Assessment of lattice strain, rotation and dislocation content using electron back-scatter diffraction

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Abstract. Cross-correlation based analysis methods have been developed for electron back scatter diffraction (EBSD) patterns that improve the angular sensitivity to ~10^{-4} rads. This enables EBSD to be used to study the much smaller misorientations and even local elastic strain fields that are typical in semiconducting materials. Mapping of the lattice rotations and elastic strain variations provides sufficient detail for quantitative analysis of the threading dislocation density through the Nye tensor. The analysis will be briefly described and applications given to GaN and Si/SiGe based systems. Measurements of tilt, twist and elastic strain variations in GaN layers on basal plane sapphire will be reported and compared to results for some epitaxial lateral over grown (ELOG) GaN samples. The effects of misfit interfacial dislocations on the spatial distribution of the full strain and rotation tensors in a partially relaxed SiGe layer will also be shown.

1. Introduction
Over the last two decades electron back scatter diffraction (EBSD) has become an established tool for microstructural characterization of structural materials, particularly metallics [1, 2]. Although EBSD has been used to identify substrate – epilayer orientation relationships [3, 4] and grain boundary types in multicrystalline Si [e.g. 5, 6] its uptake for characterization of functional and semiconducting materials has been much more limited than for metallics. To a significant extent this can be attributed to the angular resolution limit of ~0.5° (~10^{-2} rads) on lattice misorientations. Although acceptable for metallics which are most often polycrystalline and with relatively high lattice defect density, to date EBSD measurements have been too insensitive for semiconductors which are often single crystal and with relatively low lattice defect content.

Early attempts at using cross-correlation-based analysis by Troost et al [7] and Wilkinson [8-10] have recently seen significant development and improvement by Wilkinson, Meaden and Dingley [11, 12] to provide the sensitivity required for characterisation of many semiconductor samples. The approach relies on the fact that elastic strains and small rotations cause slight shifts in features, such as zone axes, within the EBSD patterns that can be measured to high precision using cross correlation techniques. The method will be described in more detail below. EBSD is now emerging as a laboratory based technique that, in some ways, is competitive with many large scale centralised synchrotron facilities for high spatial resolution strain mapping.

The cross-correlation-based analysis of EBSD patterns has been applied to unrelaxed SiGe blanket films on Si in planview [8, 10] and elastic relaxation caused by sectioning [12] and by growth on mesas of finite width [13-15]. Vaudin et al [16] have used the method to study stresses generated by
edge indents in Si. They found excellent agreement between EBSD and micro-Raman measurements and assessed the stress sensitivity to be ~10 MPa. Very recent work by Tomita et al [17] have also shown good agreement between EBSD and micro-Raman measurements for strained Si on insulator and SiN/Si systems. Villert et al [13] have made measurements across the depth of a bent Si sample finding good agreement with theory. Excellent spatial resolution of the technique is evident in studies on cross-sections of AlGaN/GaN heterostructures by Ishido et al [18] which includes a linescan across a 5 nm AlN/25 nm GaN multilayer in which the 50 repeats can be distinguished. Alternative approaches for using EBSD to analyze strain in semiconductors include the use of pattern blurring to demark regions of high elastic strain gradients [19], and a modified three parameter Hough transform [20].

2. High Accuracy EBSD Methodology

EBSD systems comprise a ~40 mm diameter scintillator screen mounted in front of a highly tilted (~70°) sample and viewed with a low light level CCD camera. In most cases, including in our systems, the CCD and screen are coupled using a fast lens, though more direct coupling with a fibre optic bundle is used in some cases. The camera is mounted outside the SEM vacuum chamber and connected to a PC via a ‘firewire’ interface. TSL OIM software was used to control the SEM beam position and acquire the EBSD patterns at the full (~1k by 1k by 12 bit deep) resolution of the camera. The patterns are recorded to hard disc for subsequent batchwise analysis for strain variation using CrossCourt software from BLG productions [21].

