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Invited Article

Rare earth ion implantation and optical activation in nitride semiconductors for multicolor emission

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Abstract

In order to understand the behavior of nitride semiconductors when submitted to ion implantation, we have used 300 keV europium at fluences from 10¹² to above 10¹⁷ ions cm⁻². Subsequently, Rutherford backscattering (RBS), x-ray diffraction (XRD), and transmission electron microscopy (TEM) were used to investigate the evolution of damage. The optical properties were investigated prior to and after annealing. It was found that the behavior of the three compounds (AlN, GaN InN) under ion implantation is rather different: whereas InN breaks down at very low fluences (∼10¹² ions cm⁻²), the damage formation mechanisms are similar in AlN and GaN. In both compounds, extended defects such as stacking faults play a critical role. However, they exhibit different stability, as a consequence, GaN transforms to nanocrystalline state from the surface at a fluence of around 2.5 × 10¹⁵ ions cm⁻², whereas AlN undergoes a chemical amorphization starting at the projected range (Rₚ), when implanted to extremely high Eu fluences >10¹⁷ ions cm⁻². As for the optical activation, the formation of highly stable extended defects in these compounds constitutes a real challenge for the annealing of heavily doped layers, and it was noticed that for a substantial optical activation, the implantation fluences should be kept low (<10¹⁵ Eu at cm⁻²).

Keywords: nitride semiconductors, rare earth emission, ion implantation, damage formation, optical activation, transmission electron microscopy, mechanisms

(Some figures may appear in colour only in the online journal)

1. Introduction

During recent years, rare earths (REs) doping of nitrides has attracted worldwide interest due to expected applications in optoelectronics and photonics [1]. Indeed, a number of results have been published on the incorporation of REs in III/N hosts which for instance demonstrated laser action in Eu-doped GaN at room temperature [2], or generation of blue-UV emission with REs in AlGaN [3] in layers grown and doped in situ by molecular beam epitaxy. Subsequently, light emitting diodes have also been reported in Eu doped GaN layers grown by metalorganic vapour phase epitaxy [4]. Obviously, for the semiconductor industry, ion implantation is one of the basic tools to tune the properties and functionalities of materials [5]. In the nitride semiconductors, early attempts have also been made using ion implantation at medium range energy (1 to a few hundred keV) in order to investigate the n or p doping with Si or Mg species [5], or to incorporate optically active rare earth elements [6]. Of course ion implantation always affects the electrical and optical properties due to generation of crystallographic disorder leading to the introduction of numerous charge carrier
recombination paths. In the case of nitride layers, the implantation damage comes on top of the numerous growth defects that may be present depending on the growth conditions and techniques. Such defects are dislocations [8–12], stacking faults which can be in the basal [13–16] or prismatic planes [16–18], and even inversion domains boundaries [19–22].

As usual, thermal treatment at high temperatures is necessary in order to anneal implantation damage and to gain some electrical and/or optical activity for the implanted ions. Interestingly, early investigations pointed out that the nitride semiconductors should be subject to either moderate (InN) or dominant (GaN, AIN) dynamical annealing and thus exhibit substantial resistance to damage formation [23].

From the pure point of view of damage formation, in GaN, it was reported that the amorphization was a layer-by-layer process starting from the surface [24] and that the extended defects that formed beyond the amorphous layer were predominantly planar defects independently of the implanted ion, energy and temperature [25]. In contrast, our recent studies showed the formation of a nanocrystalline surface layer rather than amorphization, as discussed below. For AIN, we have shown that ion implantation results in amorphization, in contrast to GaN, although, at very high fluences [26]. Early investigations of InN put the threshold for amorphization just above silicon carbide, in the $10^{14}$ at cm$^{-2}$ fluence range for 100 keV Si ion implantation at room temperature [23]. In recent years, InN material’s quality has been strongly improved using molecular beam epitaxy (MBE) [27, 28], as well as metal organic vapour phase epitaxy (MOVPE) [29, 30]. Moreover, as for the other nitride compounds, highest growth rates for this substantially low temperature (450–600 °C) material are also possible to achieve by hydride vapour phase epitaxy (HVPE), where some 4 μm h$^{-1}$ growth rates [31] can be obtained around 500 °C achieving reasonably good quality material [32]. The availability of such good quality InN layers has motivated us in carrying out the implantation of this additional nitride compound.

