Letters

Bi-refringence observations of strain and plastic deformation in GaP

While the search for the elusive "killer" centre in GaP LED material continues, structural defects have lately attracted a great deal of research effort (see for example [1, 2]). Dislocations have, in particular, emerged as a probable candidate for the dominant non-radiative centre. Various luminescence techniques have revealed dislocations as dark spots (e.g. [3–5]), i.e. regions of non-radiative recombination, thus adversely affecting the overall light emission efficiency of the material. Artificial introduction of dislocations by plastic deformation of GaP has been shown [5] to bring about a marked reduction in the luminescence efficiency of nitrogen-doped liquid-phase epitaxial (LPE) material. This importance of dislocations in LED materials has recently aroused considerable interest in a shift from the scanning electron microscope-based techniques to the development of techniques using the conventional optical microscopy for the observation of dislocations and other structural defects. Tajima and Iizuka [6] have, in particular, successfully used the 90° light scattering method for direct observation of dislocations and associated imperfections in liquid-encapsulated-Czochralski (LEC) samples of GaP, Ahearn *et al.* [7] have used bi-refringence under applied stress to study misfit dislocations in vapour-phase epitaxial (VPE) layers of III–V ternaries on mismatching substrates.

We report here observations of slip band patterns in samples of LPE GaP subjected to plastic deformation by the four-point bending technique, using bi-refringent transmission caused by the built-in strain. Observations of as-grown samples of both LPE and VPE GaP were also made and compared with those on the bent samples. This provides interesting information on the strain distribution due to growth, particularly at the epilayer–substrate interface.

Observations reported here were carried out on three types of samples:

(a) bent strips, 20 to 50 μm thick, of LPE grown, nitrogen-doped (1 0 0) GaP on $\sim 300\,\mu$m thick pulled substrates. The epitaxial layers are n-type with electron concentrations $\sim 8 \times 10^{17}\,\text{cm}^{-3}$. Observations were made on cross-sections through thickness obtained by cleavage along (1 1 0) planes;

(b) undeformed strips of the same material;

(c) undeformed strips of GaP grown by vapour-phase epitaxy on pulled substrates.

Four-point bending was carried out either in an apparatus described earlier [5], in which the sample is heated by passing an electric current through it or in a modified version* in which bending is carried out inside a small oven thus ensuring uniform heating of the strip. Bending requires temperature in the range 600 to 700° C and takes about 60 to 90 sec. Samples are examined in a Nikon microscope provided with a rotatable stage. The (1 1 0) cleaved surfaces of the samples are viewed between crossed polarizers so that the light passes through a region of GaP equal to the width of the strips ranging between ~ 1.5 and 3 mm and the stage is rotated to obtain maximum contrast. The sample orientation and geometry are illustrated in Fig. 1.

Fig. 2 shows a low magnification view of a bent GaP strip placed between crossed polarizers. The intensity of transmitted light is found to increase markedly as one moves from the ends of the sample to the middle indicating that most of the strain has been introduced in that region, the end

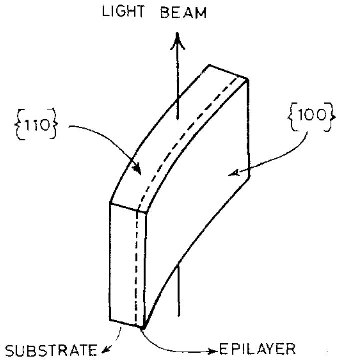

Figure 1 Geometry and orientation of a bent sample.

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*The author is indebted to E. Huang and S. M. Davidson of the University of Manchester Institute of Science and Technology, UK for making available some samples bent in this modified version.

