

## Identification of the prime optical center in GaN:Eu<sup>3+</sup>

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We identify a dominant light-emitting center in ion-implanted GaN:Eu<sup>3+</sup> for which the lattice damage has been completely healed, according to x-ray diffraction and Rutherford backscattering spectrometry measurements, by high-temperature, high-pressure annealing. This center is likely to be the isolated substitutional Eu<sub>Ga</sub> defect. It lacks a “subgap” excitation band and therefore has no state in the GaN band gap, shows threefold splitting of its <sup>7</sup>F<sub>2</sub> level, with two sublevels nearly degenerate, and exhibits a long, single-exponential luminescence decay. Competing luminescent centers of GaN:Eu involve this prime center with intrinsic lattice defects, one of which may also be responsible for the GaN yellow band.

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### I. INTRODUCTION

The identification of the microscopic structures of light-emitting centers (often misleadingly called “sites”<sup>1</sup>) in rare-earth- (RE-)doped semiconductors is a very long-standing problem of solid-state spectroscopy.<sup>2</sup> Arguably, no such identification, of many made over the last 25 years, is 100% safe. A major complication in RE spectroscopy is the so-called “site multiplicity” problem whereby several centers produce spectral lines that overlap in wavelength.<sup>3</sup> This causes more than just a practical difficulty in assigning a line or even a set of lines to a particular center. For example, Dierolf *et al.*<sup>4</sup> used combined excitation-emission spectroscopy of molecular-beam-epitaxy-doped GaN:Er<sup>3+</sup> to assign spectra to two “majority” and four “minority” centers. Even when the component lines of an individual center can be distinguished in this way, the problem of *identifying* the center responsible (i.e., specifying its microscopic structure) remains to be addressed. The situation would of course improve if the spectrum of the paradigm center, the isolated substitutional defect, RE<sub>Ga</sub>, could be *unambiguously* identified. In this paper, recent work is presented involving high-pressure (HP) annealing of ion-implanted GaN:Eu that achieves the objective of producing samples with no residual implantation damage and with essentially a single dominant species of Eu light-emitting center. On the balance of probabilities, together with direct spectroscopic indicators, we identify this as the prime optical center Eu<sub>Ga</sub> in GaN.

Ion implantation is a powerful technique for doping semiconductors. Lorenz *et al.*<sup>5</sup> have carried out a detailed study of optical activation of implanted III-N:RE, in particular, GaN:Eu, by high-temperature annealing. Optimization of the implantation conditions can also enhance the optical activation of RE ions via processes of “dynamic annealing;” Vantomme *et al.*<sup>6</sup> showed that lattice damage is reduced substantially by implanting GaN in a channeling geometry along a low-index crystallographic direction.

Two majority luminescence centers, which dominate the PL spectra of Eu-implanted GaN,<sup>7</sup> can be readily distin-

guished by the photoluminescence excitation (PL/E) spectra of partly resolved <sup>5</sup>D<sub>0</sub> → <sup>7</sup>F<sub>2</sub> Eu<sup>3+</sup> transitions near 620 nm.<sup>8</sup> One center, named Eu<sub>1</sub> by Bodiou *et al.*,<sup>9</sup> but which we prefer to call Eu1 in order to avoid confusion caused by using a subscript, appears at relatively low annealing temperatures. It can be photoexcited at energies below the GaN band gap, in a broad band peaking at 385 nm (3.22 eV); it can also be excited above gap, in a stepped spectral feature equivalent to the GaN band-edge absorption at 354 nm (3.50 eV). The other majority center, Eu2, is excited *only* by above band-edge light.<sup>8</sup> Both of these efficient *indirect* excitation processes of RE luminescence, via the generation of photoexcited electron-hole pairs or excitons in the host material, is additional to the *direct*, but very much weaker, excitation of RE transitions via intrasystem transfer within high-lying internal states of the RE ion.<sup>10</sup> Andreev *et al.*<sup>11</sup> usefully present expected *mean* values of the energies of all the observed <sup>5</sup>D<sub>0,1,2</sub> to <sup>7</sup>F<sub>0,1,2,3,4,5,6</sub> transitions of GaN:Eu<sup>3+</sup>.