The annealing out of implantation disorder in nitride compounds is not expected to be easy. As calculated by Van Vechten [33], the melting point of, for instance, GaN is quite high (2500 °C), and one would expect that annealing temperatures of around 1600 °C are necessary in order to be able to remove most of the implantation damage. At these high temperatures, annealing of nitride compounds is limited by material decomposition; i.e. by loss of nitrogen from the surface, for instance, in the case of GaN, surface decomposition becomes pronounced for anneals at temperatures above 800 °C [34, 35].

In the following, we review our results on the investigation of the damage formation in InN, GaN and AIN layers subsequent to Eu ion implantation along with the attempts to attain optical activation of the ions in relation with literature. It is pointed out that the sensitivity to damage formation by heavy ion implantation is highest for InN which presents a pronounced instability and the lowest in AIN which was found to be extremely resistant to damage formation.

2. Experimental procedures

Using GaN, AIN, and InN layers grown by MOVPE, MBE, or HVPE, the Eu implantation was carried out at room temperature (RT) at the energy of 300 keV; the fluence was varied from $10^{12}$ to the mid $10^{15}$ ions cm$^{-2}$. In order to characterize the samples, we first carried out Rutherford backscattering/channelling (RBS/C) using a 1 mm diameter collimated beam of 2 MeV He$^+$ ions. The backscattered particles were detected at 140° and close to 180° with respect to the incoming beam direction with silicon surface barrier detectors. ‘Aligned’ spectra were taken along the c-axis and ‘random’ spectra were acquired by tilting the sample by 5° and rotating it during the measurement. In parallel, XRD measurements were performed on a two-circle goniometer with the Cu Kα1 radiation (λ = 0.15405 nm) provided by a 5 kW Rigaku RU-200 generator with a vertical linear focus in combination with a quartz monochromator. Symmetric $\theta$–$2\theta$ scans were recorded around the (0004) Bragg reflection ($2\theta_B = 73.035°$) for which the penetration depth of x-rays in GaN (εN ≈ 8 μm) is significantly larger than the extension of the implanted layer. The strain along the surface normal (normal strain), $\varepsilon_N$, was directly determined by plotting the $\theta$–$2\theta$ curves vs $q[0001]/H(0004)$, where $q[0001]$ is the component of the deviation vector along the surface normal [0001], from the reciprocal lattice vector H(0004) of the (0004) planes [36]. Finally, conventional transmission electron microscopy (C-TEM) and high resolution TEM (HRTEM) were carried out on cross sections of the same samples which were previously thinned down to 100 μm by mechanical grinding and dimpled down to 15 μm. For observations, the electron transparency was achieved by ion milling at 5 keV using a GATAN precision ion polisher system (PIPS) at an incidence angle of 5° with the sample holder kept at liquid nitrogen temperature. In order to further...
minimize the ion beam damage, a final milling step was carried out with the beam energy decreased to 0.6–0.7 keV.

3. Results

In this section, we first present the results on the damage formation on GaN (section 3.1), AlN (section 3.2) and InN (section 3.3). We then discuss the damage formation mechanisms which characterize the behavior of these compounds when submitted to ion implantation (section 3.4) and we finally deal with the ways to optical activation of the Eu³⁺ ions (section 3.5).

