Direct observation of dislocations in GaP crystals

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The bi-refringent method is applied to investigate dislocations in GaP single crystals. The results are compared to those obtained by X-ray topography, electroluminescence, and electron beam induced conductivity. It is shown that the bi-refringent method reveals slip lines, lattice stresses, as well as single dislocations, with a high contrast depending on the orientation of the sample with respect to the polarization plane of the incident light. The method enables the resolution of single dislocations in crystals with high dislocation densities and the distinction between decorated and undecorated dislocations. Furthermore, dislocations in an epitaxial layer can be observed separately from those in the substrate material.

1. Introduction

Dislocations in GaP specimens used for the production of light emitting diodes (LEDs) can cause a decrease in luminescence efficiency [1]. The single crystals required for substrate material already contain grown-in dislocations. Subsequent processing techniques such as epitaxy or diffusion can form further dislocations. Therefore, it is important to have a fast and simple method which allows the observation of dislocations in GaP wafers with regard to their density and distribution.

In general, dislocations in semiconductor materials are revealed by chemical etching followed by the optical examination of the resulting etch pits. This technique is destructive and specimens examined cannot be used further for manufacturing LEDs. On the other hand, X-ray topography is a nondestructive method used to observe lattice defects in single crystals. However, for the examination of GaP with its high absorption coefficient for X-rays the exposure time necessary to make a topograph becomes very long, especially if high resolution is required. Furthermore, the dislocation density of GaP crystals commercially available is usually in the region of 10^4 cm^{-2} or even exceeds this value, resulting in an overlapping of dislocations in the topographic image if samples of the usual thickness are studied. To resolve single dislocations, specimens are needed which are less than about 100 μm in thickness.

Applying methods such as photoluminescence (PL), electroluminescence (EL), cathodoluminescenc (CL), or electron beam induced conductivity (EBIC), dislocations can be observed in the surface region as dark spots, thus indicating locally increased nonradiative recombination of carriers [2–5]. However, the EL and EBIC methods require the formation of p–n junctions.

Another technique published recently applies light scattering at an angle of 90° to reveal dislocations in GaP (ultramicroscopy) [6]. Again this method requires specially prepared samples of about 5 mm in thickness and therefore cannot be used for controlling processes.

For the detection of dislocations in silicon and germanium crystals a method was proposed based on the property of bi-refringence in these crystals when stressed [7]. Because silicon and germanium are transparent to i.r. light an i.r. image converter had to be used. The contrast and resolution of image converters are, however, limited and in general single dislocations were not detected. Since GaP belongs to the same crystallographic class as silicon and germanium it seems a reasonable assumption that it also becomes bi-refringent when

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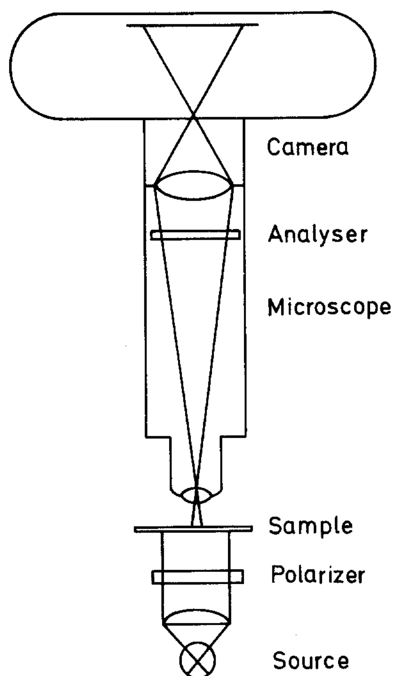


Figure 1 Experimental arrangement of the bi-refringent method.

stressed. Furthermore, because GaP is transparent far into the visible, optical microscopy can be applied with its higher perfection and resolution. This work reports on the bi-refringent method applied to investigations on GaP single crystals. The performance of the method is discussed and it is demonstrated that single dislocations in GaP crystals can be easily detected. The optimum experimental conditions are specified and the results are compared to those of X-ray topography, EBIC and EL.

2. Experimental method

The experimental arrangement of the bi-refringent method is demonstrated in Fig. 1. The wafer, mounted on a revolving table, is illuminated from behind with condensed linearly polarized light. For the examination of the sample an optical arrangement or a microscope containing an analyser is used. Polarizer and analyser are crossed.