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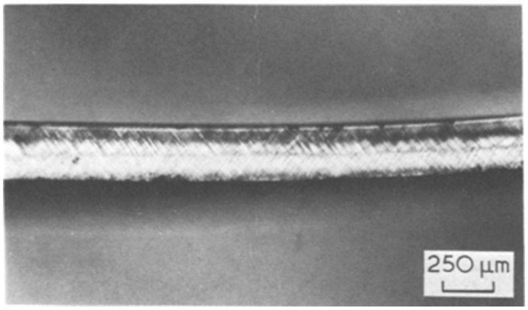

Figure 2 A bent sample viewed in transmission between crossed polarizers.

regions remaining practically unbent. A cross-hatched pattern of dark and bright bands is revealed in the bent region on closer examination, as shown in Fig. 3. The dark band running along the length of the strip is the trace of the neutral plane which shows no bi-refringent transmission because no strain occurs on this plane. The dark and bright bands forming the cross-hatched pattern are identical to the pattern observed in cathodoluminescence [5] and are interpreted to be the traces of (1 1 1) slip planes of GaP. An interesting fact is that this pattern can also be observed when the polarizer is removed (Fig. 4); of course no feature is seen to distinguish the neutral plane, as one would expect. The fact that slip bands should be visible even with unpolarized light is rather surprising; we suspect this might be caused by impurity segregation at the dislocations composing the slip bands, which would bring about a change in the refractive index locally, thereby causing a variation in the transmitted intensity.

Unbent samples are featureless in unpolarized light transmission except for the sharply defined epilayer–substrate interface, the epilayer appearing generally brighter than the substrate. Between the crossed polarizers, however, these samples show interesting features against a back-

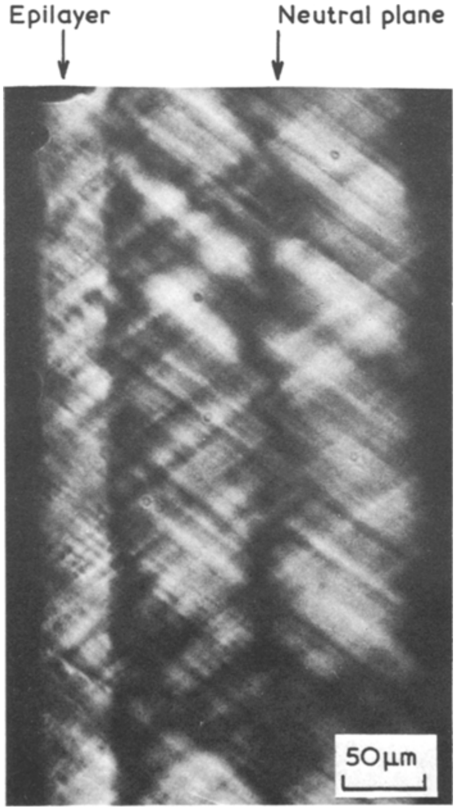

Figure 3 Slip-band pattern seen in bi-refringent transmission through a bent sample.

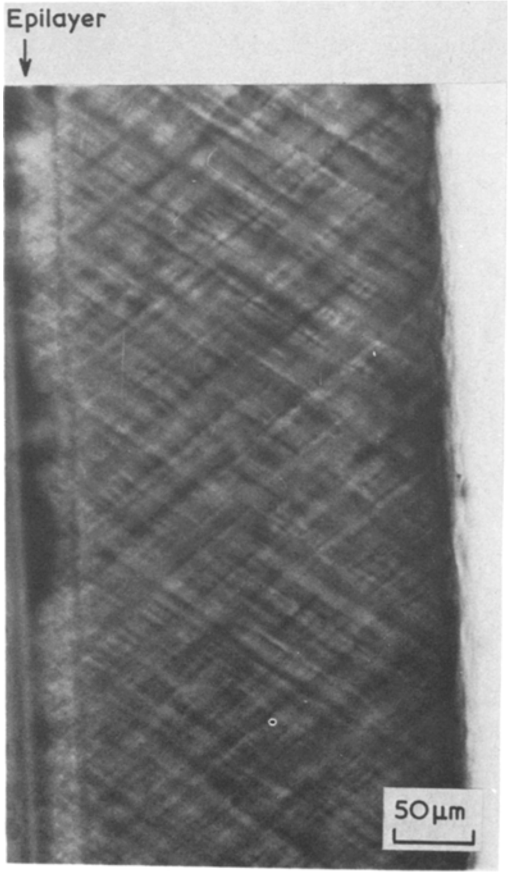

Figure 4 Transmission view of a heavily bent sample with unpolarized light.