3.1. Gallium nitride

In contrast to earlier reports [23], our detailed investigations by TEM, RBS and XRD subsequent to ion implantation in GaN have shown that the damage build up does not take place as a layer-by-layer amorphization which proceeds from the surface. Indeed, from RBS investigations (figure 1), it can be pointed out that the notable damage build up which is clearly starting at the fluence of $6 \times 10^{14}$ Eu cm$^{-2}$ proceeds simultaneously in the bulk (around 100 nm) and at the very surface of the implanted layer ($\leq$20 nm depth). At $10^{15}$ Eu cm$^{-2}$, the relative defect concentration inside the bulk saturates, whereas the surface peak increases rapidly, eventually saturating at a relative defect level of $\sim$0.9 starting at $\sim 2.5 \times 10^{15}$ Eu cm$^{-2}$. When the fluence is further increased, the highly defective layer extends towards the bulk, but the relative defect concentration does not reach 1, which means that the surface layer does not become completely amorphous. This is confirmed by the TEM observations, as shown in figure 2 which is an HRTEM image of the GaN surface layer recorded on the $4 \times 10^{15}$ Eu cm$^{-2}$ implanted sample. As can be seen, for a depth of some 50 nm below the surface (black arrow), the layer exhibits a nanocrystalline structure with randomly misoriented crystallites as shown by the corresponding diffraction pattern. The average size of the nanocrystals is about 3–4 nm as attested by the size of the diffraction rings (inserted diffraction pattern). The interface of this surface layer with the underlying damaged part which is still monocrystalline (below the two white arrows) is rough, as has been underlined by a black line in the figure. At lower fluences, the predominant defects have been shown to be the basal [15] and prismatic stacking faults [18]; they start to form at the lowest fluences mostly in the bulk and constitute a network which propagates towards the surface, reaching it at around $2 \times 10^{15}$ Eu cm$^{-2}$ [37–39]. From the combination of the XRD data with TEM and RBS data it was possible to identify four regimes in the build-up of the damage in GaN [38]: (i), below $2 \times 10^{15}$ Eu cm$^{-2}$, the normal strain (XRD) increases progressively with the formation of both point defect clusters and stacking faults in the implanted layer. (ii), then from $2 \times 10^{14}$ to $1 \times 10^{15}$ Eu cm$^{-2}$, a first saturation of strain related to a satellite peak around $\varepsilon_{\text{Sat}} = -0.6\%$ takes place along with a continuous increase of the maximum strain up to 1.6%. In this regime, bulk and surface damage increase slowly up to $5 \times 10^{14}$ Eu cm$^{-2}$ and then rapidly. (iii), from approximately $1 \times 10^{15}$ to $2 \times 10^{15}$ Eu cm$^{-2}$ and above, there is no more evolution of the normal strain. This fluence ($2 \times 10^{15}$ Eu cm$^{-2}$) corresponds to the point when the stacking faults network reaches the surface. The strain saturation occurs simultaneously with the saturation of the damage in the bulk part of the implanted area, whilst the surface damage still increases. (iv), above $2 \times 10^{15}$ Eu cm$^{-2}$, the surface damage level saturates at a relative defect level of $\sim$0.9 (figure 1), which is consistent with the formation of the nanocrystalline layer at the surface (figure 2).

3.2. Aluminum nitride

In the report by Williams on amorphization threshold fluences for semiconducting materials [23], it was estimated that under 100 keV Si ion implantation, GaN, AlN and Si would amorphize starting at $2 \times 10^{16}$ Si cm$^{-2}$, $6 \times 7 \times 10^{16}$ and $10^{17}$ Si cm$^{-2}$, respectively. In a comparative investigation, we recently showed that the amorphization of Si took place below the fluence of $10^{14}$ Eu cm$^{-2}$ for 300 keV implantation at room temperature, whereas the damage accumulation in AlN is much slower [40, 41]. Indeed, at the medium range fluences ($5 \times 10^{15}$ cm$^{-2}$), when the GaN surface layer has already been transformed to nanocrystalline state, we see by TEM that, as in GaN, the implanted AlN layer also exhibits stacking faults inside the highly damaged area (figure 3), however the stacking faults network in AlN is buried beyond a 40 nm thick surface layer of good crystalline quality. When the implantation fluence was increased past $10^{15}$ Eu cm$^{-2}$, an amorphous layer formed in place of the SFs layer which falls around Rp and the surface layer remained monocrystalline. Moreover, EDS analysis revealed that this amorphous layer contained high concentrations of Eu (16%) in contrast to the case of GaN where Eu concentration stays below the EDS detection limit at the threshold of the nanocrystallization. Eventually, an attempt was made to anneal this highly damaged system; this was done under nitrogen overpressure.
As can be seen in figure 4, this resulted in the formation of a buried layer which has crystallized with an irregular morphology and has entirely substituted the previous amorphous part. The EDS analysis (inset) and HRTEM imaging have allowed us to identify this intermediate phase as EuO which formed after annealing. Indeed, significant nitrogen depletion in InN has already been reported after irradiation, implantation or ion beam analysis experiments [43–45]. During the implantation, nitrogen release seems to be the most probable reason for the formation of this buried layer with such a typical morphology. Moreover, the InN dissociation could explain the presence of indium oxide nanograins in the N-depleted region. As may be expected, an important N depletion should lead to the formation of metallic In clusters. Subsequently, the oxidation of the metallic In phase may have occurred when the sample was removed from the implantation chamber to air. Indeed, as the formation energy of In₂O₃ (−221.27 kcal mol⁻¹), at room temperature, is significantly lower than that of InN (−4.2 kcal mol⁻¹), metallic indium would exhibit a strong preference to oxidation [46].

