Structure factor determination on Gallium nitride, GaN.

T. Lippmann[1], J. Waliszewski[2], L. Dobrzynski[2], S. Porowski[3] and J. Jun[4]

[1] *HASYLAB/DESY, Notkestr. 85, 22603 Hamburg*
[2] *Institute of Physics, University of Bialystok, 41 Lipowa str., 15-424 Bialystok, Poland*
[3] *High Pressure Research Center UNIPRESS, 29 Sokolowska str., 01-142 Warsaw, Poland*
[4] *The Soltan Institute for Nuclear Studies, 05-400 Otwock-Swierk, Poland*

GaN has recently attracted much attention due to its potential for the use in lasers and blue light emitting diodes. It crystallizes both in the hexagonal wurtzite (β–ZnS) and the cubic sphalerite (α–ZnS) structure. Here the hexagonal structure (space group $\mathrm{P6_3mc}$, $\mathrm{a_0}$ = $\mathrm{b_0}$ = 3.185 Å,$\mathrm{c_0}$ = 5.18 Å, Z = 2) has been examined by high energy synchrotron radiation of 100 keV, which was chosen in order to reduce both absorption and extinction [1].

The experiment was performed on the Triple–Axis Diffractometer at beamline BW5 using parameters as shown in Tab. 1. The crystal – a cube of $\approx$ 100 μm length of edge – was mounted on a Eulerian cradle, and the data were recorded using a Ge solid state detector. Since earlier powder measurements performed at Nagoya, Japan [2] had shown that some of the low order reflections were affected with long tails in reciprocal space due to stacking faults, profile analyses were carried out during the experiment, and intensities of reflections with improper profiles were sorted out manually. A couple of these 'bad profile' reflections were additionally remeasured at different Ψ–settings and compared to symmetry–equivalent reflections in order to achieve an identification of outliers. Outliers identified that way were sorted out and averages of the remaining intensities at different Ψ were taken for the further data processing, while dubious reflections were completely neglected. Finally, 1156 reflection intensities were recorded in four octants.

The lattice constants were calculated from the Bragg angles of a couple of stronger high order reflections. The constants $\mathrm{a_0}$ = $\mathrm{b_0}$ = 3.175 Å and $\mathrm{c_0}$ = 5.192 Å are in good agreement with literature data.

monochromator	Si 111 flat Laue crystal (FWHM: 5.8")
energy	100.57 keV
$\frac{\sin\Theta}{\lambda}_{max}$	$\approx$ 1.25 Å^{-1}
beam size	1 $\times$ 1 mm
sample – detector dist.	1200 mm
detector aperture	12 $\times$ 12 mm
scan mode	ω step scans (continuous)
steps / scan	151
$\Delta\omega$	0.01°
time/step	1 sec
number of reflections	1156
number of unique	132

Tab. 1: Scan parameters.

Ga	$\mathrm{U_{11}}$	332.0(3.4)
	$\mathrm{U_{33}}$	361.1(7.6)
	$\mathrm{U_{12}}$	166.0
N	z	0.37750(34)
	$\mathrm{U_{11}}$	382.7(16.4)
	$\mathrm{U_{33}}$	371.0(52.2)
	$\mathrm{U_{12}}$	191.3
	GoF	3.2
	R(F)	0.0098
	$\mathrm{R_w(F)}$	0.0102

Tab. 2: IAM–refined parameters of GaN. $\mathrm{U_{ij}}$: temperature factor coefficients $\times$ 10^5Å^2, GoF: goodness of fit.

