Focused ion beam micromilling of GaN and related substrate materials (sapphire, SiC, and Si)

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Micromilling of GaN films has been obtained using a Ga^+ focused ion beam (FIB). The GaN micromilling has been investigated over a range of energies (15–70 keV), incident angles (0°–30°), and number of scans (10–50). At normal incidence, increasing the Ga^+ energy up to 50 keV increases the milling rate, while higher energies produce the same (or a slightly decreased) milling rate. Increasing the angle of incidence increases the milling rate at all energies. The highest GaN milling rate of 0.6 μm^3/nA s (corresponding to an average yield of 6.6 atoms/ion) has been obtained at 50 keV, 30° incidence, and 50 scans. The milling rate of current substrate materials (sapphire, Si and SiC) for GaN thin film growth is shown to be 2–5 times lower. The sputtering yield is found to vary inversely with the strength of the chemical bond in the materials investigated. Distributed Bragg reflection air/GaN gratings for short cavity lasers were fabricated to show the capability of FIB micromilling to produce optoelectronic devices based on GaN. © *1999 American Vacuum Society.* [S0734-211X(99)04702-2]

I. INTRODUCTION

The III-N semiconducting compounds are of particular interest[1] because of their direct band gap and high level of optical activity. Currently, laser diodes fabricated using GaN-based structures[2] grown primarily on sapphire and SiC substrates cover nearly the entire visible spectrum and have rapidly reached various stages of commercialization. Among the aspects of GaN laser fabrication which can benefit from improvement is the process of forming the facets of the laser cavity. Cleaving of the end facets, which is used in conventional semiconductor laser fabrication, is made more difficult in this case by the various degrees of misalignment[3] between the GaN and sapphire cleavage planes. Use of plasma etching to define the laser cavity leaves the facets with a rough surface.[4]

Focused ion beam (FIB) techniques of micromilling[5] and ion-induced mixing[6] have been successfully used in the fabrication of a wide variety of photonic components (waveguides, mirrors, gratings, laser cavities, etc.) in GaAs/AlAs and InP materials systems. Recently, FIB micromilling has been reported[7,8] to produce flat and smooth single mirror facets for GaN laser diodes. However, no quantitative evaluation of GaN micromilling has been reported to date. In this article, we present results on FIB micromilling of GaN layers under various conditions of energy, incident angle and scan frequency. In addition, we report similar results for materials currently utilized as substrates for GaN growth.

II. EXPERIMENTAL CONDITIONS

The micromilling experiments were carried out in a FIB NanoFab 150 system using a Ga liquid metal ion source (LMIS). The Ga^+ total emission current was typically set at 3 μA and the target current was ~170 pA. The total Ga^+ dose used in all experiments reported here was kept constant at $1\times10^{18}\,cm^{-2}$, which was found appropriate[9,10] for Si removal by Ga^+ FIB micromilling. To achieve this dose, the FIB was scanned multiple times (ranging from 10 to 50) over the GaN surface. The GaN micromilling was investigated using Ga^+ ions with the energy ranging from 15 to 70 keV. The FIB pixel dwell time for the 50 scan case ranged from 1.35 ms at 15 keV to 60 μs at 70 keV. At 50 keV, the beam scan speed was 42 μm/s in the 10 scan case and 208 μm/s in the 50 scan case. The angle of incidence of the ion beam was varied between 0° (normal incidence) and 30°. The primary GaN material[11] used for the milling experiments was grown by hydride vapor phase epitaxy (HVPE) on sapphire substrates. The GaN film was relatively thick (~20 μm) which allowed the milling experiments to be performed without encountering any substrate effects. However, since in practice thinner GaN layers are grown on various substrates for fabricating devices, we have separately investigated the FIB Ga^+ micromilling of several substrate materials: (100) Si, *c*-face sapphire, and 6H-SiC.

III. RESULTS

The common pattern that was FIB micromilled into GaN and the substrate materials is shown in Fig. 1(a). The total area of the pattern was 200 μm^2, which requires an approximately 30–35 min exposure for a $1\times10^{18}\,cm^{-2}$ dose. The milling depth for the GaN experiments ranged between ~0.6 to 1 μm. The ''writing'' strategy is that of a serpentine scan, where the scan direction is reversed after each pass to minimize redeposition effects.[5] Figure 1(b) contains a scanning electron microscopy (SEM) microphotograph of a GaN pattern micromilled using a 50 keV Ga^+ FIB with a dose of $1\times10^{18}\,cm^{-2}$, at normal incidence, and using 10 FIB scans. A sharply defined milled region is obtained that has uniform

