Optical transitions in Eu^{3+} ions in GaN:Eu grown by molecular beam epitaxy

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We report on the photoluminescence, photoluminescence excitation, and cathodoluminescence studies of Eu-doped wurtzite-phase GaN grown by plasma-assisted molecular beam epitaxy. Intra-4*f*-transitions of Eu^{3+} ions starting from the $^5D_2$, $^5D_1$, and $^5D_0$ excited states have been identified and show different thermal quenching in photoluminescence. The $^5D_0 \rightarrow {}^7F_2$ transition at around 620 nm exhibits well-resolved Stark-split emission lines. Depth-sensitive cathodoluminescence and photoluminescence experiments have put in evidence two different sites of Eu^{3+} ions, one near to the sample surface and the other deeper in the volume, characterized by different crystal-field splitting, thermal quenching, and dependence on optical and electron beam excitations. It is shown that Eu^{3+} ions located deeper in the volume can be selectively excited by below-band-gap excitation.

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I. INTRODUCTION

Rare-earth- (RE-)doped group III nitride semiconductors are promising materials for visible light emitters. The wide band gap allows efficient energy transfer from the host to the RE ions, resulting in RE luminescence visible at room temperature. In the specific case of Eu, intense red luminescence has been reported from Eu-doped GaN layers by several groups (see for instance Refs. 1–9). Eu was introduced either by implantation or during molecular beam epitaxy (MBE) growth.

As it is well known, in trivalent RE ions inner-4*f*-shell transitions are dominant and only weakly perturbed by the surrounding host. The resulting narrow trivalent RE ion transitions are an ideal optical probe to identify different RE sites since their Stark splitting should depend on the local crystal-field symmetry. Along these lines different sites have been identified for Er^{3+} in MBE-grown GaN layers[10] as well as for Nd^{3+} in implanted GaN layers.[11] Recently we showed that Tm^{3+} and Eu^{3+} transitions in (In)GaN quantum dots are shifted and broadened due to the existence of an internal electric field of several MV/cm.[12,13] Because different sites could exhibit different optical properties their identification is important for the optimization of RE-based emitting devices.

The aim of this paper is to study optical transitions of Eu^{3+} ions in a GaN:Eu layer by spectroscopic methods, namely, photoluminescence (PL), photoluminescence excitation (PLE), and cathodoluminescence (CL). Special attention was paid to the $(^5D_0 \rightarrow {}^7F_2)$-related transitions at around 620 nm, which are the most intense in the PL and CL spectra. It will be shown that at least two different Eu^{3+} sites are present in our MBE-grown layers.

II. EXPERIMENTS

The growth was performed using 1-μm-thick AlN pseudosubstrates deposited by metal organic chemical vapor deposition (MOCVD) on *c*-sapphire.[14] After a standard chemical degreasing procedure and acid cleaning, the pseudosubstrate was fixed with indium on a molybdenum sample holder, and introduced in a MBE chamber equipped with Ga and Eu effusion cells and a radio-frequency plasma cell to produce monatomic nitrogen. A 200 nm undoped GaN buffer was grown in Ga-rich conditions on the AlN pseudosubstrate. Then, on this relaxed GaN buffer, about 200 nm GaN:Eu was grown also in Ga-rich conditions. The growth temperature for both GaN buffer and GaN:Eu was around 720 °C. The growth was controlled using reflection high-energy electron diffraction (RHEED). During growth of the sample, the RHEED pattern showed lines typical of the smooth surface associated with Ga-rich growth conditions. Also a 2×4 reconstruction was observed, as a result of the Eu surfactant properties previously discussed in Ref. 15.

The Eu concentration in the sample was between 0.2% and 0.3% as measured by Rutherford backscattering spectroscopy. After the growth, the sample was chemically etched with HCl in order to remove possible segregated Eu from the sample surface.[15]

PL and PLE measurements were carried out using a tunable excitation source consisting of a 500 W high-pressure Xe lamp equipped with a Jobin-Yvon high-resolution double-grating monochromator (Gemini 180). The PL was analyzed by another Jobin-Yvon grating monochromator (Triax 550) and detected by either a charge-coupled device (CCD) camera operating at liquid nitrogen temperature or a photomulti-