We assume that Eu1 and Eu2 have different, but probably related, microscopic structures.<sup>1</sup> RE lattice location studies, by electron-emission channeling and Rutherford backscattering spectrometry/channeling (RBS/C),<sup>12</sup> and x-ray absorption fine structure,<sup>13</sup> agree that RE<sup>3+</sup> tends to substitute for Ga in wurtzite GaN. (In other words, the *site* occupation is preferably substitutional, denoted RE<sub>Ga</sub>. In zinc-blende GaN, on the other hand, Eu prefers to be interstitial.<sup>14</sup>) Moreover, the integrated RE luminescence intensity correlates well with the Eu<sub>Ga</sub> substitutional fraction in cases where the recovery of implantation damage is not complete.<sup>5</sup> These observations suggest the existence of a *family* of defects, RE<sub>Ga</sub>-X, where X is a lattice defect, presumably a point defect, *which alters the spectroscopic signature* of RE<sub>Ga</sub> only slightly through the effect of a slight modification of the crystal field.<sup>1,3</sup> By controlling the implantation conditions (beam energy, fluence, crystal orientation) and postimplantation annealing (ambient pressure and sample temperature) we are able to engineer the relative abundances of Eu1 and Eu2. We show here that Eu2 dominates in HP-annealed samples with little or no residual lattice damage. By virtue of this circumstance, together with

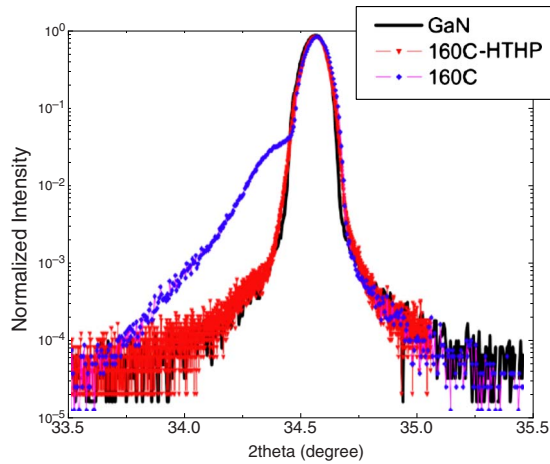


FIG. 1. (Color online) HRXRD  $2\theta$ - $\theta$  scans of virgin GaN compared with as-implanted (160C) and annealed (160C-HTHP) GaN:Eu samples.

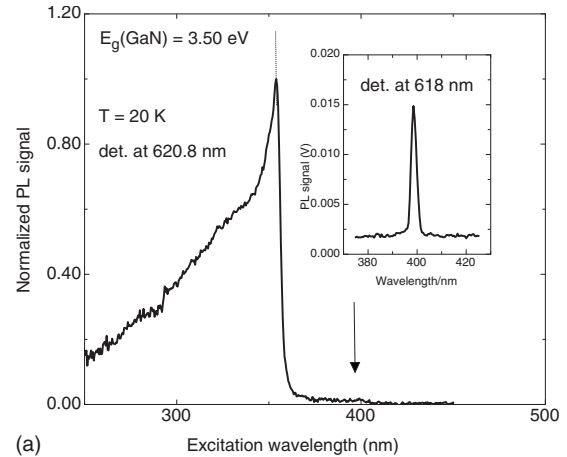
its specific spectral properties, the Eu2 species is identified as  $\text{RE}_{\text{Ga}}$ . The Eu1 center is then likely to be  $\text{RE}_{\text{Ga}}$  associated with an intrinsic lattice defect, which further evidence suggests to be the defect also responsible for the well-known GaN “yellow band” (YB). Other minority centers are then likely to be similar variations on the main theme.

