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Structural characterization of 6H- and 4H-SiC polytypes by means of cathodoluminescence and x-ray topography

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Abstract

The cathodoluminescence (CL) technique is used to analyse the radiative recombination properties of four distinct silicon carbide (SiC) samples: a 6H-SiC n^+-type Lely wafer, two off-axis 4H-SiC epitaxial layers of n type and p type, and a $(11\bar{2}0)$-oriented 4H-SiC n^+-type substrate. The CL spectra, recorded at various temperatures and at various excitation conditions, show strong differences between the polytypes, indicating a better homogeneous distribution of radiative centres inside the 6H polytype than in the 4H one, and also between the different orientations. For the $(11\bar{2}0)$-oriented 4H sample, luminescence features decrease when the excitation intensity increases, probably due to a more significant indirect transition band. The CL spectra also vary for the same sample, due to the impurity and the microscopic defect density variations. Comparisons between two local spectra taken in two distinct areas of the $(11\bar{2}0)$-oriented 4H sample, and with images obtained by x-ray topography in the same areas, allow us to establish that some structural defects are involved in luminescence centres. A deep centre involved in green luminescence (at 1.80 eV) is found to be associated with basal plane dislocations with the Burgers vector $b = (1/3)\langle 11\bar{2}0\rangle$.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Silicon carbide (SiC) is a wide band gap semiconductor, whose opto-electronic properties make it suitable for many applications, especially in high power and high frequency devices. Some structural defects due to crystal growth and/or doping technologies are commonly present in the epitaxial layers or substrates of SiC. It is therefore necessary to investigate the properties of these defects, in order to determine their influence on the optical and electrical properties of the material.

Detection and identification of impurities creating radiative centres in the band gap have been carried out on the basis of the photon emission arising from the recombination of electron–hole pairs in the volume. The study of deep levels is easier with cathodoluminescence (CL) than with photoluminescence (PL), thanks to a higher intensity excitation level, since one electron can indirectly generate thousands of photons. Moreover, high spatial and spectral resolution can be obtained at once. Until now, CL studies of the (0001) Si faces 6H- and 4H-SiC have been essentially performed to investigate the localization of stacking faults [1] or the behaviour of background impurities [2].

It is the $(11\bar{2}0)$ SiC face that is mainly studied today, due to there being fewer negative charges at the 4H-SiC oxide interface compared with the (0001) SiC face, leading to better channel mobilities and lower threshold voltages measured for planar MOSFETs [3]. Another advantage of an orientation perpendicular to the (0001) face is a better crystal reordering during the post-ion implantation annealing, achieved by avoiding site competition between cubic and hexagonal sites [4]. Studies of the structural and optical properties of $(11\bar{2}0)$-oriented 4H-SiC wafers are then crucial. This paper presents CL and x-ray topography (XRT) characteristics obtained for SiC samples. The defects observed on (0001)-oriented 4H samples are compared with those detected on $(11\bar{2}0)$-oriented 4H samples, in order to check whether the structural defects observed on these two faces produce the same radiative properties.

2. Experiment

Four different samples of SiC have been investigated in this study: off-axis Si face 4H-SiC epitaxial layers of n type (4.9 μm, 5.4×10^{15} cm^{-3}) and p type (3.0 μm, 2.0×10^{15} cm^{-3}), $(11\bar{2}0)$-oriented 4H-SiC n$^+$-type substrate (216 μm, 5.0×10^{18} cm^{-3}) and a 6H-SiC Lely n$^+$-type wafer (450 μm, (3–5) $\times 10^{18}$ cm^{-3}). The CL measurements were performed in a Hitachi S-2500 scanning electron microscope, equipped with a computer-controlled Oriel 77200 monochromator and a Hamamatsu R298 photomultiplier as a detector. The spectral resolution was better than 10 nm. The microscope is equipped with a temperature controller system that permits regulation from 77 K. Details of the experimental set-up for spectral and panchromatic CL measurements are presented elsewhere [5]. Different acceleration voltages in the range of 10–20 kV were used. The estimated depth of the maximum electron penetration R_e for this last energy is estimated as about 3.3 μm [6], but the effective zone analysed is reduced due to a partial absorption of the light emitted by SiC between the electron–hole pair generation zone and the surface. This zone is estimated as $0.3R_e$ by Toh and Phillips [7], giving $\sim$1 μm.

X-ray topographs were registered in Lang transmission geometry using the Ag Kα wavelength.