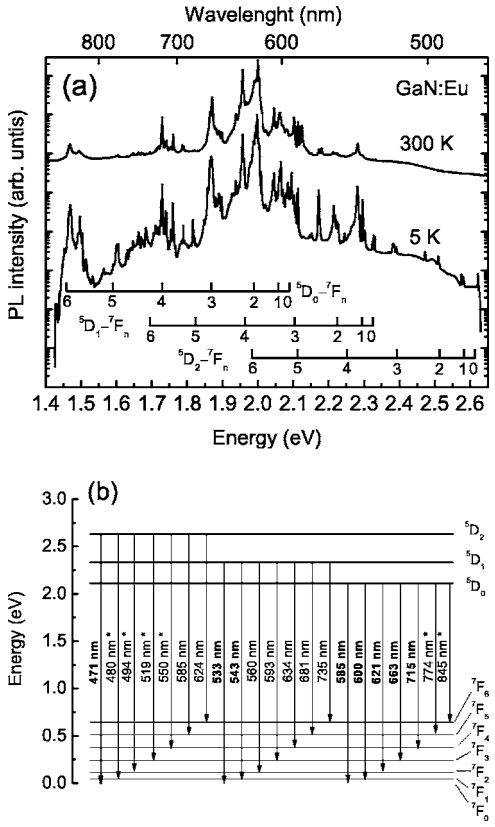

FIG. 1. (a) PL spectrum in logarithmic scale of a GaN:Eu layer measured at 5 K with a frequency-doubled Ar-ion laser line emitting at 244 nm. The position of the transitions is indicated in the spectrum where ^7F_n means the ground state with $n=0$ to 6. (b) Energy diagram and observed transitions of Eu^{3+} ions in GaN host. Bold plotted emission lines have been already found in the literature. Transitions marked by an asterisk are found here for the first time to our knowledge. For other lines only the expected emission wavelength is given. The indicated wavelengths present mean values for transitions.

plier tube operating in the photon-counting mode. The excitation power density was about 200 $\mu W/cm^2$ at 350 nm. The sample was mounted on the cold finger of a microcryostat which enabled us to record PL and PLE spectra at various temperatures from 5 to 380 K. Additional low-temperature PL was performed using a frequency-doubled Ar-ion laser emitting at 244 nm. To achieve very high optical excitation the fourth harmonic (266 nm) of a pulsed neodymium-doped yttrium aluminium garnet (Nd:YAG) laser (0.5 ns pulse width, 8 kHz frequency, and 1.17 kW peak power) was used. The diameter of the focused Nd:YAG laser and Ar-ion laser beam was smaller than 0.4 mm. CL at liquid helium temperature was carried out with a FEI Quanta 200 SEM equipped with a Jobin-Yvon HR460 monochromator and a CCD camera operating also at liquid nitrogen temperature.

III. RESULTS AND DISCUSSION

PL spectra at 5 and 300 K of a GaN:Eu layer, excited at 244 nm, are shown in Fig. 1(a). At 5 K the PL spectrum is characterized by sharp Eu^{3+} emissions extending from 470 to

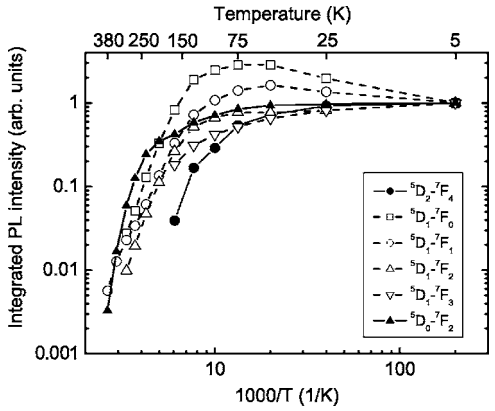

FIG. 2. Temperature dependence of the PL intensity of a GaN:Eu layer in double logarithmic scale. The absolute intensity was measured for each transition. Excitation source: 244 nm line of a frequency-doubled Ar-ion laser with 10 mW.

