High-angle transmission electron diffraction in a scanning electron microscope

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Keywords: scanning transmission electron microscopy, electron diffraction, image plates, quantification

Scanning transmission electron microscopy (STEM) is well suited to achieve sensitive material (Z-)contrast. This applies especially in high-angle annular dark-field (HAADF) mode where contrast is mainly attributed to incoherently scattered electrons. In this work it is shown that diffraction contrast cannot be neglected even at large scattering angles which are covered by a HAADF STEM detector as already exploited by Keller and Geiss in transmission electron backscatter diffraction [1].

We use a FEI Strata 400S scanning electron microscope equipped with a segmented semi-conductor STEM detector at electron energies below 30 keV. This detector has a bright-field segment and ring-like segments for dark-field and HAADF imaging covering scattering angles up to 700 mrad. The HAADF ring is further divided in twelve azimuthal segments which can be selected separately for image acquisition. Due to negligible knock-on damage at these low energies radiation sensitive materials can be investigated.

Molecular beam epitaxy (MBE) was applied to grow the sample examined in this work. A Si(111) wafer was covered with a 120 nm thick AlN layer followed by a GaN layer of 140 nm thickness. Thin TEM lamellae were prepared by focused-ion-beam (FIB) milling to investigate the sample material in a cross-sectional perspective. The lamellae were thinned with a wedge-shaped thickness profile which is useful for thickness determination.

Figure 1 shows a HAADF STEM cross-section image of the Si-substrate, AlN and GaN layers and the Pt-rich protection layer on top and was taken at 25 keV. Due to the wedge-like thickness profile, the sample thickness increases from left to right and remains then constant at 120 nm. Both epitaxially grown layers show various contrasts. Dislocations (indicated by arrows in Figure 1) as known from conventional TEM images can be observed. Additionally, intensity variations can be found in the GaN layer between block-like regions (indicated by dashed lines in Figure 1). To exclude that this contrast stems from compositional changes a tilt series and a series of images with different azimuthal HAADF segments were recorded. Both image series show change and even contrast inversion of the layers. Since material contrast should not be affected by sample tilting, this is a strong indication that coherent scattering contributes to the image intensity.

To investigate possible coherent electron scattering a conventional image plate was inserted into the microscope chamber right below the retractable STEM detector. The latter is used to shadow the image plate during beam alignment and recording of a reference image. Before the exposure of the image plate the STEM detector is retracted. The beam has to be in spot mode because only one image plate can be used at a time.

Figure 2a shows the inner region of an image taken at 25 keV at the position marked by ‘X’ in Figure 1. A diffraction pattern corresponding to GaN in [1-100] zone axis is visible. Figure 2b depicts the whole illuminated area with adjusted contrast and brightness. The centre with the diffraction pattern is oversaturated but in the outer regions Kikuchi lines are visible. Moreover, the dark shadow of the STEM detector can be recognized. It is possible to identify these Kikuchi lines by comparison with simulated patterns.

The dashed circles in Figure 2b illustrate the radii of the STEM detector segments. It can be seen that the Kikuchi lines and poles are not confined to the bright-field segment but occur on the whole detector area – even at large scattering angles. The comparison of diffraction patterns taken at different positions along the GaN layer shows shifts of the Kikuchi line positions. These can be attributed to orientation changes which cause the observed contrast modifications in the HAADF

STEM images. The HAADF intensity can be calculated by integrating the intensity measured in the diffraction pattern over the area of the corresponding detector segment.

The acquisition of diffraction patterns with image plates reveals Bragg reflections and Kikuchi lines over the whole scattering range covered by the STEM detector. This explains intensity contributions to the HAADF signal by coherent scattering which has to be considered while interpreting low-energy STEM images. [2]

References

[1] RR Keller and RH Geiss, Journal of Microscopy **245** (2012), p. 245.

[2] This work was funded by the Deutsche Forschungsgemeinschaft (DFG).

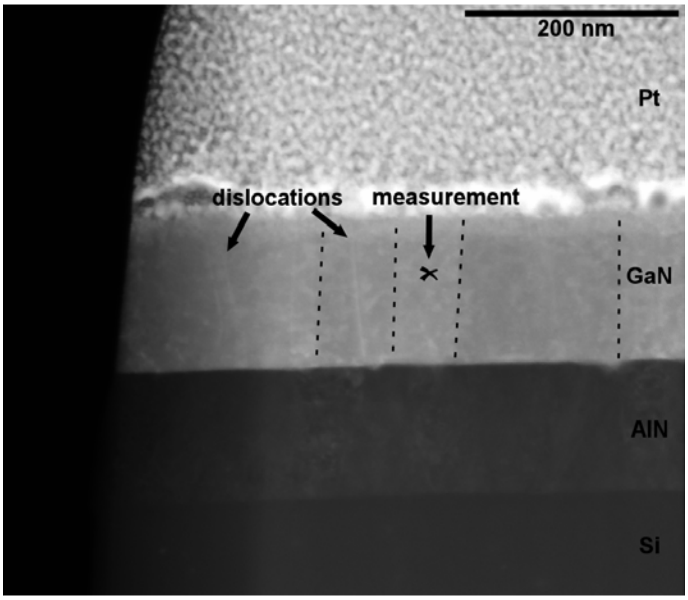

Figure 1. *HAADF STEM image taken at 25 keV showing the layer system in cross-section with dislocations marked by arrows and different HAADF STEM intensity in regions separated by dashed lines. The cross indicates the position where the diffraction pattern in Figure 2 was taken.*

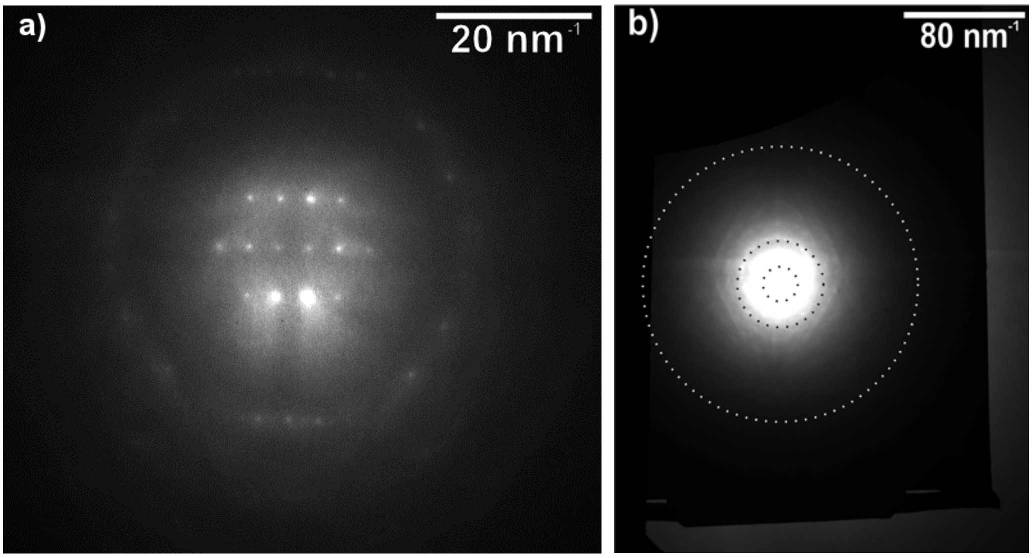

Figure 2. *a) Inner region of 25 keV diffraction pattern taken at marked position in Figure 1 showing Bragg diffraction of GaN [1-100]. b) Contrast-adjusted complete diffraction pattern showing Kikuchi lines with layout of the STEM detector indicated by dashed-line circles.*