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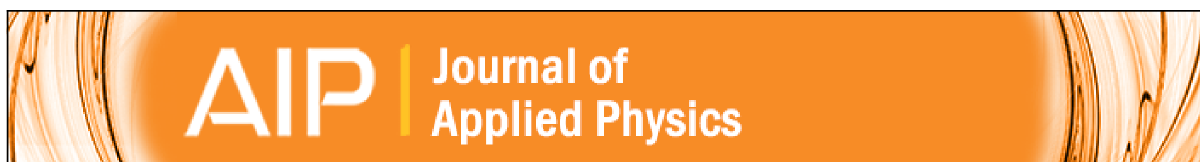

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Blistering of H-implanted GaN

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Mechanisms of blistering of wurtzite GaN films implanted with H ions are studied. In particular, we report on the influence of the following parameters on the blistering process: (i) ion energy (from 20 to 150 keV), (ii) ion dose (up to 1.2×10^{18} cm^{-2}), (iii) implantation temperature (from −196 to 250 °C), and (iv) annealing temperature (up to 900 °C). Results show that both the onset of blistering and blistering surface patterns strongly depend on implant conditions. This study may have significant technological implications for ion slicing and "etching" of GaN using high-dose implantation with H ions. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1430533]

Blistering of solids bombarded with ions which can form gas bubbles has been a subject of ongoing research over the past decades.[1] Early work was focused on blistering of metals bombarded with He ions, which was a significant problem in the components of nuclear reactors.[1] More recently, blistering of semiconductors has received research interest due to technological applications of ion slicing (or "ion cut").[2] Ion slicing, which involves high-dose H ion implantation in combination with wafer bonding and postimplantation annealing, provides a method to transfer thin surface layers onto other substrates. Ion slicing has been studied in a number of semiconductors such as Si, Ge, GaAs, InP, SiC, and even diamond (see, for example, Refs. 3–7). It has been found that different semiconductors respond differently to H ion implantation.[3–7]

Studies of ion cut are usually made in two steps: (i) investigation of surface blistering and (ii) studies of wafer bonding and layer splitting during postimplantation annealing. Previous results have shown that both processes of blistering and layer splitting have the same activation energy.[4] Moreover, implantation and annealing conditions resulting in the formation of surface blisters and layer splitting (after wafer bonding) are similar.[4,5,7]

In this communication, we report on the influence of implant and annealing parameters on blistering of GaN. This study may have important technological implications for a transfer of GaN thin films onto foreign substrates using H ion slicing. In particular, ion slicing is a rather attractive tool for the integration of GaN-based electronics (with GaN epilayers being usually grown on sapphire or SiC substrates) with Si-based integrated circuit technology.

The ~2 μm thick wurtzite undoped GaN epilayer used in this study was grown on a *c*-plane sapphire substrate by metalorganic chemical vapor deposition at Ledex Corp. Implantation with H ions was carried out with the ANU 180 kV ion implanter under conditions given in Table I. Ion beam flux was typically $1-3\times10^{13}$ cm^{-2} s^{-1}. During irradiation, samples were tilted by ~7° relative to the incident ion beam to minimize channeling.

After implantation, samples were characterized at room temperature (RT) by Rutherford backscattering/channeling (RBS/C) spectrometry using an ANU 1.7 MV tandem accelerator (NEC, 5SDH) with 1.8 MeV $^4He^+$ ions incident along the [0001] direction and backscattered into a detector at ~170° relative to the incident beam direction. Rapid thermal annealing was carried out in a nitrogen ambient at atmospheric pressure with a temperature ramp rate of ~200 °C/s.[8] Surface morphology was studied by tapping-mode atomic force microscopy (AFM), scanning electron microscopy (SEM), and optical microscopy. Selected samples were studied by cross-sectional transmission electron microscopy (XTEM) in a Philips CM12 transmission electron microscope operating at 120 keV.