The image of the complete wafer or small details such as dislocations can be photographed with normal equipment. Magnifications of up to 300 were carried out with exposure times between 1 and 200 sec.

In order to observe slip lines at low magnifications the backside of the wafer has to be etched free of damage. For an examination at higher magnification it is necessary to polish both sides of the wafer to obtain good resolution.

X-ray topography by the Lang technique [8] was carried out with AgKα radiation at the diffraction planes (2 2 0) and (0 0 2). Since the dislocation density of the GaP crystals investigated usually exceeded 10^4 cm^{-2} the samples were thinned to about 70 μm in order to prevent overlapping of single dislocations in the topographs.

3. Experimental results and discussion

The dislocation pattern observed on half of a GaP substrate wafer (1 0 0)-oriented is shown in Fig. 2. The slip lines crossing one another cause a sudden contrast inversion indicating highly stressed zones. Extended areas of equal brightness are regions with a certain direction of stress which are free of slip lines, but not necessarily free of single dislocations. The stress is mainly caused by the large number of surrounding slip lines.

Rotating the wafer in the plane of the polarizers with respect to the direction of the polarization changes the contrast. In general it was found that (1 0 0)-oriented wafers reverse the contrast with a rotation angle of 90°. Maximum contrast is observed if a ⟨1 0 0⟩ direction in the surface plane of the crystal is parallel to the direction of the electric field of the polarized light (polarization plane). After a turn of 45° (⟨1 1 0⟩-direction parallel to the polarization plane) nearly all slip lines and stress regions are invisible. It is therefore possible to determine the direction of stress by this method. A similar relation was found for (1 1 1)-oriented wafers. In this case maximum contrast appears if one of the ⟨1 0 0⟩-directions (or a ⟨1 1 2⟩-direction in the surface plane) equals the polarization plane. In these wafers, however, the contrast never disappears completely.

Single dislocations can be observed under the same experimental conditions. Due to additional local stresses the contrast of the dislocations can often be increased by rotating the slice by up to 10°. The dislocations appear as dark or bright lines, compared to the background, according to the orientation of the crystal with respect to the polarization plane, the local stress direction and the direction of the dislocation and its Burgers vector.

Grown-in dislocations in a (1 0 0)-oriented GaP substrate wafer are shown in Fig. 3a. The wafer was thinned to about 70 μm for comparison with

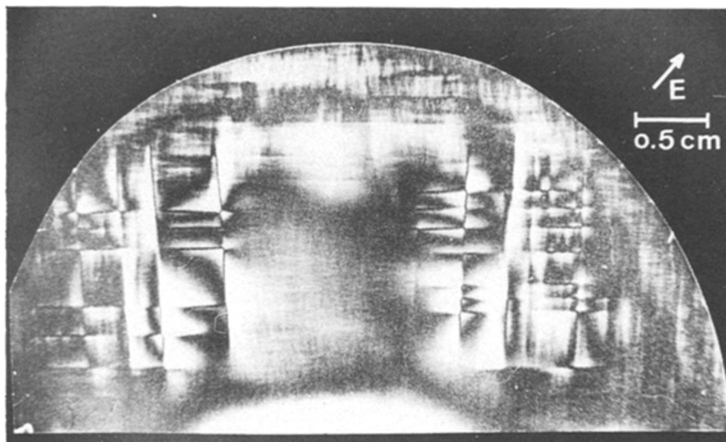


Figure 2 Slip line pattern of half a (1 0 0) GaP wafer. E indicates the direction of electric field of the polarized light.