3.4. Damage formation processes

As can be observed, during RE implantation at room temperature in GaN and AlN, the highest density of SFs forms in a layer whose width scarcely evolves versus the fluence [39, 47]. This area corresponds to the depth of maximum nuclear energy deposition. Therefore, small SFs and/or precursors like dislocation loops may form during the collision cascade process possibly by the overlapping of the dense cascades due to the energy deposited by the heavy ions. In this layer, additional defects provided during the implantation should either recombine, form clusters or be trapped by SFs which grow in length. This explains our observations as well as those of Kucheyev et al. [48] in GaN which showed that the average size of SFs is larger than the lateral size of cascades, meaning that SFs may not be formed in a single cascade but should instead result from cumulative effects. With increasing fluence, the SF density strongly increases creating a network which can act as preferential diffusion paths for point defects. Interstitials and vacancies in excess migrate through the SFs network towards the bulk and the surface. These processes are common to AlN and GaN implanted in the same conditions as shown above, and the damage formation processes are

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Figure 3. An AlN layer after Eu implantation at 300 keV, and fluence of 5×10¹⁷ at cm⁻², the surface is indicated by the black arrow, the high density stacking fault area is located between the two white arrows.

Figure 4. AlN nitride layer after Eu implantation at 10¹⁷ at cm⁻² and annealing, a EuO continuous layer has formed as identified by its diffraction lines (inset: the zone axis is [110], as well as EDS analysis).

As can be seen in figure 4, this resulted in the formation of a buried layer which has crystallized with an irregular morphology and has entirely substituted the previous amorphous part. The EDS analysis (inset) and HRTEM imaging have allowed us to identify this intermediate phase as EuO which formed after annealing. As can be seen in the image recorded along the [1120] AlN zone axis along with the associated diffraction pattern (inset), there is an almost preferential growth of the EuO precipitates with one of the (111) axes almost parallel to [0001] AlN.

3.3. Indium nitride

At Eu fluences as low as 5×10¹² Eu cm⁻², the InN layer which at the beginning contained numerous defects such as basal stacking faults (figure 5(a)) with an undulating surface, has been highly been damaged (figure 5(b)). Two clearly different contrasts can be seen inside surface layer (1 and 2) which extends to around 130 nm in depth. One can notice a bright contrast at the top part of this layer (1) meaning that in this area the layer has kept some crystalline state, in depth (2), the contrast is darker, meaning that the layer is either amorphous, or is made of misoriented crystallites. This dark buried layer is centred on Rρ and embedded between areas which are defective but exhibit a crystalline contrast. Imaged along the InN [1120] zone axis (figure 6), area 2 is not uniform: at large scale, it is made of a non-homogeneous distribution of round shaped bright features which at higher magnification exhibit small misoriented nanograins embedded in an amorphous matrix (inset). Within our detailed investigation of the electron diffraction patterns recorded in this area, we have shown that the grains exhibit In₂O₃ bixbyite structure [41, 42]. The large bright areas can be identified either as amorphous pockets, voids or N₂ gas bubbles resulting from the dissociation of InN induced by the ion implantation. Indeed, significant nitrogen depletion in InN has already been reported after irradiation, implantation or ion beam analysis experiments [43–45]. During the implantation, nitrogen release seems to be the most probable reason for the formation of this buried layer with such a typical morphology. Moreover, the InN dissociation could explain the presence of indium oxide nanograins in the N-depleted region. As may be expected, an important N depletion should lead to the formation of metallic In clusters. Subsequently, the oxidation of the metallic In phase may have occurred when the sample was removed from the implantation chamber to air. Indeed, as the formation energy of In₂O₃ (−221.27 kcal mol⁻¹), at room temperature, is significantly lower than that of InN (−4.2 kcal mol⁻¹), metallic indium would exhibit a strong preference to oxidation [46].
probably similar. However, the above observations show that the particular damage build up in GaN is due to the fact that the ejection of defects out of the SF layer is accelerated towards the surface. As suggested by our TEM observations and underlined by Wang et al. [49], the basal stacking faults (BSFs) observed in GaN and AlN result from the preferential condensation of interstitials on the basal plane. Moreover, the same authors [49] reported that vacancies preferentially condense on pyramidal planes leading to the formation of prismatic stacking faults (PSFs). Indeed, as has been shown [14, 41, 50], the basal and prismatic stacking faults have nearly equal formation energies in GaN, whereas in AlN, the PSFs have higher formation energies than those of the BSFs, especially I1, which are the main SFs encountered in our samples. However, the amorphization of AlN cannot be considered as a purely mechanical process which leads to a structural breakdown as one usually expects from the ion implantation. In figure 7, we present a micrograph of the subsurface area of the AlN layer which was implanted at $10^{17}$ at cm$^{-2}$, as can be seen, the amorphous layer (between the two horizontal white arrows) is buried under a surface crystalline part. EDS was carried out on this layer, and the results are inserted for three representative positions. In the surface the Eu concentration is below the EDS detection limit (3), towards the middle of the amorphous layer, the Eu