Table I summarizes data on the observation of blistering for different implant and annealing conditions. It is seen from Table I that, without postimplantation annealing, no blistering occurs in GaN after implantation at RT with 100 keV H ions to a dose as large as 1.2×10^{18} cm^{-2} (the maximum dose used in this study). Table I also shows that annealing at temperatures ≳300 °C is required for surface blistering in the case of samples implanted at RT to doses $\geqslant 5\times10^{17}$ cm^{-2}. Implantation at RT with 100 keV H ions to doses $\leqslant 3\times10^{17}$ cm^{-2} does not result in the formation of surface blisters during annealing at temperatures ≤900 °C for up to ~1 h (the maximum annealing time used in this study). Hence, the critical H dose required for surface blistering during annealing of GaN (~5×10^{17} cm^{-2} for 100 keV H ions implanted at RT) is considerably larger than the critical dose in the case of metals and other semiconductors

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TABLE I. Implant and annealing conditions of this study. *B* and *N* indicate surface blistering or the absence thereof, respectively, after postimplantation annealing for 5 min.

Energy (keV)	Dose (10^{17} cm^{-2})	Implantation temperature (°C)	Annealing temperature (°C)					
			20	300	350	500	800	900
20	3.3	20	*N*	*N*	*N*	*B*	*B*	*B*
50	4.4	20	*N*	*N*	*B*	*B*	*B*	*B*
100	⩽3	20	*N*	*N*	*N*	*N*	*N*	*N*
100	5	20	*N*	*N*	*B*	*B*	*B*	*B*
100	8	20	*N*	*B*	*B*	*B*	*B*	*B*
100	12	20	*N*	*B*	*B*	*B*	*B*	*B*
100	5	−196	*N*	*N*	*N*	*N*	*N*	*B*
100	8	−196	*N*	*N*	*N*	*N*	*B*	*B*
100	5	100	*N*	*B*	*B*	*B*	*B*	*B*
100	5	200, 250	*B*	*B*	*B*	*B*	*B*	*B*
150	5.9	20	*N*	*N*	*B*	*B*	*B*	*B*

studied previously (Si, Ge, GaAs, InP, and SiC), where blistering during annealing occurs after H doses of $\sim 1-10 \times 10^{16}$ cm^{-2} for similar ion energies.[1,3–7]

Compared with the behavior in other materials,[1,3–7] a large critical dose for blistering of GaN revealed in this study may be attributed to one or more of the following factors: (i) interaction and trapping of H atoms with implantation-produced defects, (ii) very efficient dynamic annealing in GaN compared to that in other semiconductors[10] (i.e., pronounced defect migration, interaction, and annihilation processes *during* ion bombardment), and/or (iii) specific mechanical properties of GaN, particularly given that implantation-produced defects have been shown to suppress the plastic component of deformation.[9,10] However, additional studies are needed to better understand the physical and chemical mechanisms responsible for the difference in the critical dose for blistering of different materials.

An example of an exfoliated surface and an illustration of the dependence of blistering on ion energy are given in Fig. 1. This figure shows typical optical micrographs of GaN implanted at RT with 20, 50, 100, or 150 keV H ions to doses resulting in ~30 at. % of H atoms in the maximum of the ion distribution profile and subsequently annealed at 900 °C for 5 min. Figure 1 reveals that, with increasing ion energy from 20 to 100 keV, blister size also increases. Interestingly, for the largest ion energy studied (150 keV), Fig. 1(d) shows that blisters coallesce, resulting in large, irregular areas of exfoliated surface. The dependence of blister size on ion energy revealed by Fig. 1 is consistent with previous extensive studies of blistering in metals.[1] In addition, our AFM and SEM imaging has revealed that craters left after blister bursting in GaN have flat floors, which is again consistent with previous observations of blistering in other materials.[1]

Figure 2 shows RBS/C spectra taken from samples implanted with the same implant conditions as for Fig. 1 (but without an annealing step). It is seen from Fig. 2 that significant dechanneling and ion scattering occur at the ion end-of-range region. In addition, a small shoulder and a dip are observed in the random spectrum for the 50 keV H case, due to ~30 at. % of H atoms implanted into the GaN lattice. Figure 2 also reveals a very low level of lattice disorder (with a minimum RBS/C yield of ~3%) in the near-surface region.

The effect of ion dose on the blistering process is illustrated in Figs. 1(c), 3(a), and 3(b) for the case of 100 keV H ion implantation at RT. A comparison of Figs. 1(c) and 3(a) shows that, with increasing dose of 100 keV H ions at RT, surface blisters become elongated in shape. At a very large dose of 1.2×10^{18} cm^{-2} [Fig. 3(b)], blisters appear to grow and overlap to produce large exfoliated areas. Hence, Figs. 1(c), 3(a), and 3(b) clearly illustrate that the pattern of surface blistering of GaN strongly depends on ion dose.

