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Cathodoluminescene study of Mg implanted GaN: the impact of dislocation on Mg diffusion

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Magnesium (Mg) ion implanted homoepitaxial GaN layers is investigated by cathodoluminescence (CL) and secondary ion mass spectrometry (SIMS). The impact of dislocations on Mg diffusion is clarified by CL monitoring the Mg-related donor-acceptor pair (DAP) emission on novel angle cutting specimen. CL results suggest that: (1) there exist high concentration of nonradiative defects in a Mg implanted layer; and (2) Mg shows pipe diffusion along threading dislocations throughout epilayer to substrate. To achieve successful Mg doping by ion implantation, it is necessary to suppress the formation of a dead region in the Mg implanted layer and the pipe diffusion along threading dislocations.

Wide bandgap semiconductor materials attract a great deal of attention for their potential application in future power electronics.[1] Among them, GaN-based power devices is one of the promising candidates from the material based figure of merit. Lateral structured devices such as AlGaN/GaN high electron-mobility transistors have been practically applied to high-frequency power devices.[2,3] However, the performance and reliability of lateral devices is limited by the presence of high density dislocations (10^8–10^9 cm^{-2}) due to large lattice mismatch with foreign substrates. With the progress in free-standing GaN substrates with low dislocation density (10^4–10^6 cm^{-2}), vertical structures attract more interest due to higher breakdown voltage. p-n diodes fabricated on a homoepitaxial GaN epilayer grown on pseudo bulk GaN substrates demonstrate the approach possibility of high breakdown voltage up to 4 kV.[4] By introducing multiple n-GaN drift-layer structures fabricated on free-standing GaN substrates, vertical GaN p-n junction diodes with high breakdown voltage over 4 kV have been achieved.[5]

The achievement of conductive p-type GaN is important for GaN-based power devices. Mg doping is so far the only available method to obtain p-type conductivity in GaN. In 1989, p-type GaN has been realized for the first time in GaN: Mg film grown by metalorganic vapor phase epitaxy (MOVPE) followed with low-energy electron-beam irradiation.[6] Due to the presence of Mg-H complexes, post-growth thermal anneal is necessary to activate Mg as an acceptor in films grown by metalorganic chemical vapor deposition.[7] Mg atoms substitute Ga site and form shallow acceptor Mg_{Ga} with the ionization energy about 170 ~ 220 meV.[8–10] Mg doped GaN shows the characteristic donor–acceptor pair (DAP) emission around 3.27 eV at low temperatures as revealed by photoluminescence (PL) studies.[11,12] In heavy Mg-doped GaN, broad blue luminescence exists at 2.7 ~ 2.9 eV due to a deep donor to Mg acceptor.[13–15] To realize precise p-type doping in vertical devices, Mg ion implantation is an important technique. However, Mg ion implantation processes is still under development and need optimization. It is reported that a high density of point defects and vacancy clusters in Mg implanted layers could act as non-radiative centers and suppress p-type conductivity.[16]

Most optical characterizations have limitations in the spatial resolution, it is difficult to know the distribution of activated and non-activated Mg. On the other hand, for vertical structures, the presence of crystal defects like dislocations may bring potential detrimental effects leading to device failure. At present, dislocations still remain in GaN substrate and epitaxial growth, the effect of dislocations on Mg diffusion should also be explored. Cathodoluminescence (CL) is a light emission based on electron beam injection. It can be used for the characterization for not only the nature of defects or impurities but also their distributions in the material.[17] It is extensively used for defect characterization in GaN, such as dislocations,[18] stacking faults,[19] as well as pit defects.[20] In this study, we perform CL observation on Mg ion implanted homoepitaxial GaN layers by adopting a novel angle cutting cross-sectional sample preparation method. By this method, the spatial distribution of DAP emission with in-depth analysis is obtained, and the dislocations either in epilayer or substrate could be visualized in the angle cutting plane.