850 nm. Note that the PL intensity is presented in a logarithmic scale to make visible weak emission lines. The near-band-gap emission of GaN at 353 nm has been found to be quenched in spite of a relatively low Eu concentration of about 0.2%. This is a qualitative indication that the Eu-doped layer is of high crystalline quality, associated with a large carrier diffusion length and an efficient energy transfer to RE ions. The sharp Eu^{3+} emissions in GaN originate from the three excited states $^5D_2$, $^5D_1$, and $^5D_0$, which have been already identified in a GaN host using direct Eu excitation.[3] Based on the PL spectra shown in Fig. 1(a) an energy diagram can be established, as shown in Fig. 1(b).[16] It should be noticed that this diagram does not take into account either splittings of Eu levels by the local crystal field or the possible existence of different sites. Also note that transitions of electrons within the $4f$-shell are restricted by the selection rules applicable to electric and magnetic dipole or quadrupole, vibronic and phonon processes. The $^5D_0 \rightarrow {}^7F_2$ transition resulting in strong emission lines around 620 nm is J-allowed electric dipole radiation that has been identified by previous authors.[1–9] Some weaker lines [identified by an asterisk in Fig. 1(b)] are identified here that originate from intra-f partially allowed transitions. However, some transitions are overlapped, as for example the $^5D_0 \rightarrow {}^7F_4$ and $^5D_1 \rightarrow {}^7F_6$ lines and the levels are split by the local crystal field so that alternative methods have to be used for further identification. It has to be remarked that the positions of Eu energy levels with respect to the GaN band gap are not clear at this stage. In particular, there is no reason to make the $^7F_0$ ground state coincide arbitrarily with the top of the valence band. As a matter of fact no emission from the $^5D_3$ excited state has been reported, although the energy separation between the $^5D_3$ excited state and the $^7F_0$ ground state is smaller than the band gap of GaN. In the 300 K PL spectrum [Fig. 1(a)], hardly any transition from the $^5D_2$ level can be found. To address this issue and to confirm the assignment of the transitions, the temperature dependence of the PL has been analyzed in Fig. 2. The thermal quenching is different for transitions belonging to different excited states. The temperature dependence of all transitions originating from the $^5D_2$ excited state is rather similar, characterized by the stron-

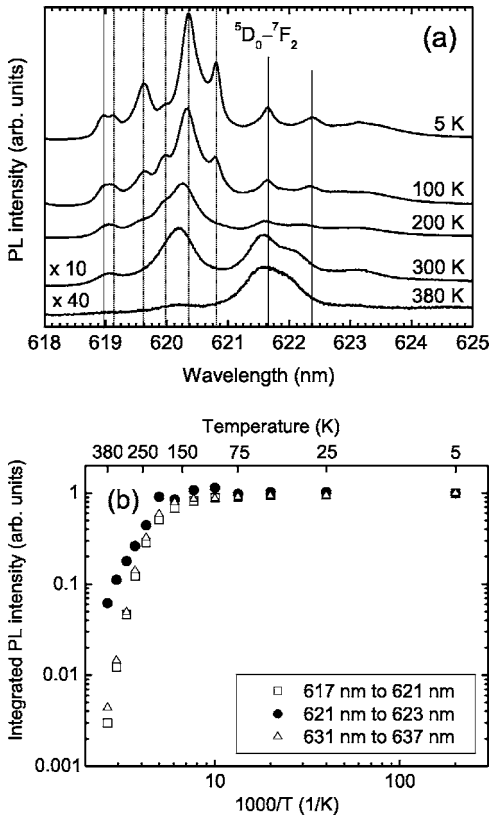

FIG. 3. (a) PL spectra from GaN:Eu measured at various temperatures between 5 and 380 K, as indicated. The spectra are normalized by the excitation power density and the integration time for detection. The spectra at 300 and 380 K are multiplied by factors of 10 and 40, respectively, for clarity. The excitation wavelength was 350 nm. The vertical lines are guides to the eye. (b) Integrated PL intensity between 5 and 380 K of $^5D_0 \rightarrow {}^7F_2$ transition from 616.0 to 621.2 nm (unfilled square), from 621.2 to 622.8 nm (filled dot), and from 630.0 to 637.0 nm (unfilled triangle).

gest quenching. Consequently, the emission intensity falls under the detection limit of our setup for temperatures higher that 200 K. Energy transfer from host carriers to RE ions usually takes place via a RE-related trap. The strong quenching of $^5D_2$ transitions could imply an energy back-transfer process from $^5D_2$ to this trap level.[17,18] Emissions originating from the $^5D_1$ excited state show higher thermal stability and emissions from the $^5D_0$ excited state are the most stable. This specific behavior of each excited state strongly supports the above transition assignment, although details of the thermal quenching are not fully understood yet, e.g., the increase of the $^5D_1 \rightarrow {}^7F_0$ emission intensity from 5 to 75 K.

