



# Effect of ion species on implantation-produced disorder in GaN at liquid nitrogen temperature

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## Abstract

The effect of ion species on the damage buildup behavior in wurtzite GaN under bombardment at liquid nitrogen temperature (LN<sub>2</sub>) is studied by a combination of Rutherford backscattering/channeling spectrometry and transmission electron microscopy. Results show that both the density of collision cascades and chemical effects of implanted species affect the damage buildup behavior during bombardment at LN<sub>2</sub>. In particular, an increase in the density of collision cascades (with increasing ion mass) strongly enhances the level of pre-amorphous lattice disorder in the crystal bulk at LN<sub>2</sub>, which is similar to the situation during bombardment at room temperature. © 2002 Elsevier Science B.V. All rights reserved.

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## 1. Introduction

Extensive studies of ion-beam-damage processes in GaN have been undertaken for the past several years (see, for example, recent reviews [1,2]). It has been shown that GaN exhibits a rather complex behavior under ion bombardment, with very strong dynamic annealing of ion-beam-generated point defects even at liquid nitrogen temperature (LN<sub>2</sub>) [1,2]. In addition, our more

recent studies [3,4] have revealed a strong influence of the density of collision cascades on the production of stable damage in GaN by ion bombardment. In our previous report [3], we focused on such cascade density effects during room-temperature (RT) ion bombardment. In this paper, we concentrate on cascade density effects in GaN irradiated at LN<sub>2</sub>. In particular, we will present (i) the damage buildup behavior in GaN, as measured by Rutherford backscattering/channeling (RBS/C) spectrometry; (ii) the effect of the density of collision cascades on the level of implantation-produced disorder; and (iii) cross-sectional transmission electron microscopy (XTEM) data illustrating the amorphization behavior as

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well as microstructural and chemical effects arising from extremely large ion doses required for amorphization in the case of light ions.

## 2. Experiment

The  $\sim 2 \mu\text{m}$  thick wurtzite undoped GaN epilayers used in this study were grown on *c*-plane sapphire substrates by metalorganic chemical vapor deposition at Ledex Corp. Samples were implanted at  $\text{LN}_2$  using the ANU 180 kV ion implanter and an ANU 1.7 MV tandem accelerator (NEC, 5SDH-4) with 60 keV  $^{28}\text{Si}^-$ , 130 keV  $^{63}\text{Cu}^-$  and 200 keV  $^{107}\text{Ag}^+$  ions over a wide dose range to study the damage buildup. During implantation, samples were tilted by  $\sim 7^\circ$  relative to the incident ion beam to minimize channeling.

After implantation, samples were taken from the implanter chamber and characterized ex situ at RT by RBS/C using an ANU 1.7 MV tandem accelerator (NEC, 5SDH) with 1.8 MeV  $^4\text{He}^+$  ions incident along the  $[0001]$  direction and backscattered into a detector at  $98^\circ$  relative to the incident beam direction. All RBS/C spectra were analyzed using one of the conventional algorithms [5] for extracting depth-profiles of the effective number of scattering centers, which will be referred to below as “relative disorder”. Selected samples were studied by XTEM in a Philips CM12 transmission electron microscope operating at 120 keV.

## 3. Results and discussion

### 3.1. Damage buildup at $\text{LN}_2$

Fig. 1 shows RBS/C spectra illustrating the damage buildup in GaN bombarded at  $\text{LN}_2$  with 60 keV Si (Fig. 1(a)), 130 keV Cu (Fig. 1(b)) and 200 keV Ag (Fig. 1(c)) ions. It is seen from Fig. 1 that, in this case of intermediate-mass ions, apparent amorphization (as suggested by the RBS/C yield reaching the random level) proceeds only from the sample surface, while damage in the bulk saturates at some level.