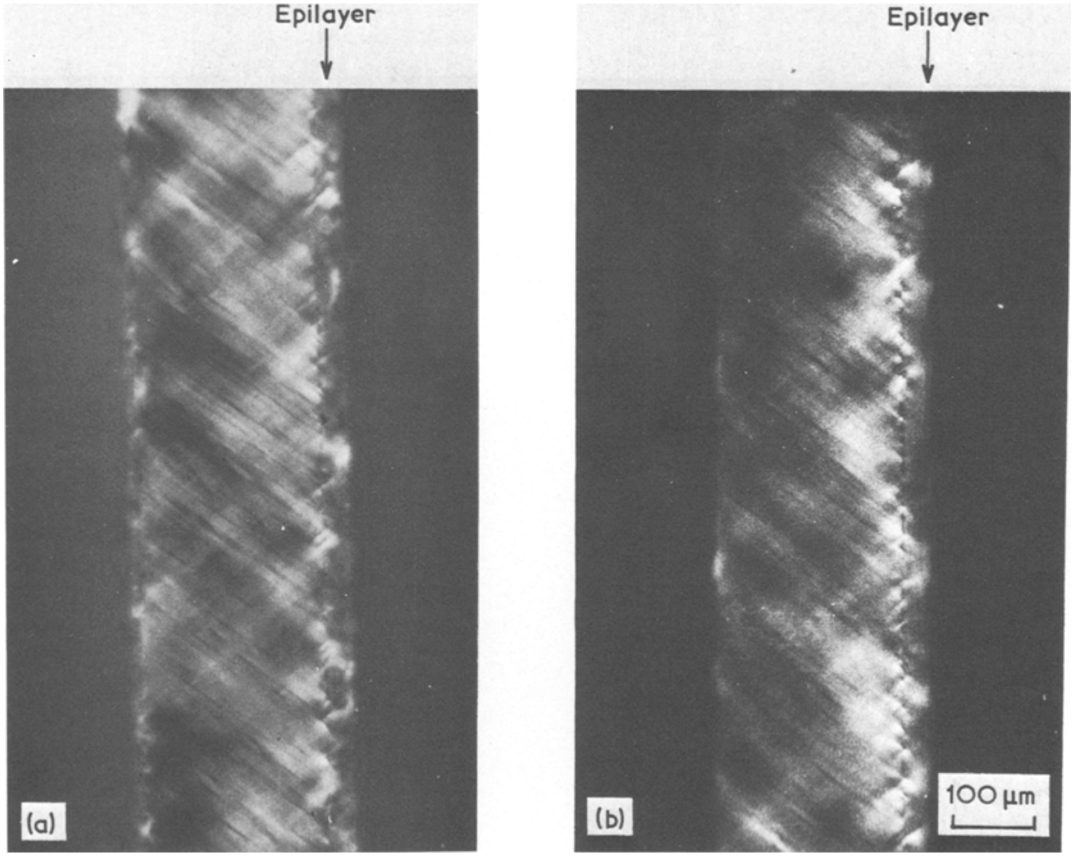

Figure 5 Transmission (a) and reflection (b) views of as-grown LPE GaP between crossed polarizers.

ground transmitted intensity which is far below that transmitted by the bent samples. This is also true for reflection between crossed polarizers. Fig. 5a and b show the most striking examples of the features observed in transmission and reflection, respectively. A criss-cross pattern of bands having a surprising resemblance with those observed in the bent samples ending at sharply defined positions at the epilayer–substrate interface are generally observed in all the unbent samples with varying density. They can be brought into focus somewhat below the cleaved surface so that they can be clearly distinguished from the cleavage steps, etc. In fact, in samples subjected to very light bending one finds only a little difference in the pattern as compared to the unbent sample obtained from the same strip.