A Mg ion implanted GaN sample structure is shown in Fig. 1(a). The substrate is *c*-plane free standing GaN substrate grown by the hydride vapor-phase epitaxy (HVPE) method. The substrate is n-type (Si doped) with a carrier concentration of 1.75×10^{18} cm^{-3}. Homoepitaxial GaN layer with a thickness of 4 μm is grown on the substrate by MOVPE. The epilayer is Si doped GaN with a carrier concentration of 1.5×10^{16} cm^{-3}. Mg ions are implanted into this epilayer with a 500 nm deep box profile at room temperature. The detail procedure of Mg ion implantation has been reported in Ref. 16. The implanted Mg concentration is of 1×10^{19} cm^{-3} as characterized by secondary ion mass spectrometry (SIMS). The activation of Mg is performed by high temperature annealing at 1300 °C for 5 min with AlN protection layer, then AlN is chemically removed. Figure 1(b) shows the SIMS depth profile of Mg concentration in as-implanted and annealed. After annealing, in the depth about 600 ~ 1000 nm, the Mg concentration is slightly increased. The diffusion of Mg is limited in the upper part (<3 μm) of the epilayer.

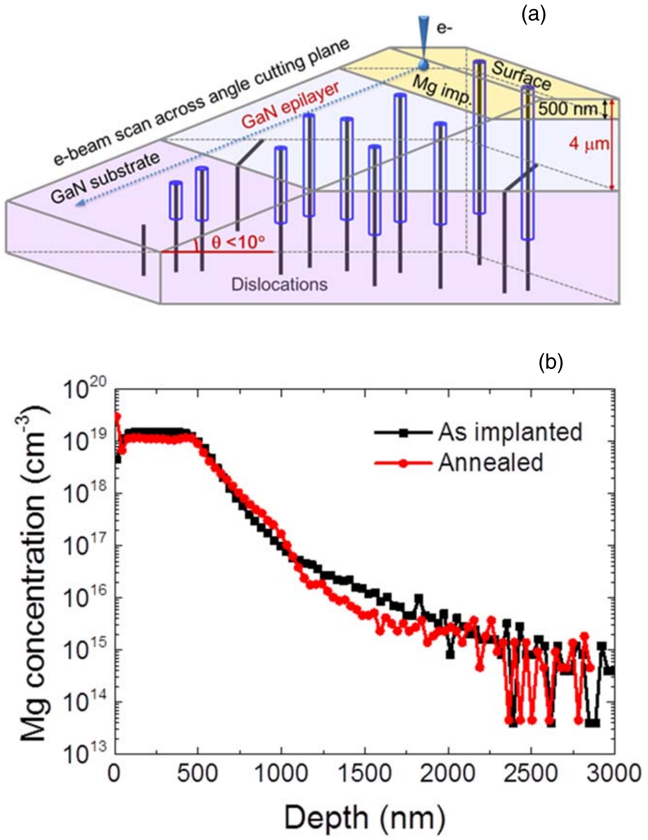

Fig. 1. (Color online) (a) Mg ion implanted GaN sample structure. For CL observation, the e-beam scans across the angle cutting cross-section plane. Schematic of Mg pipe diffusion along threading dislocations. (b) SIMS depth profile of Mg concentration.

Cross-sectional observation is necessary to get the depth distribution of Mg and dislocations. Regarding general cross-sections perpendicular to the top surface, the width of Mg implanted layer is only around 500 nm. Since the electron range of 3 kV is around 100 nm, it is difficult to improve the depth resolution. We introduce an elongated cross-section with a small inclination angle (less than 10°) to the surface, as shown in Fig. 1(a). This angle cutting method enlarges the specimen depth into the lateral direction. The distance of the cross-section is increased by a factor of 5 ~ 10, and threading dislocations could be imaged by e-beam scanning across the angle cutting plane. The angle cutting is performed by a cross-section polisher (JEOL SM-09010), which used an Ar ion beam at low beam energies (4 kV) with a shield plate. By using low energy ion beam, it is able to prepare cross-section samples with less surface damage and degradation in the luminescence. CL observations were performed with an e-beam of 3 kV and specimen temperature of 78 K, by a HORIBA MP32 CL system attached to a Hitachi SU6600 field-emission scanning electron microscope (FE-SEM). The GaN substrate used in this study contains regions with low- or high-density dislocations due to facet growth or the dislocation elimination technique. The density of dislocation is estimated based on CL images. It is in the order of 10^7 cm^{-2} for high density region, and about 10^6 cm^{-2} for low density region.