## II. EXPERIMENTAL DETAILS

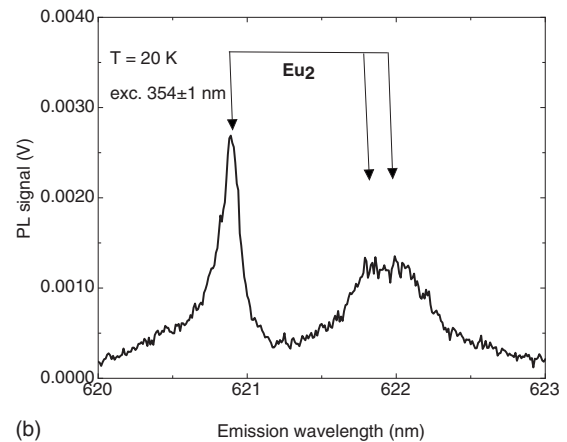
GaN epilayers grown on (0001) sapphire substrates by metal organic chemical-vapor deposition were implanted either off-axis (so-called random implantation, denoted R in sample identifiers) or along the  $c$  axis (channeled implantation, indicator C) with 160 or 300 keV  $\text{Eu}^+$  ions to fluences of  $10^{13}$ ,  $5 \times 10^{14}$ , or  $10^{15}$   $\text{at}/\text{cm}^2$ , as specified by case below. Postimplantation high-temperature HP (HTHP) annealing was performed at temperatures in the range 1000–1450 °C for 30 min under 1 GPa ( $10^4$  bar) of nitrogen with the sample surface covered by bulk GaN crystallites. High-resolution x-ray diffraction (HRXRD)  $2\theta$ - $\theta$  scans of the 0002 reflection were acquired using Cu  $K\alpha 1$  radiation in a Bruker D8 Discover system equipped with a monochromator with four Ge (002) crystals and a scintillator detector. RBS/C was performed in order to study both the recovery of lattice damage after annealing and the lattice location of Eu. An overview of the sample luminescence at room temperature (RT) was obtained by hyperspectral cathodoluminescence (CL) imaging in an adapted Cameca SX100 Electron Probe Microanalyser.<sup>15</sup> Low-temperature (LT) PL/E spectra were obtained for samples mounted in a helium cryorefrigerator using a 1000 W Xe lamp/1/4 m monochromator combination with a spectral resolution of about 0.5 nm in the excitation arm and a 2/3 m monochromator with a limiting spectral resolution of  $\sim 0.01$  nm in emission.

## III. EXPERIMENTAL RESULTS

XRD measurements (Fig. 1) evidence a complete removal of implantation-induced lattice damage for 160 keV (C) Ga-



(a)



(b)

FIG. 2. PL (a) excitation and (b) emission spectra of sample 160C-HTHP. The inset of Fig. 2(a) shows a detail of PLE of the 618 nm “broad” band from a sample implanted at 300 keV to a fluence of  $10^{13}$   $\text{Eu cm}^{-2}$  and annealed at 1000 °C.

N:Eu as a result of annealing a  $5 \times 10^{14}$   $\text{cm}^{-2}$  sample (160C-HTHP hereafter) at 1400 °C at HP. Figure 1 compares the XRD profile of 160C-HTHP with that of a virgin layer of GaN. The damage peak or shoulder found at low angle in as-implanted material (160C in the figure) persists in those samples annealed at low pressure and lower temperature (XRD not shown here.) This peak originates in the expansion of the lattice due to introduced defects.<sup>16</sup> Samples implanted in random geometry with a lower dose of  $10^{13}$   $\text{at}/\text{cm}^2$  at 300 keV (R) also show complete recovery of the lattice damage after HP annealing at 1000 °C but a high-fluence  $10^{15}$   $\text{cm}^{-2}$  300 keV (R) sample does not recover completely even when annealed at 1450 °C. Full angular RBS/C scans, not shown,<sup>17</sup> are even more revealing: for 160C-HTHP, the RBS/C angular scan of Eu is indistinguishable from that of the Ga host atoms along two major crystallographic directions. The Eu lattice location is thus identical to that of Ga and Eu is 100% substitutional on Ga sites.  $\chi_{\text{min}}(\text{Ga})$  and  $\chi_{\text{min}}(\text{Eu})$ , the minimum backscattering yields for Eu and Ga, respectively, are each about 4% for this sample, which is comparable to the Ga minimum yield of virgin GaN.

The low-temperature PL/E spectra of the principal  $^5\text{D}_0 \rightarrow ^7\text{F}_2$  red transition for 160C-HTHP are shown in Fig. 2. Compared with previous spectroscopic results<sup>2,7,8</sup> the excita-

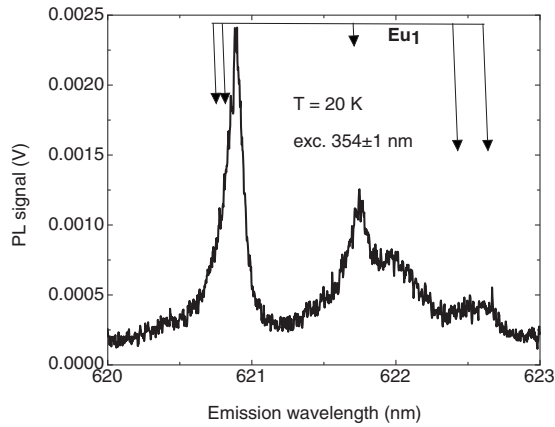


FIG. 3. LTPL of 300 keV (R)  $10^{13}$   $\text{cm}^{-2}$  sample annealed under 1 GPa of  $\text{N}_2$  at 1000 °C. Note additional, compared to Fig. 2(b), weak lines of Eu1 center.

tion spectrum of the 620.8 nm line [Fig. 2(a)] shows little evidence of a broad subgap feature (associated with underlying unresolved Eu1 emission lines) and the emission spectrum simplifies.