The following describes the conceptual basis of strain analysis using EBSD. Changes in elastic strain and lattice rotations cause small shifts in the positions of zone axes and other features in the EBSD patterns. The magnitude and direction of these shifts vary with position across the pattern in a systematic way that has been related to the size and nature of the strain and rotation state within the diffracting volume [10-12]. One pattern from the series to be analyzed is chosen as a reference pattern. Ideally the reference pattern should come from a location on the sample for which the strain is known, or in many cases is known to be zero. A set of square sub-regions are defined across the patterns. The shifts relative to positions in the reference pattern of diffraction features in each of these sub-regions is then determined using cross-correlation analysis for each of the test patterns. The cross-correlation functions are calculated via the Fourier domain, with a weighting function used to prevent issues with aliasing. Within the Fourier domain band pass filtering is applied to remove high frequency noise, and low frequency background intensity variations. Details of the significance of these image analysis procedures on the resulting pattern shift measurements are given in [12]. The pattern shifts in 256 by 256 pixel sub-regions can be measured to better than ±0.05 pixels though of course pattern quality and signal to noise influence this noise floor. The magnitude of the cross-correlation function peaks are also recorded. These are normalized to give unity for an autocorrelation and a geometric mean over all sub-regions on a pattern is calculated and used to assess the quality of the pattern matching. Typically for deformed metallics peak heights below ~0.3 are considered unsatisfactory and the corresponding data are discarded as unreliable. For most semiconductor applications the peak heights are generally considerably higher due to the lower lattice defect content.

To distinguish the effects of strains and tilts it is necessary to determine the shifts in the positions at multiple sub-regions of the EBSD patterns. We generally use at least 20 sub-regions combined with either least square error, or iterative robust fitting methods to obtain the strain and rotation tensors that best fit the measured pattern shifts. Infinitesimal deformation theory is used in the analysis. The quality of this best fit solution can be assessed by calculating the (weighted) mean angular error in the shifts actually measured at each sub-region of the pattern compared to shifts expected on the basis of the best fit solution for strain and rotation. If the measured strains and rotations are larger than the mean angular error there is good confidence in the measurements, but if they are smaller then they are below the noise limit and should be disregarded.
Variations in the lattice rotations about three orthogonal axes and the three shear strains are found directly, however the analysis is insensitive to hydrostatic dilatation and as a result only the differences between pairs of normal strains are determined. To separate these normal strains we can assume that the sample surface (normal along $x_3$) is traction free so that we can write

$$\sigma_{33} = 0 = C_{13} \varepsilon_{11} + C_{23} \varepsilon_{22} + C_{33} \varepsilon_{33} + C_{34} \varepsilon_{23} + C_{31} \varepsilon_{31} + C_{36} \varepsilon_{12}$$

(equation 1)

in which $C_{ij}$ and $\varepsilon_{ij}$ are the known single crystal elastic constants and elastic shear strains in the sample reference frame. This equation along with measurements of the differences ($\varepsilon_{11} - \varepsilon_{33}$) and ($\varepsilon_{22} - \varepsilon_{33}$) allows all 6 strain terms to be isolated. This traction free surface condition is taken to be acceptable in most instances in the structural metallics, however, for measurements very near abrupt interfaces in semiconductors it is possible for the stress (and/or elastic strain) field to vary significantly within the sampling depth of EBSD (a few tens of nanometers). In any such instances the separation of the normal strains should be considered in more detail. The elastic constants often simplify when measurements are made on a sample with surface normal along a symmetry axes.

3. Applications

3.1. Epitaxially Lateral Over Grown GaN

Epitaxial lateral overgrowth (ELOG) [22] and pendeoepitaxy [23] have been shown to give a significant reduction in the density of threading dislocations in GaN layers grown on sapphire and SiC substrates. In the laterally grown wing regions dislocation densities of $10^6$ cm$^{-2}$ can be achieved.

![Example EBSD pattern from GaN grown directly above the central seed region of an ELOG sample.](image)
compared to $10^{10}$ cm$^{-2}$ in the seed regions. In ELOG GaN the crystal quality (defect density), crystal orientation, stress and strain thus all vary with position relative to the underlying mask structure. EBSD measurements were conducted on an ELOG GaN sample grown by Dr Ian Watson and co-workers at the Institute of Photonics, University of Strathclyde [24]. The sample studied (STR129/3) consisted of a (0001) sapphire substrate on which a 2.5 µm planar GaN seed layer was grown. Over the seed layer a 4 µm thick ELOG GaN layer was grown through a mask of 9 µm wide 200 nm thick SiO$_2$ stripes repeated with a period of 16 µm. The sample studied had been grown to a point just beyond coalescence. An example pattern from the seed region is shown in figure 1, and preliminary measurements on the sample were reported in [25]. The measurements were taken on a JEOL 6500F, operated at 20 keV and an estimated probe current of ~3 nA with sample tilted 60° from normal incidence. A line scan was made on the top surface of the ELOG GaN, using a step size of 80 nm and spanning ~3 repeats of the structure. A reference pattern was selected within the central seed region and used within CrossCourt to conduct the cross-correlation measurements at 50 256 by 256 pixel sub-regions and to make the subsequent analysis for lattice rotations and strain variations. Figure 2 shows some of the outputs from the analysis. Two data quality parameters described in section 2 above are routinely used to assess the data. The first of these is the geometric mean of the cross-correlation peak heights, which remains above 0.9 throughout but shows lowest values near the wing-wing coalescence boundaries.