3. Results and discussion

3.1. Off-axis Si face 4H-SiC epitaxial samples

Three characteristic broad bands were observed in all spectra of n-type and p-type (0001)-oriented 4H-SiC samples, centred on the following photon energies:

(a) 3.25 eV, corresponding to the free exciton recombination band related to the edge luminescence (EL) [8];

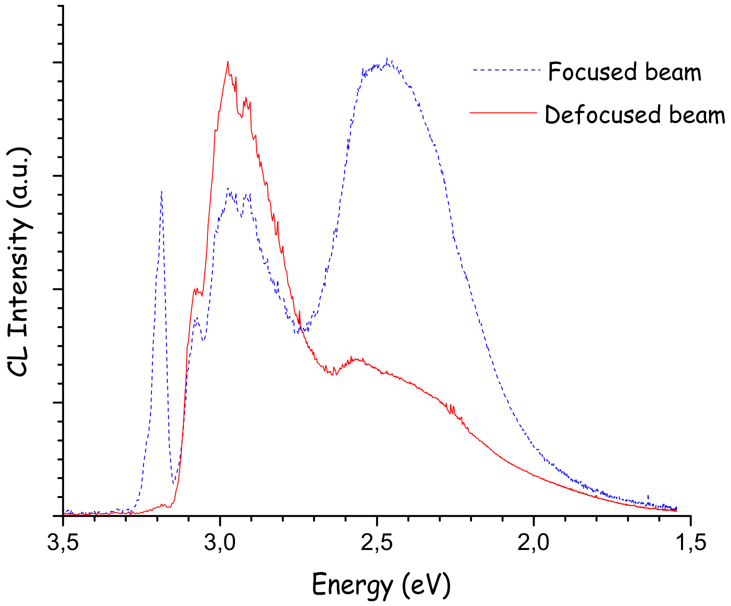

Figure 1. CL spectra of a (0001)-oriented p-type 4H-SiC epitaxial layer, with the electron beam focused and defocused. The accelerating voltage is 20 kV and the sample temperature is 77 K.

(b) 2.95 eV, called blue luminescence (BL) and attributed to Al–N acceptor–donor pairs [9];

(c) 2.45 eV, called green luminescence (GL) and resulting from recombinations at radiative centres involving boron (B) [10].

A comparison between the overall CL spectra at 77 K of a p-type epitaxial layer obtained with a focused and a defocused electron beam is given in figure 1, for an accelerating voltage of 20 kV. Defocusing the beam leads to reduction of the EL and GL intensity bands, and enhancement of the BL band. This difference in behaviour between emission via Al–N acceptor–donor pairs and via boron-related centres is related to a competition between the presence of Al and that of B in the material [10]. The number of impurities acting as recombination centres is more significant when the electron beam is defocused, normally resulting in an increase of the corresponding luminescence radiation. Figure 1 proves the cross-section of BL-related centres to be higher than that of the GL-related centres.

A saturation of deep levels may appear when the intensity of the electron excitation rises (that is the case for a focused beam), leading to a higher intensity of the EL peak. When the accelerating voltage varies from 20 to 10 kV, the BL intensity band appears to be higher unlike the GL one, indicating that Al-related centres may be situated closer to the surface than B-related ones.

A proper deconvolution of the GL band evidences two Gaussian fits. The corresponding energies are 2.50 and 2.26 eV (with decreasing height), proving the existence of at least two deep levels related to B. Some authors attribute their presence to boron on carbon sites B_C and/or to complexes involving B atoms and vacancy clusters [10].

Figure 2 shows CL normalized spectra of an n-type epitaxial layer obtained with a focus beam at 20 kV (and a temperature of 77 K). A comparison is made between two areas detected in a CL panchromatic image. For this sample, the EL peak is the most visible. The GL signal intensity is rather less significant than for the p-type layer, denoting a lower concentration of

