955) beams with the (200) beam was destructive and resulted in a black cross observed in the (200) disc. This black cross was correlated with the Ga planes (Fig. 4a). These observations were confirmed on sample II of the same cross section (from Fig. 2a). Again, the white cross was correlated with the As plane, as shown schematically in Fig. 2a. The identical experimental configuration was applied for two cross-section samples with two Ga planes glued together (Fig. 2b). Now, the same (755) and (955) beams interacted destructively with the (200) beam and produced a black cross correlated with the Ga planes (Fig. 4c), and the (755) and (955) beams interacted constructively with the (200) beam resulting in a white cross correlated with the As planes (Fig. 4d). If we consider sample I in Figs. 2a and 2b, we can explain the change of the cross contrast in the
(200) disc only if the positions of the As and Ga atoms in sample I of Fig. 2b are exchanged. Following Tafto's notation\(^7\) and using the same notations in all our diffractograms, for sample I in Fig. 2a the Ga atoms would be placed at \(\frac{1}{8} \frac{1}{8} \frac{1}{8}\) and the As atoms at \(\frac{1}{8} \frac{1}{8} \frac{1}{8}\). The positions would be reversed in sample I in Fig. 2b. The same remarks held for sample II in Fig. 2a (it has the same atomic positions as sample I in Fig. 2b). Therefore, the structure factor (for sample I in Fig. 2a) for 

\[ F_{200} = 8\Delta \exp\left(\frac{\pi}{2} i\right), \]

and 

\[ F_{\overline{2}00} = 8\Delta \exp\left(-\frac{\pi}{2} i\right), \]

assuming that \(f_{As} = \bar{f} + \Delta\) and \(f_{Ga} = \bar{f} - \Delta\). Using the polynomial expression given by Cowley and Moodie\(^8\) for the phase change in the thin crystal approximation,

\[ \omega = \frac{n\pi}{2} + \sum_{i=1}^{n} \omega_i, \]

The phase change by reflections close to the Bragg position for sample I in Fig. 2a are: \(000 \rightarrow 200 = -\frac{\pi}{2} - \frac{\pi}{2} + \omega_{200} = 0\) and \(000 \rightarrow \overline{2}00 = -\frac{\pi}{2} + \omega_{\overline{2}00} = -\pi\). The phase change for sample I in Fig. 2b is reversed because the atom positions in this sample are reversed. The same is true for sample II in Figs. 2a and 2b.

Our samples consisted of either (\(\overline{1}1\overline{1}\)) As or Ga planes glued together. Therefore, the diffraction spot reflected from the particular (\(\overline{1}1\overline{1}\)) plane is rotated 55° clockwise from the (200) spot and is placed at the characteristic distance from the central beam. Knowing the position of the white (black) cross in the (200) CBED disc and the position of the (\(\overline{1}1\overline{1}\)) spot rotated 55° from the \(\overline{2}00\) reflection (according to our notation), the particular reference As or Ga (\(\overline{1}1\overline{1}\)) plane used in our experiment was immediately determined. In practice, it is adequate to
take two CBED patterns from the (200) and the (200) systematic rows to
determine the position of the (111) As (Ga) plane. Once the position of
these two planes is known, the other planes can be determined from the
crystal symmetry.

Similar information about the Ga and As planes can be obtained from
only one <011> zone axis CBED pattern. The only mirror plane is present
in this pattern, and the (200) and (200) discs contain different informa-
tion. The same samples described above were used to observe the [011]
zone axis CBED pattern. For sample I in Fig. 2a, three arrows were
observed in the (200) disc correlated with the Ga planes, and one arrow
was observed in the (200) disc correlated with the As planes (Fig. 5a).
The positions of these arrows in the (200) and (200) discs were reversed
for sample II from Fig. 2a, and they were the same as for sample I from
Fig. 2b (see Fig. 5b). The information in the (200) discs is very sen-
sitive to the sample thickness. For very thin or very thick sample
areas the arrow pattern is blurred and the CBED pattern possesses 2mm
symmetry, but the three and one arrow pattern is never reversed. One
must choose the proper thickness to observe the difference in the (200)
and (200) discs. It is not difficult to choose the proper thickness in
a wedge-shaped sample because one can observe the symmetry of the (200)
and (200) discs directly on the microscope screen. It is preferred that
the CBED pattern is taken simultaneously with the lattice image, but the
latter is not necessary to obtain all information. One needs a reliable
rotation angle for a particular image magnification and specific camera
length to determine the plane in the sample connected with the respec-
tive diffraction pattern.
The valuable information obtained from the CBED pattern can be directly applied to the study of defects and dislocation types in bulk and epitaxial compound semiconductors.

This novel use of the observed CBED pattern has already helped to determine and eliminate faceting mechanisms of GaAs grown on (110) GaAs by molecular beam epitaxy. It is expected that the white cross in the 200 systematic row disc and the three arrow arrangement in the 200 disc of the full low index zone axis CBED pattern can be correlated with the element plane of the higher atomic number. Further work is in progress to combine the CBED pattern information about the As and Ga planes in the GaAs unit cell with a computer simulation of the lattice image.

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References


FIGURE CAPTIONS

Fig. 1. Scanning electron micrograph of (111) GaAs after preferential chemical etching; a) As plane, b) Ga plane.

Fig. 2. Schematic of GaAs cross-sectional samples with the respective diffraction patterns in the (011) orientation. The pattern arrangement in the (200) disc of the systematic (200) CBED pattern and in the (011) zone axis CBED pattern, depends on the specific positions of the As and Ga planes in the sample. a) GaAs with (111) As planes glued together. In sample I, in the (200) systematic disc, the white cross appears correlated with the As plane. In the (200) disc, the black cross appears correlated with the Ga plane. The same is true for sample II: the white cross is always correlated with the As plane. The (011) zone axis CBED pattern shows three arrows in the (200) disc which correlate with the As plane and one arrow in the (200) disc which correlates with the Ga plane.

b) GaAs with (111) Ga planes glued together. It is a confirmation of the results obtained with the previous sample (see Figs. 2a, 4, and 5). Different information exists in the 200 discs due to As planes compared to the Ga planes.

Fig. 3a. Schematic drawing of the reflections on the Ewald sphere fulfilling the Bragg condition and the scattering paths from the central beam to the 200 reflection.

Fig. 3b. GaAs diffraction pattern from sample I in Fig. 2a showing the four beams on the Ewald sphere during electron beam exposure.
The correct sample image orientation with respect to the diffraction pattern is shown in the right lower corner.

**Fig. 4.** CBED pattern showing the 200 and $\bar{2}00$ reflections at the Bragg position: (a and b) from sample I, Fig. 2a; (c and d) from sample I, Fig. 2b.

a) black cross in the $(200)$ disc correlates with the Ga plane  
b) white cross in the $(200)$ disc correlates with the As plane.

c) white cross in the $(200)$ disc correlates with the As plane  
d) black cross in the $(200)$ disc correlates with the Ga plane

**Fig. 5.** $(011)$ CBED zone axis patterns:

a) From sample I in Fig. 2a; three arrows in the $(200)$ disc correlates with the As plane and one arrow in the $(\bar{2}00)$ disc correlates with the Ga plane.

b) From sample I in Fig. 2b; one arrow in the $(200)$ disc correlates with the Ga planes and three arrows in the $(\bar{2}00)$ disc correlates with the As planes.
Fig. 1

Fig. 2

Fig. 3
Fig. 4

XBB 867-5383
(lower)

Fig. 5

XBB 867-5485
(lower)
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