CATHODOLUMINESCENCE AND X-RAY TOPOGRAPHY OF HPHT DIAMONDS

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ABSTRACT

Early X-ray topographic experiments on synthetic diamonds combined diffraction contrast observations, which detected dislocations and the strain fields of inclusions, with low-voltage monochromatic microradiography, which revealed sheets of sub-micrometre metallic inclusions. The larger crystals now available facilitate combining assessments by cathodoluminescence, X-ray topography, birefringence, and absorption of ultraviolet, visible and infrared wavelengths for mapping distributions of impurity, internal strains and the relative development of the different growth forms, (100), (111), etc. X-ray topography, by non-destructively revealing the trajectories of dislocations within a cut and polished specimen, can also disclose the specimen's geometrical relationship to its parent seed crystal. The new, powerful synchrotron X-ray sources facilitate application of sensitive double-crystal X-ray topographic methods to record coherency strains between different growth sectors and measure their lattice parameter differences. The topographic study of cathodoluminescence discovered the growth-sectorial dependence of the type of CL spectrum and its polarization (e.g. the ambient temperature green emission from (100) and the 1.4 eV system emission from (111)). New observations, to be described, have detected a dependence of CL properties upon the direction of advance of growth steps lying on a given growth surface.

INTRODUCTION

This paper reports some recent experiments on large synthetic diamonds (0.1 carat and greater) grown by the reconstitution technique. The two experimental methods illustrated differ greatly, but complement each other excellently. A simplified distinction between them would be that cathodoluminescence examines crystal surfaces and X-ray topography examines crystal interiors. Recall, however, that a classic way of examining a crystal interior is by a sequence of polishing and etching experiments on the specimen (and such studies performed on natural diamonds, e.g. [1], have stimulated many subsequent X-ray and cathodoluminescence experiments, e.g. [2-4]). Substitute etching by cathodoluminescence, and one sees how the latter technique could also be used to examine crystal volumes, albeit in a laborious and specimen-destructive fashion. Conversely, whereas X-ray topographic techniques are particularly noted for their ability to probe crystal interiors non-destructively, it is possible to tailor them to be especially sensitive to defects at or close below the specimen surface, as will be explained below.

The concentration here on only X-ray diffraction and cathodoluminescence techniques does not imply disregard of the power of other methods, especially those involving optical microscopy. Thus, highly relevant to our present topic are the excellent photomicrographic studies by Shigley et al. concerning large synthetic diamonds grown by Sumitomo Electric Industries [5] and by de Beers [6]. Nor to be neglected is the informativeness of differential abrasion resistance patterns on polished diamond surfaces, revealed by Nomarski interference reflection microscopy [7-9], and of refractive index variations within specimen volumes, revealed by Schlieren transmission microscopy [9].

In the following sections we review some recent case studies of specific features in large synthetic diamonds, preceding the presentation of the X-ray and cathodoluminescence (CL) observations by brief descriptions of the particular embodiments of the X-ray and CL techniques that were employed in the experiments. The motive for our studies was general interest in diamond physics, but it is hoped that similar investigative methods might find

practical application in looking for causes of premature wear and failure of tools and dies made from large synthetic diamonds. In particular, now that there is evidence that synthetic diamond can rival natural diamond in performance as anvils in HP cells [10], some increased interest in sensitive ways for locating and identifying the causes of internal stress concentrations in large synthetic diamonds may be anticipated.

