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Citation: *Applied Physics Letters* **78**, 156 (2001); doi: 10.1063/1.1335552

View online: <http://dx.doi.org/10.1063/1.1335552>

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Deformation behavior of ion-beam-modified GaN

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(Received 31 July 2000; accepted for publication 31 October 2000)

The deformation behavior of wurtzite GaN films modified by ion bombardment is studied by nanoindentation with a spherical indenter. Results show that implantation disorder significantly changes the mechanical properties of GaN. In particular, GaN amorphized by ion bombardment exhibits plastic deformation even for very low loads with dramatically reduced values of hardness and Young's modulus compared to the values of as-grown GaN. Implantation-produced defects in crystalline GaN suppress the plastic component of deformation and significantly change the values of hardness and Young's modulus. In addition, implantation disorder in crystalline GaN suppresses both "pop-in" events during loading and the appearance of slip traces on the sample surface as a result of indentation. This strongly suggests that slip nucleation (rather than a phase transformation) is the physical mechanism responsible for the pop-in events observed during loading of as-grown crystalline GaN. © 2001 American Institute of Physics. [DOI: 10.1063/1.1335552]

Gallium nitride is currently the subject of intensive research because of the very important technological applications of this material.¹ Several studies of the mechanical properties of as-grown GaN have been reported in the literature.²⁻⁸ However, to our knowledge, the effects of implantation damage on the deformation behavior of GaN have not yet been studied. This is not surprising since damage processes in GaN exposed to ion bombardment are still not well documented in the literature. However, understanding the deformation behavior of ion-beam-modified GaN is not only important for contact damage issues in the GaN industry but is also necessary for understanding the evolution of the structural characteristics of GaN under ion bombardment.⁹

In this letter, we present results on the mechanical properties of (i) crystalline GaN with a relatively high concentration of implantation-produced defects and (ii) GaN amorphized by ion bombardment. Results show that, unlike the situation for Si,¹⁰ implantation damage dramatically changes the deformation behavior of GaN.

A $\sim 2 \mu\text{m}$ thick wurtzite undoped GaN epilayer was grown on a *c*-plane sapphire substrate by metalorganic chemical vapor deposition in a rotating disk reactor at Ledex Corporation. All samples used in this study were cut from the same GaN wafer. For this indentation study, two samples were modified by ion bombardment—a GaN sample with implantation-produced defects (referred below as "ion-damaged" GaN) and GaN amorphized by ion implantation. All implants were done using the Australian National University (ANU) 1.7 MV tandem accelerator (NEC, 5SDH).

To prepare an amorphous layer, GaN was bombarded with 2 MeV $^{197}\text{Au}^+$ ions at liquid nitrogen temperature with a beam flux of $5 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ to a dose of $1.5 \times 10^{16} \text{ cm}^{-2}$. Such implantation results in the formation of a $\sim 0.6 \mu\text{m}$ thick, completely amorphous surface layer, as discussed in detail elsewhere.¹¹ It should be noted that GaN amorphized by ion bombardment exhibits some degree of porosity,¹¹ which may also affect the deformation behavior of amorphized GaN discussed below.

The ion-damaged sample, with a relatively high concentration of implantation-produced defects (but not amorphous), was prepared by multiple-energy bombardment with ^{197}Au ions at 300 °C. The implant conditions to prepare this sample are given in Table I. Given the present understanding of ion-beam-damage processes in GaN, such multiple-energy implantation at an elevated temperature results in the formation of lattice defects, including some planar defects, in the implanted layer (up to $\sim 1 \mu\text{m}$ from the surface).⁹ It should be noted that, for the ion doses used in this study, the concentration of implanted Au species ($< 0.06 \text{ at. } \%$) is expected to have a negligible effect on the mechanical properties of GaN. Rather, the deformation behavior of ion-beam-modified GaN should be determined by implantation-

TABLE I. The implant conditions used to prepare the ion-damaged sample by multiple-energy bombardment with ^{197}Au ions at 300 °C.

| Energy (MeV) | Dose (10^{14} cm^{-2}) | Beam flux ($10^{12} \text{ cm}^{-2} \text{ s}^{-1}$) |
|-----------------|---------------------------------------|---|
| 6.6 | 5 | 5.2 |
| 2 | 7 | 18 |
| 0.45 | 3 | 17 |