**Figure 5.** Indium nitride. (a) Starting layer before ion implantation, the visible horizontal features are basal stacking faults. (b) After Eu implantation, at the fluence of $10^{12}$ at cm$^{-2}$: an embedded highly damaged layer is marked 2.

**Figure 6.** The Eu implanted InN layer area 2, is made of irregular white features, the top inset exhibits the details inside the white feature: lattice fringes are visible, meaning that there are nanocrystals, the diffraction pattern of the area shows that the main dots which belong to InN are superimposed with more elongated features belonging to the In$_2$O$_3$ bixbyte phase, as can be seen on the compound simulated rings.

**Figure 7.** AlN nitride layer after Eu implantation at $10^{17}$ at cm$^{-2}$ and, the amorphous layer contains about 16% Eu, this concentration drops rapidly to zero towards the surface and the bulk parts of the layer.
concentration is around 16% (2), and it drops rapidly towards the bulk (1). As also can be noticed, this high concentration of Eu is also associated with the presence of oxygen. Therefore, the final degradation stage which leads to the amorphization of a region that contains the highest concentration of the implanted species is most probably a chemically assisted process as the resulting phase is an Eu (∼16%) rich alloy. In GaN the observed formation of nanocrystalline material is in agreement with the work of Budde et al [51] who suggested that amorphous GaN stoichiometric films were not stable, and attempts to deposit amorphous GaN led, instead, to the formation of randomly stacked nanocrystals of about 3 nm diameter (see figure 2).

For indium nitride, the above observations show that high quality InN is not stable when submitted to ion implantation, especially with such heavy ions as Eu. As extensively discussed elsewhere [41, 42], this may be a limitation for the processing of indium rich compounds for electronic devices, for instance in the possible fabrication of photovoltaic devices where it was reported that InGaN, could be resistant to high energy radiation damage when bombarded with electrons or protons [52].