X-RAY TECHNIQUES APPLICABLE TO SYNTHETIC DIAMONDS

In early X-ray topographic studies of synthetic diamonds [11], absorption microradiographic observations were fruitfully combined with diffraction experiments. In those days most synthetic diamonds were profusely populated with metallic inclusions, which occurred both as globules up to some tens of micrometres in diameter and also as sheets of sub-micrometre-sized particles. Microradiography with radiation monochromatised by Bragg reflection from a germanium crystal provided beams of either CuKα or CuKβ radiation. The difference in absorption of these radiations by the inclusions enabled the nickel content of the inclusions to be estimated, since the nickel K absorption edge falls between the wavelengths of CuKα and CuKβ. Also, by setting the specimen at different angles to the parallel monochromatic X-ray beam stereo-pairs of radiographs were obtained, and disclosed sheets of sub-micrometre metallic particles that had apparently been occluded in growth sector boundaries. This powerful technique of monochromatic X-ray absorption micrography has not been needed in assessing the present specimens since their much sparser inclusion population could be adequately mapped using diffraction topographs alone.

In X-ray diffraction topographs one may observe both "orientation contrast" and "extinction contrast". The latter contrast refers to the change in X-ray reflecting power of a crystal volume element that results from a strain gradient within it, such as that generated by a dislocation, or a crack, or a surface indentation. The relative sensitivity to orientation contrast and extinction contrast in an X-ray topographic set-up varies widely according to the nature of the X-ray source, its collimation, and other experimental parameters under the experimenter's control [2,13]. It will suffice here to note some particular cases. With an X-ray source emitting a continuum of wavelengths (a synchrotron source is the prime example) given lattice planes in the crystal will satisfy Bragg's Law over a range of glancing angles of incidence on them. The topographs then obtained are insensitive to (small) relative misorientations of parts of the specimen, but do show local variations in X-ray reflecting power, i.e. extinction contrast. At the other extreme, the beam incident on the specimen may be made both highly parallel and highly monochromatic by prior Bragg reflection at two or more perfect crystals. Then sensitivity to misorientations in the specimen becomes very high, but in practice it is changes in the specimen's angular setting for peak Bragg reflection that are detected, and such changes include the joint effects of lattice tilt and of Bragg angle change resulting from change of lattice spacing. The sensitive "double-crystal" topographic technique [12,13] has been recently applied to large synthetic diamonds in order to measure the small differences in lattice parameter between different growth sectors that result from their different concentrations of nitrogen impurity [14,15]. In this report we will chiefly discuss experiments using standard X-ray topographic techniques, employing conventional X-ray tubes as sources of characteristic X-rays such as MoKα (wavelength 0.071 nm) and CuKα (wavelength 0.154 nm). The basic experimental arrangements involved and some applications to diamond are described in [16].

In the present work both experimental operations and image interpretation were simplified by the absence of significant misorientations (i.e. those exceeding a few seconds of arc) within the large synthetic crystals examined. (In this respect they compared most favourably with many natural diamonds, in particular Australian ones.) Consequently, the incident beam collimation employed was chosen not with reference to the range of misorientation in the specimen but by the practical need to prevent simultaneous reflection of both the α1 and α2 components of the Kα doublet. Such simultaneous reflection would cause a highly undesirable doubling of the topograph image. The needed collimation to a beam divergence not exceeding 1 to 2 minutes of arc was effected by combined use of an X-ray tube giving a small apparent focus size, a distance of 0.5 to 1.0 m between source and specimen, and the placing of a narrow slit close to the specimen. Freedom from gross misorientations in
the specimen also meant that the photographic blackening patterns that appeared on our
topograph images could be taken as direct representations of the patterns of extinction contrast
due to local strain gradients in the specimen.