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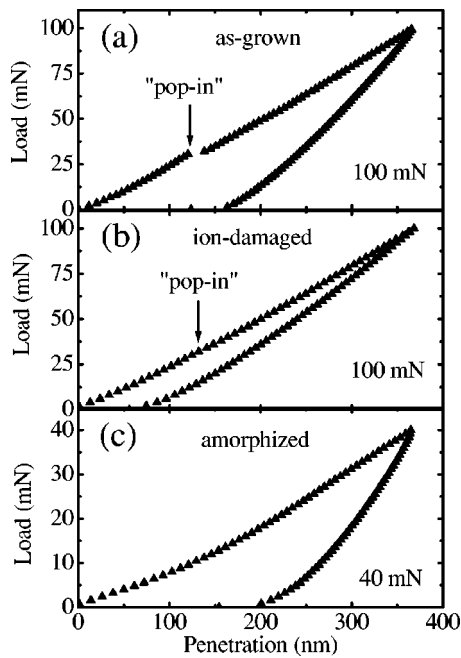


FIG. 1. Typical continuous load–unload curves of (a) as-grown, (b) ion-damaged, and (c) amorphized GaN films. Note that the maximum load is 100 mN for (a) and (b) and is 40 mN for (c).

produced lattice disorder and its consequences.

As-grown and implanted GaN films were subjected to indentation using an ANU UMIS-2000 nanoindentation system with an $\sim 4.2 \mu\text{m}$ radius spherical indenter. The shape of the indenter tip was characterized by scanning electron microscopy. The indentation system and indenter tip were carefully calibrated by indenting fused silica. A series of both partial and continuous load–unload indents was carried out on each of the above three GaN samples at loads up to 100 mN (in the case of as-grown and ion-damaged GaN) and up to 40 mN (in the case of amorphized GaN). All indents were performed at room temperature. The load–unload data was analyzed using the method of Field and Swain¹² to extract the hardness and elastic modulus as a function of indenter penetration.

After indentation, residual impressions of all indents were examined by tapping mode atomic force microscopy (AFM) to check for the evidence of slip, cracking, and pileup/sink-in.¹³ The AFM study was performed under ambient conditions with a Nanoscope III scanning probe microscope using commercial single-beam Si cantilevers with force constants of $30\text{--}120 \text{ Nm}^{-1}$.

Figure 1 shows typical continuous load–unload force–displacement curves of as-grown [Fig. 1(a)], ion-damaged [Fig. 1(b)], and amorphized [Fig. 1(c)] GaN films. Figure 1(a) clearly illustrates a discontinuity (or pop-in) of the yield response occurred during loading of as-grown GaN. This result is in agreement with nanoindentation data previously reported for GaN.^{3,6,8} Interestingly, Fig. 1(b) reveals that in ion-damaged GaN, the pop-in event is still present but significantly less pronounced than in as-grown GaN. Finally, Fig. 1(c) shows no discontinuities in the load–displacement curve of amorphous GaN, in full agreement with the expected deformation behavior of an amorphous solid.¹⁰

Shown in Fig. 2 are typical amplitude-mode AFM images of as-grown [Fig. 2(a)] and ion-damaged [Fig. 2(b)]

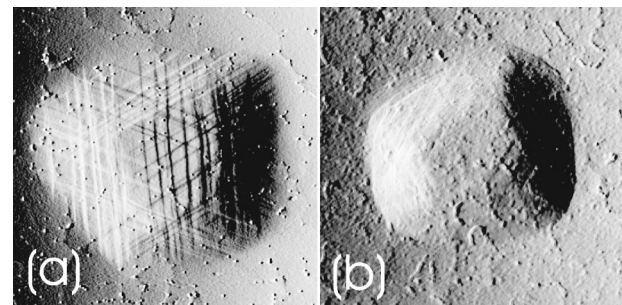


FIG. 2. Typical amplitude-mode AFM images of (a) as-grown and (b) ion-damaged GaN films indented at a maximum load of 100 mN. [Horizontal field width = $4 \mu\text{m}$ for both images (a) and (b).]

GaN films indented at a maximum load of 100 mN. These AFM images clearly illustrate that slip, which occurs during loading of as-grown GaN, is strongly suppressed in ion-damaged GaN. A comparison of nanoindentation data [see Figs. 1(a) and 1(b)] and AFM data (see Fig. 2) gives a compelling argument that slip nucleation (rather than a phase transformation) is the physical mechanism responsible for the pop-in events observed during loading of GaN. Therefore, the deformation behavior of GaN is somewhat similar to that of sapphire.¹⁴

Shown in Fig. 3 are the curves of the average contact pressure [or (Meyer) hardness] H [Fig. 3(a)] and Young's modulus E [Fig. 3(b)] as a function of indenter penetration below the circle of contact, as determined from the partial load–unload data. This figure, showing data for as-grown, ion-damaged, and amorphized GaN, reveals that ion bombardment dramatically modifies H and E . It is seen that, for shallow penetration depths of the indenter (prior to the pop-in event), the H and E values of ion-damaged GaN are