The features observed in the unbent LPE strips are suggestive of plastic deformation, similar to that occurring during slip, being incorporated in the as-grown LPE–LEC substrate system largely at the interface. Considering the temperatures used during epitaxial deposition, it seems possible that enough stress develops at the interface to cause this plastic deformation. In order to investigate this point further we examined some as-grown vapour-phase epitaxial samples which are obviously subjected to a different heat treatment during growth. These samples were found to show much weaker transmission between crossed polarizers than the unbent LPE samples. Fig. 6 is a micrograph of an as-grown VPE sample. The dark and bright bands are still in evidence but the contrast is rather poor and their density low in comparison with that found in LPE samples. These observations suggest a lower overall strain in these samples and show that some amount of plastic deformation does exist – this may be frozen in during the substrate growth or may have been caused during vapour-phase epitaxy.

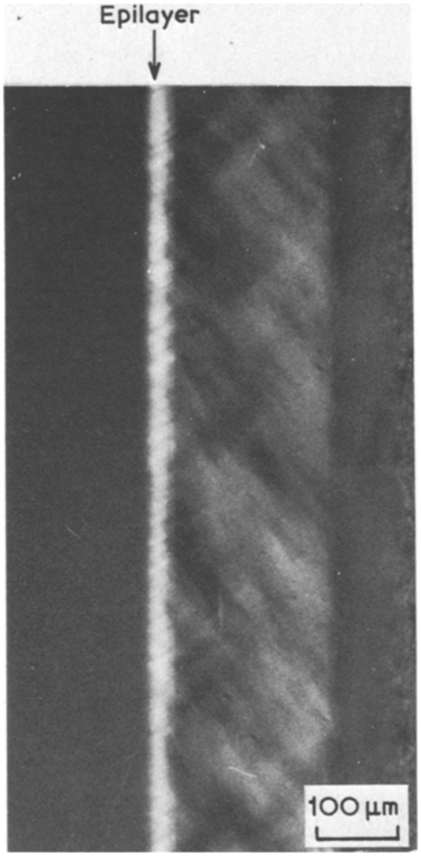

Figure 6 Bi-refringent transmission through an as-grown VPE sample.

Thus we have found that stress-induced bi-refringence provided the same information on the plastic deformation in GaP as revealed by SEM cathodoluminescence showing that the polarizing microscope provides a much more convenient and inexpensive substitute for the SEM for this purpose. Additional information has been obtained on the as-grown material. The sharp features observed at the epilayer–substrate interface and the accompanying strain may account for the sharp drop in the minority carrier lifetime at the interface observed by Blenkinsop *et al.* [8].

Acknowledgement

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Selected-area diffraction ring patterns in Al—Zn—Mg powders

Thin-foil observations of as-produced pre-alloyed aluminium powders have raised the question as to the nature of the microstructure of these powders [1–5]. The interpretation of selected-area electron-diffraction ring patterns ranges from the presence of non-crystalline phases [1, 4] to either an amorphous carbon artifact introduced during the splat-sequence process or an oxide formation introduced during ion-beam thinning [3]. The purpose of this letter is to report on a different type of artefact discovered while attempting to prepare thin foils of an as-produced Al–Zn–Mg* alloy manufactured by four different commercial vendors. Based upon average dendrite arm-spacing measurements [6] (see Fig. 1), the solidification rates varied between 10^4 to 10^5 K sec^{-1} [7]. As mentioned, these cooling rates are five orders of magnitude slower than those calculated for the powders where an amorphous structure was reported [1, 4].

TEM thin foils were initially prepared in a

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*Composition: 6.5 wt% Zn, 2.5 wt% Mg, 1.5 wt% Cu, 0.40 wt% Co, balance Al.