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Optical properties of gallium nitride bulk crystals grown by chloride vapor phase epitaxy

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The optical properties of bulk crystals of gallium nitride grown by chloride vapor-phase epitaxy are investigated. It is shown that these crystals exhibit exciton luminescence bands. Analysis of the energy positions of the band maxima imply certain conclusions about the presence or absence of mechanical stresses in the bulk crystals of GaN obtained. Analysis of the luminescence spectra also reveals that the temperature dependence of the width of the GaN band gap E_g in the temperature range $T=6-600$ K is well described by the expression $E_g(T)=3.51-7.4\times10^{-4}\ T^2(T+600)^{-1}$ eV. It is estimated that values of the free electron concentration in these crystals do not exceed $10^{18}\,\mathrm{cm}^{-3}$. The optical characteristics of the bulk GaN crystals are compared analytically with literature data on bulk crystals and epitaxial layers of GaN grown by various methods. © *1999 American Institute of Physics.* [S1063-7826(99)00510-4]

1. INTRODUCTION

The creation of high-efficiency blue and green light-emitting diodes based on gallium nitride[1] and the fabrication of a violet injection laser[2] have advanced this material to the ranks of the most intensely studied semiconductors. At this time, an important factor that retards progress in developing this material is the lack of an ideal substrate material, although single crystals of GaAs, ZnO, MgO, $MgAl_2O_4$, and especially Al_2O_3 and SiC are actively used as substrates for heteroepitaxial growth of GaN.[3] The cardinal solution to the mismatch problem (with regard to crystal lattice parameters and thermal expansion coefficient) between the epitaxial layer of GaN and the substrate is obviously to use homoepitaxial methods to make device structures based on GaN. For this reason, methods for obtaining GaN substrates have been intensely pursued.[3] However, until now only a few publications have addressed the problem of obtaining and characterizing bulk single crystals of GaN,[4–10] a problem which, it must be asserted, is accompanied by serious technological difficulties.

In this paper we discuss the optical properties of bulk GaN crystals obtained by the chloride version of halide vapor-phase epitaxy (HVPE). We compare the properties of bulk GaN crystals grown in this way with structurally perfect epitaxial layers of GaN grown by the same method on substrates of $6H$-SiC, and also published data on gallium nitride.

2. TEST SAMPLES AND MEASUREMENT PROCEDURE

Samples of GaN were obtained at atmospheric pressure in a horizontal reactor placed in a multizone furnace with resistive heating. As substrates we used crystals of silicon carbide in its $6H$ polytype. Growth temperatures were in the range $T_g=950-1050\,^\circ\mathrm{C}$. The growth rate, depending on the fabrication regime, was varied from 1 to 60 μm/h. A description of the distinctive features of this technology can be found in Refs. 6–11. In order to investigate optical properties we used single crystals of GaN with thicknesses $100-200$ μm separated from the substrate by plasma-chemical etching of the latter,[12] and also epitaxial layers of GaN with thicknesses of 1–3 μm. Prior investigations of structural properties of bulk GaN crystals using x-ray diffraction[6] showed that the samples obtained consist of block single crystals of the wurtzite polytype $(2H)$. Half-widths of rocking curves from the N-facet [i.e., from the (0002) plane] obtained in the ω-scanning regime were less than 130 arc-sec. Detailed studies of the structural properties of GaN/SiC layers obtained by chloride HVPE and published by us previously[13] also showed that they consist of bulk single crystals.

Photoluminescence (PL) of the samples was investigated in the temperature range $T=6-600$ K. At low temperatures (6–50 K) the PL was excited by a DRSh-250 mercury lamp (with UFS2 and ZhS3 filters). Comparatively low intensities were used in order to exclude the effect of concentration saturation of the optical transitions with participation of impurities and defects. The samples were placed in a helium cryostat, which allowed us to obtain a minimum sample temperature of 4.2 K. In the high-temperature range we excited the photoluminescence with a nitrogen laser for convenience (wavelength 337.1 nm), and the samples were placed in a nitrogen cryostat, where the sample temperature could be regulated from 77 to 900 K. By defocusing the laser beam we were able to decrease the excitation pulse power density to $100\ \mathrm{W/cm^2}$ (corresponding to an average pump power density of $10^{-4}\ \mathrm{W/cm^2}$), which also ensured a low level of excitation. The spectrum of the luminescence was recorded using an MDR-23 monochromator with a dispersion of 1.3 nm/mm. The spectral resolution of the apparatus was 0.1 nm or better. In order to obtain absorption spectra in the ultra-

