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Effects of Beam-Induced Carbon Deposition on Electron Energy-Loss Spectroscopy Analysis of Compositional Fluctuations in InGaN/GaN Quantum Well LEDs

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Electron energy-loss spectroscopy (EELS) performed in an aberration-corrected STEM has a higher signal-to-noise ratio compared to EELS in a conventional STEM, allowing for more information to be extracted from the ionization edges. Additionally, the higher resolution in aberration-corrected STEM translates to higher resolution EELS maps. However, the higher current densities in aberration-corrected STEM increase the possibility of developing beam-induced artifacts, and the ability to collect angstrom-level EELS maps further increases the sample exposure. One such effect that can become exacerbated from high exposure is beam-induced deposition of carbon-rich condensates, which arises from interaction of electrons with hydrocarbons in the sample vicinity [1]. The presence of carbon deposition is indicated by the appearance of the C K-edge at 284 eV in the EELS spectrum (Figure 1a). For many materials systems, the C K-edge can be disregarded because it does not overlap with other ionization edges of interest. However, in the case of indium or nitrogen, the C K-edge is close in energy to the N K-edge at 400 eV and the In M-edge at 480 eV (Figure 1a), a combination of particular importance for EELS studies of the technologically relevant InGaN alloy system.

Here, we investigate the effect of beam-induced carbon deposition on the EELS analysis of InGaN/GaN quantum wells (QW) for light emitting diodes (LEDs) (Figure 1b). While InGaN/GaN LEDs have achieved over 70% efficiency and are the standard for blue/green inorganic LEDs, the extent of indium fluctuations and their impact on device performance is still under debate [2,3]. EELS is routinely used for investigating material fluctuations in InGaN because of its high resolution and ability to provide compositional information and electronic properties [4]. Although the effect of beam-induced artifacts such as knock-on damage on the accuracy of EELS analysis of InGaN have been previously investigated [5], the influence of carbon deposition resulting from hydrocarbon contamination on the sample or in the microscope column has not been addressed.

We use an aberration-corrected STEM at low accelerating voltage to map the composition of InGaN/GaN QWs using EELS with high spatial resolution. By performing EELS scans with varying pixel sizes (distances between consecutive spectra on the linescan) and dwell times, we investigate the impact of beam-induced carbon deposition on the compositional analysis obtained through EELS (Figure 1c,f). A variation in indium signal along the EELS scan is observed when the spectrum image is collected at small pixel sizes corresponding to high electron doses (Figure 1g). We demonstrate that this variation is not due to intrinsic InGaN composition, but rather an artifact of unavoidable carbon contamination. Here, we study spectroscopic parameters to reduce carbon deposition and produce pristine EELS spectra unaffected by artifacts. Although carbon contamination particularly impacts characterization of InGaN due to the spectral proximity of the C signal to the In signal, this work applies to other high-resolution EELS studies that require precise quantification [6].

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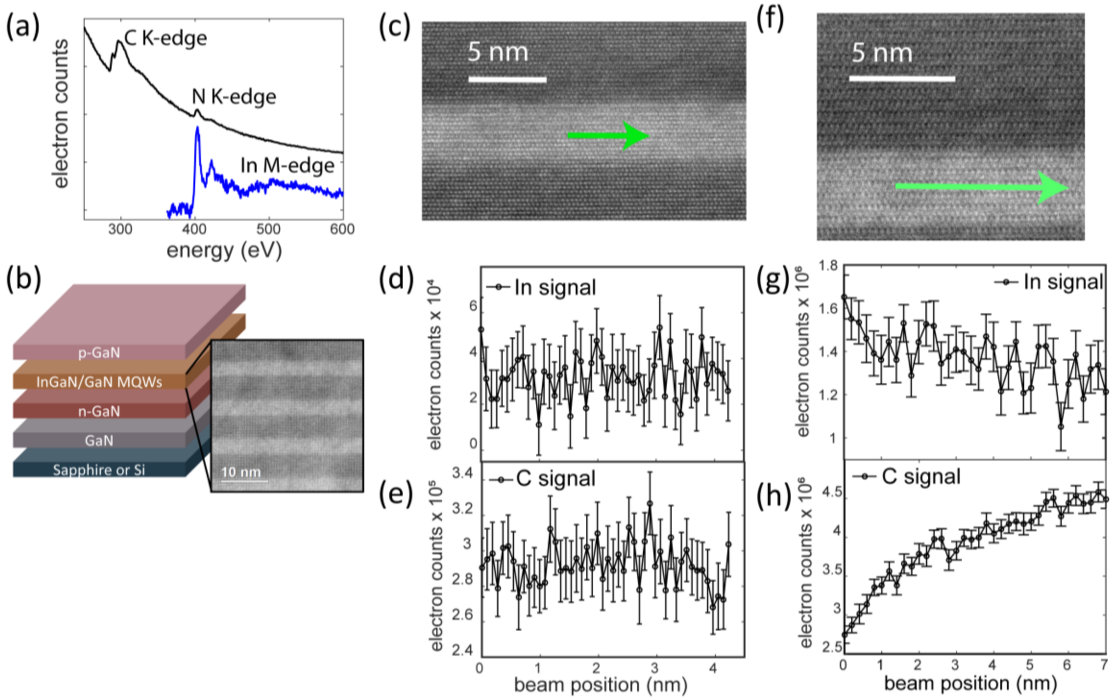

Figure 1. (a) Blue – background-subtracted InGaN EELS spectrum showing N K-edge at 400 eV and In M-edge at 480 eV. Black – raw InGaN EELS spectrum showing carbon K-edge at 284 eV. (b) Schematic and dark-field STEM image of QW heterostructure. (c) Dark-field STEM image showing region in which EELS linescan presented in (d) and (e) was collected. (d) Profile of indium signal along EELS linescan in (c). Profile is flat with some fluctuations. (e) Profile of carbon signal along EELS linescan in (c). (f) Dark-field STEM image showing region in which EELS linescan presented in (g) and (h) was collected. This linescan was collected using a different dwell time and pixel size than the scan shown in (c). (g) Profile of indium signal along EELS linescan in (f). Indium signal decreases along length of linescan. (h) Profile of carbon signal along EELS linescan in (f). Carbon signal increases in intensity along the linescan, indicating that decrease in indium signal is an artifact resulting from beam-induced carbon deposition rather than an intrinsic property of the sample.

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