Near band edge (NBE) emission at 3.47 eV is observed in the deeper n-type region in both as-implanted and annealed samples. Mg-related DAP emission at 3.28 eV is only detected in annealed samples, which suggests that the as-implanted state is not activated in an as-implanted samples. The post-annealing process not only reduces the heavy damage induced by ion implantation, but also activates Mg as an acceptor by substituting Ga-sites.[21] Figure 2 shows the monochromatic CL images of DAP and NBE emission obtained from annealed sample. This region is with low density dislocations. Several dark spots in the right bottom are surface dust particles. The 4 μm thick epilayer is extended to 28 μm due to the cutting angle θ about 8.1°. The regions of surface, epilayer and substrate are denoted in Fig. 2(a). In this DAP emission image, the Mg-implanted layer is of weak luminescence due to the presence of high density nonradiative centers. The nonradiative centers are associated with a complex Ga vacancy (V_{Ga}) and nitrogen vacancy (V_N), which change to vacancy clusters $(V_{Ga})_3(V_N)_3$ after annealing above 1000 °C, as reported by recent PL and positron annihilation spectroscopy (PAS) studies.[22,23] Beneath the implanted layer, these exists a bright zone with significant DAP

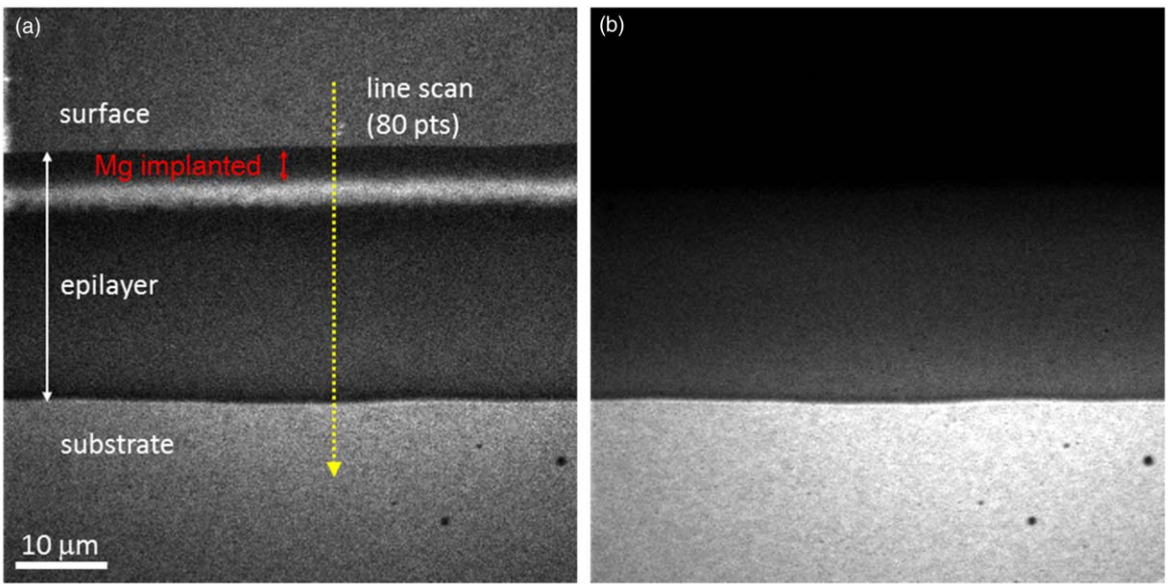

Fig. 2. (Color online) The monochromatic CL images of a region with few dislocations taken at DAP (a) and NBE (b) emissions.

