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Determination of the Structural and Luminescence Properties of Nitrides Using Electron Backscattered Diffraction and Photo- and Cathodoluminescence

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In this paper we describe the use of electron backscattered diffraction (EBSD) for the characterisation of nitride thin films, and report its use in the study of the spatial variation of strain across an epitaxially laterally overgrown GaN (ELOG) thin film. We also discuss the combination of luminescence and EBSD measurements to enable luminescence properties of samples to be directly correlated with their crystallographic properties. We compare photoluminescence spectra and EBSD measurements from a set of GaN thin films grown on off-axis sapphire substrates, revealing the tilt of a GaN thin film grown on a 10° off-axis sapphire substrate to be responsible for the observation of luminescence defect bands in this film. We finally report on the use of EBSD to identify zinc blende regions in a predominantly wurtzite MBE film, with cathodoluminescence used to obtain correlated luminescence spectra.

1. Introduction The acquisition of electron backscattered diffraction (EBSD) patterns in the scanning electron microscope is a very powerful method for the microstructural characterisation of crystalline materials. EBSD is presently used predominantly in metallurgy [1], being applied to the measurement of *texture* [1–2], and for the identification of different crystalline phases. Wilkinson [3] and Troost et al. [4] have applied EBSD to the measurement of strain in SiGe epilayers, while Baba-Kishi [5] has used EBSD to investigate crystallographic polarity in non-centrosymmetric materials. Their results make EBSD an attractive technique to adapt for the characterisation of nitride thin films.

We can for example use EBSD to measure the orientation between nitride thin films and their substrates and measure tilt in nitride thin films [6], we may identify zincblende inclusions in a predominantly wurtzite film and vice-versa. We may also map the strain in nitride films due to the lattice mismatch between the nitride films and their substrates, in particular investigate lateral variations in strain in epitaxially laterally overgrown GaN (ELOG) films. Finally, we can investigate film polarity [7].

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EBSD is a particularly attractive technique because its lateral spatial resolution is of order 100 nm [8] and because it can be combined with other analytical techniques in the SEM, e.g. EDX and cathodoluminescence. Combining luminescence measurements with EBSD allows the luminescence properties of materials to be directly correlated with their crystallographic properties. For example in multiphase material luminescence transitions associated with different phases may be identified. Comparison of EBSD results with luminescence spectra may also help identify the origin of luminescence defect bands.

2. The Electron Backscattered Diffraction Technique In electron backscattered diffraction an electron beam is incident on a sample which is tilted at an angle typically $\geq 70°$. The impinging electrons are scattered elastically through high angles forming a spherical source of diffracted electrons. Electrons that satisfy the Bragg condition for a given plane emanate in diffraction cones from both the upper and lower surfaces of the plane [1]. When these cones intersect the phosphor screen Kikuchi lines are observed (Fig. 1). The Kikuchi lines appear as almost straight lines because the cones are very shallow, as the Bragg angle θ_D is of order 1°. Each Kikuchi band is effectively the trace of the plane from which it is formed, the EBSD pattern is therefore a 2-D projection (the gnomic projection) of the crystal structure [4].

3. Strain Variation in a GaN ELOG Layer Methods have been developed to allow the spatial variations of elastic strain and lattice rotation to be determined from a series of EBSD patterns [3]. For strain measurement the EBSD patterns are recorded at large specimen to screen distance so that only a single zone axis is recorded in the pattern. This geometry gives higher angular resolution and so allows small shifts in the zone axis position to be determined. When the pattern shifts are recorded at two or more zone axes, strains can be distinguished from lattice rotations.

A series of EBSD patterns were recorded along a line scan on the top surface of a MOCVD GaN ELOG sample [9]. The sample comprises a ≈ 4 μm thick fully coalesced GaN layer grown over 9 μm wide SiO_2 stripes repeated with a period of 16 μm. Fig. 2a shows that the linescan is at right angles to the direction of the stripes. EBSD pattern