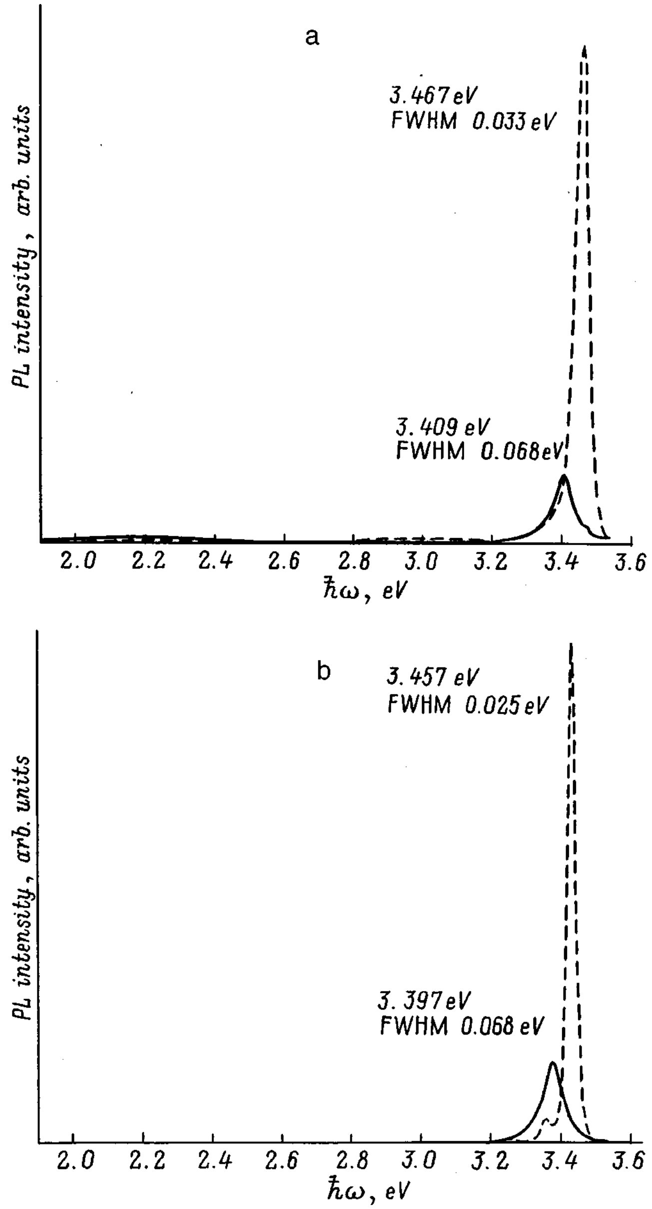

FIG. 1. Photoluminescence spectra of a bulk GaN crystal (a) and a thin (1.5 μm) epitaxial layer of GaN (b) at a temperature of 77 K (dashed curve) and 300 K (solid curve). The excitation light comes from a nitrogen laser.

violet range we used the MDR-23 monochromator and a DDS-30 deuterium lamp. Room-temperature infrared reflection spectra in the range 2.5–25 μm were measured using an IKS-29 two-beam IR spectrometer and two IPO-22 reflection attachments. As a reflection reference we used a mirror layer made of aluminum deposited on glass.