Along with the thermal quenching of the emission, a systematic blueshift with increasing temperature was observed. This blueshift was different for each emission line. It was as large as 0.7 nm for emission at 622.4 nm as shown in Fig. 3(a). The origin of this blueshift is not understood. It is opposite to the usual redshift related to the temperature dependence of the band gap and the redshift observed for $LaCl_3:Gd^{3+}$.[19]

We will now focus our attention on the luminescence lines in the 617–628 nm wavelength range. According to the energy diagram in Figs. 1(a) and 1(b), they could be assigned to the $^5D_0 \rightarrow {}^7F_2$ and $^5D_2 \rightarrow {}^7F_6$ transitions. The emission intensity from the emission lines from the $^5D_2 \rightarrow {}^7F_6$ transition is expected to be much lower, especially at room temperature [see Fig. 3(a)]. Therefore, only the $^5D_0 \rightarrow {}^7F_2$ transition is considered here. The $^7F_2$ state ($J=2$) could be split by local symmetry C_{3v} (hexagonal GaN) into degenerate doublets and singlets or only singlets in lower symmetry. Thus, the $^5D_0 \rightarrow {}^7F_2$ transition is expected, giving rise to three transition lines in GaN material if there exists only one site in C_{3v} symmetry. However, as seen in Figs. 1(a) and 3(a), extra lines are observed, as evidence of local symmetry lowering and/or that several Eu^{3+} sites are present. Actually, at least 15 emission lines were identified in the spectral range of 617 to 628 nm resulting from the $^5D_0 \rightarrow {}^7F_2$ transition. However, for the lowest site symmetry of the Eu^{3+} ion, the $^7F_2$ level can be split into five sublevels corresponding to the maximum fivefold emission from the $^5D_0 \rightarrow {}^7F_2$ transition. This points out the existence of different Eu^{3+} sites in our MBE-grown GaN:Eu, as previously considered also in Ref. 20.

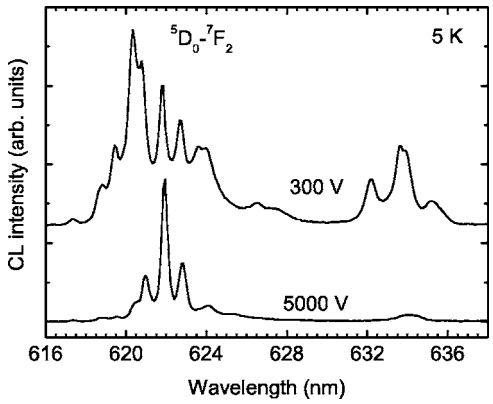

FIG. 4. CL at different depths of GaN:Eu measured at 5 K. 300 and 5000 V were used for excitation near the surface and for the inner sample, respectively. For better clarity the emission intensity has been normalized to its value at 623 nm.

To characterize the different sites of Eu^{3+} ions, the PL of the $^5D_0 \rightarrow {}^7F_2$ transition has been measured from 5 to 380 K [Fig. 3(a)]. Two different sets of Eu^{3+} ions can be identified by comparing the thermal quenching of the various emission lines: one set with lines at 621–623 nm exhibiting a thermal quenching of about one order of magnitude from 5 to 380 K, and another set with lines at around 617–621 nm and a quenching of nearly three orders of magnitude [Fig. 3(b)]. Detailed analysis reveals a much more complicated behavior. Emissions at 617.7 and 619.9 nm show, for instance, an increase of the intensity between 5 and 100 K, followed by a decrease for higher temperatures, which is not yet understood.

Clues as to the origin of the different Eu^{3+} sites have been obtained by probing luminescence as a function of depth through CL experiments performed for different accelerating voltages.[21–23] Figure 4 shows two CL spectra measured for accelerating voltage of 5000 and 300 V, which correspond to carrier generation depths of about 100 and 5 nm, respectively. The spectra are remarkably different and allow us to

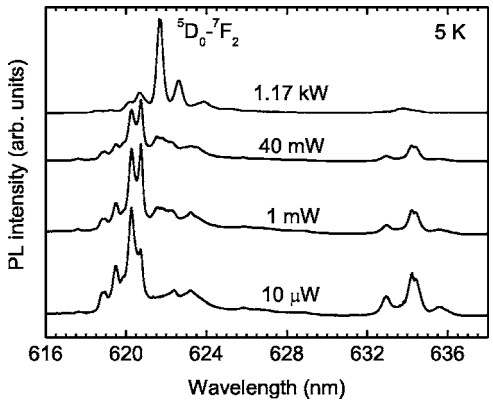

FIG. 5. (a) PL spectra of a GaN:Eu layer measured at 5 K with different excitation powers as indicated in the figure. The spectra are normalized to the emission at 622 nm. The excitation source was a frequency-doubled Ar-ion laser line emitting at 244 nm. The radius of the focused laser spot was 0.2 mm. The spectrum with 1.17 kW excitation power has been measured with a pulsed Nd:YAG laser emitting at 266 nm.