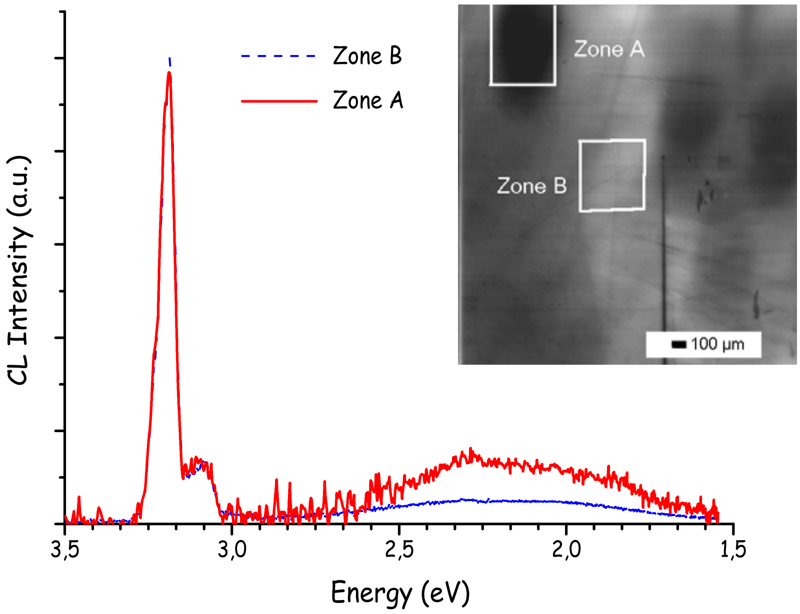

Figure 2. CL normalized spectra of a (0001)-oriented n-type 4H-SiC epitaxial layer, with the electron beam focused at an accelerating voltage of 20 kV and a temperature of 77 K. A comparison is made between two zones detected in a CL panchromatic image.

B-related centres. We should note that the GL band is broader than in the p-type layer, due to a new emission appearing at ~1.8 eV. Zone A, darker than the zone B, contains more radiative centres associated with the GL band.

A small peak is observed at about 3.1 eV. Luminescence situated near this energy is attributed by some authors to inclusions of 15R-SiC polytype [11]. Since monochromatic images at 3.1 eV showed a uniform distribution of the emission, this peak should here be a phonon replica of the EL band.

3.2. The (0001)-oriented 6H-SiC n^+-type Lely substrate

Two major CL broad bands appear, centred at 2.64 and 1.76 eV; they are respectively attributed to the free exciton recombination band (EL) and to B-related recombination centres (GL). Figure 3 presents the luminescence intensity with the electron beam energy varying (in a defocused way). The steady shape of the bands with respect to the depth analysed, as well as the nearly perfect superposition of focused and defocused signals, indicates a homogeneous distribution of radiative centres and/or structural defects throughout the material analysed. The only defects observed in XRT images were growth basal dislocations, with a low defect density of ~10 cm^{-2}. These dislocations may partially give rise to the GL band, and their density is probably correlated with the signal homogeneity.

Radiative recombination due to Al–N acceptor–donor pairs is only visible with a high excitation level (focused electron beam) and at low temperature (around 77 K). The intensity of this band is very weak, although the concentration of nitrogen is (3–5) × 10^{18} cm^{-3}.

3.3. The $(11\bar{2}0)$-oriented 4H-SiC Lely modified n^+-type substrate

XRT shows the strain fields developed around three kinds of structural defect (see figure 4). Defects labelled as 1 are screw dislocations (Burgers vector $b = c$ along [0001]) with a

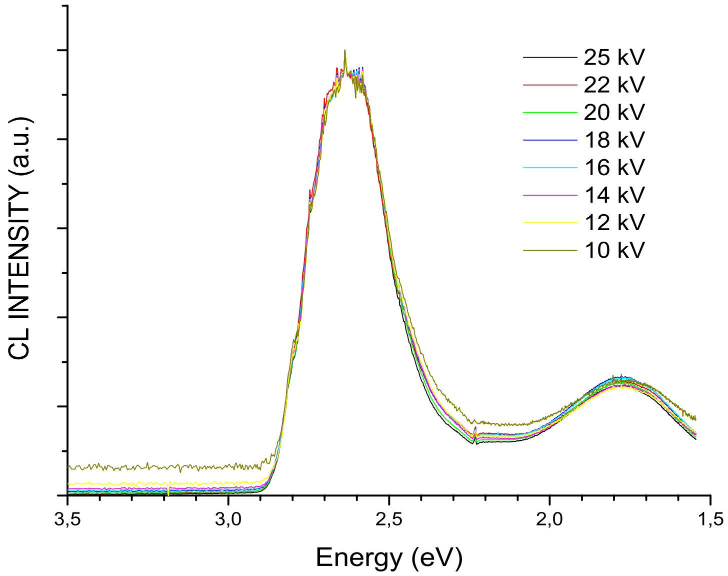

Figure 3. CL spectra of a (0001)-oriented n^+-type 6H-SiC Lely substrate, with the electron beam defocused and a sample temperature of 86 K. The accelerating voltage varies from 10 up to 25 kV.

density of 7×10^2 cm^{-2}, defects 2 are perfect or partial dislocations on the basal plane ($b = (1/3)\langle 11\bar{2}0\rangle$) with a density of 9×10^3 cm^{-2} and defects 3 are low angle boundaries. The Lang transmission geometry means that this image comes from an integration over all volume.