3. EXPERIMENTAL RESULTS AND DISCUSSION

During photoexcitation the GaN crystals luminesced intensely, with a luminescence that was distributed uniformly over the sample area. The luminescence spectrum of bulk GaN crystals, like that of epitaxial GaN layers, is dominated by the exciton luminescence band (Fig. 1). In the low-temperature region, the dominant band of luminescence arises from excitons bound at a neutral donor, while at high temperatures the dominant band is from recombination of free excitons.[14,15] The ratio of intensities of the ''yellow'' band caused by defects, with a maximum at an energy $\hbar\omega \simeq 2.2$ eV, to the intensity of the exciton peak was less than 0.01. It is important to note that for our very best GaN samples the value of the full widths at half-maximum (FWHM) of the exciton bands agreed closely with values quoted in the literature for structurally perfect epitaxial GaN layers (see, e.g., Ref. 16), which in turn indicates the rather high structural perfection of our bulk crystals. This also follows from Figs. 1a and 1b, where we compare PL spectra from our best bulk GaN crystal with that of a structurally perfect epitaxial GaN layer, also grown by us, at two temperatures 77 and 300 K. As for the positions of the exciton band maxima, it is obvious from Figs. 1a and 1b that the peaks of the bulk crystal spectra are shifted by ~ 0.01 eV towards higher energies compared to those of the epitaxial GaN layer (for different samples of epitaxial GaN layers on SiC substrates with different thicknesses and technological growth regimes, this shift can have values from 0.01 to 0.02 eV). A similar shift in the PL spectra of GaN/SiC layers compared to bulk GaN crystals was reported previously by Buyanova *et al.*,[15] where it was explained by residual tensile mechanical stresses that arise along the axis of the epitaxial layer (i.e., along a plane perpendicular to the principal crystallographic axis **c**) when the samples are cooled after epitaxial growth, primarily due to differences in values of the thermal expansion coefficients of GaN and SiC.[3] The GaN layers used in Ref. 15 were obtained by metallorganic chemical vapor deposition (MOCVD) with a buffer layer, in contrast to the layers investigated by us, which may explain the somewhat lower values of residual mechanical stresses in them, and consequently the lower values of the energy shift, observed in that study,[15] in the position of the exciton lines within the layer compared to bulk material (~0.008 eV). Using the average value of the energy shift of exciton lines obtained in Ref. 17 for a biaxial mechanical stress of 1 GPa in the plane of the GaN epitaxial layer (~24 meV/GPa), we can estimate a value of ~0.5 GPa for the average tensile mechanical stress in the best epitaxial layers obtained by us.

We observed the presence of both free and bound excitonic states in the low-temperature luminescence shifts. Figure 2 shows an example of low-temperature photoluminescence spectra containing the largest number of features for one of our samples of bulk GaN in the temperature range 6–45 K. At $T = 6$ K this sample exhibits a peak corresponding to an exciton bound to a neutral donor (DBE) with an energy maximum $\hbar\omega = 3.472$ eV. In addition, it is easy to see two other exciton peaks, which we assume are from an exciton bound to an acceptor (ABE) with energies $\hbar\omega = 3.449$ and 3.421 eV. On the long-wavelength side we see the first two donor-acceptor recombination peaks—a zero-phonon peak (3.263 eV) and its *LO*-phonon (energy ~92 meV) replica (3.171 eV). On the short-wavelength side, in the region 3.48–3.49 eV we see a weakly expressed shoulder whose energy position corresponds to the free exciton A $(n = 1)$.[14,15] It is clear from Fig. 2 that the donor-bound exciton line at 3.472 eV, which corresponds to the lowest binding energy (~ 4 meV), quenches most rapidly with increasing temperature. The two emission lines of the acceptor-bound

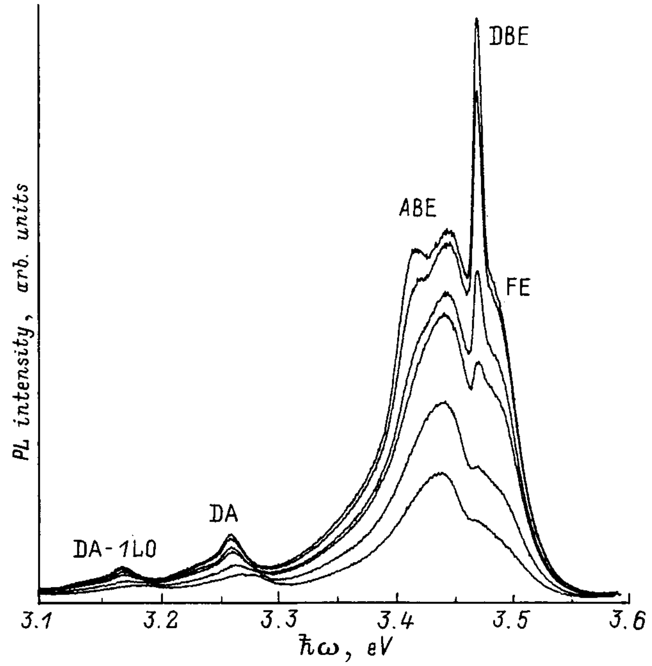

FIG. 2. Low-temperature photoluminescence spectra of a sample of bulk GaN crystal at temperatures (from top to bottom) of 6, 10, 15, 20, 30, and 45 K. Excitation light comes from a DRSh- 250 lamp.