Table I and Fig. 3 illustrate that implantation temperature has a very strong effect on blistering. Indeed, annealing at ≳900 °C is required for blistering of GaN bombarded with 100 keV H ions at liquid nitrogen temperature (LN_2) to a dose of 5×10^{17} cm^{-2}, while, after identical implantation at RT, surface blistering occurs at much lower annealing temperatures (≳350 °C). Table I also shows that bombardment with 100 keV H ions at 200 and 250 °C to a dose of 5×10^{17} cm^{-2} results in surface blistering *during implantation*, while blistering of specimens implanted under identical implant conditions but at RT or LN_2 requires annealing at temperatures of ≳350 or ≳900 °C, respectively. Moreover, Figs. 1 and 3 reveal that surface blistering patterns are markedly different for RT and LN_2 bombardment regimes. Hence, an increase in implantation temperature not only enhances blistering but also changes the pattern of surface exfoliation during postimplantation annealing.

Finally, three GaN samples implanted with 100 keV H ions to a dose of 5×10^{17} cm^{-2} at −196, 20, and 100 °C have been studied by XTEM. Interestingly, XTEM has revealed that the defect structure in GaN after implantation at these three temperatures is similar, although the blistering

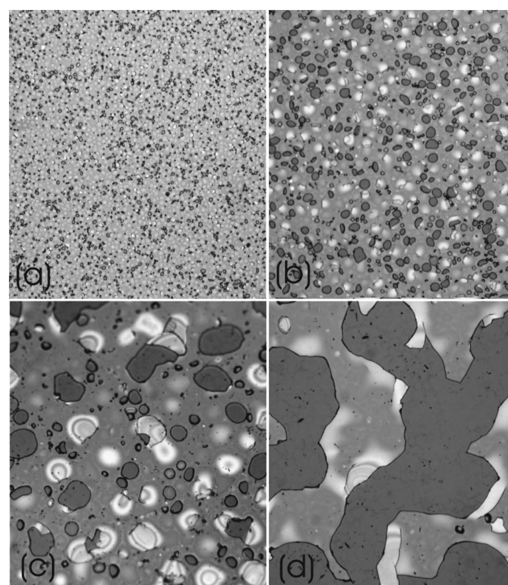

FIG. 1. Typical optical micrographs of GaN samples implanted at 20 °C with 20 keV H ions to a dose of 3.3×10^{17} cm^{-2} (a), 50 keV H ions to a dose of 4.4×10^{17} cm^{-2} (b), 100 keV H ions to a dose of 5×10^{17} cm^{-2} (c), and 150 keV H ions to a dose of 5.9×10^{17} cm^{-2} (d). After implantation, samples were annealed at 900 °C for 5 min in a nitrogen ambient. The horizontal field width of each image is 250 μm.

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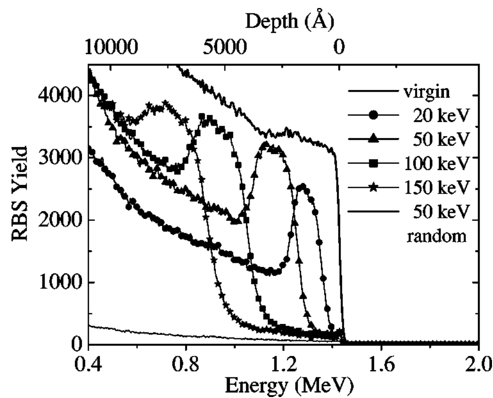

FIG. 2. RBS/C spectra of GaN bombarded at 20 °C with 20 keV H ions to a dose of 3.3×10^{17} cm^{-2}, 50 keV H ions to a dose of 4.4×10^{17} cm^{-2}, 100 keV H ions to a dose of 5×10^{17} cm^{-2}, and 150 keV H ions to a dose of 5.9×10^{17} cm^{-2}, as indicated in the legend.