The effect of ion species on the damage buildup behavior in GaN at  $\text{LN}_2$  is illustrated in Fig. 2,

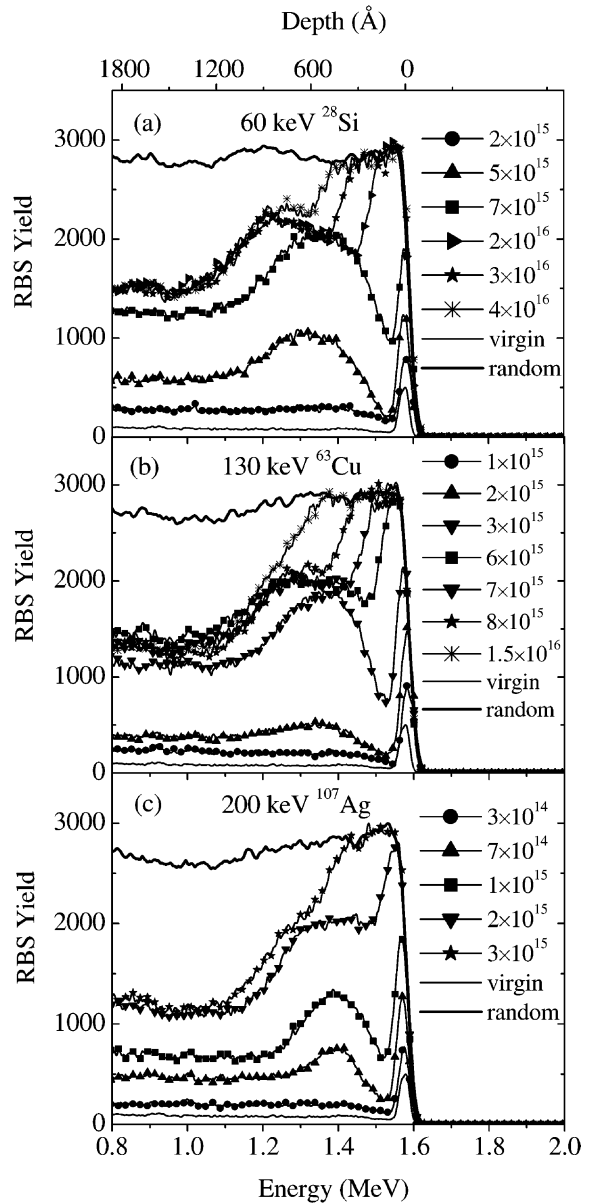


Fig. 1. RBS/C spectra showing the damage buildup in GaN bombarded at  $\text{LN}_2$  with 60 keV Si ions with a beam flux of  $1.6 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$  (a), with 130 keV Cu ions with a beam flux of  $9.4 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$  (b) and with 200 keV Ag ions with a beam flux of  $\sim 6 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$  (c). Implantation doses (in  $\text{cm}^{-2}$ ) are indicated in the figure.

which shows the dose dependence of maximum relative disorder in the bulk for several ion species (extracted from RBS/C spectra from Fig. 1 and

also from spectra previously reported in [3,6]). It is seen from Fig. 2 that GaN exhibits bulk amorphization at LN<sub>2</sub> (with the formation of buried amorphous layers, as suggested by RBS/C data) only for very heavy ions (such as <sup>197</sup>Au and <sup>209</sup>Bi) and for light <sup>12</sup>C and <sup>16</sup>O ions. In the case of bombardment with intermediate-mass ions studied (<sup>28</sup>Si, <sup>63</sup>Cu and <sup>107</sup>Ag), Fig. 2 shows that the damage level in the bulk saturates, and amorphization appears to proceed only from the surface, as suggested by RBS/C spectra shown in Fig. 1.

To confirm amorphization suggested by RBS/C in the case of light ions, the bright-field XTEM image shown in Fig. 3(a) indeed reveals the presence of two amorphous layers (buried and surface layers) in GaN bombarded at LN<sub>2</sub> with 40 keV C ions to a dose of  $2 \times 10^{16}$  cm<sup>-2</sup>. In addition, Fig. 4 shows the formation of a buried amorphous layer in GaN bombarded at LN<sub>2</sub> with 50 keV O ions to a dose of  $6 \times 10^{16}$  cm<sup>-2</sup>. Hence, RBS/C data on amorphization is in good agreement with XTEM results.<sup>1</sup>

The bulk amorphization of GaN bombarded with light C and O ions at LN<sub>2</sub> is attributed to strong chemical effects arising from extremely large ion doses required for amorphization in the case of light ions ( $\sim 10^{16}$ – $10^{17}$  cm<sup>-2</sup>, which roughly corresponds to  $\sim 1$ – $10$  at.% of C or O atoms implanted into the GaN matrix). Possible physical mechanisms of such chemical effects on the damage buildup behavior have been discussed in detail in [3] for RT implantation.<sup>2</sup>

<sup>1</sup> Note that the  $g = 1\bar{1}00^*$  XTEM image shown in Fig. 4(b) illustrates the formation of extended (planar) defects in GaN bombarded to high ion doses. These planar defects, discussed in more detail in [2,3,6], are characteristic defect structures for GaN implanted to high ion doses.