exciton at $\hbar\omega$= 3.449 and 3.421 eV quench much more slowly, so that at $T=45$ K the predominant line for this sample is still the emission line at 3.449 eV, the most intense line in the spectrum. The emission peaks due to donor-acceptor pairs shift towards shorter wavelengths as the temperature increases, which corresponds to the decay of pairs with the lowest binding energy (i.e., the most distant pairs). The transition from donor-acceptor emission to emission via band-acceptor transitions occurs at a temperature $T \simeq 80$ K, since at $T > 80$ K the observed emission is no longer described by the donor-acceptor mechanism. The energy position of an exciton bound to a neutral donor is in rather good agreement with the energy position of the donor-bound exciton published in Refs. 14 and 15 for bulk GaN without mechanical stresses (Table I). On the other hand, the donor-bound exciton appears at an energy position that is ~ 10 meV lower in the PL spectra of thin (~1μm) epitaxial layers of GaN grown by the same method in our apparatus, and not detached from a SiC substrate.[18] The energy positions of excitons bound at an acceptor (Fig. 2) are likewise close to the numbers published in Ref. 19 for mechanically unstressed bulk GaN crystals. In our view, all these results point to the absence, or at least weakness, of mechanical stresses in our samples after their removal from the substrate. At the same time, the ratio of intensities of lines from bound excitons for our samples differ somewhat from those reported in Ref. 19, e.g., for the sample in Fig. 2, the acceptor-bound exciton lines are rather intense and broad. A possible explanation for this could be stoichiometric nonuniformity of our sample, and also local electric fields which cause fluctuations in charged impurities leading to Stark shifts. We note that the more structually perfect the samples of GaN are, the weaker are the inhomogeneous broadening and acceptor-bound exciton lines and the narrower the exciton spectrum (for example, even at high temperatures the values of the FWHM for the bulk sample of GaN shown in Fig. 1a are considerably smaller— 0.033 eV at 77 K and 0.068 eV at 300 K— than for the sample shown in Fig. 2, i.e., 0.086 eV at 6 K). The temperature broadening of the exciton spectrum we obtained for the highest-quality samples of bulk GaN is well described by a linear law with a slope of 1.8×10^{-4} eV/K in the temperature range 77–600 K.

When we increased the temperature to 300 K, we observed an additional temperature- induced quenching of the luminescence spectrum for all the bands, with the bound-exciton bands decaying more rapidly than the free-exciton bands due to the considerably larger binding energy of the latter (~30 meV for the ground state of the A-exciton[14,20,21]). At higher temperatures (T= 500–600 K) the temperature-induced quenching of the exciton luminescence bands occurs more rapidly than we would expect, starting from values of the binding energy for free A-excitons mentioned above; therefore, we assume that nonradiative recombination processes play a dominant role.

Figure 3 shows the temperature dependence of the width of the bandgap E_g obtained by analyzing the position of the free-exciton luminescence band for bulk crystals of GaN and epitaxial GaN layers.[22] There we plot data from Ref. 23 on the temperature dependence of the band gap calculated from the optical absorption spectra of bulk crystals of GaN obtained at high pressure (Fig. 3, curve *2*). Using the standard empirical expression for the temperature dependence of the band gap of semiconductors to approximate the experimental data shown in Fig. 3 (Ref. 24).

$$E_g = E_g(0) - \gamma T^2 (T+\beta)^{-1}, \tag{1}$$

where $E_g(0)$ is the band gap at 0 K, and β and γ are em-

TABLE I. Parameters of low-temperature exciton luminescence from epitaxial layers and bulk single crystals of GaN.