The second data quality parameter is the mean angular error which is significantly below $10^{-4}$ rads throughout the dataset but shows distinct peaks above the wing-wing coalescence boundaries, and lower values of 2-3x$10^{-5}$ rads above the seed regions. Visual inspection of the patterns indicates a very subtle loss of acuity near the wing-wing coalescence boundaries though the effect is very minor and possibly subjective and steered by knowledge of the trend shown in figure 2a.

Selected components of the lattice rotation and strain variations are shown in figure 2b. The terms not shown all had only minor variations along the line scan, with the exception of the out of plane normal strain $\varepsilon_{33}$ which varied in the opposite sense to $\varepsilon_{11}$ and to a lesser magnitude as expected from equation 1. These near zero results are all expected from the traction free surface boundary condition, and the plane strain like conditions expected from the long extent of the stripe structure along the $x_2$ axis.

There is good reproducibility in the results from the ~three periods of the mask pattern. The tilts $w_{12}$ about the $x_3$ axis parallel to the stripe direction show marked changes at the seed-wing interface and wing-wing coalescence boundary (figure 2b). At seed-wing interfaces tilts of ~0.006 rad (~0.3°) occur, the sense of the tilt being opposite for the wings grown from either side of a given seed region. As a consequence the tilt changes by ~0.012 rads (~0.6°) at wing-wing coalescence boundaries. The change in tilt occurs more abruptly at the wing-wing coalescence boundary than at the seed-wing interface. There is also a significantly smaller and unexpected variation of lattice rotations $w_{12}$ about the surface normal (i.e. twists) which again show a gradual increase in size across the wing regions and a more abrupt change at the wing-wing coalescence boundaries.

The strain variations are all relative to the unknown strain state at the reference point. The 'in plane' strain $\varepsilon_{11}$ perpendicular to the stripes varies considerably and becomes progressively less compressive as we move from the centre of a seed region toward the wing regions. However near the wing-wing coalescence boundary there is a very pronounced but localized increase in the 'in plane' compression.
3.2. GaN Buffer Layer on Sapphire

The second example application is to the analysis of tilt and twist mosaics in a sample of 900 nm thick GaN film grown on (0001) oriented sapphire single crystals by metalorganic vapour phase epitaxy (MOVPE). The sample was one of a set grown by Prof Peter Parbrook and co-workers while he was at the EPSRC National Centre for III-V Technologies, University of Sheffield. EBSD analysis was performed on a JEOL JSM 6500F using a beam energy of 15 keV, and current of approximately 10 nA. The sample was tilted toward the EBSD detector by 70° from normal incidence about the [101 ¯ 0]GaN axis. A 6 µm by 6 µm area was mapped using a square grid with a 100 nm step size. Pattern shifts were again measured at 50 sub-regions across each pattern and were used to determine a least square error best fit strain and rotation tensor. A pattern toward the top left hand
corner of the map was used as a reference pattern. The mean of each strain and rotation component was determined over the map and these values then subtracted so that the maps shown in figure 3 show the deviations from the mean value. The mean peak height map (figure 3a) shows some reduction from the top to bottom which corresponds to a loss of pattern contrast due to a build-up of carbonaceous contamination on the sample surface during the scan. The mean angular error (figure 3b) gives an average value of $5 \times 10^{-5}$ rads for the entire dataset, and shows a slight decrease in the data quality towards the lower part of the map due to a build-up of surface contamination during the scan.