<sup>2</sup> In addition, Fig. 3(b) shows a bright-field image of GaN bombarded at RT with 40 keV C ions to a dose of  $5 \times 10^{16}$  cm<sup>-2</sup>. It is seen from Fig. 3(b) that bulk amorphization (with the formation of a buried amorphous layer) also occurs in GaN bombarded with C ions at RT. This supports the above assertion that a relatively large concentration of C atoms implanted into the GaN matrix strongly influences dynamic annealing processes, resulting in the formation of buried amorphous layers even during bombardment at RT, where damage saturation in the bulk has been observed for all the other ion species studied (i.e., O, Si, Cu, Ag, Au and Bi).

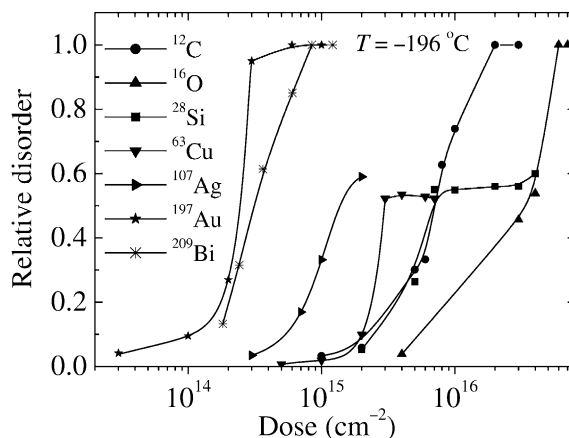


Fig. 2. Maximum relative disorder (extracted from RBS/C spectra) in the bulk defect peak as a function of dose for 40 keV C, 50 keV O, 60 keV Si, 130 keV Cu, 200 keV Ag, 300 keV Au and 500 keV Bi ions implanted at LN<sub>2</sub>, as indicated in the legend. The following beam fluxes were used:  $1.4 \times 10^{13}$  (for C),  $1.9 \times 10^{13}$  (for O),  $1.6 \times 10^{13}$  (for Si),  $9.4 \times 10^{12}$  (for Cu),  $\sim 6 \times 10^{11}$  (for Ag),  $3.1 \times 10^{12}$  (for Au) and  $\sim 1 \times 10^{11}$  cm<sup>-2</sup> s<sup>-1</sup> (for Bi ions).

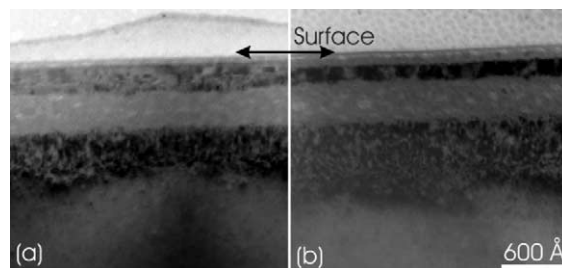


Fig. 3. Bright-field XTEM images of GaN bombarded with 40 keV C ions with a beam flux of  $\sim 1.4 \times 10^{13}$  cm<sup>-2</sup> s<sup>-1</sup> at LN<sub>2</sub> (a) and RT (b) to doses of  $2 \times 10^{16}$  (a) and  $5 \times 10^{16}$  cm<sup>-2</sup> (b). Images (a) and (b) are of the same magnification. The formation of buried and surface amorphous layers is demonstrated.