Thickness, μm	Substrate	T_g ,°C	Method	FE $\hbar\omega$, eV (T, K)	DBE $\hbar\omega$, eV (T, K)	ABE $\hbar\omega$, eV (T, K)	Mechanical stress	Literature
100−200	None	~1000	HVPE	3.48−3.49 (6)	3.472 (6)	3.449 (6) 3.421 (6)	None	Data from Refs.
>500	None	~1000	HVPE	3.479−3.480 (2)	3.473−3.474 (2)		None	14,15,19
2	6H-SiC	~1000	HVPE	3.472 (6)	3.464 (6)		Tension along layer	18
<10	6H- SiC	~1000	MOCVD	3.472 (2)	3.466 (2)		Tension along layer	14 and 15

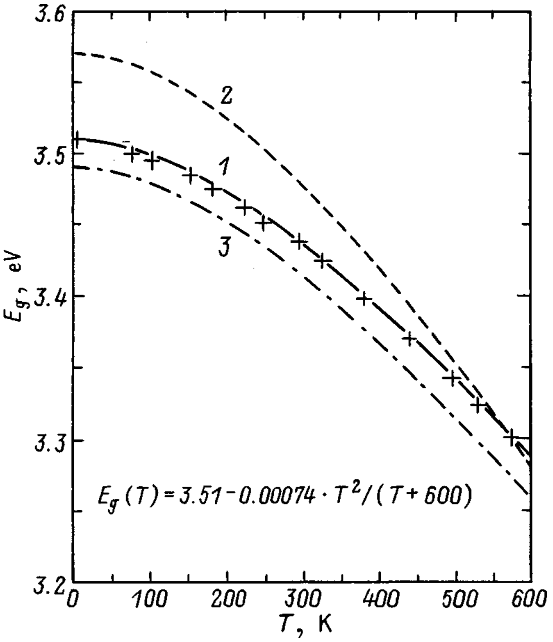

FIG. 3. Temperature dependence of the width of the optical band gap $E_g(T)$ for bulk crystals of GaN grown by chloride HVPE (*1*). For comparison we show the dependence of the energy position of the optical absorption edge for bulk crystals of GaN obtained at high pressure (Ref. 23) (*2*) and the function $E_g(T)$ obtained from the exciton luminescence data of Ref. 22 for thin ($\sim 1\mu$m) epitaxial layers of GaN grown by MOCVD on a 6 H-SiC substrate (*3*).

pirical constants (here the parameter β is often connected with the Debye temperature), we obtain the following values for the coefficients in Eq. (1) that give us the best agreement with the experimental data (Fig. 3, curve *1*): $E_g(0)=3.51$ eV, $\gamma=7.4\times10^{-4}$ eV/K, and $\beta=600$ K. It is clear from Fig. 3 that, ignoring the effect of residual mechanical stresses, the agreement between the curves for our bulk crystals and epitaxial layers of GaN is good. On the other hand, the bulk crystals of GaN grown in Ref. 23 at high pressure (Fig. 3, curve *2*) exhibit a considerably larger value for the band gap compared to our samples of GaN. This is most likely due to the Burstein–Moss effect,[25] given the high concentration of free electrons in these crystals [on the order of $10^{20}\,\mathrm{cm}^{-3}$ (Ref. 23)]. In contrast, the concentration of free electrons is relatively low in the samples of GaN we investigated; in fact, it never exceeds the Mott limit[26] (this is confirmed by the existence of an excitonic luminescence band), an estimate for which is $n_M=(0.26/a_B)^3=7\times10^{17}\,\mathrm{cm}^{-3}$ (here $a_B=2.9$ nm is the Bohr radius of an exciton). The values reported above for the nonlinear coefficients in the temperature dependence of E_g are in satisfactory agreement with those obtained previously for epitaxial GaN layers[22] (Table II). The difference in values of the nonlinear coefficients β and γ for bulk crystals of GaN obtained at high pressure (Table II) can be qualitatively explained[23] by the temperature dependence of the Burstein-Moss effect.

The most structurally perfect of all the GaN crystals obtained by us was visually colorless and in the visible region possessed a rather high optical transparency (around 60% for a thickness of 100 μm). The value of the band gap obtained by measuring the optical absorption edge at $T=300$ K (assuming a classical parabolic band and ignoring exciton effects) lies in the range 3.31–3.32 eV for various samples of GaN, which is roughly 0.13 eV lower in energy than the value expected from the photoluminescence results [as indicated by the data shown in Fig. 1a, the value of the band gap at $T=300$ K should be $E_g(300\,\mathrm{K})=3.436$ eV].

TABLE II. Nonlinear coefficients of the temperature dependence $E_g(T)$ obtained for bulk crystals and epitaxial layers of gallium nitride.