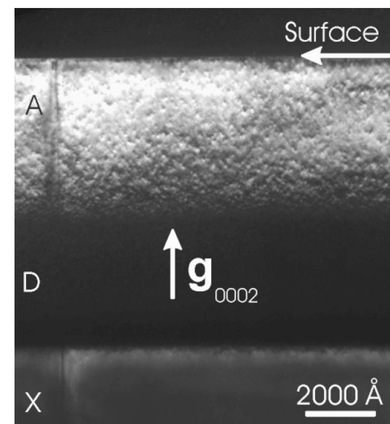

FIG. 4. Dark-field XTEM image ($\mathbf{g}=0002^*$) of GaN implanted at −196 °C with 100 keV H ions to a dose of 5×10^{17} cm^{-2}. Unimplanted GaN, beyond the ion penetration range, is labeled X.

behavior during annealing is rather different for these three cases. As an example, Fig. 4 shows an XTEM image of GaN bombarded at LN_2, which illustrates that implantation with H ions results in the formation of a defect band (labeled as D) at the ion end-of-range, while the near-surface region (labeled as A) is essentially free from implantation-produced structural defects. This XTEM result is consistent with RBS/C data from Fig. 2, discussed above.[11] A low level of implantation-produced disorder in the near-surface region is due to extremely efficient dynamic annealing in GaN even at LN_2.[10] Interestingly, no H_2 gas bubbles have been revealed by XTEM in any of the above three samples (bombarded at −196, 20, and 100 °C). In all three samples, the dense defect band (labeled as D in Fig. 4) consists of coarse defect complexes (presumably, including H-defect complexes) and some planar defects, as revealed by XTEM. Different blistering behavior of samples implanted at −196, 20, and 100 °C strongly suggests that the nature of H trapping at defects and subsequent release of H during annealing depend on implantation temperature. Our more detailed XTEM study of the nature of these defects is in progress.

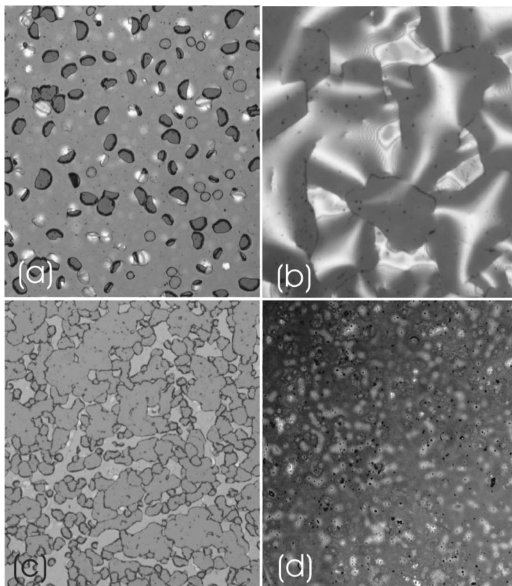

FIG. 3. Typical optical micrographs of GaN samples implanted at 20 °C [(a), (b)] and at −196 °C [(c), (d)] with 100 keV H ions to doses of 8×10^{17} [(a), (d)], 1.2×10^{18} (b), and 5×10^{17} cm^{-2} (c). After implantation, samples were annealed at 900 °C for 5 min in a nitrogen ambient. The horizontal field width is 250 μm for (a), (c), and (d) and is 500 μm for (b).

In conclusion, blistering of H-implanted GaN films has been studied. Results have shown that the blistering behavior strongly depends on both implant and annealing conditions. A knowledge of the influence of implant and annealing conditions, presented in this communication, is essential for the development of ion slicing and ''etching'' of GaN using implantation with H ions.

This research was supported in part by the ARC.

[1] See, for example, early reviews by G. M. McCracken, Rep. Prog. Phys. **38**, 241 (1975); B. M. U. Scherzer, in *Sputtering by Particle Bombardment II*, edited by R. Behrisch (Springer, Berlin, 1983), p. 271.
[2] It should be noted that research interest in implantation-produced blistering of solids has been driven not only by technological importance but also by the fact that the blistering process is rather spectacular and easy to observe.
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[11] It should be noted that the depth of the defect band revealed by XTEM (Fig. 4) is in good agreement with such a depth measured by RBS/C (Fig. 2) as well as the depth of surface craters left after blister bursting, as measured by AFM.

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