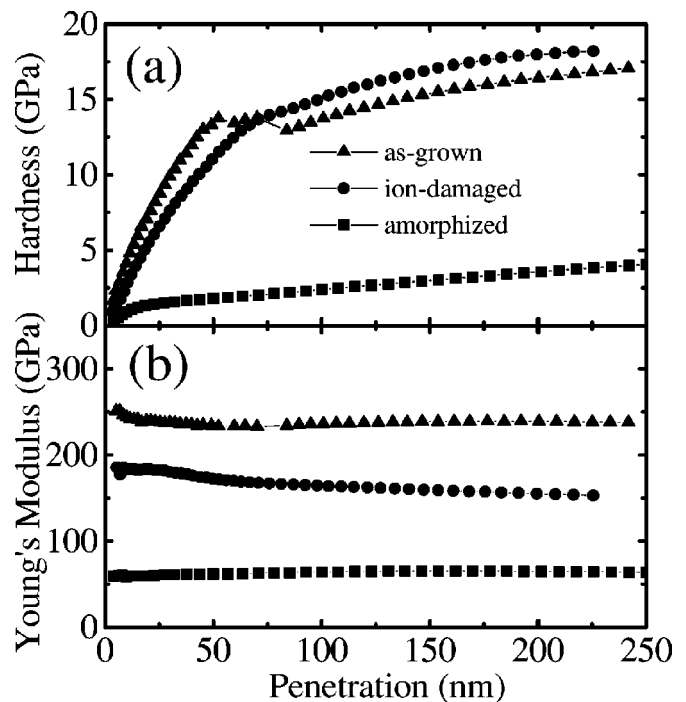


FIG. 3. The curves of (a) the hardness and (b) Young's modulus as a function of indenter penetration below the circle of contact, as determined from the partial load–unload data. Figures show data for as-grown, ion-damaged, and amorphized GaN, as indicated in the legend in (a).

TABLE II. The values of hardness H and Young's modulus E at a plastic penetration depth of 100 nm for the three GaN samples used in this study.

| GaN sample | H (GPa) | E (GPa) |
|-------------|-----------|-----------|
| as-grown | 13.4 | 233 |
| ion-damaged | 15.1 | 164 |
| amorphized | 2.4 | 65 |

lower than those of as-grown material. As expected, the value of E is not affected by slip and remains essentially constant for the whole indenter penetration depth.¹⁵ However, slip significantly affects H of as-grown GaN, while in ion-damaged GaN slip is strongly suppressed, and, for larger depths of indenter penetration, H becomes larger than that of as-grown GaN. Figure 3 also shows that the values of H and E of amorphized GaN are significantly lower than those of as-grown and ion-damaged GaN; i.e., amorphous GaN is very soft. Table II gives the values of H and E for the three GaN samples used in this study at a plastic penetration depth of 100 nm, a depth where the substrate effects are expected to be small.

Finally, a close examination of partial load–unload curves reveals that in ion-damaged GaN the purely elastic regime extends to higher loads and penetration depths than in as-grown GaN. This result indicates that implantation disorder in crystalline GaN somewhat suppresses pressure-induced nucleation and/or propagation of extended defects, the processes which are most likely responsible for the plastic deformation of crystalline GaN.^{7,8} In contrast, in amorphized GaN, deformation response is elastic–plastic even for very low loads, and, with increasing load, plastic deformation dominates with pileup around the impression (as revealed by AFM), typical for indentation of amorphous solids. Such a difference in the deformation modes of as-grown, ion-damaged, and amorphized GaN is also reflected by the depths of residual depression (see Figs. 1 and 2) and the depths of maximum plastic penetration (see Fig. 3) of the above three samples.

In conclusion, wurtzite GaN films modified by ion bombardment have been studied by nanoindentation with a spherical indenter. Implantation-produced defects suppress

both pop-in events and slip during loading. This result suggests that slip nucleation (rather than a phase transformation) is responsible for pop-in events. In addition to suppressing slip, implantation disorder in crystalline GaN also suppresses the plastic component of deformation, which may suggest that slip is the major contributor to the plastic component of deformation of crystalline GaN. The deformation behavior of amorphous GaN is very different from that of as-grown crystalline GaN. In particular, amorphous GaN exhibits plastic flow even for very low loads. In addition, the values of hardness and elastic modulus of amorphous GaN are much lower than those of as-grown GaN, as indicated in Table II. Finally, this study may have significant technological implications for the estimation of contact damage in ion-beam-processed GaN.

¹ See, for example, a recent review S. J. Pearton, J. C. Zolper, R. J. Shul, and F. Ren, *J. Appl. Phys.* **86**, 1 (1999), and references therein.

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¹⁵ Slightly nonconstant behavior for E of ion-damaged GaN with increasing penetration depth [see Fig. 3(b)] may be attributed to a nonuniform distribution of implantation disorder and/or to the substrate effect.