Sample type	Experimental method	γ, 10^{-4} eV/K	β, K	Literature
Layer GaN/Al$_2$O$_3$	Luminescence	7.2	600	(Ref. 30)
Layer GaN/Al$_2$O$_3$	Optical absorption	9.39	772	(Ref. 23)
Layer GaN/SiC	Luminescence	7.7	600	(Ref. 22)
Bulk GaN crystal	Optical absorption	10.8	745	(Ref. 23)
Bulk GaN crystal	Luminescence	7.4	600	This work

In the infrared reflection spectrum, the lattice resonance (reststrahl band), which extends from 13.7 to 18.9 μm and whose boundaries correspond to energies for the transverse (TO) and longitudinal (LO) optical phonons,[3] is quite intense, which also attests to the relatively high structural quality of the crystals obtained (Fig. 4). In Fig. 4 we show for comparison the reflection spectrum of a structurally perfect epitaxial GaN layer on a SiC substrate (dashed curve). We note the good agreement between the two spectra in Fig. 4 in the lattice resonance (reststrahl band) of GaN, and also the appearance in the spectrum of the epitaxial layer of an additional resonance in the region 10.3−12.8 μm, which corresponds to the silicon carbide substrate.[27] Estimates of the values of the TO- and LO-phonon frequencies (560 and 730–749 cm^{-1}, respectively) from the IR reflection spectra of the bulk GaN crystals are in good agreement with results obtained previously by Raman spectroscopy,[6] and for the

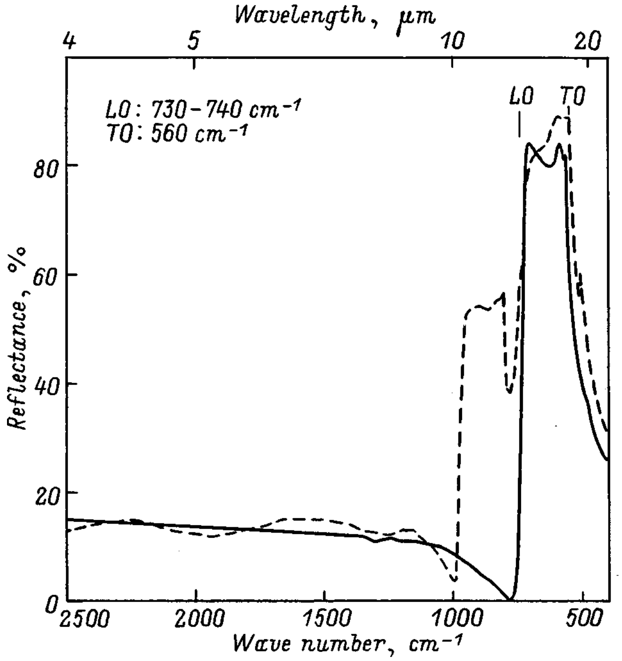

FIG. 4. IR reflection spectra for a bulk crystal of GaN (solid curve) and an epitaxial layer of GaN with thickness 3.3 μm on a SiC substrate (dashed curve). $T=300$ K.

LO-phonon there is good agreement with the photoluminescence data (see the text above and Fig. 2). The location of the minimum in the reflection near the resonance, which is sensitive to the concentration of free carriers,[28] corresponds to a relatively low concentration of free electrons (less than $10^{18}\,\mathrm{cm}^{-3}$; see Ref. 29), which again agrees with luminescence data.

Thus, we have investigated several features of the optical properties of bulk GaN crystals obtained by using the chloride version of HVPE. We have established that these crystals have a bright excitonic luminescence, and that their spectra are similar to those of structurally perfect thin epitaxial layers of GaN. What differences there are primarily involve exact energy positions of excitonic bands, which in the case of bulk crystals correspond to mechanically unstressed states of gallium nitride. We have shown that the temperature dependence of the band gap for these GaN crystals in the temperature range 6–600 K can be described by the expression $E_g(T)=3.51-7.4\times10^{-4}\ T^2(T+600)^{-1}$, eV. Using photoluminescence and IR reflection, we have established that, in contrast with bulk crystals of GaN grown at high pressure, the concentration of free electrons in the bulk crystals is no more than $10^{18}\,\mathrm{cm}^{-3}$, i.e., far from degeneracy. We found that the band gap determined from the optical absorption edge for our bulk crystals is shifted (by about 0.1 eV towards lower energies) relative to the value obtained from photoluminescence measurements.

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