As is explained in [13], there are both theoretical and practical reasons for preferring to
take topographs with longer wavelengths rather than shorter, e.g. with CuKα rather than
MoKα or AgKα, when possible. In fact the higher absorption of CuKα precludes its use for
transmission topography for all but thin specimens of material containing only light elements.
Fortunately diamond falls in the latter category and CuKα is found useful for taking
transmission topographs of diamonds up to 1-1.5 mm thick. The basic techniques of the
'section topograph' and the 'projection topograph' are described in [12,13,16]. Figs. 1a and b
are simplified representations of their diffraction geometries. The drawings show ray paths in
the plane of incident and diffracted rays. Thus the specimen slab ABCD, its reflecting Bragg
planes (shown by light lines), and the ribbon-shaped incident X-ray beams 1,2,3 and 4 all
extend normal to the plane of the drawing. A plate-shaped diamond specimen, with major
surfaces in cube orientation, and the use of CuKα radiation are assumed; and the ray directions
drawn correspond to some frequently employed diffraction geometries with this combination.
In the section topograph arrangement, shown in fig. 1a, the crystal is stationary during the
exposure, but it can be positioned accurately with respect to the ribbon incident beam (typically
15 μm wide) so that the latter cuts through the crystal to reveal a section (indicated by the heavy
line XX') exactly in the position desired. The photographic plate (not shown) is placed so that
diffracted rays m,n fall perpendicularly upon it. Simple geometric calculation gives the ratio of
the image width mm' to the distance XX' as a function of Bragg angle and inclination of the
Bragg planes to the specimen surfaces. The upper part of fig. 1a (incident ray 1) represents a
220-type reflection of CuKα in symmetrical transmission by (110)-type planes normal to the
cube-orientation plate specimen (Bragg angle 37.65°). Incident ray 2 illustrates a case of
asymmetric transmission when Bragg reflection occurs from one of the four equivalent (111)
planes that make 54.74° with the specimen plate (Bragg angle 21.96°).

In projection topographs the specimen and photographic plate are mounted together on a
platform that is translated back and forth during exposure so that the entire specimen area of
interest is scanned by the ribbon incident beam. Fig. 1b shows two of the projection topograph
geometries used with cube-orientation plates. The specimen is translated parallel to itself, as
indicated by the doubled-headed arrow. (For simplicity, not shown are the photographic plate,
which is set normal to the diffracted beam m, and the absorbing screens needed to prevent
unwanted rays falling on the plate.) As fig. 1b indicates, projection topographs can be taken in
transmission (incident ray 3) or using a surface reflection (incident ray 4). The latter case is a
symmetrical 004-type reflection, an example of which is reproduced in fig. 3a. Incident ray 3 is
producing the reverse reflection to ray 2 of fig. 1a, i.e. it is incident on the reverse side of the
same set of (111)-type planes shown reflecting in fig. 1a. Rays 2 and 3 illustrate an important
practical point. Generally one desires sections obtained with the incident beam making a small
angle with the normal to the specimen surface. But to obtain a projection topograph of a plate-
shaped specimen with minimum foreshortening of the image one chooses the diffracted beam to
make a small angle with the specimen normal. Thus, using similarly inclined Bragg planes,
incident beam direction 2 would be preferred for section topographs and direction 3 for
projection topographs. The factor by which the length BC is foreshortened in projection along
diffracted ray m in the latter case is cos(90-54.74 - 21.96°) = cos13.30° = 0.97.

X-RAY TOPOGRAPHIC IMAGES AND THEIR INTERPRETATION

In order to analyse fully the geometry of all the defects generating contrast on X-ray
topographs of synthetic diamonds it may be necessary to record them in up to dozens of
different specimen settings. Fig. 2 is a 111-type projection topograph of a cube-orientation
slab, 3 mm x 1.75 mm in area and 1 mm thick, cut from a Sumitomo Electric Industries
synthetic. The diffraction geometry is like that with ray 3 in fig. 1b, but in the case of fig. 2
synchrotron radiation, wavelength 0.125 nm, Bragg angle 17.75°, was used, not CuKα.
(Another 111-type reflection from this specimen is shown and discussed in [9].) The arrow
Fig. 1.

Diffraction geometry: explained in text.