[The small, fairly narrow ( $\sim 2$  nm, 15 meV) excitation band near 400 nm resembles a feature first reported by Nyein *et al.*<sup>18</sup> Closer inspection reveals that this excitation is not in fact associated with the *sharp-line*  $\text{Eu}^{3+}$  luminescence lines of the centers Eu1 and Eu2 but instead with a 15 meV broad emission which peaks at 618 nm, shown in the inset of Fig. 2(a). The nature of these semibroad transitions is uncertain at present but they are perhaps due to a small number of Eu ions stabilized in highly distorted environments.]

The emission spectrum of Fig. 2(b) shows three main sharp lines, at 620.8, 621.8, and 622.0 nm, all assigned to Eu2 by virtue of their identical excitation spectra. The closely spaced doublet near 622 nm was not resolved in previous work, partly in consequence of the overlapping of Eu2 and Eu1 components. The smallest measured linewidth of the singlet at 620.8 nm is  $\sim 0.1$  nm, an energy bandwidth of 0.4 meV. It is worth noting that in previous work<sup>8</sup> on samples annealed at low pressures (LP) of  $\text{N}_2$  ( $\sim 1$ –5 bar), the equivalent linewidth was estimated to be  $\sim 0.4$  nm; however, the HTHP samples are much brighter than the previous ones; the difference in estimated linewidths is therefore partly instrumental in origin.

The use of high pressure does more than simply allow higher annealing temperatures: Eu2 also dominates the spectrum of a sample irradiated at 300 keV (R) to a low fluence of  $10^{13}$   $\text{cm}^{-2}$  and HP annealed at 1000 °C (Fig. 3). In strong contrast to this result, the spectrum of Eu1 dominates most LP anneals at this temperature. While dependent to some extent on template quality, the ratio of intensities of the 620.8 nm (mainly Eu2)/622.5 nm (Eu1) emission lines increases for LP annealing from near zero for 1000 °C anneals to near unity at 1150 °C and about 3 for the highest practical temperature of 1300 °C.<sup>19</sup> Only a trace of the Eu1 line at 622.5 nm remains in the spectrum of the 300 keV (R),  $10^{13}$   $\text{cm}^{-2}$  sample, annealed at HP at 1450 °C, which displays the strongest PL of all samples measured to date. Figure 4 shows the rise and decay of the 620.8 nm PL line for this sample,

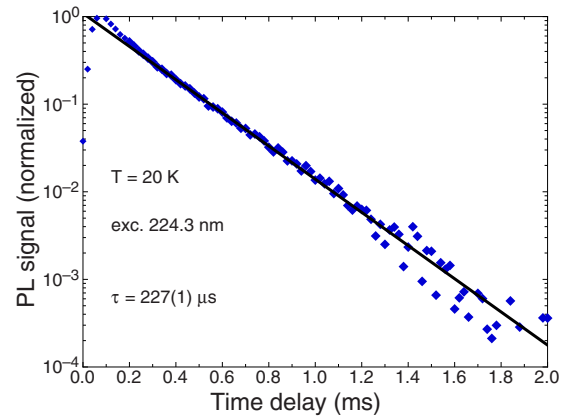


FIG. 4. (Color online) PL rise and decay of the  ${}^5\text{D}_0 \rightarrow {}^7\text{F}_2$  620.8 nm emission of Eu2 at 20 K.

excited at 224.3 nm using a HeAg30–224SL laser set to its minimum pulse width of 10  $\mu\text{s}$ . The best fit to the decay tail in the figure yields a single-exponential decay time of 227(1)  $\mu\text{s}$ .