The axes system used to describe the strains and rotations are given in figure 3c. The twist rotations $w_{12}$ about the surface normal $x_3$ are clearly larger than either of the tilt rotations $w_{23}$ and $w_{31}$ about the two in plane axes $x_1$ and $x_2$. Elastic strain variations are generally smaller than the lattice rotations, though this difference is not nearly as marked as for deformed metals [26, 27]. In this crystal orientation the two out of plane shear strains $\varepsilon_{23}$ and $\varepsilon_{31}$ simply scale linearly with the corresponding stress terms $\sigma_{23}$ and $\sigma_{31}$, which should go to zero at the traction-free surface. The results do indeed show that variations in $\varepsilon_{23}$ and $\varepsilon_{31}$ are noticeably smaller than the other strain components in accord with the expected boundary condition. Figure 4 gives histograms showing the distribution of different rotation components about the mean value. Comparison is made to rotations measured in a similar way in a near perfect GaAs single crystal which effectively show the noise level on the measurements. For the GaAs data the histogram shows the distribution averaged over the three rotation components. These ‘pseudo-rocking curves’ show quantitatively the greater spread in the twist mosaic compared to the tilt mosaics that was visually noticeable in the maps (figure 3). The distributions of elastic strains around the mean value are seen to be significantly smaller than for the rotation and only just above the noise level indicated by data from the GaAs sample. A greater spread for the twist mosaic than for the tilt was also seen in the Hough-transformed based EBSD measurements by Rao et al [28] though in the current analysis the angles are an order of magnitude smaller indicating a lower defect density in the GaN film.

Nye [29] gave an elegant geometrical construction allowing the lattice curvature to be linked to the density of geometrically necessary dislocation (GND) content within the crystal. Given the densities, line and Burger’s vectors of a set of dislocations the lattice curvature of the crystal can be calculated directly and unambiguously, however, the reverse process of determining the dislocation content from the curvatures leads to ambiguities in most cases due to there being too many possible dislocation types. However, a lower bound GND density can be established which supports the measured curvatures and minimizes some other term such as the total dislocation line length or energy (an L1 minimization). Kroner [30] extended this analysis to include the elastic strain gradients that Nye had assumed to be small (which we have found to be the case in deformed metals). In the GaN sample although the elastic strain variations are smaller than the rotations the difference is not so great that the strain field can be ignored in the analysis. When elastic strain gradients are included in the analysis only three of the nine terms in the Nye dislocation tensor can be found and these relate to dislocation types that thread through the sample surface [31]. For this GaN sample we assume that the three measured lattice curvatures (rotation and elastic strain gradients) are accommodated by combinations of screw dislocations along [0001], and three possible edge dislocation types with [0001] line direction and $\frac{1}{3}<11\bar{2}0>$ Burgers vectors. Even with this small and simple set of possible dislocation types there is ambiguity in solving for the dislocation densities which arises from the non-independence of the three edge dislocations. Of the many solutions that support the measured curvature we select one that also minimizes the total line energy in which we account for the differing magnitudes of the Burgers vectors and differentiate between screw and edge types. Maps showing the density distributions of the four dislocation types are given in figure 5. As expected the larger twist mosaic propagates through the GND density analysis to result in a greater density of $<a>$ type edge dislocations.
Figure 3. High accuracy EBSD maps of a 900 nm thick GaN layer grown on sapphire. (a) cross-correlation peak height, (b) mean angular error (rads), (c) axis system and colour scale for strains and rotations, (d), (e), (f) rotations about the $x_3$, $x_1$, and $x_2$ axes respectively, (g), to (l) variations from the mean of elastic strains $\epsilon_{11}$, $\epsilon_{22}$, $\epsilon_{33}$, $\epsilon_{12}$, $\epsilon_{23}$, and $\epsilon_{31}$ respectively. The scalebars are 2 $\mu$m long and the step size was 100 nm.
Figure 4. Distributions of (a) tilt and twist rotations, (b) normal strains, and (c) shear strains in a 900 nm thick GaN layer on sapphire. Solid bars show similar distributions for a GaAs single crystal averaged over the selected rotation or strain components.

Figure 5. Maps showing a low energy solution for the threading dislocation density distribution in GaN region shown previously in figure 4. Top row shows three edge dislocation types, bottom row the screw type. Red/Green lines in the ‘clockface’ at top-left of each image indicate the Burgers vector direction. Colourscale gives dislocation density in lines/m².
3.3. SiGe/Si – plastic relaxation

The final illustration to be given here is from a series of SiGe/Si samples supplied a considerable time ago by the B. T. Research Laboratories (Martlesham Heath) by Tuppen, Gibbings and Hockly [32]. The particular sample studied consisted of a 1.2 µm thick Si$_{1-x}$Ge$_x$ alloy grown by molecular beam epitaxy on a Si (001) substrate. The nominal Ge content was 10%, for which the epilayer thickness is well above the critical value required for strain relaxation by interfacial misfit dislocations [32]. SEM conditions employed were 15 keV beam energy, a beam current of ~3 nA, with a relatively long exposure time for the EBSD pattern acquisition of approximately 1 second. An 80 µm by 80 µm map was made with a 0.5 µm step size. The data was collected on a LaB$_6$ SEM and a slight scan instability can be seen in the maps but does not detract from the discussion and striking comparison with the GaN data. The cross-correlation analysis was undertaken in an off-line, batchwise, automated process using CrossCourt 3 software [21], using 32 sub-regions for the analysis. A reference pattern was selected at the centre of the map. Within the mapped area the mean angular error was found to have an average value of 1.1x10$^{-4}$ rads, with 5% of points having values less than 0.9 x10$^{-4}$ rads, and 95% being below 1.3x10$^{-4}$ rads.