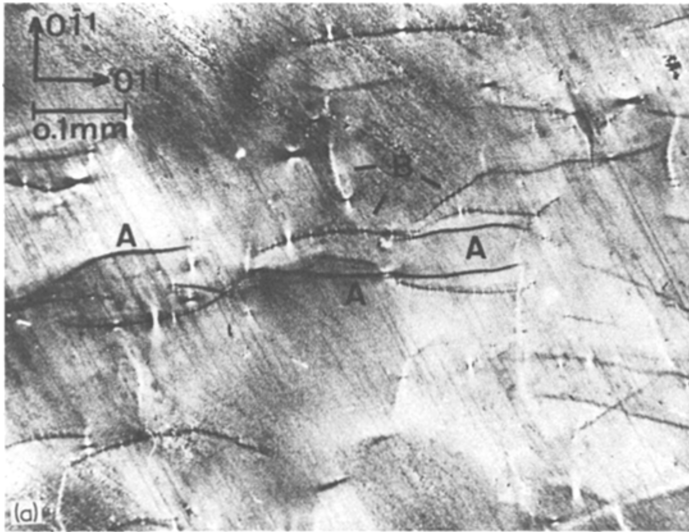
X-ray topography. In general the dislocations are parallel to the surface with orientations in the [0 1 1] and [0 $\bar{1}$ 1] directions causing dark and bright contrast respectively. X-ray topography of the same area is shown in Fig. 3b. All dislocations visible in X-ray topograph also appear when the bi-refringent method is applied. However, it is clear from these images that the resolution of the bi-refringent method is much better. The high magnification also enables the discovery of different dislocation types in this sample. Nearly all dislocations in Fig. 3a resemble chains of points, marked B, and are here called B-dislocations. The remaining 3 dislocations, forming straight lines, are called A-dislocations.

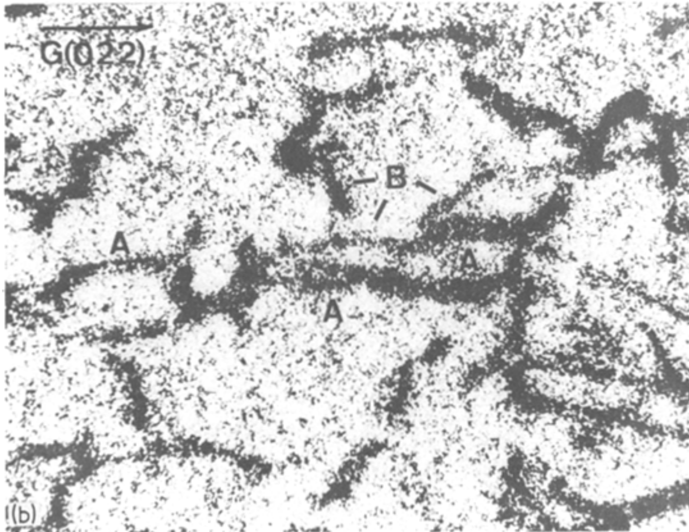
In Figs. 4a and b the two dislocation types are shown in different samples at higher magnification. The B-dislocations have stress components in all crystallographic directions (see below). For this reason they are assumed to be decorated by impurities, while the A-dislocations are not. X-ray topography was applied to verify this assumption. It is known that the contrast of dislocations disappears in X-ray topography if the reciprocal lattice vector of the reflecting plane is orthogonal to the Burgers vector of the dislocation. In this case the reflecting plane is not disturbed by the dislocation. The distortion of a decorated dislocation, however, includes all planes yielding a contrast for all orientations of the reciprocal lattice vector with respect to the Burgers vector of the dislocation.

In Figs. 3b and c X-ray topographs of the same area are shown. They were obtained by diffraction on (0 2 2) and (0 $\bar{2}$ 2) lattice planes respectively. In Fig. 3b all dislocations are observed, whereas the A-dislocations disappear in Fig. 3c. The Burgers vector of the A-dislocations is $\frac{1}{2}$ [0 1 1]. Because of their disappearance they may be only weakly decorated or not decorated at all. This experimental result was verified in further topographs and the contrast of the B-dislocations never disappeared. In general, crystals with dislocation densities exceeding 10^4 cm^{-2} contain mainly A-dislocations; very rarely were both types observed. They also show a large number of slip lines consisting of A-dislocations. In contrast to this, B-dislocations are found either in areas of crystals without slip lines or with low dislocation densities, thereby supporting the assumption that the B-dislocations are much more decorated than the A-dislocations. A verification by means of electron microscopy is in progress.

Because of the high magnification, the depth of view of the microscope is rather small. Therefore, only dislocations positioned in a layer of about 20 μm in thickness are well focused, whereas others outside are not resolved. As a further advantage of this method, overlapping dislocations can be separated and profiles of dislocations with increasing penetration depth can be determined. One example is demonstrated in Figs. 4a and b. The ends of the dislocations disappear because they bend up or down. The trace of these dislocations can be studied by refocusing.

