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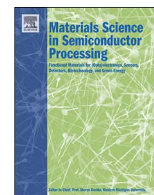

Reprint of: Electron channelling contrast imaging for III-nitride thin film structures

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ABSTRACT

Electron channelling contrast imaging (ECCI) performed in a scanning electron microscope (SEM) is a rapid and non-destructive structural characterisation technique for imaging, identifying and quantifying extended defects in crystalline materials. In this review, we will demonstrate the application of ECCI to the characterisation of III-nitride semiconductor thin films grown on different substrates and with different crystal orientations. We will briefly describe the history and the theory behind electron channelling and the experimental setup and conditions required to perform ECCI. We will discuss the advantages of using ECCI, especially in combination with other SEM based techniques, such as cathodoluminescence imaging. The challenges in using ECCI are also briefly discussed.

1. Introduction

III-nitrides are the only class of commercially available inorganic semiconducting materials with the potential to emit light from the infrared to the ultraviolet (with commercial devices available in the green to the ultraviolet part of the spectrum) with direct band gaps ranging from 0.7 eV for InN to 6.2 eV for AlN [1]. In the last 25 years, the development of a wide variety of nitride–based photonic and electronic devices has opened a new epoch in the field of semiconductor research. Nitride semiconductors are used in light emitters, photodiodes and high–speed/high–power electronic devices [2,3]. For these reasons nitride semiconductors have attracted much attention from both the consumer product industries and the defence sector, engendering intensive research with the aim of improving device efficiencies and reducing their costs.

One method for improving the performance of nitride–based devices is the reduction of the polar and piezoelectric fields which are a result of the wurtzite crystal structure and strain induced in device structures, respectively. This can be achieved by growing on nonpolar, *m*-plane (1–100) and *a*-plane (11–20), or semipolar, (11–22) and (20–21), planes. Growth of semipolar InGaN/GaN quantum well structures also enables the effective incorporation of higher concentrations of InN [4], improving the efficiency of amber and red nitride based light emitting diodes (LEDs).

A range of crystal growth technologies is being developed for the realisation of GaN substrates with large size (2–6 in.) and high quality, especially for the polar (0001) *c*-plane GaN [5]. However, the sizes of nonpolar and semipolar GaN substrates remain small and their cost is too high [6–8].

Heteroepitaxial growth on sapphire and silicon substrates is cheaper than growth on bulk GaN. However, heteroepitaxially grown nitrides suffer from a high density of extended defects such as threading dislocations (TDs), basal plane stacking faults (BSFs) and associated partial dislocations (PDs) mainly due to the large lattice mismatch between the heteroepitaxial substrate and the epilayer [9]. In addition to lattice mismatch, differences in thermal expansion coefficients cause biaxial stress to the epitaxial layer; for example GaN is compressively strained when *c*-plane sapphire is used as a substrate material [10]. Irrespective of the substrates, growth plane or growth techniques employed, extended defects are always present in the as-grown layers and have proven to be detrimental to device performance [11–14]. In order to optimise the growth and thereby improve the crystal quality, we require a rapid, non-destructive and cost-effective structural characterisation technique for detailed understanding of extended defects and their formation.

Recent advancements have made x-ray diffraction (XRD) a powerful tool for characterising nitride semiconductors, but there are several limitations, especially in using XRD to characterise non-polar nitrides [15]. Modified Williamson-Hall analysis is a widely used method to estimate stacking fault densities in nonpolar GaN thin films [16].

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However, the accuracy of this technique is limited to line densities above 10^4 cm^{-1}. Moreover, the applicability of this method is questionable as other superimposing effects, such as surface morphology and wafer bowing, may produce unphysical results [17]. At present, transmission electron microscopy (TEM) is the best known and most widely used technique for characterising individual threading dislocations and stacking faults [18–20] in nitride semiconductors. The need for sample preparation and the localised nature of the information acquired from TEM make other microscopic imaging techniques such as atomic force microscopy (AFM) – and the subject of this review, electron channelling contrast imaging (ECCI) – attractive complementary techniques to TEM.

We will begin this review by providing a brief history of electron channelling followed by theoretical and practical aspects of the technique. Through results from a wide range of nitride thin films grown by metalorganic vapour phase epitaxy (MOVPE), we will illustrate that ECCI can be used to reveal (i) individual dislocations, (ii) atomic steps, (iii) low angle tilt and rotation boundaries, and (iv) basal plane stacking faults and associated partial dislocations. We will also show that the range of magnifications and resolution afforded by ECCI in the SEM allows dislocation densities to be measured over a wide range of densities. The large field of view also allows the distribution of dislocations to be studied; for example we have observed long range ordering as well as clustering of dislocations. We will also discuss the advantages of using ECCI with other techniques in the SEM, especially in combination with cathodoluminescence (CL) imaging. Finally we will summarise our results and discuss a few remaining challenges to using ECCI for characterising nitride semiconductor thin films.

