

Imaging Extended Defects in Low Z materials using Electron Channelling Contrast Imaging – New Approaches and Challenges

G. Naresh-Kumar*, A. Alasamari, Ben Hourahine and C. Trager-Cowan

Department of Physics, SUPA, University of Strathclyde, Glasgow, UK.

* Corresponding author: naresh.gunasekar@strath.ac.uk

Metrology of crystalline defects is crucial for the understanding and development of a wide range of novel materials. Hence there is a huge demand from both academia and the semiconductor industry for extended defect characterisation tools which are simultaneously rapid to use, non-destructive and capable of sampling larger areas on the nanoscale. Electron Channelling Contrast Imaging (ECCI) provides the required capability for various materials through use of scanning electron microscopes [1-3]. While images of extended defects, similar to those observed in plan-view transmission electron microscopy, can be obtained using ECCI (understandably with lower spatial resolution), the uptake of the technique has not been widespread. One of the major challenges limiting the use of ECCI in the wider material science community is challenges associated with characterising low atomic weight, topography-dominated and insulating samples. In the present work, we demonstrate a much wider applicability of ECCI, including for low atomic weight and insulating materials for defect metrology using gaseous secondary electron detectors (GSEDs) [4] in a field emission variable pressure scanning electron microscope. We show the advantage of performing ECCI in the gaseous environment in this case water vapour which plays a dual role in reducing the surface charges as well as acting as an amplification medium, thus providing high signal to noise micrographs. We show example from technologically important semiconductor material such as AlN, a low atomic weight and a wide-band gap material.

Gaseous secondary electron detectors collect all the electrons produced by the ionised gas, hence they can produce images with topographic as well as diffraction/channelling contrast. Figure 1a shows an ECCI micrograph from an AlN thin film collected using an off-axis gaseous secondary electron detector revealing surface steps (similar to those observed using atomic force microscopy) and threading dislocations due to diffraction/channelling effects. Figure 1b shows the ECCI micrograph simultaneously acquired from the same area using a conventional solid-state forescatter detector. There is no difference observed for the dislocation contrast from the ECCI micrographs acquired using the GSED and forescatter detectors, although the latter collects only the high energy backscattered electrons whereas the former collects secondary electrons and backscattered electrons. However, careful inspection reveals minor differences between the images. Surface steps are not so clear in Fig. 1b, since the forescatter detector (collecting only backscattered electrons) is not in an optimised position to enhance the topographic contrast. The total threading dislocation density in the AlN thin film estimated through ECCI is $1.2 \pm 0.3 \times 10^9 \text{ cm}^{-2}$. Since the AlN thin film is a single crystal, it was also possible to acquire electron channelling patterns using the GSED, as shown in Fig. 1c. The solid yellow circle shows the pattern centre corresponding to the optical axis of the SEM, which allows us to determine the diffraction conditions (g vector) for classifying various extended defects [2]. The image in Fig. 1d shows the electron backscatter diffraction pattern (EBSD) pattern acquired using the same camera/detector position as the ECCI micrograph shown in Fig. 1b.

While the use of ECCI in variable pressure scanning electron microscope is particularly useful for characterising low Z, insulating and wide band gap semiconductor materials, we stress that the methodology we have demonstrated is applicable to all kinds of crystalline samples provided appropriate imaging conditions are used [5].

References:

- [1] Zaefferer, S. and Elhami, N-N., *Acta Materialia* **75** (2014), p. 20.
 [2] Naresh-Kumar, G. et al., *Materials Science in Semiconductor Processing* **47**, (2016), p. 44.
 [3] Kriaa, H., Guitton, A. and Maloufi, N. *Scientific Reports* **7**, (2017), p. 9742.
 [4] Danilatos, G. D. *Journal of Microscopy*, **245** (2012) p. 171.
 [5] The authors acknowledge support from the EPSRC grant; “Quantitative non-destructive nanoscale characterisation of advanced materials” (EP/P015719/1).