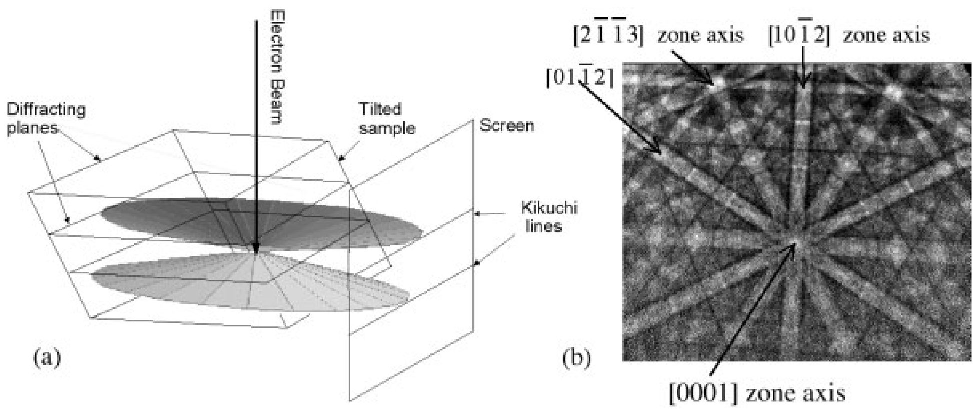

Fig. 1. a) Illustrating formation of Kikuchi lines, b) EBSD pattern from GaN

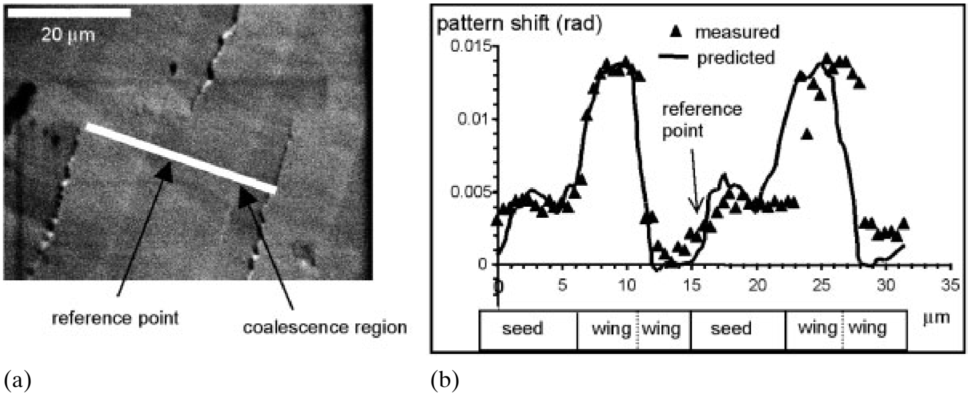

Fig. 2 a) SEM image of ELOG showing position of line scan, b) pattern shift along line scan

shifts were measured at [$2\bar{1}\bar{1}3$], [$01\bar{1}2$], and [0001] zone axes (see Fig. 1b). Data measured for the first two of these were used to calculate strains and rotations assuming that generalised plane strain conditions applied. Note the method measures a strain variation relative to some reference point as marked in Fig. 2. As a consistency check the strain variation was used to predict the pattern shift expected at the third zone axis (i.e., at [0001]). Fig. 2b compares this prediction with the experimental measurements. There is reasonable agreement over the first half of the line scan, in the second half the data sets show some discrepancy. This may be due to some sample charging distorting the line scan.

As is seen from Fig. 2 these preliminary results show the strain variation across the ELOG sample to be complex. Further work is required before final conclusions can be drawn.

4. Comparing EBSD and PL Results for GaN Thin Films Grown on Off-Axis Sapphire Substrates PL spectra from a 3 µm MOCVD GaN thin film grown on a 10° off-axis sapphire substrate (off-axis by 10° from $(0001)_{\text{sapphire}}$ towards $(10\bar{1}0)_{\text{sapphire}}$) is found to be markedly different from the PL spectra acquired from GaN thin films grown on an on-axis sapphire substrate and a 4° off-axis sapphire substrate respectively (see Fig. 3 opposite). Additional bands are observed for the GaN thin film grown on the 10° off-axis substrate which we attribute to structural defects (see [10]). EBSD measurements reveal a tilt of $13 \pm 1°$ towards $[10\bar{1}0]_{\text{GaN}}$ for the GaN thin film grown on the 10° off-axis substrate [6], but no discernable