1.1. Brief history of ECCI.

The first observation of dislocation networks in thin foils of aluminium and copper recorded with backscattered electrons (BSEs) in a scanning electron microscope (SEM) was performed by Clarke in 1971 [21] followed by Stern and Kimoto in 1972 (imaging dislocations in molybdenite) [22]. The importance of using a field emission gun scanning electron microscope (FEG–SEM) was realised from their work and the first observation of dislocations (also in molybdenite) using a FEG–SEM was obtained by Pitaval et al. in 1976 [23–25]. The term ECCI was first used by Morin et al. in 1979 [26] who were able to image extended defects in semiconductors (Si) using ECCI. Seminal work from Joy and Newbury et al. for characterising metals [27], Wilkinson et al. in imaging misfit dislocations in $Si_{1-x}Ge_x$ thin films, and improvement in detector geometries opened up the possibilities of using ECCI for variety of materials [28,29]. Trager-Cowan and co-workers [30] were the first to apply ECCI to the imaging of threading dislocations in nitride semiconductors followed by Picard et al. [31] who used ECCI to investigate SiC [32], and $SrTiO_3$ [33]. Recently Carnevale and co-workers used ECCI to image misfit dislocations in GaP [34] and Yan et al. applied the technique to image antiphase domains boundaries in $LaSrMnO_3$ thin films [35]. Some of our own work [36–40] and recent work from our collaborators [41–43] have taken ECCI a step further as a quantitative technique for characterising nitride semiconductor thin films by resolving individual dislocation types over statistically significant dislocation distributions, opening up new possibilities for advanced materials characterisation.

1.2. Principle of electron channelling

Contrast from electron channelling can be used in two modes of operation [44,45]. The first of these is the acquisition of electron channelling patterns (ECPs) which allows the selection of the set of planes from which the electrons are diffracted. This procedure is referred to as selecting a diffraction vector, $\boldsymbol{g}$. This is analogous to choosing diffraction conditions in TEM. Detailed description of ECPs is beyond the scope of this review; more information on this topic can be found in the references [27,44,45]. The second mode of operation is obtaining ECC images, the main focus of this review. When the SEM is operated at a very high magnification, the angle between the scanned beam and the surface remains constant. As a result, changes in crystallographic orientation or changes in lattice constant due to local strain are revealed by changes in contrast in a channelling image constructed by monitoring the intensity of BSE as an electron beam is scanned over the suitably oriented sample. Images with resolution of the order of tens of nanometres can be obtained by ECCI. Extremely small changes in orientation (≈0.01°) [42] and strain are detectable, revealing for example low angle tilt and rotation boundaries and atomic steps and enabling extended defects to be imaged.

The conditions required to resolve individual dislocations in an ECC image are quite stringent: a small (nanometres), high brightness (nanoamps or higher), low divergence or high convergence beam (of order of a few mrad) electron beam is required [46,47]. These conditions are necessary to obtain good quality channelling contrast and they are met only in a field emission SEM (FEG–SEM). All the ECC images in the present work were acquired using an FEI Sirion 200 Schottky FEG–SEM with an electron beam spot of ≈4 nm, a beam current of ≈2.5 nA and a beam divergence of ≈4 mrad. It is also necessary to use a detection system that allows discrimination between those electrons leaving the sample which carry channelling information and those which have been diffusely scattered by the sample. An amplifier which can offset the diffuse background signal and a preamplifier to amplify the channelling signal is required. In the present work, the forescatter Si diodes, preamplifier and a signal amplifier were provided by KE Developments Ltd., now Deben, UK.

ECC images can be acquired in either forescatter geometry (sample tilted to between 30° and 70° to the impinging electron beam and the forescatterd electrons detected by a diode placed in front of the sample) [30] or the backscatter geometry (sample at approximately 90° degrees to the incident electron beam with the BSE detected by an electron–sensitive diode or diodes placed on the pole piece of the microscope) [48]. Fig. 1a and b shows the schematic of forescatter and the backscatter geometries respectively and Fig. 1c and 1 d shows the corresponding ECCI respectively. The ECC image shown in Fig. 1c is acquired by tilting the sample to 70° whereas the ECC image shown in Fig. 1d is acquired with sample approximately flat (not tilted). Note the images are not from the same part of the sample. The acquisition time required to obtain each image is typically less than a minute.

The backscatter geometry has the advantage that large samples, e.g., a full semiconductor wafer (depending on the size of the SEM chamber), could be imaged and the results obtained may be more easily compared to a TEM diffraction image. The forescatter geometry requires tilt correction of the acquired images but provides a larger channelling signal and therefore channelling images with superior signal to noise. The forescatter geometry is the one used in our present work.

2. Results and discussion

2.1. ECCI of GaN thin films

In ECCI, vertical threading dislocations appear as spots with black–white (B–W) contrast; this is shown in Fig. 2a, an ECC image acquired from a 1600 nm thick GaN thin film grown on a sapphire substrate in which a typical threading dislocation is highlighted by a black circle [49]. The (B–W) contrast is basically due to strain fields around a dislocation. For materials with a wurtzite crystal structure such as GaN, we have previously developed a simple geometric procedure to identify a given threading dislocation as edge, screw, or mixed type by exploiting differences in the direction of the black–white contrast between two ECC images acquired under 2–beam conditions from two symmetrically equivalent crystal planes whose diffraction vector

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