A panchromatic CL image at 20 keV, obtained from the area situated on the left side of the XRT, is given in figure 5. Dark lines can be distinguished at the border, whereas no contrast appears in the centre. These dark lines are similar (same orientation) to the screw dislocations observed in XRTs. Only part of the length is visible here for these dislocations, although they are extended along the entire surface. The reason is that the surface orientation θ is given within $\pm 1°$ from $\langle 11\bar{2}0\rangle$. The observed length of the defects, estimated at $\sim$500 μm from figure 5, and a value of the depth analysed of 1 μm, give a value of surface orientation $\theta = 0.11°$.

Unlike for the (0001)-oriented 4H- and 6H-SiC samples, the luminescence features decrease in strength with increasing excitation intensity. This phenomenon could be related to a more significant indirect transition from the band gap for the $(11\bar{2}0)$-oriented 4H-SiC crystal, which requires more energetic phonons accompanying photon emissions. In this case conditions of low injection have to be used for avoiding any saturation of radiative centres. This is achieved with a defocused electron beam at an energy of 20 keV.

The basal plane dislocations are not visible in CL images because of the weak excitation conditions. The low angle boundaries are not seen either, since they may be situated rather deeper than the depth analysed.

Figure 6 compares CL spectra for the border and the centre of the sample. In both cases, the BL band is very weak (probably due to a low concentration of Al-related impurities or due to the low excitation conditions), but the GL broad band is very significant. A deconvolution of this last band leads to the presence of at least three radiative emissions, centred at about 2.50, 2.25 and 1.80 eV. The two former energies were already detected in the (0001)-oriented p-type 4H-SiC epitaxial layer. The emission centred at 1.80 eV was previously observed in the

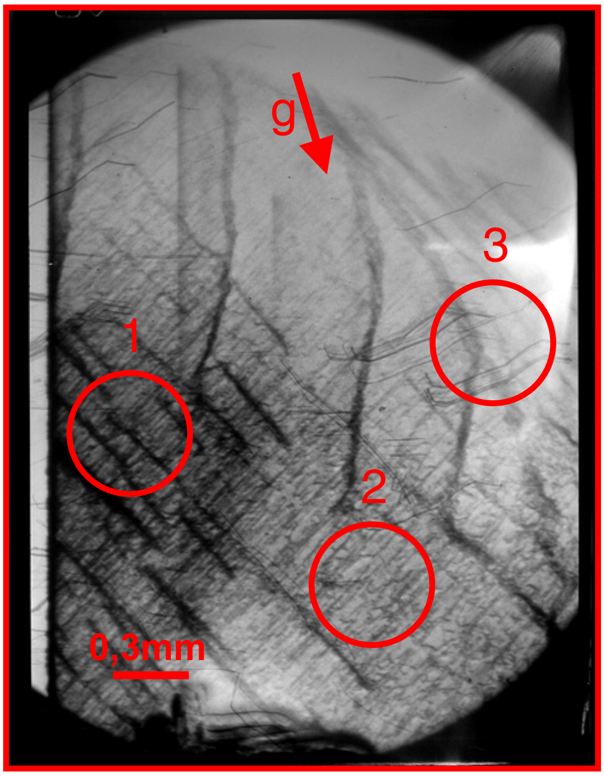

Figure 4. An x-ray topograph for Lang transmission of a $(11\bar{2}0)$-oriented n^+-type 4H-SiC substrate $g = 1\text{–}102$.

(0001)-oriented n-type 4H epitaxial layer, in an area appearing dark in a CL panchromatic image, and also in the Lely 6H wafer containing only basal dislocations.

Since these dislocations are not seen in the centre of figure 5, and as the two spectra of figure 6 are quite similar, it is difficult to decide whether the 1.80 eV level occurs directly due to recombination of carriers at centres associated with screw dislocations (defect 1 in figure 4). But this defect very often crosses the basal plane dislocations, which appear to be uniformly distributed over the whole area in figure 4 with a more significant density than screw dislocations. It is thus possible that the recombinations discussed take place at these basal dislocations.