3.5. Annealing and optical activation

Due to the behaviour of InN as discussed in section 3.3, it is clear that it will be difficult if not impossible to process using ion implantation, furthermore its bandgap (0.6 eV) is too small to allow the visible red Eu emission. Therefore, the following discussion will be limited to AlN and GaN. In contrast to Si where the ion implantation leads to the rapid breakdown and formation of an amorphous layer at substantially low fluences, in GaN and AlN, one obtains, instead, extended defects [25, 37, 41, 47]. During subsequent annealing the low concentration doped amorphous layer is easily regrown by solid phase epitaxy on the underlying silicon matrix and the dopants are activated at reasonably low temperatures (800°–900 °C). As already mentioned above, when brought to more than 800 °C, GaN starts to decompose at the surface, and special protection needs to be used for annealing at higher temperatures. Following the proposal of Pearton et al [53] who showed that coating GaN with AlN cap layer in order could help to avoid decomposition up to temperatures above 1300 °C, we carried out Eu implantation through an optimized 10 nm AlN layer which had been grown by MOVPE on GaN. As a consequence, the threshold for the nanocrystallisation of GaN during Eu implantation was pushed from $2.5 \times 10^{15}$ Eu cm$^{-2}$ to over $2 \times 10^{16}$ Eu cm$^{-2}$ as can be seen in figure 7. Obviously, capping GaN influences its behaviour during the ion implantation. Moreover such protected layers were submitted to thermal annealing in a furnace for 20 min at 1100 °C and 1300 °C [54], as can be seen in figure 8, an increase the of Eu cathodoluminescence emission of about one order of magnitude was then obtained. However, even in that case, it was not possible to remove the stacking faults or to recover the layer in which the nanocrystallization has started to take place. In such cases, no regrowth is possible, and the nanocrystalline part decomposes when the temperature is increased above 800 °C [55]. In AlN, we have investigated the optical behaviour of the AlN sample series from the lowest fluences to the highest; as was noticed, all the samples present Eu photoluminescence emission already in the as-implanted state [40]. However, after annealing at 1300 °C in a furnace, a strong increase in the Eu related light emission was achieved (more than 3 orders of magnitude for a sample at $10^{15}$ Eu cm$^{-2}$), although there was a negligible structural recovery. Despite low Eu concentrations, highest PL intensities were measured for the samples within the low fluence regime and the PL decreased strongly when the fluence reached a critical value of $10^{15}$ Eu cm$^{-2}$ which is an indication that implantation damage strongly affects the optical activation.

4. Discussion and conclusion

Although the three nitride compounds have the same wurtzite structure, it is clear that they exhibit different behaviour when
submitted to ion implantation of RE atoms such as Eu in this work. From the above observations, it appears that InN is obviously not stable during rare earth implantation even at very low fluences (5 × 10^{12} \text{Eu cm}^{-2}), but the damage formation appears to be conventional and the InN crystalline system breaks down around Rp, where the largest part of atomic displacements takes place. Such fluences correspond to negligible displacements per atom (0.05 dpa) and very low rare earth concentrations (0.001%). This is an indication that the ballistic effects may not be dominant and InN thus exhibits an intrinsic metastable character. As for GaN, from the lowest fluences, an increase of strain along [0001] characterized by different saturation steps has been observed [38]. Around $2 \times 10^{14}$ \text{Eu cm}^{-2}, a first saturation of the normal strain arises from an embedded region containing both point defects and stacking faults. The regime preceding this saturation was mainly attributed to an efficient trapping of point defects by the SFs, leading to their lateral extension. Subsequently, as the SFs network becomes denser, the migration of point defects results in the formation of a highly strained region beyond the SFs network due to point defects clustering and in the expansion of the layer with planar defects towards the surface leading to the nanocrystallization [42, 47]. For AlN, at low fluence, there appears a single satellite peak in XRD curves which was attributed to a buried layer located in a region where interstitials are in excess [47]. Such a layer contains both clusters of point defects and SFs which partially relax the strain induced by interstitials. These SFs are formed from precursors generated in the dense collision cascades that grow with fluence by trapping migrating point defects. With increasing fluence, the strain increases in this layer with the increase of defect concentration and then saturates at 0.7% [46]. From this saturation, two spatially separated strained layers in contraction and in dilatation on each side of the buried layer are formed. It then appears that, whilst SFs continue to grow by trapping point defects, defects are ejected from the strain-saturated layer of SFs towards the surface and the bulk, where they form clusters and generate large strains. Similar processes take place in Eu-implanted GaN where the same three-layer structure forms; however, they do not lead to the same final microstructural state at high fluence. As shown above, the lower stability of SFs, especially PSFs in AlN constitutes an obstacle for easy migration of point defects towards the surface thus impeding the nanocrystallization, as observed in GaN. For the optical activation of RE emission, it is clear that high temperature annealing is necessary, however, during this work, accessible temperature values of around 1300°C are not enough to anneal out the created damage made of extended defects in GaN and AlN. Moreover, extreme care should be taken for the protection of the GaN surface, and in this instance, the use of AlN can allow reaching reasonably high temperature. Unfortunately, it should also be taken into account that the cap layer will always be strained on GaN, and it is not clear that it is going to be still stable when temperatures are going to exceed 1300°C. One good point is that the highest emission is obtained at low fluences where the accumulated implantation damage is still low.

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