distinguish the same two groups of lines: emission lines at around 617–621 nm are as strong as those at 621–623 nm for low accelerating voltage, but become much weaker for higher accelerating voltage. Emission lines at 634 nm follow the same trend as those at 617–621 nm. From the energy diagram in Fig. 1, they should be assigned to the $^5D_1 \rightarrow {}^7F_4$ transition. However, their other optical properties (thermal quenching, saturation effect, PLE; see below) are also found to be very similar to those at 617–621 nm, so that their assignment is not clarified yet. Strictly speaking in a CL experiment, increasing the accelerating voltage should generate free carriers deeper in the sample volume, but also increase their number, which roughly scales as $V/3E_g$, where V is the accelerating voltage and E_g the band-gap energy. Since both the number of free carriers and the extension of the generation volume are varied, the CL result can be interpreted by assuming two types of Eu sites which differ either by their concentration, i.e., small or large numbers of sites randomly distributed in the sample, or by their location, i.e., sites located near the surface or deeper in the volume. In the first case, emissions at 617–621 nm and 634 nm should be related to sites with a small concentration, so that their relatively weaker intensity for higher accelerating voltage could be induced by a saturation effect. In the second case, these emissions should be associated with Eu sites located near the sample surface. Then their intensity should decrease when exciting deeper in the volume with higher accelerating voltage.

It is interesting to note that both CL interpretations imply that emissions at 617–621 nm and 634 nm could be saturated since the number of "surface" Eu ions is also limited. Figure 5 shows PL spectra obtained at 5 K, using excitations at 244 and 266 nm for different powers. These excitations are well above the band gap of GaN (around 354 nm at 5 K), and are mostly absorbed within a surface layer of 10–20 nm thick, which allows probing Eu centers located near the sample surface. With increasing excitation powers, it can be seen that emissions at 617–621 nm and 634 nm become

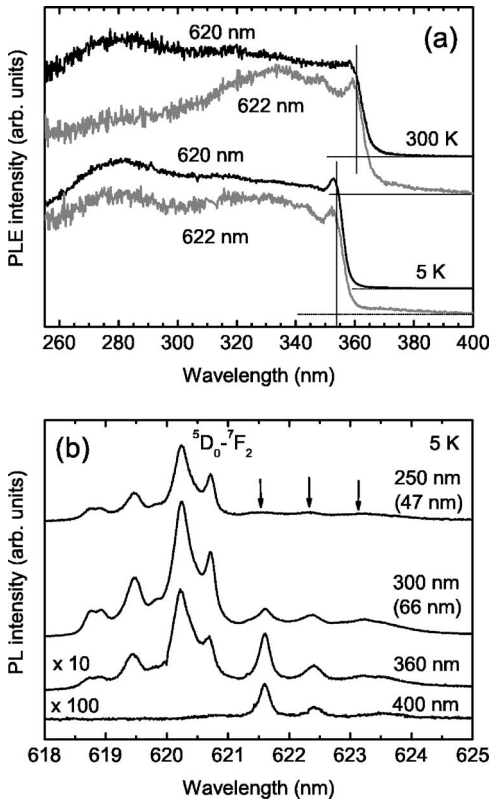

FIG. 6. (a) PLE spectra of a GaN:Eu layer for Eu emissions at 620 and 622 nm (measured at 300 K) and at 620 and 622.0 nm (measured at 5 K). The horizontally dotted lines are PLE base lines. The vertically dotted lines show the band-gap energy of GaN. (b) PL spectra of a GaN:Eu measured at 5 K with different excitation wavelengths (indicated in the curves). The penetration depths until $1/e$ as indicated in the figure are calculated using values from Ref. 24. The spectra are normalized to the excitation power density and the integration time for detection. The spectra under 360 and 400 nm excitation are multiplied by factors of 10 and 100, respectively, for clarity. The arrows indicate the 622 nm lines assigned to Eu^{3+} ions from the volume.

weaker with respect to emission at 621–623 nm and eventually saturate when exciting above a power of 1 kW.