Fig. 2. Synchrotron X-ray projection topograph of polished slab of synthetic diamond. Specimen width 3mm. Arrow shows projection of normal to Bragg plane reflecting.
below fig. 2 points along the projection of the normal to the Bragg plane that is reflecting. Strong extinction contrast (more generally referred to as diffraction contrast) is generated by the strain gradients associated with tips of cracks and with indentations, the latter being composed of ring cracks in various stages of development, and X-ray topographs are extremely sensitive to such defects. In fig. 2 strong contrast comes from burrs and chips along the specimen edges, and also from a couple of elliptical patches of damage where micro-abrasion tests had been carried out (near centre, and lower right of centre of the image). Abrasion damage is particularly severe on the right-hand lateral surface (corresponding to AB), which had been coarsely polished. Streaking of diffracted rays from this surface shows evidence of misorientations up to 0.2° in this surface. As for internal defects, a number of bundles and fans of dislocations are evident. The great majority of dislocations diverge from the seed crystal, which had been situated about 1 mm externally from the far surface of the slab as viewed in fig. 2.

Another Sumitomo specimen, a square cube-orientation plate, 3.2 mm wide and 0.73 mm thick, is illustrated in fig. 3. Fig. 3a is a 004-type surface reflection, taken with CuKα1 radiation and the geometry of ray 4 shown in fig. 1b. This topographic mode offers a good combination of information-richness and easy geometrical interpretability. With CuKα1 radiation, the Bragg angle for the 004 reflection is 59.74°, and the foreshortening of the width BC by the factor sin 59.74° = 0.86 needs to be borne in mind when geometrically interpreting features such as images of growth sector boundaries outcropping on the polished face BC. Fig. 3b is a section topograph taken through the specimen with CuKα1 radiation and a 004-type reflection in symmetrical transmission. The diffraction geometry is analogous to that with incident ray 1 in fig. 1a, but has Bragg angle 59.74° rather than 37.65°. Below the diagram fig. 3c arrows show where the section topograph incident beam enters the far surface AD at X, and leaves the near surface BC at X'. In the case of the surface reflection image fig. 3a, if the specimen were ideally perfect then most of the incident X-ray energy would be totally reflected from a surface layer extending to a depth of not more than about 5 μm below BC. In actuality, numerous internal defects, principally dislocations, but also some growth sector boundaries, reflect rays incident outside the range of total reflection (which is only 2.5 seconds of arc), and their additional reflected intensity produces strong local blackening on the image. (The component of total reflection from the surface BC explains the greater overall intensity of its image in fig. 3a compared with the weak image of the lateral surface AB which is formed by X-rays that have travelled into the crystal.) As in fig. 2, here in fig. 3a strong contrast comes from burrs and chips along the specimen edges; and all surfaces are peppered with indentations, which show up as small black dots or rings on fig. 3a, being especially intense in the case of damage on the reflecting surface BC. Images of surface scratches are in evidence: examples of these are the black zig-zag lines seen in the upper part of fig. 3a. The profusion of diffraction contrast features in fig. 3a renders interpretation of a single projection such as this extremely difficult. In practice, major amelioration of the problem is achieved by taking stereo topographs. With these specimens the Haruta technique is appropriate [17]. In this method the stereo effect is produced by a small rotation of the crystal about the normal to the Bragg plane that is reflecting. For specimen dimensions and the Bragg reflection used in fig. 3a, a rotation of 8° was very effective. Inspection of the resulting image pair using twin microscopes provided dramatic clarification, showing clearly what contrast arose from surface damage, and from which surface, and revealing the three-dimensional configuration of the dislocations and some growth-sector boundaries.