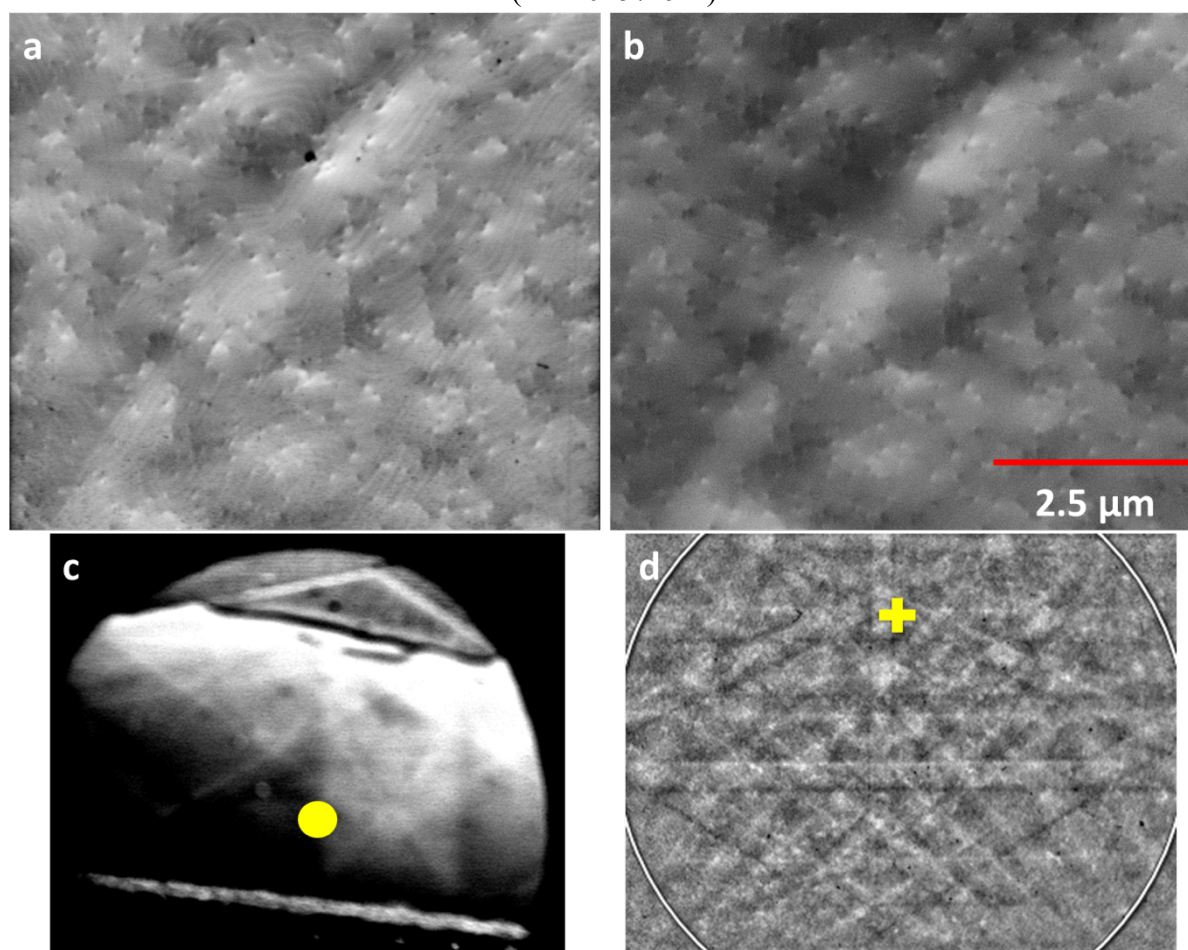


Figure 1. (a) ECCI of AlN thin film acquired at 64.6° sample tilt using the off-axis gaseous secondary electron detector (b) ECCI using conventional forescatter detectors underneath the electron backscatter diffraction detector, (c) electron channelling pattern corresponding to the ECCI micrographs, (d) EBSD pattern revealing the estimated pattern centre (yellow cross). The solid yellow circle in the electron channelling shows the diffraction conditions at which the ECCI micrograph is acquired. The images are acquired using 20 keV incident beam energy with a working distance of with a working distance of ≈ 11 mm and a gas pressure of 0.6 mbar.