

Novel Applications of Electron Channeling Contrast Imaging

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Microstructural characterization plays a critical role in crystalline semiconductor materials research, and such work has traditionally been performed using transmission electron microscopy (TEM). However, sample preparation for TEM work can be time intensive, and TEM specimen preparation can often damage the sample and features of interest, compromising their integrity. Electron channeling contrast imaging (ECCI) can serve as an alternative to traditional TEM in many instances, and because it is performed in a scanning electron microscope (SEM), ECCI effectively avoids many of these sample preparation issues. ECCI has a broad range of applications still to be exposed, and in this contribution, two such applications are discussed: quantum dot (QD) visualization and phase separation detection.

We have demonstrated ECCI to be an ideal characterization tool for visualizing of defects within heteroepitaxial semiconductor structures [1], providing unprecedented access to valuable information regarding the formation and evolution of defects. We present here preliminary results regarding the further expansion of ECCI as applied to other epitaxial (opto)electronic materials systems. This includes, to our knowledge, the first demonstration of the use of ECCI for the visualization of subsurface epitaxial III-V QDs (InAs/GaAs) and the detection and characterization of crystallographic defects and phase separation in a III-V alloy (InGaP). All ECCI in this work was performed, on as-grown samples, using a FEI Sirion field-emission SEM, fitted with a pole piece mounted annular back scatter electron (BSE) detector with an accelerating voltage of 30 kV, a spot size of 5 (2.4nA), and a working distance of 5 mm.

Application to Complex III-V Alloys: The $\text{In}_x\text{Ga}_{1-x}\text{P}$ ternary alloy system is of major importance to nearly all III-V based PV technologies. Nonetheless, InGaP is also a material system fraught with complex issues, such as atomic ordering and significant phase instabilities, making it a prime candidate for rapid microstructural characterization methods. Figure 1 presents preliminary data from an InGaP epilayer calibration growth nominally lattice-matched to $\text{GaAs}_{0.9}\text{P}_{0.1}$. The X-ray diffraction (XRD) reciprocal space map in Fig. 1(a) indicates a missed compositional target, and the splitting of the InGaP peak suggests phase separation. Low magnification ECCI, Fig. 1(b), shows a strong mottled appearance with no apparent correlation to any surface structure. Given the indication of phase separation provided by XRD, and the high sensitivity of ECCI to strain, the most likely source of this contrast is indeed phase separation, where the two lattice-mismatched phases possess different levels of strain, both with respect to the underlying substrate and each other; two-beam TEM yields similar contrast appearance in the presence of phase separation, depending upon the degree and extent of the separation [2]. Work is in progress to verify the identification of phase separation via ECCI in this sample and others.

Of additional note here is that in the high magnification micrograph in Fig. 1(c) a large number of crystal defects – threading dislocations and stacking faults – are visible. The ability to visualize these defects despite the apparent phase separation, which in addition to general alloy scattering contributes to the degradation of the channeling quality, provides demonstration that ECCI is still a useful characterization technique in crystalline materials that are highly non-uniform.

Application to Epitaxial Quantum Dots: Epitaxial QDs within the III-V compound semiconductor materials system typically form via the Stranski-Krastanov mechanism. Upon encapsulation, the QDs exert a high degree of strain on the surrounding material, producing strong strain fields that should strongly scatter the channeling electrons. Therefore, ECCI should enable the imaging of embedded QDs in plan-view geometry. To this end, Figure 2 presents ECCI micrographs of a single layer of InAs QDs embedded within a GaAs host at multiple imaging conditions. Clearly visible in Figs. 2(b) and 2(c) are small, round features consistent with the appearance of QDs (or their strain fields), while the standard BSE image in Fig. 2(a) shows no significant surface features. With the combination of ECCI's capabilities for imaging crystallographic defects and this demonstration of QD imaging, ECCI could be a powerful tool in QD research, enabling rapid characterization of such aspects as embedded QD density, size/shape, and uniformity, as well as investigation of potential QD-induced defect formation.

Conclusion: Here, we demonstrate the use of ECCI for nondestructive analysis of, to our knowledge, two new areas: phase separation in complex alloys and subsurface epitaxial QDs. In both cases the results are comparable to those achieved via TEM analysis, but with the advantage of no specimen preparation and the use of SEM instrumentation. These extended applications of ECCI prove it to be an invaluable technique for PV characterization.

[1] S. Carnevale, J. Deitz, *et al*, Appl. Phys. Lett. **104** (2014), p. 232111.