4. Conclusions

CL measurements were carried out on several SiC samples, differing in surface orientation, polytype and doping type. Three main bands are detected as characteristics of the 4H and 6H polytypes: the EL related to free exciton recombinations, the BL related to Al–N acceptor–donor pairs and the GL due to recombination of carriers at B-related centres. The BL band is not visible for n-type samples (4H- or 6H-SiC), except in the case of 6H-SiC in very high excitation conditions. But the BL band is prominent for p-type 4H-SiC epitaxial samples (having cross-sections more significant than those of B-related centres). These latter centres are situated deeper than the ones involved in the BL band. A deconvolution of the GL band related to B shows two different emission peaks (situated at 2.50 and 2.25 eV) which can be

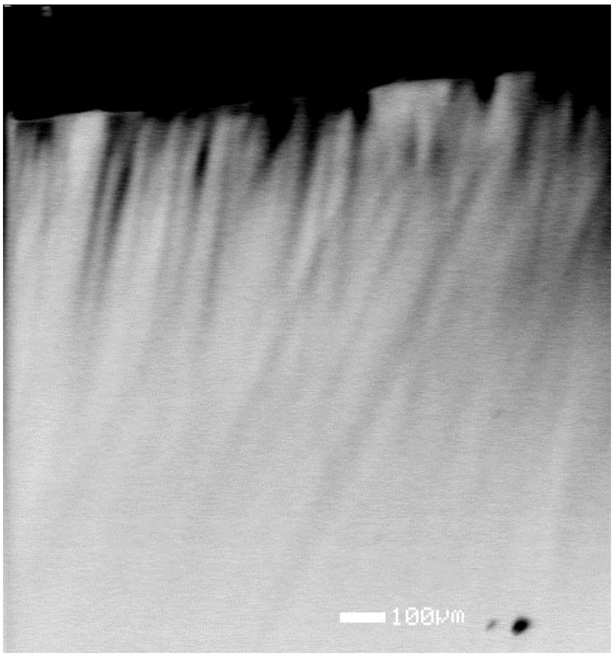

Figure 5. A CL panchromatic image of a (11$\bar{2}$0)-oriented n$^+$-type 4H-SiC substrate, at an accelerating voltage of 20 kV.

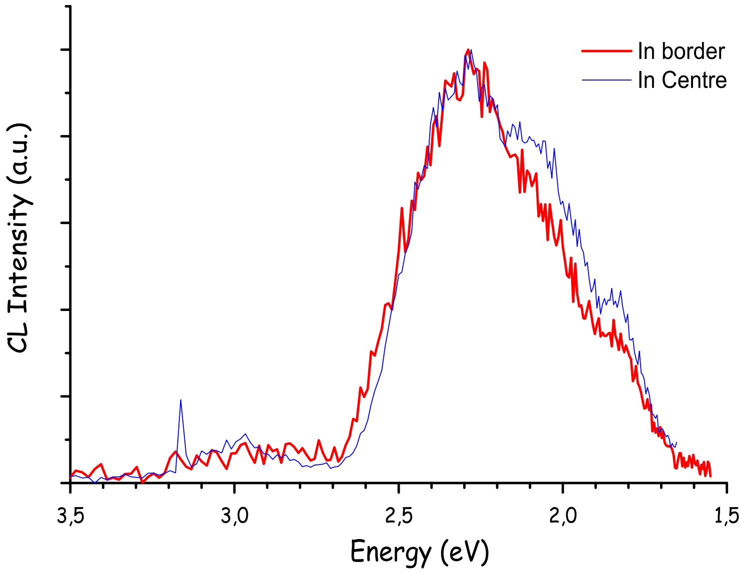

Figure 6. CL spectra of a (11$\bar{2}$0)-oriented n$^+$-type 4H-SiC substrate, with the electron beam defocused, an accelerating voltage of 20 kV and a sample temperature at 86 K. A comparison is made between the border and the centre of the sample.

related to screw dislocations, since these defects are present in epitaxial layers at a high density ($>10^5$ cm^{-2}).

The CL spectra of an n-type (0001)-oriented 4H-SiC epitaxial layer indicate the presence of a third emission peak in the GL band (at 1.80 eV), with an increasing intensity in the sample area appearing darker in the CL panchromatic image. A study of a $(11\bar{2}0)$-oriented 4H-SiC n^+-type substrate containing three kinds of structural defect (revealed by XRT observations) leads to attribution of this peculiar energy level to basal plane dislocations with the Burgers vector $b = (1/3)\langle 11\bar{2}0\rangle$.

This is confirmed by measurements on a 6H-SiC Lely wafer, where XRT images only reveal the presence of basal growth dislocations. The GL band is sharp and centred around 1.76 eV, bearing out the supposed correlation.

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