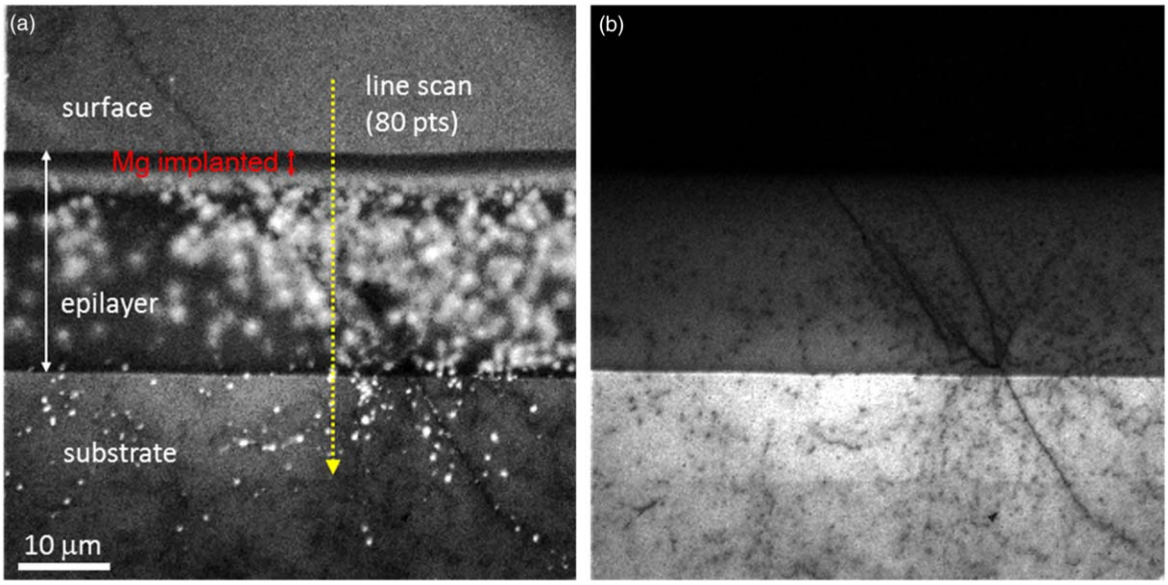

Fig. 3. (Color online) The monochromatic CL images of a region with high density dislocations taken at DAP (a) and NBE (b) emissions.

emission. This region is located below implantation BOX with minor implantation damage and few nonradiative defects, thus strong DAP emission is dominant in this region. Figure 2(b) shows the NBE emission from substrate is stronger than that from epilayer due to a higher carrier concentration.

Figure 3 shows the DAP and NBE emission images of a high-density dislocated region. The epilayer is extended to 24 μm with a cutting angle θ about 9.4°. In the DAP emission image shown in Fig. 3(a), the Mg-implanted region is also dark, and the p-type region beneath the Mg-implanted layer is also a bright zone. Unexpectedly, many bright spots are observed in epilayer and substrate. By comparing with the NBE emission image shown in Fig. 3(b), almost all the bright spots in the DAP emission image corresponds to the dark spots in the NBE emission image. The threading dislocations are visualized as dark spots or lines due to their non-radiative centers for NBE emission. This indicates that DAP emission is enhanced around dislocation.

Compared to the small and sharp bright spots in the substrate region, the bright spots are obviously large and blurred in the epilayer. This may be explained by the following reasons. The first consideration is the variation of minority carrier diffusion length in-between epilayer and substrate. Since Si doping concentration in the substrate is two orders of magnitude higher than that in the epilayer, the substrate tends to have a shorter diffusion length of minority carrier compared with the epilayer. Minority carriers injected into substrates recombine quickly at the dislocations accompanied with radiative recombination centers (activated Mg acceptors in this study), and thus these dislocations are visualized as smaller bright spots. On the other hand, minority carriers into the epilayer undergo longer distance before recombination. The second reason is that there may be Mg out-diffusion from dislocation core to the matrix, and it may happen in the epilayer more significantly.