Although the saturation effect is consistent with both CL interpretations, several observations are in favor of the surface vs volume interpretation. First, a spectral shift between "surface" and "volume" Eu centers is expected due to some specific surface effects such as strain relaxation at the surface or the Fermi level band bending induced by surface defects, which is observed between the two sets of lines of interest. Second, Eu centers located near the surface should be more sensitive to thermal quenching through nonradiative channels because of the higher defect densities at the surface. Figure 3 shows that the thermal quenching is stronger for lines at 617–621 nm and 634 nm, which suggests that they are related to surface centers.

In order to study the excitation mechanism of the Eu^{3+} ions in our sample, PLE was carried out for emission at 620, 622, and 633 nm at 5 and 300 K. Figure 6(a) presents the PLE spectra, which are vertically shifted for clarity. In general, the spectra are characterized by strong above-band-gap

absorption with a steep decrease at around the band edge (at 356.2 and 362.4 nm at 5 and 300 K, respectively), followed by a very weak absorption tail at the long-wavelength side. No well-resolved trap absorption at around 400 nm (Refs. 3 and 9) could be resolved in our GaN:Eu sample. In fact, PLE at 400 nm and longer wavelength (not shown) is more than 100 times weaker than for above-band-gap excitation, and only exists in the case of the emission at around 622 nm, indicating that most Eu^{3+} centers in our sample are excited by an energy transfer mechanism via the host band gap and/or very shallow traps close to the band edge. The PLE at 633 nm shows similar characteristics to those at 620 nm, in agreement with previous studies.[12] A small redshift from 5 to 300 K of the near-band-edge resonance is assigned to the usual band-gap thermal narrowing effect.

One possible indication of the sample quality is the linewidth of the Eu emission lines. We found for the $^5D_2 \rightarrow {}^7F_0$ transition a linewidth of 0.1 nm and for the $^5D_0 \rightarrow {}^7F_2$ transition a linewidth of about 0.3 nm. These small linewidths are indicating an unperturbed local environment of Eu atoms. Results in literature on the $^5D_0 \rightarrow {}^7F_2$ transition are rather scattered: for example, a linewidth of 1.6 nm is reported in Ref. 3 for a sample grown by MBE on a *p*-type Si(111) substrate and a linewidth of 0.4 nm for an Eu-implanted MOCVD-grown layer of GaN.[25] These results show that growth conditions, such as cell and substrate temperatures, as well as dislocation densities of substrates, have a strong influence on the linewidth of optical transitions, and thus on the sample quality. Moreover the Eu content can play a significant role since too high Eu concentrations can yield polycrystalline growth of GaN in MBE.[5]

It is interesting to notice that the implantation method allows producing doped samples with unperturbed Eu environments comparable to those found in MBE-grown samples.

The strongest argument for the surface vs volume interpretation is provided by selectively excited PL measurements shown in Fig. 6(b). The tunable excitation source was a 500 W Xe lamp filtered by a double-grating monochromator, producing typical excitation densities below 200 $\mu W/cm^2$. Therefore these measurements at very low excitation should not induce any saturation effect (as shown in Fig. 5), and the only varying parameter was the absorption depth of the excitation beam. This depth depends on the chosen excitation wavelength, which would be smaller than 10–20 nm for above-band-gap excitation at 250–300 nm (because of the strong optical absorption), and would extend deeper in the volume for below-band-gap excitation at 360–400 nm. Data in the figure show that above-band-gap excitation produces intense lines at 617–621 nm (and at 634 nm) which should be assigned to Eu centers close to the sample surface. By contrast, lines at 621–623 nm become stronger for below-band-gap excitation, and should come from Eu centers located deeper in the volume.

It is interesting to notice that a GaN layer grown by MOCVD implanted with Eu atoms shows no emission at around 620 nm, which is understandable because Eu ions are implanted typically deeper inside the sample,[25] leading to the absence of lines identified in the present work as being related to sites close to the surface.

IV. CONCLUSION

In conclusion, photoluminescence, photoluminescence excitation, and cathodoluminescence studies of MBE-grown GaN:Eu layers have put in evidence two different sites of Eu^{3+} located either near the surface or inside the volume. Theses sites exhibit markedly different crystal-field splitting, thermal quenching, and dependence on optical and electron beam excitations. The sharpness of Eu^{3+} emission lines has enabled the observation of a systematic spectral blueshift with increasing temperature.

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