The section topograph image, fig. 3b, reveals directly that dislocations are radiating into the specimen from the face BC, that closer to the seed. Dislocation images dominate this topograph (and mask contrast from growth sector boundaries that one can recognise on section topographs of synthetic diamonds with lower dislocation density). The dislocation images show two types of contrast. Where the dislocation line is cut by the incident beam XX' its associated lattice tilts allow extra X-ray intensity to be reflected, and the dislocation appears as a black dot or streak according to its obliquity to XX'. Dislocation segments that are not cut by XX', but lie in the crystal volume contained between XX' and the diffracted ray Xm act to divert energy from the diffracted beam back into the incident beam direction, thus forming an 'extinction shadow' (often referred to as the 'dynamical image'). Such shadows appear as unsharp white streaks in fig. 3b.
Fig. 3. (a), top left, X-ray projection topograph; (b), top right, X-ray section topograph; and (c), bottom, basic growth sector map of near face of polished cube-orientation slice of synthetic diamond, face width 3.2 mm.
Information from several X-ray topograph reflections, and in particular from CL, was used for identification of growth sectors intersected by the polished face BC (fig. 3c). The central area is occupied by cube-facet growth in the direction of the inward normal to the surface. This growth is separated from other cube growth sectors (above and below, and to right and left) by thin sheets of \( \{110\} \) growth sectors. \( \{111\} \) growth sectors (weakly developed) are identified by hatching parallel to their facet traces on BC. They are surrounded by \( \{113\} \) facets of variable width, but generally quite narrow.

CATHODOLUMINESCENCE TOPOGRAPHIC TECHNIQUE

The value of CL microscopy as an adjunct to conventional optical microscopy in mineralogy and petrology is demonstrated by the variety and detail found in CL images recordable with simple CL accessories attached to optical microscopes [18]. Optical imaging, aided sometimes by IR-sensitive and low-light-level TV systems [19], was used in all the CL work reported here. The physical processes relevant in understanding diamond CL, and the observational techniques available, are described in reviews [20-22]. Excitation conditions controllable and emission characteristics recordable are a) bombarding electron energy, b) specimen current density, c) specimen temperature, d) emission spectral distribution, e) polarisation of emission, and f) emission decay times. All but f) are accessible using the SEM with optical imaging and photographic recording; and indeed e) has been a special focus of CL work on diamond at Bristol University. For most diamond CL studies performed there the specimen is placed in the specimen chamber of a standard SEM, its electron-bombarded surface is viewed either directly or via a lens in the specimen chamber, and the recording camera or electronic imaging device is placed outside a chamber window. Polarisation and other optical filters are placed between chamber window and camera. Use of the SEM affords easy independent control of electron energy and specimen current density: the former being variable between 500 V and 40 kV, typically, and the latter ranging over 7 orders of magnitude, from \( 1 \text{ nA mm}^{-2} \) to \( 10 \text{ mA mm}^{-2} \), say. Variation of electron kV has been used to obtain some three-dimensional information on luminescent features. For example, in the case of a luminescent horizon outcropping at a polished crystal surface, its dip relative to the surface can be found by observing how its image shifts when switching from low kV (shallow electron penetration) to, say, 40 kV (deeper penetration with half the emission coming from depths 5 \( \mu \text{m} \) or more below the diamond surface). Varying the specimen current density, like varying the specimen temperature, causes large changes in the relative intensities of emission from various optical centres, and is a valuable diagnostic tool for distinguishing between emissions from different centres. With lens imaging and photographic recording one is free to choose between scanning the specimen with a small electron probe or flooding the whole area of interest with a stationary, defocussed electron beam. The CL images shown here were obtained in the latter mode.

THE MALTESE CROSS PHENOMENON

Using the optical imaging methods described above, a number of CL polarisation phenomena have been discovered in natural and synthetic diamond. None of the findings was accidental: all resulted from deliberate search for polarisation effects. The polarisation phenomena can be categorised according to their associations, viz. with 1) linear defects, i.e. dislocations in natural and synthetic diamonds [4,8,23,24], 2) platelet defects on cube planes in natural diamonds [4,25], 3) the orientation of the growth facet in synthetic diamonds [8,26], and 4) the direction of advance of growth steps on a given growth facet [9]. Here we summarise the most recent observations, those in the category last-mentioned above. For fuller description of what we term the Maltese Cross phenomenon, reference may be made to [9].