For detailed analysis, CL line-scan is done from the surface to substrate in both low and high dislocation density regions as illustrated by the dotted lines in Figs. 2(a) and 3(a), respectively. In low dislocation density regions, Fig. 4(a) shows the color-code CL intensity dependence of photon energy and depth position. The obviously weak DAP emission is observed in the deeper epilayer region. Typical spectra of different regions are shown in Fig. 4(b). The substrate (n^+-GaN) shows stronger NBE and its phonon replica emissions than the epilayer (n-GaN) due to higher carrier concentration. The region beneath the Mg-implanted layer exhibits significant intensity of DAP emission (3.28 eV) with its phonon replica (DAP-LO 3.20 eV). It is regarded as active p-type region. The Mg implanted layer (Mg imp.) also shows DAP emission, but the intensity is relatively weak due to the presence of large account non-radiative recombination centers. Note the Mg implanted layer shows broad green emission around 2.4 eV, which is attributed to V_N and V_N-related vacancy complex.[16]

Figure 4(c) shows the CL spectral variation at dislocated region. The intensity of NBE emission is discontinuous, with some breakpoints, corresponding to the presence of dislocations. Unexpectedly, there exist Mg-related DAP emission with DAP-LO from the corresponding points. Figure 4(d) shows the typical spectra corresponding with the marked positions in Fig. 4(c). The intensities of DAP emission from dislocations are comparable to that from p-GaN region. This suggests that these dislocations accommodate activated Mg with the concentration close to the p-GaN region.

For the effective p-type doping by Mg implantation, post-annealing process is the crucial issue for Mg activation by substitution of Ga-site. Thus, DAP emission is only observed in an annealed sample. Meanwhile, high temperature annealing also promotes the diffusion of Mg atoms. The activation energy of Mg diffusion in GaN is derived to be 1.3 eV.[24] In the dislocation free case, the concentration of Mg exponentially decreases from a Mg-implanted layer to the deeper region, as shown by the SIMS profile of Mg in Fig. 1(b). As a result, the DAP emission is intense beneath the implanted layer, and weak in the deeper epilayer and substrate. In high dislocation density region, some dislocations are bright in the DAP emission image in the deeper