The above results show that the density of collision cascades affects not only the level of implantation-produced lattice disorder but also the main features of the damage buildup behavior in GaN at LN<sub>2</sub>. Indeed, Fig. 2 shows that, with increasing density of collision cascades (with increasing ion mass from <sup>28</sup>Si to <sup>197</sup>Au), the damage buildup behavior changes from surface amorphization (and damage saturation in the bulk) to bulk amorphization (with the formation of buried

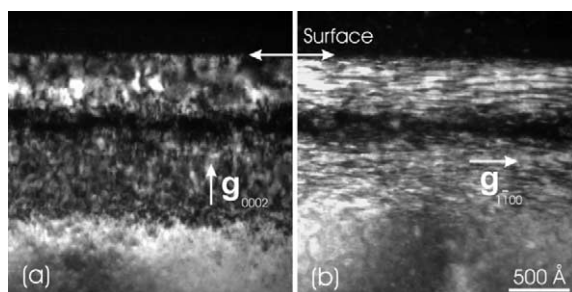


Fig. 4. Dark-field XTEM images ((a)  $g = 0002^*$  and (b)  $g = 1\bar{1}00^*$ ) of GaN bombarded at LN<sub>2</sub> with 50 keV O ions with a beam flux of  $1.9 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$  to a dose of  $6 \times 10^{16} \text{ cm}^{-2}$ . Images (a) and (b) are of the same magnification.

amorphous layers). Hence, an increase in the density of collision cascades appears to stabilize an amorphous phase in GaN.

### 3.2. Effect of cascade density on the level of pre-amorphous disorder

To illustrate the effects of collision cascade density on the level of pre-amorphous disorder in GaN at LN<sub>2</sub>, Fig. 5 (the left axis) shows the ion mass dependence of the ion doses required to produce 30% relative disorder,  $\Phi_{0.3}$ . Such ion doses

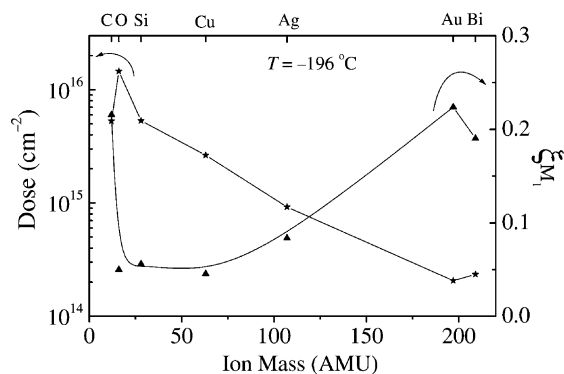


Fig. 5. Left axis: the ion mass dependence of the ion dose necessary to produce relative disorder of 0.3 (as measured by RBS/C) by implantation at LN<sub>2</sub>. Right axis: the dependence of  $\xi_{M_1}$  ( $\xi_{M_1}$  = the ratio of the level of lattice disorder measured experimentally to the damage level predicted based on ballistic calculations) on ion mass. See the caption of Fig. 2 for the details of implant conditions.

( $\Phi_{0.3}$ ) have been found by interpolation of damage buildup curves from Fig. 2. The level of relative disorder of 0.3 has been chosen since (i) the analysis used to extract relative disorder [5] is not valid for very low levels of implantation-produced damage and (ii) this level is below the saturation level of damage for all ions used. It is seen from Fig. 5 (the left axis) that  $\Phi_{0.3}$  generally decreases with increasing ion mass. Interestingly, a similar dependence has been reported for bombardment at RT and discussed in [3]. This qualitative trend of  $\Phi_{0.3}$  is expected since the number of ion-beam-generated atomic displacements increases with ion mass for ion energies used in this study. However, two deviations from the trend are clearly seen in Fig. 5 for the cases of <sup>12</sup>C and <sup>209</sup>Bi ions. Indeed,  $\Phi_{0.3}$  for <sup>12</sup>C ions is ~2.6 times smaller than  $\Phi_{0.3}$  for <sup>16</sup>O ions, despite the fact that <sup>16</sup>O ions produce ~1.6 times more vacancies than <sup>12</sup>C ions in the maximum of the nuclear energy loss profile. This is a somewhat extreme example of chemical effects of implanted species, consistent with the effect of C on the damage buildup behavior in GaN at RT discussed in [3]. The fact that irradiation with <sup>209</sup>Bi ions produces less stable lattice damage than bombardment with <sup>197</sup>Au ions (see Figs. 2 and 5) may be attributed to the lower beam flux value in the case of Bi ions compared with the beam flux value of Au ions, as has been discussed in [3] for the RT case.