The CL patterns in figs. 4a and b illustrate the essence of this phenomenon. This specimen, one of whose X-ray topographs was shown in fig. 2, was also studied by birefringence, Schlieren photography and Nomarski interference microscopy of its differential abrasion resistance patterns on its major polished cube-orientation surfaces, that surface closer to the seed being shown in fig. 4. The growth sector configuration is analogous to that in the
Fig. 4. The Maltese Cross polarised CL phenomenon. Double-headed arrows show direction of E-vector transmitted by polarising filter.
map fig. 3c. In these room temperature CL patterns the \{113\} sectors appear brightest. The \{111\} sectors (together with very thin sheets of \{110\} growth separating cube growth sectors) are non-luminescent. Spatially and spectrally variable green-yellow luminescence comes from the cube growth sectors. These are seen edge-on left, bottom and right in this view. The central sector represents cube-direction growth perpendicularly into the specimen surface, which is in the direction indexed \{001\}.

Looking through the crystal along \{001\} one could see a cross-like distribution of deeper yellow colouration in the central \{001\} growth sector, its arms radiating in the diagonal ± \{110\} and ± \{110\} directions, corresponding geometrically to the cross pattern in fig. 4. The infrared absorption was probed locally by a Fourier transform infrared spectrometer fitted with a reflecting-objective microscope and it was found that these diagonal arms contained 25% to 30% more substitutional nitrogen impurity than the paler arms of the cross in between them, the measurements being made at a probe centroid distance of 600 µm from the cross centre. When viewing the CL pattern without a polarising filter, significantly brighter green-yellow emission from the diagonal nitrogen-rich arms was observed. It was guessed that some radial-direction-dependent polarisation of a emission from the cross pattern would be detectable in this \{001\} view, and such was indeed found, as figs. 4a and b testify. The double-headed arrows below these figures indicate the direction of E-vector transmitted by the polariser. In the bright diagonal arms the dominant E-vector is radial. On the other hand, in the interdigitating less bright arms, which point towards ± \{100\} and ± \{010\} in \{001\}, no intensity change was observed upon rotation of the polarisation filter, in this \{001\}-direction view. Several variants of this Maltese Cross pattern have been observed in numerous encounters with it in face-on views of \{001\} growth facets sectioned on polished synthetic diamonds. This phenomenon provides a striking demonstration of CL polarisation that is dependent on the orientation of growth steps on the growth face concerned. There can be little doubt that the differences in level of impurity concentration and of optical properties observed arise from differences in surface structure on vicinal slopes of a low-elevation growth pyramid. In this specimen, and in other examples, a single such feature (or a small number of them) have dominated growth over the entire facet surface (or a substantial fraction of its area) for sufficiently long epochs in the crystal’s growth history to generate a high probability of their being intersected, and hence made visible by CL, on polished sections. In [9] an explanation in principle of the polarisation phenomena is given, it being shown that the diamond structure fourfold screw symmetry axis parallel to \{001\} allows a growth step model that can account for the unpolarised emission from the cross arms pointing towards ± \{100\} and ± \{010\}, and the 90° rotation of dominant E-vector direction between the ± \{110\} and ± \{110\} directed arms.