The data reduction stage was carried out using the CRYSTAL software package and consisted of a Lorentz– and polarization correction. A polarization factor of 0.9 was assumed for all corrections. The value turned out to be a good approximation for experiments performed at BW5 in earlier investigations [3]. Absorption was not corrected due to the small absorption coefficient (1.7 cm^{-1} at 100 keV) and the small crystal size. TDS was also not corrected, since no significant TDS was visible in the background of higher order reflections. Special care was taken during data averaging, and only 5% intensity deviation from the mean value was

allowed. Finally, after sorting out of further outliers the data were averaged to 132 reflections with an internal consistency of 0.014. Compared to earlier experiments on cuprite [3] the consistency is worse by a factor of 2, although the same experimental setup and conditions were used. Hence, this result must be ascribed to the crystal quality. Nevertheless, the data were considered to be well–suited for accurate electron density studies.

Independent atom model refinements were carried out using VALRAY [4]. Anomalous dispersion corrections were omitted, but an extinction model was included into the calculations. Tab. 2 presents the refined parameters, i.e. the z coodinate of the nitrogen and temperature factors from both Ga and N. The extinction found for the strongest reflection 002 (Y_{ext} = 0.86) was approximately of the same order of magnitude as found for the strongest cuprite reflections, and was therefore considered not to impair the IAM refinement results.

The data was well–refined as shown by the R-factor of 0.0098. The goodness of fit (3.2), however, is not sufficiently small and suggests either a modification of the weighting scheme or the use of another strucure model, i.e. a multipole model.

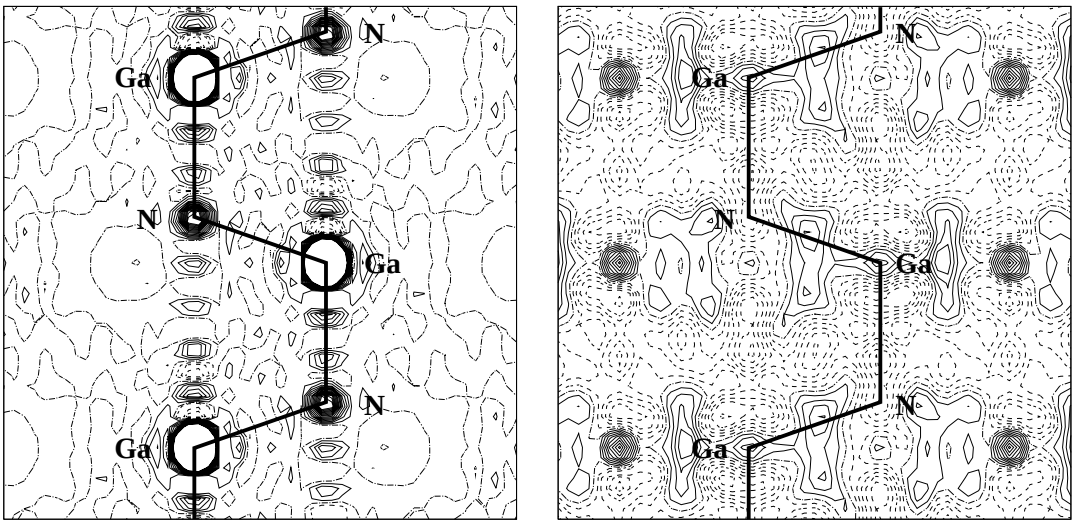

Figure 1: Charge density maps of the (11.0) plane of GaN derived by an IAM refinement. Horizontal axis: $[\bar{1}1.0]$, vertical axis: $[00.1]$, center at -0.5,0.5,0. Dimensions: 7.2 $\times$ 7.2 Å^2. a) Fourier summation, contours at 10 eÅ^{-3}, b) residual density, contours at 0.1 eÅ^{-3}, negative broken, positive solid lines.

Fig. 1a) shows the electron density in the (11.0) plane, which contains both Ga and N, derived by a Fourier summation of F_{calc}. The residual density in Fig. 1b) shows additional density, especially surrounding the Ga atoms. Hence, according to these results a further multipole evaluation of the data seems worthwhile. Additionally, the map shows spherical density in the voids on the left and right side of the atoms. These features have not been understood up to now.

The data has also been investigated by the Maximum Entropy Method, which is presented elsewhere in this report.

References

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