A further insight into the effect of ion mass on the formation of stable lattice disorder in GaN at LN<sub>2</sub> is given in Fig. 5 (the right axis), which shows the ion mass dependence of  $\xi_{M_1}$ , the ratio of the level of relative lattice disorder of 0.3 (as measured by RBS/C) to the damage level predicted based on ballistic calculations [7]  $\Phi_{0.3} N_{\text{vac}}^{\text{max}} / n_{\text{at}}$ , where  $N_{\text{vac}}^{\text{max}}$  is the number of lattice vacancies in the maximum of the nuclear energy loss profile, and  $n_{\text{at}}$  is the atomic concentration of GaN. In the first approximation, the parameter  $\xi_{M_1}$  reflects the effectiveness of the production of stable lattice disorder ( $N^{\text{def}}$ ) by ion bombardment under particular implant conditions:  $N^{\text{def}} = \xi_{M_1} \Phi N_{\text{vac}}^{\text{max}}$  for relatively low levels of lattice disorder, where  $\Phi$  is ion dose. If stable post-implantation lattice damage were the same as that predicted based on ballistic calculations (such as the TRIM code [7] used in this

study),  $\xi_{M_1}$  would be equal to unity and independent of ion mass.

In contrast to such expectations, Fig. 5 (the right axis) shows a rather complex dependence of  $\xi_{M_1}$  on ion mass. First of all, for all ion species used,  $\xi_{M_1}$  is significantly below unity even for this case of low-temperature bombardment. This is a direct consequence of strong dynamic annealing processes when, even at LN<sub>2</sub>, a large fraction of ion-beam-generated point defects experiences annihilation. It is also seen from Fig. 5 (the right axis) that, for ions from <sup>16</sup>O to <sup>63</sup>Cu,  $\xi_{M_1}$  is essentially independent of ion mass. For ions heavier than <sup>63</sup>Cu,  $\xi_{M_1}$  shows a strong increase with increasing ion mass.<sup>3</sup> In the case of <sup>12</sup>C, the value of  $\xi_{M_1}$  is much larger than that for the other light and intermediate-mass ions studied (O, Si, Cu and Ag).

Such a complex behavior of  $\xi_{M_1}$  appears to be a result of (i) chemical effects of implanted species, which are particularly pronounced in the case of light and intermediate-mass species due to large ion doses and (ii) nonlinear cascade density effects, which dominate in the case of heavy ions (such as Au and Bi). The physical mechanisms responsible for these effects have been discussed in detail in [3,6]. Here, we will only mention that, as compared to the RT case, chemical effects of O and Si ions seem to be less pronounced at LN<sub>2</sub>, presumably due to smaller ion doses  $\Phi_{0.3}$  in the LN<sub>2</sub> case. It is also somewhat surprising that the main features of ion mass dependences of  $\Phi_{0.3}$  and  $\xi_{M_1}$  shown in Fig. 5 for bombardment at LN<sub>2</sub> are similar to those in the RT case discussed in [3]. Indeed, the rate of thermally activated defect interaction processes is reduced at LN<sub>2</sub> as compared to the RT case, and a different behavior of  $\Phi_{0.3}$  and  $\xi_{M_1}$  can be expected at different implantation temperatures. It is clear that, at present, additional studies are highly desirable to better understand complex cascade density effects in GaN under ion bombardment.

<sup>3</sup> As discussed above, a legitimate explanation for a low value of  $\xi_{M_1}$  in Fig. 5 in the case of <sup>209</sup>Bi ion bombardment is the effect of beam flux.

#### 4. Summary

The damage buildup and amorphization behavior of wurtzite GaN bombarded at LN<sub>2</sub> with a wide range of ion species (from <sup>12</sup>C to <sup>209</sup>Bi) of keV energies have been discussed. Results have shown that, at LN<sub>2</sub>, GaN exhibits bulk amorphization under heavy-ion bombardment (such as <sup>197</sup>Au and <sup>209</sup>Bi) and irradiation with light ions (such as <sup>12</sup>C and <sup>16</sup>O), while irradiation with lighter <sup>28</sup>Si, <sup>63</sup>Cu and <sup>107</sup>Ag ions appears to result in surface amorphization and damage saturation in the bulk. Bulk amorphization of GaN by bombardment with light C and O ions has been attributed to strong chemical effects of implanted species. Results have also shown that an increase in the density of collision cascades strongly increases the level of pre-amorphous lattice disorder in the bulk at LN<sub>2</sub>, which is similar to the case of RT ion bombardment previously discussed in [3].

#### Acknowledgements

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