

Supplementary Material

Identification and thermal healing of focused ion beam-induced defects in GaN using off-axis electron holography

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1. Thicknesses of the TEM lamellas

The table below summarizes the TEM lamellas' crystalline thicknesses (using CBED), the amorphous and the overall thicknesses (using SEM) including the respective errors. Note that the crystalline thickness of lamella B2 (marked *) was determined by subtracting the average amorphous layer thickness of samples A1, A2, and B1 (marked **) from the overall thickness measured by SEM, since no CBED measurement could be done for lamella B2.

lamella	Crystalline thickness [nm]	Overall thickness [nm]	Amorphous layer thickness [nm]
A1	278 ± 2	296 ± 10	9 ± 5.1
A2	363 ± 6	380 ± 10	8.5 ± 5.8
B1	339 ± 8	360 ± 10	10.5 ± 6.4
B2	352 ± 12 *	371 ± 10	9.2 ± 3.3 **

Table 1: Thicknesses information of TEM lamellas used in this work.

The thickness of the amorphous surface layer depends on the material and especially on the preparation recipe (i.e., ion acceleration voltage, incident angles, and ion dose). A lot of care has been taken to ensure that the same recipe was applied to all four lamellas. This resulted in almost equal amorphous layer thickness for all lamellas, with an average of 9.2 ± 3.3 nm.

2. Sample tilt geometry

An image of the double-tilt holder along with the installed grid is shown in Fig. S1. The α and β tilts of the double tilt holder are in parallel with the crystallographic axis [0001] and [1-210], respectively. During the experiment at all annealing temperatures, the δ -doped GaN layer is always kept edge-on. That means β was kept constant at 0-0.3° relative to the [10-10] zone axis, whereas α was tilted between 5 and 7° to avoid diffraction contrast.

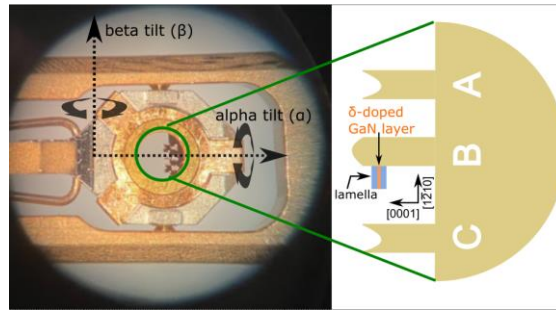


Figure S1: Definition of the rotation axes α and β of the double tilt sample holder and orientation of the δ -doped GaN layer relative to that. No broadening of the δ -doped GaN layer's phase peak can occur, if only an alpha tilt is conducted.

3. Simulation of stopping and range of ions (SRIM) and transport of ions in matter (TRIM)

In order to investigate the distribution of implanted C as well as Ga and N in form of vacancies and interstitials into GaN layer, SRIM/TRIM simulations [1] with a 2 nm thick carbon layer on top of GaN are carried out. Note that the thickness of the carbon layer does not represent the much thicker carbon protection layer deposited on the sample before lamella cutting, but resembles a thin C film that is permanently deposited on the lamella by the electron beam and residual C in the FIB chamber. For the Ga⁺ ion beam, we applied the same incident angle (85°) and energy (5 kV), as well as the ion dose rate (16 pA), as those applied during final polishing step of the FIB process. As demonstrated in in Fig. S2, a Ga⁺ ion beam with an energy as low as 5 kV and rather flat incident angles recoils C sufficiently deep into the subsurface region of the GaN crystal in concentrations significantly larger than the doping concentration. Hence, the recoiled C present is able to pin the Fermi level fully.

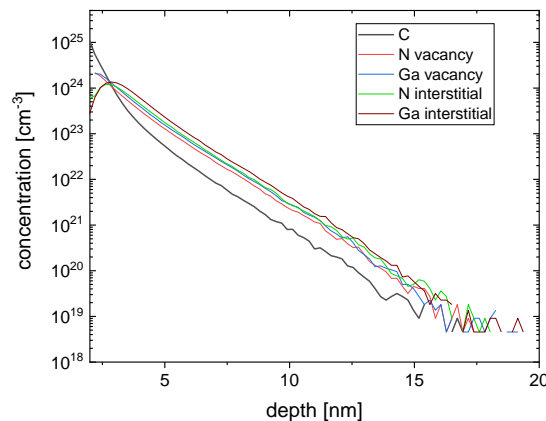


Figure S2: TRIM results of the recoil distribution of C, Ga, N in form of vacancies and interstitials in the GaN layer for a total of 999999 incoming Ga⁺ ions at 5 kV acceleration voltage and an incident angle of 85°, as well as a carbon layer thickness of 2 nm. The thickness of the amorphous layer can be estimated on basis of the displaced atoms. At a thickness of 6 nm (incl. the 2 nm C layer) 100% of atoms were displaced and one can anticipate that amorphization occurred certainly. This thickness is in good agreement with the measured thickness of the amorphous layer of about (9.2 ± 3.3) nm. Beyond this the crystalline structure is preserved. Note, for the interpretation of the large vacancy and interstitial concentrations one needs to recall that the simulation does not consider recombination. If recombination is taken into account, most of the N and Ga vacancies will be annihilated already at room temperature in the GaN layer remaining crystalline due to the low diffusion barriers. Thus, the dominating defect will be C impurities in line with our experiments.

[1] J. F. Ziegler *et al.*, Nucl. Instrum. Methods Phys. Res. B **268**, 1818–1823 (2010)