In this way epitaxial layers can be examined separately from the substrate material. In Fig. 5a one part of an LED is shown. This represents a vapour phase epitaxial layer of GaP with a large number of defects. The dislocations in the substrate material are invisible. There is a pronounced difference between the defects observed in epitaxial layers and dislocations in the





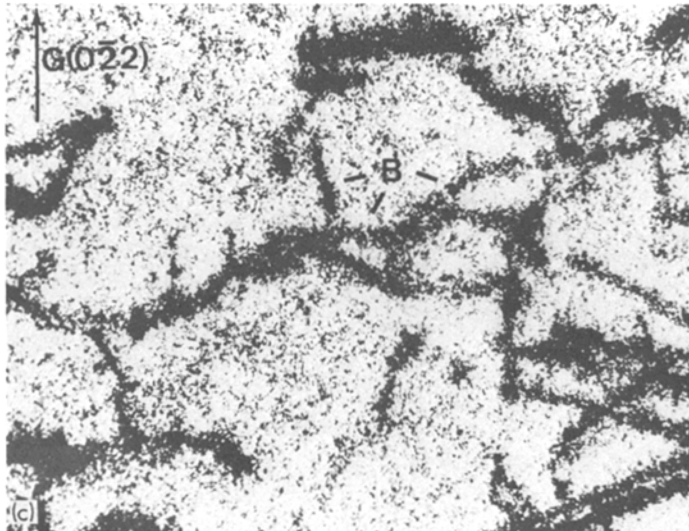
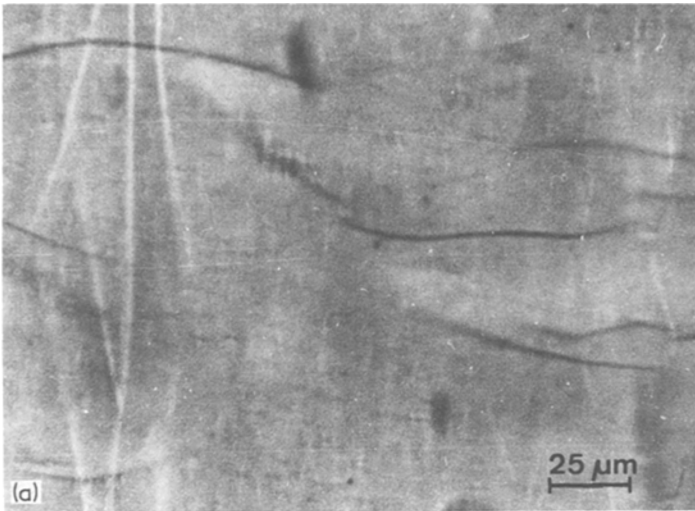


Figure 3(a) Grown-in dislocations in GaP registered by the bi-refringent method. (b) X-ray topography of the same area. Diffraction at (0 2 2). (c) X-ray topography of the same area. Diffraction at (0 $\bar{2}$ 2). Dislocations denoted A in Figs. 3a and b are invisible in c.



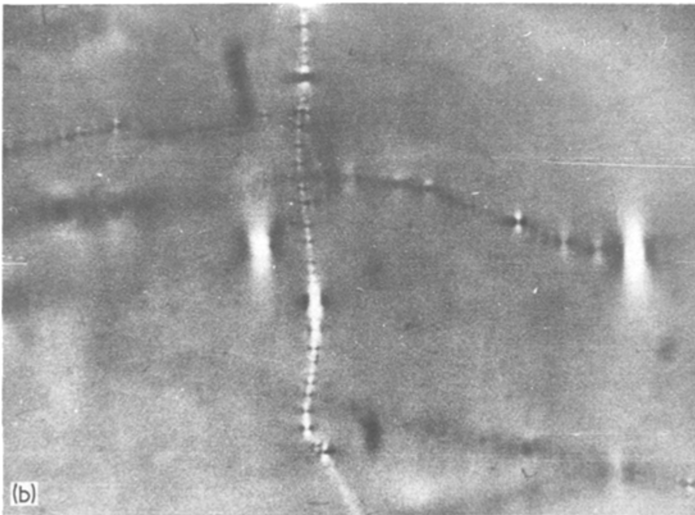


Figure 4(a) A-type dislocations in GaP crystal. (b) B-type dislocations in GaP crystal.

substrate material (compare with Fig. 3a). From detailed examinations it is deduced that generally these defects in the epitaxial layer do not correspond to dislocations crossing the surface of the substrate material. As already mentioned above, dislocations in epitaxial layers may act as centres of enhanced nonradiative recombination, giving rise to dark spots in EL, CL and EBIC examinations. Fig. 5b represents the same area as Fig. 5a showing a photograph in the light of the EL emission. Obviously there is a strong correspondence between the defects in both figures.