#### IV. DISCUSSION

As described in Ref. 5, conventional thermal annealing at low pressures of nitrogen has limited success in optically activating implanted GaN:Eu samples. The annealing temperature at which RTCL emission is maximized increases with the ion fluence, but the activation level eventually saturates or disimproves, accompanied in some cases by the appearance of macroscopic sample damage.<sup>20</sup> Coincident with the onset of activation saturation, we observe, above 1000 °C, an increase in the intensity of the 570 nm YB, which makes the brighter GaN:Eu samples appear more orange than red to the eye. The use of HP annealing allows the optimal temperatures for RTCL to increase to about 1300 °C and  $>1400$  °C for  $10^{13}$  and  $10^{15}$   $\text{cm}^{-2}$  samples, respectively. At the same time, YB emission increases only slightly for the  $10^{15}$   $\text{cm}^{-2}$  sample (and by about a factor of 7 for the  $10^{13}$   $\text{cm}^{-2}$  sample). The LTPL intensities show completely different behavior from RTCL, due to the very different temperature dependences of Eu1 and Eu2 emission:<sup>13</sup> while the  $10^{15}$   $\text{cm}^{-2}$  sample appears to saturate above 1300 °C, the integrated LTPL emission intensity of the less strongly doped (and therefore initially less damaged sample) continues to increase exponentially and in fact exceeds the more strongly doped sample at 1450 °C annealing.

Theoretical studies of RE tend to agree that the isolated ions do not, when substituting for Ga, form deep states in the gap of GaN, unless they are associated with another defect.<sup>21</sup> In principle, this allows us to eliminate Eu1 as the  $\text{Eu}_{\text{Ga}}$  species, an assignment recently made by Bodiou *et al.*,<sup>22</sup> since the presence of subgap excitation strongly suggests the presence of a deep level that sequentially traps an electron and hole during excitation.<sup>10</sup> Furthermore, the spectral splitting pattern of Eu2, Fig. 2(b), is identically the three lines expected from the splitting of  ${}^7\text{F}_2$  in the wurtzite crystal field; in a tetrahedral geometry, crystal field splitting would yield two lines<sup>23</sup> and this is very nearly the case here. The

long single-exponential decay over many lifetimes (Fig. 4) confirms the dominance of a single Eu center at the detection wavelength of 620.8 nm. An absence of nonradiative shunt processes at low temperature yields a high value for the decay time, expected to equal the radiative lifetime. Taken together with the absence of lattice damage in samples annealed at HTHP, all of the available evidence points to the identification of Eu2 with  $\text{Eu}_{\text{Ga}}$ .

Having identified  $\text{Eu}_{\text{Ga}}$  as the product of secondary annealing, we can speculate about the defect reactions which lead to the interesting competition between the populations of the two majority Eu centers in ion-implanted samples. Eu1 persists for low pressure anneals even at high temperatures up to 1300 °C. In contrast it appears to be almost eliminated in low-fluence (low-damage) samples by high-pressure annealing at the relatively “low” temperature of 1000 °C. In actual fact the concentration of Eu1 may saturate while that of Eu2 increases more quickly in such a case. It is clear that the emergence of Eu2 in LP annealing coincides with an increase in YB luminescence, and that we see a simultaneous increase in YL and Eu2 in the low-fluence HP sample with increasing annealing temperature. These observations suggest that a defect reaction of the general type



controls the relative populations of the different Eu species.

If Eu2 is  $\text{Eu}_{\text{Ga}}$ , the unassociated substitutional defect, then Eu1 is likely to be  $\text{Eu}_{\text{Ga}}\text{-X}$ , where X is a lattice defect. Equation (1) then suggests that the defect X is also responsible for or involved in the YB luminescence. Although the nature of X is unknown, the dominance of Eu2 for anneals at high nitrogen overpressure, even at relatively low temperatures,

may suggest a dearth of nitrogen vacancies,  $V_{\text{N}}$ , in material that shows essentially a single-emitting center,  $\text{Eu2}=\text{Eu}_{\text{Ga}}$ . In LP-annealed samples where  $\text{Eu1}=\text{Eu}_{\text{Ga}}\text{-X}$  dominates, its lowered symmetry may manifest in the apparent average displacement of Eu from the Ga site, which has been reported in some earlier lattice location studies.<sup>24</sup> No such apparent displacements are found in fully recovered HP-annealed samples. It is also interesting to note that the electroluminescence of the recently reported GaN:Eu red-emitting light-emitting diode<sup>25</sup> appears to be dominated by Eu2.

## V. CONCLUSION

In conclusion, we report the identification of a dominant Eu center, Eu2, in GaN:Eu after annealing ion-implanted samples at extremely high temperatures and pressure. Spectroscopic considerations confirm the *prima facie* inference that the center involved, in a GaN lattice free from residual implantation damage, is the isolated substitutional impurity,  $\text{Eu}_{\text{Ga}}$ . This paradigm defect may be regarded as the essential component of all Eu-related centers,  $\text{Eu}_{\text{Ga}}\text{-X}$ , in wurtzite GaN. Preliminary studies suggest further that in at least one other center, Eu1, the associated defect X is also responsible for the YB luminescence of GaN.

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