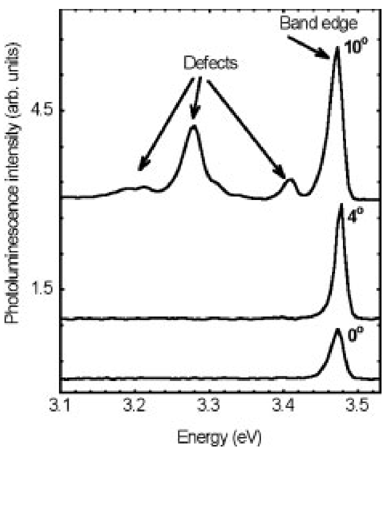

Fig. 3. Low temperature PL spectra from GaN epilayers grown on an on-axis sapphire substrate (0°), a 4° off-axis (4°), and a 10° off-axis (10°) sapphire substrate, respectively

tilt is observed for the other two films. We may therefore conclude that the observed luminescence defect bands must be related to the tilt.

5. Correlation of EBSD and CL Measurements for an MBE GaN Thin Film EBSD was used to identify zinc blende regions in a predominantly wurtzite 5.5 μm thick MBE GaN thin film [11]. Figure 4 shows EBSD patterns and corresponding room temperature CL spectra acquired at an electron beam energy of 15 keV with the sample tilted at an angle of ≈ 70°. The EBSD pattern from the zinc blende region (Fig. 4c) contains some faint features associated with wurtzite material, e.g., the $[10\bar{1}2]$ zone axis can just be discerned. This may be due to the presence of adjacent or underlying wurtzite material and will be the subject of further work. Figure 4b shows the expected wurtzite GaN band edge emission at 3.39 eV [11]. The shoulder at 3.35 eV we attribute to the presence of threading dislocations [12]. Figure 4d shows the 3.2 eV zinc blende band edge emission [11] as well as a peak at 3.35 eV. The shoulder at 3.39 eV we attribute to underlying wurtzite material. Note that CL originates from deeper in the sample (estimated electron penetration depth ≈ 1.5 μm)[2]) than backscattered electrons contributing to the EBSD pattern (typically 10 nm)[2]). The lateral spatial resolution of the CL measurements is also ≈ 1.5 μm compared to ≈ 100 nm for EBSD [8]. Further measurements are in progress to evaluate the full potential of correlated EBSD and CL measurements.

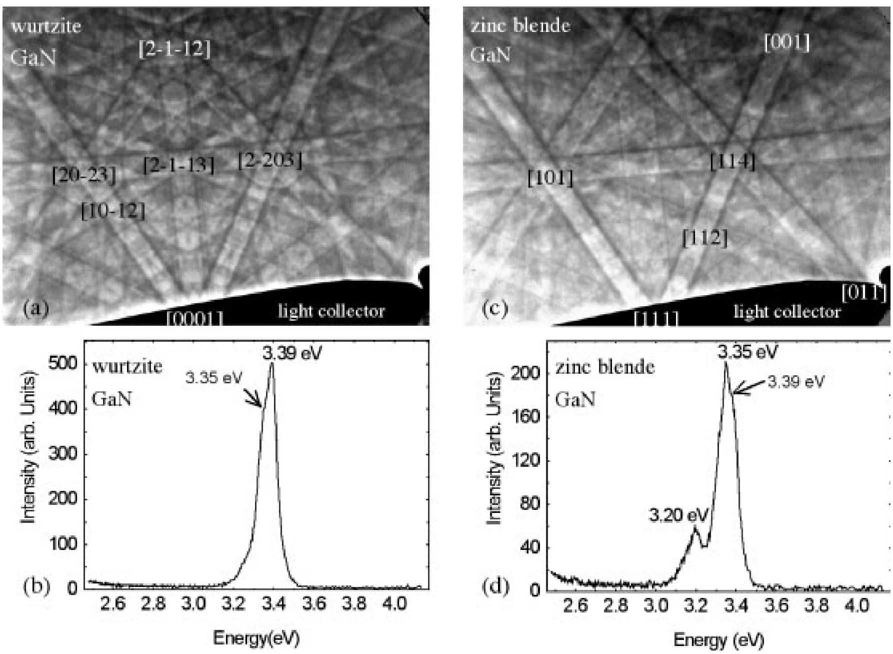

Fig. 4 a) EBSD pattern from wurtzite GaN, b) CL spectrum from same region of sample as a), c) EBSD pattern from zinc blende GaN, d) CL spectrum from same region of sample as c)

[2]) Monte-Carlo simulations of the electron trajectories [13]

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