A further correspondence between defects and dark spots was found by comparing the bi-refringent method with the EBIC technique. However, it should also be noted that not all defects in the epitaxial layer gave rise to dark spots in EBIC and EL. Those which do not correspond to dark spots in EL or EBIC were identified as lying near to the interface of the epitaxial layer–substrate material without reaching the surface of the LED. Because of the good agreement in the results obtained by applying EBIC, EL and the bi-refringent method, it may be deduced that the defects in the epitaxial layers in the present work are of the same type as the dislocations studied by Titchmarsh *et al.* by means of electron microscopy and published recently [4, 5].

4. Conclusion

It has been shown that the bi-refringent method reveals defects in GaP crystals with high resolution.

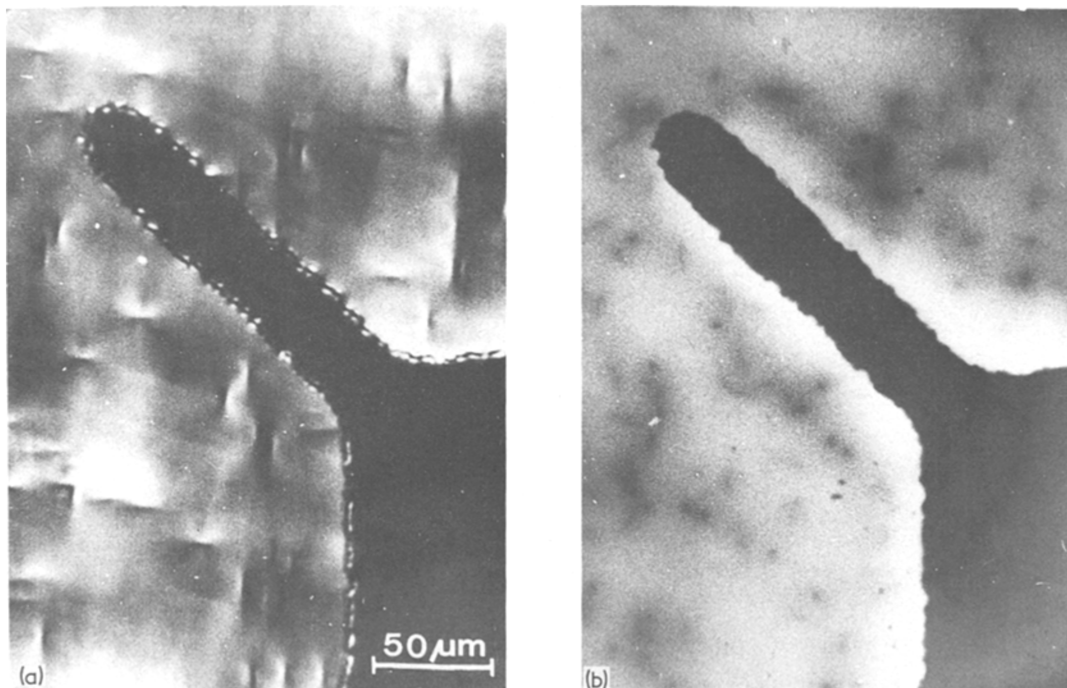


Figure 5(a) Dislocation pattern of a LED registered by the bi-refringent method. (b) Dislocation pattern of the same LED as observed in the light of EL-emission.

The method is applicable in most states of LED device production and it is nondestructive. No special preparation of specimens is required except surface polishing if high resolution is desired. The method is independent of conductivity type and carrier concentration of the GaP crystal. Although the contrast of the dislocations depends on crystal orientation with respect to the polarization plane, sufficient contrast is obtained from the (1 0 0) and (1 1 1)-oriented crystals commonly used in device production. The high resolution of optical microscopy enables specimens with high dislocation densities to be studied by this method. It can be deduced from the appearance of a dislocation whether it is decorated or not. The route of a dislocation through the crystal can be traced by refocusing at high magnification. In this way defects in epitaxial layers can be separated from those in the substrate material. The reported technique is fast and simple and the equipment required is simply a transmission microscope containing polarizers.

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