STUDIES OF THE 1.40 eV AND 2.56 eV EMISSION SYSTEMS

After it had been discovered that the green CL from synthetic diamonds was emitted from cube growth sectors, and that this emission was strongly polarised with dominant E-vector lying in the cube-plane growth surface of the sector concerned [8], it was logical to look for analogous behaviour by other emissions. An important CL emission system observed in diamonds, both natural and synthetic, containing substitutional nitrogen impurity lies in the near infrared, with zero-phonon energy 1.40 eV, 884 nm [27]. Applying CL topographic techniques developed for this wavelength [19], it was discovered that the 1.40 eV system was emitted only from the \{111\} growth sectors of synthetic diamonds, and was also partially polarised, the dominant E-vector lying parallel to the octahedral facet concerned [26]. The specimen studied in [26] has recently been re-examined by Collins [28] who has made spectroscopic studies of the zero-phonon doublet at temperatures down to 4K. A specimen more favourably, indeed ideally oriented for studies of polarisation of the 1.40 eV system, is shown in figs. 5a and b. It is a (110)-orientation polished plate of synthetic diamond, maximum width 5 mm, and is specimen no. 1 from a suite whose impurity content, CL patterns and emission and absorption spectra have recently been described [29]. (The authors are most grateful to Dr. C.M. Welbourn, DTC Research Centre, Maidenhead, Berkshire, England for loan of this specimen.) In [29], CL topographs at room temperature and near 77K are displayed in colour, and a growth sector map of this specimen is given. The topographs in fig.
Fig. 5. Partial polarisation of 1.40 eV system infrared CL from octahedral growth sectors of synthetic diamond plate polished parallel to (110). Double-headed arrows show direction of E-vector transmitted by infrared polarising filter.
were taken with specimen temperature near 77K using Kodak High Speed Infrared film type 2481 plus a Kodak-Wratten infrared-passing filter type 87 (see [19] for the wavelength response of this combination), together with a Polaroid Type HR polarising filter. Two pairs of (111) planes are seen edge-on: (111) and (111) upper left and lower centre right, and (111) and (111) upper right and lower left. An octahedral sector, indexed (111) in [29], inclined 35° to the specimen surface, lies lower right of centre. The only infrared-emitting areas are the (111) growth sectors. Comparing differently exposed topographs taken with the two polarising filter orientations gave 2.5:1 as a rough value of the ratio of intensities with E-vector parallel to those with E-vector normal to the growth surface, applying in all (111) sectors viewed edge-on, but there was a range approaching 2:1 in average (all E-vector directions) emission from different (111) sectors. There were also noteworthy point-by-point intensity variations, not polarisation dependent, in each (111) growth sector [30].

Another optical centre found only in (111) growth sectors of synthetic diamonds gives rise to the 2.56 eV, 484 nm vibronic system, and is apparently associated with the 1.40 eV system [31]. The 2.56 eV system makes little contribution to CL at room temperature, but becomes the dominant visible emission at 100K or below [29,30]. Comparisons of the distribution of intensity of the 1.40 eV and 2.56 eV systems at low-temperature (approx. 77K) from the specimen shown in fig. 5, while agreeing that, taken sector by sector, the strengths of these emissions went hand in hand, revealed that their detailed patterns showed a remarkable complementarity. This is illustrated in the comparison of visible and infrared patterns presented in figs. 6a and b, which show the sector indexed (111) in [29] under higher magnification. (The width of the sector at the top of its image is 0.49 mm.) The complementarity of the 1.40 eV and 2.56 eV emissions applies not only to the features showing approximately three-fold rotational symmetry about the growth facet normal (referred to as 'trigonal features') but also to many fine lamellae that cross the image horizontally. The trigonal features are analogous to the
four-fold symmetric Maltese Cross patterns described above in so far as there can be little doubt that the six sectors, alternatively light and dark, are images of vicinal slopes on low-elevation, six-sided pyramids on the growth facet of mean (111) orientation, as discussed in [30]. However, the trigonal intensity patterns shown in 6a and b are invariant under change of orientation of polarisation filter.

A valuable adjunct to CL topography is CL microspectrography [4]. In its simplest embodiment, the entrance slit of the spectroscope or spectrograph is placed in the CL image plane, with relative position and orientation of slit and image controllable. In fig. 7 the entrance slit is set roughly normal to the growth layers viewed edge-on in the (111) growth sector, upper left in fig. 5. The specimen is held near 77K, and the complex spectrum of the 484 nm system is recorded, together with Hg arc calibrating lines.

REFERENCES