[2] N. J. Quitoriano and E. A. Fitzgerald, J. Appl. Phys. **102** (2007), p. 033511.

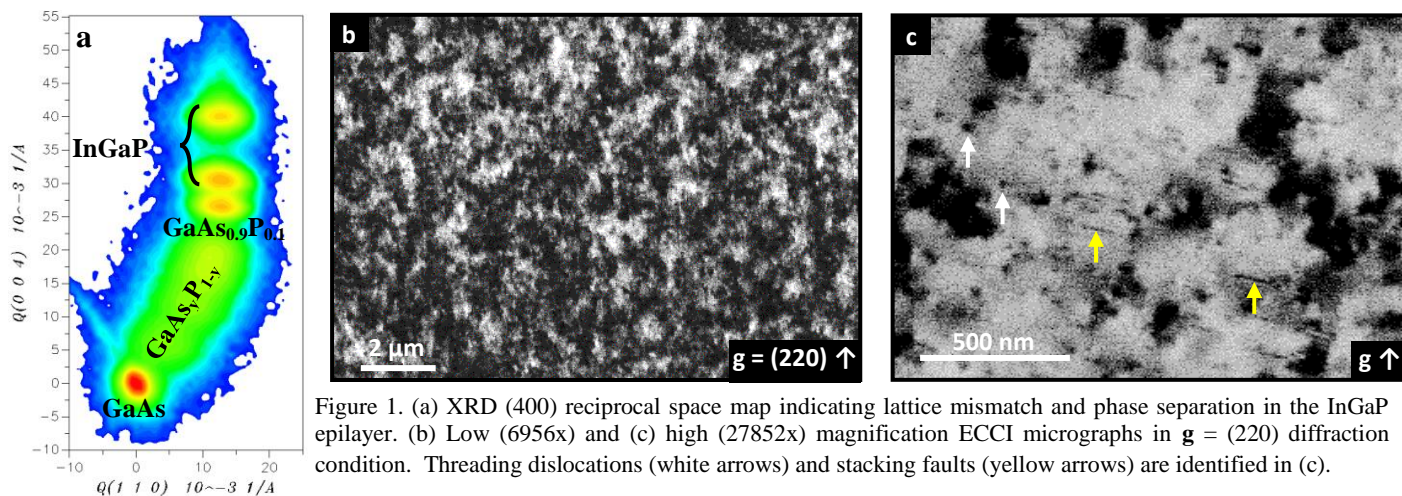


Figure 1. (a) XRD (400) reciprocal space map indicating lattice mismatch and phase separation in the InGaP epilayer. (b) Low (6956x) and (c) high (27852x) magnification ECCI micrographs in $g = (220)$ diffraction condition. Threading dislocations (white arrows) and stacking faults (yellow arrows) are identified in (c).

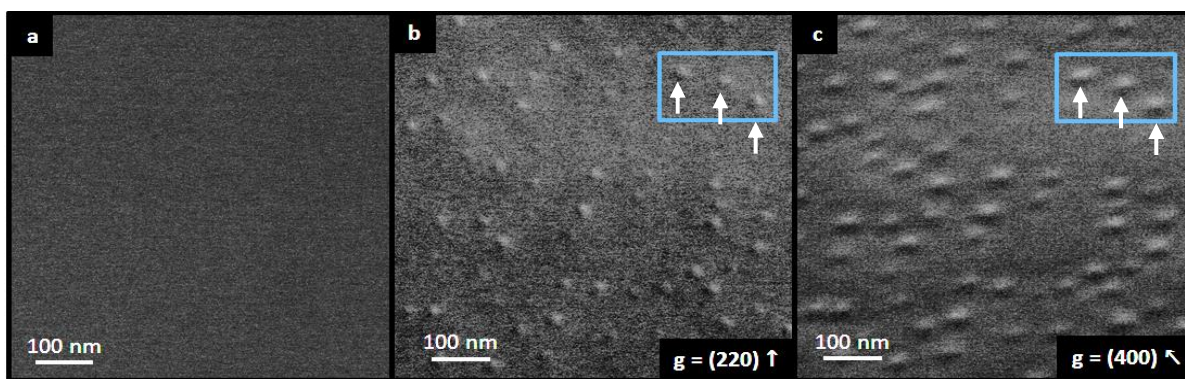


Figure 2. Single-layer InAs QDs in GaAs host as imaged via (a) standard surface-normal BSE, (b) $g = (220)$ ECCI, and (c) $g = (400)$ ECCI. The same region is picture in (b) and (c), and the blue rectangle surrounding three QDs, marked with arrows, provides frame of reference.