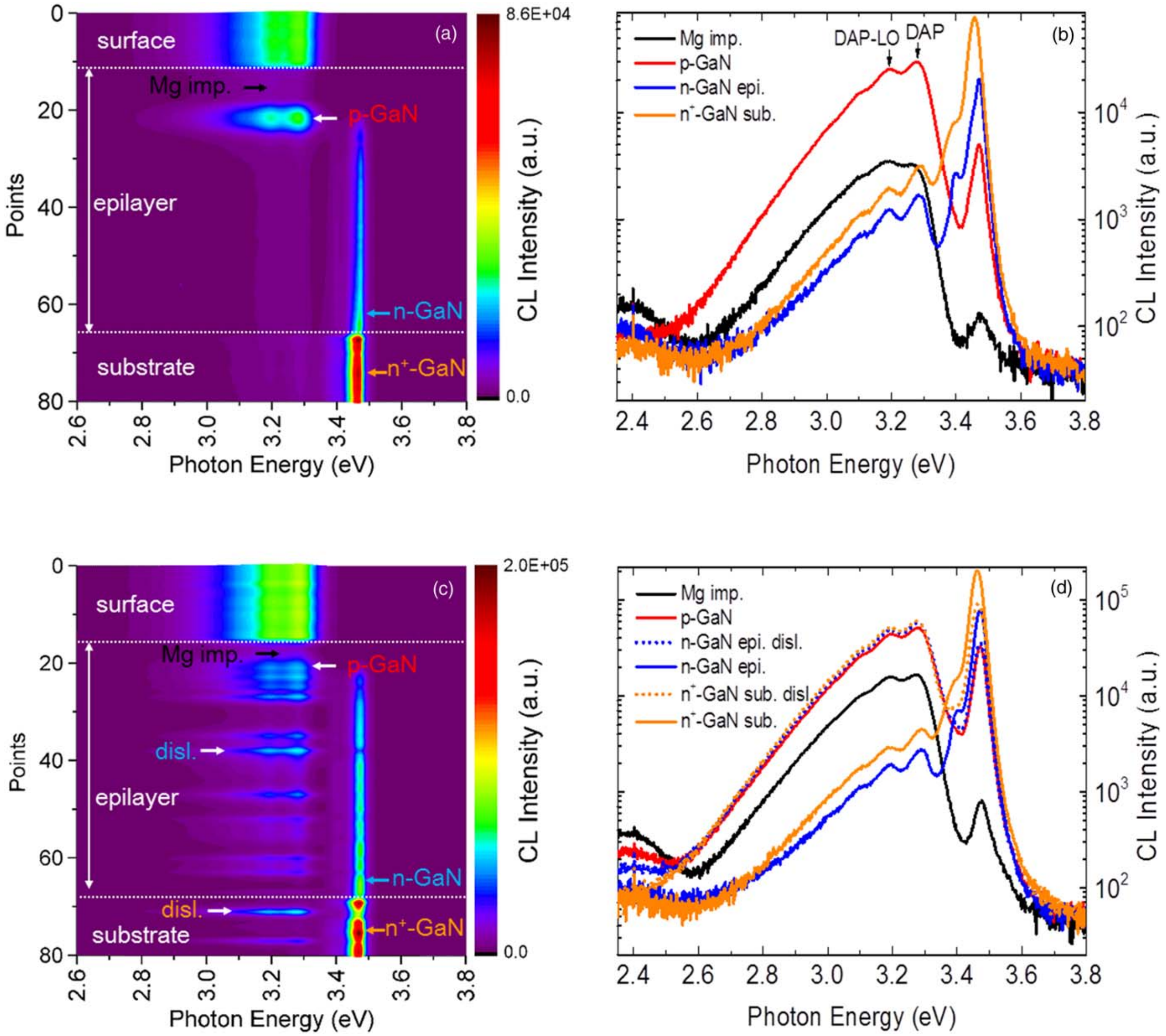

Fig. 4. (Color online) CL line-scan mapping (a) and resolved spectra of different layers (b) in low dislocation density region; CL line-scan mapping (c) and resolved spectra of different sites (d) in high dislocation density region.

epilayer and upper substrate. It clearly shows that active Mg exist with high concentration along the dislocation.

The enhanced Mg at dislocations could be explained in terms of pipe diffusion along dislocations, as illustrate in Fig. 1(a). Theoretical studies suggest that the diffusivity of interstitial Mg at the dislocation core is three orders of magnitude larger than the outside region at 1000 K. The enhanced diffusion is due to the smaller interactive atomic forces along dislocation and Mg atoms are more mobile along dislocation.[25] Some dislocations exist as dark in the DAP emission image implying that not all dislocations act as a diffusion path of Mg. It should be noted that bright dislocations are only observed in heavy Mg-doped (over 10^{18} cm^{-3}) samples that have undergone high temperature annealing.

Previous electron-beam-induced current studies on Mg-doped GaN p-n junction for laser diode applications reported that Mg may heavily diffuse into the n-type region when grown on sapphire substrate with high density threading dislocations.[26] Our study indicates that the presence of dislocations is harmful for Mg implantation. The pipe diffusion of Mg along dislocation will result in the inhomogeneity and degradation of the junction. Dislocations with p-type conductivity may act as current shunts and lead to the leakage and/or breakdown of devices. To avoid the potential devastating effects, it is necessary to reduce the numbers of threading dislocations, and to optimize the activation of Mg for implantation. Considering the growth of GaN substrate, to reduce the density of dislocations, special techniques have been developed such as epitaxial lateral overgrowth from opened window area of the mask film,[27] and dislocation elimination by epitaxial growth with inverse-pyramidal pits in which dislocations converge to the center of hexagonal pits.[28] These methods can help to eliminate dislocations within a large area, except for the center regions of pits. The dislocation distribution is not uniform; the center region tends to contain a high density of dislocations. For such substrates, the impact of dislocation on Mg diffusion should be taken into account for fabricating GaN power devices.

In summary, Mg implanted GaN layer and the impact of dislocation on Mg diffusion have been studied. The Mg implanted layer shows relatively weak DAP emission due to the presence of many non-radiative recombination centers introduced by implantation and high temperature annealing. Significant DAP emission is found from the region beneath the Mg implanted layer. Threading dislocations show an enhanced DAP emission comparable to the p-type region. It is speculated that a certain amount of activated Mg atoms exist around dislocations. This abnormal fast diffusion of Mg along dislocations is due to pipe diffusion occurred during high temperature annealing.

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