As the strain and rotation at the reference point are not known the results are presented in figure 6 as maps showing the deviation of the individual strain and rotation components from their (unknown) mean values. X-ray diffraction methods could be used to establish the mean strain values. The map of lattice rotations ($w_{12}$) about the surface normal (figure 6a) show two orthogonal sets of features running along the near vertical [110] and near horizontal [11 ¯ 0] axes. The rotations about the two in-plane axes are larger and only show the features running near parallel with the rotation axis, i.e. vertical features for the $w_{31}$ component and horizontal for the $w_{23}$ component. The features are clearly generated by the two orthogonal sets of misfit dislocation lines generated by the partial relaxation of the large misfit stress initially present in the epilayer. The edge component of these dislocations generate rotations about their line direction, so that each set of dislocations is only present in either the $w_{31}$ map or the $w_{23}$ map, but not present in the other. Rotations about the surface normal are generated by the screw components of any dislocation with line direction parallel to the surface plane and so both sets of dislocations appear in the $w_{12}$ map. The screw components of the dislocations also cause a (tensor) shear strain $\varepsilon_{12}$ in the surface plane (figure 6g), but do not contribute to the normal strains. Again the effects of the two sets of dislocations are separated in the maps of the two in plane normal strains ($\varepsilon_{11}$ and $\varepsilon_{22}$) with the dislocations not affecting the strain along their line directions. As the edge components of the interfacial dislocations relieve the misfit strain there is a concomitant relaxation of the tetragonal distortion which is seen as a change in the out of plane strain $\varepsilon_{33}$ which is affected by both sets of dislocations.
Figure 6. High accuracy EBSD maps of a 1.2 µm thick SiGe layer grown on Si. (a), (b), (c) rotations about the $x_3$, $x_1$, and $x_2$ axes respectively, (d), (e), (f), (g), (h), (i) variations from the mean of elastic strains $\varepsilon_{11}$, $\varepsilon_{22}$, $\varepsilon_{33}$, $\varepsilon_{12}$, $\varepsilon_{23}$, and $\varepsilon_{31}$ respectively. The scale bars are 5 µm long and the step size was 500 nm.
4. Summary

The development of cross-correlation based analysis of EBSD patterns has generated a step change in the sensitivity of the method and dramatically increases its scope for characterising many semiconductor samples. The residual elastic strains and lattice rotations can be measured and mapped to approximately $\pm 10^{-4}$ (rads). The method is now very straightforward to use and data sets of the size presented here take up to an hour or two to collect and a similar time to analyse to the standard used here. Quicker analysis is possible by using fewer sub-regions for cross-correlation which can be useful if a faster preliminary look at the data is required. All 9 components of the displacement gradient tensor (strain and rotations) are provided with minimal experimental complexity, so that the fullest representation of the strain state is generated, from which standard tensor analysis can be used to provide, for example, principal strains, or maximum resolved shear stresses (though for stresses single crystal elastic constant are required). A lower bound estimate of the dislocation density is also possible using the Nye/Kroner framework.

The main challenges for further development of the method for applications to semiconductors are (i) spatial resolution, and (ii) absolute (as opposed to relative) strain measurement. Spatial resolution will continue to be a major concern in applying EBSD to some device structures. Although in some instances the goals seem unattainable, in others there is already sufficient spatial resolution available. Improvements in spatial resolution are likely to come from improved detectors allowing operation at lower beam energies, and further into the future with energy filtering/discrimination. The need to have a reference pattern from a region of known strain is an issue receiving considerable attention at the current date [33]. It seems likely that dynamical diffraction simulations of EBSD patterns [34, 35] may have sufficient fidelity to the real patterns to allow their use as reference patterns (simple kinematic simulations are not sufficient). The challenge has now become an experimental one of determining the detector geometry and distortions with sufficient accuracy so as to avoid introducing 'phantom strains' through small artifacts in the cross-correlation measurement of shifts. This challenge is likely to be met in the next year or so and will give a further step change in the scope of problems that can be tackled with EBSD.

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