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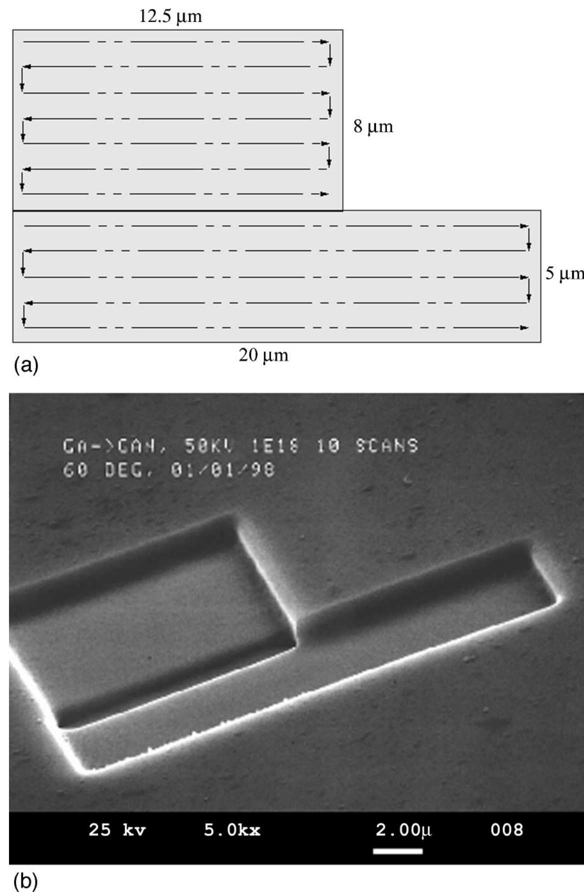


FIG. 1. Ga^+ FIB micromilled pattern: (a) schematic of the pattern and the serpentine scan; (b) SEM photograph of the GaN pattern micromilled using a 50 keV Ga^+ FIB with a dose of 1×10^{18} cm^{-2}, at normal incidence, over 10 scans.

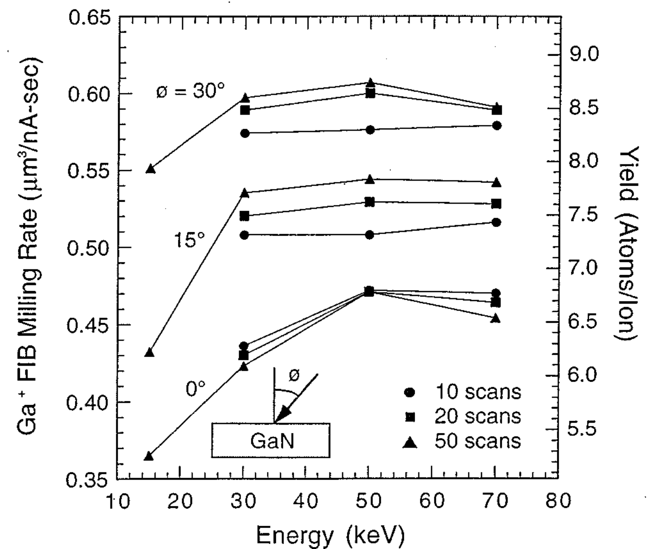


FIG. 2. FIB Ga^+ milling rate of GaN vs energy for different scan conditions and angles of incidence (dose: 1×10^{18} cm^{-2}).

depth, a smooth milled surface, and a minimum redeposition of material. A deeper trench is observed where the two rectangles of the pattern have a slight overlap.

Figure 2 shows the GaN micromilling rate as a function of Ga^+ ion energy for several angles of incidence (0°, 15°, 30°) from the surface normal. For each angle and for all but one energy (15 keV), the experiments were performed using 10, 20, and 50 FIB scans. As expected, the off-axis implants produced a higher milling rate. As the Ga^+ energy is first increased from 15 to 30 keV, the GaN milling rate increased for all angles of incidence. For a further energy increase to 50 keV, the normal incidence milling rate continued to increase, whereas for the 15° and 30° incidences the rate stayed essentially constant. The final energy increase to 70 keV yielded either no change or a decrease in the milling rate under all conditions. The effect of scan speed (i.e., the number of scans) on the milling rate is relatively minor over the range investigated, with a stronger effect being observed under off-axis conditions. In the off-axis milling, the effect of increasing the number of scans is to increase the milling rate slightly.

From the data of Fig. 2 we can take a rough value of the GaN milling rate of 0.5 μm^3/nA s when using Ga^+ ions. This number is useful in approximating the milling time required for various structures. The milling rate can be converted into a milling yield value of the number of substrate atoms removed per incident ion. This assumes an equal rate of removal for Ga and N atoms. The sputtering yield increased by approximately 30% between normal incidence and the 30° incident angle. This closely matches the calculation for the relative sputtering yield as a function of angle reported by Santamore *et al.*[10] In general, for our GaN milling experiments the effective ion yield ranged from approximately 5–9 atoms/ion.

We also performed Ga^+ milling experiments on materials commonly used as substrates for GaN thin (and sometimes thick) film growth. A comparison of the milling rate of GaN, 6H-SiC, Si, and sapphire as a function of Ga^+ energy from 30 to 70 keV is shown in Fig. 3. Under the same milling conditions for 10 scans, the GaN milling rate is nearly five times that of sapphire, three times that of SiC and two times that of Si at all Ga^+ energies. The fact that the GaN milling rate is always higher than that of the likely substrates is of assistance in terminating the milling process without removing excessive amounts of substrate material. The sputtering yields obtained from the milling rates of GaN and various substrates are shown in Fig. 4. The sputtering yield of GaN is approximately three times larger than that of all three substrate materials, which had sputtering yields ranging from 1.9 to 2.5 atoms/ion. Interestingly, the results of Pellerin, Griffis and Russell[12] and of Khamsepour and Davis[13] for lower FIB energies (included in Fig. 4) are consistent with our findings at higher FIB energies. Furthermore, the data of Young, Cleaver and Ahmed[14] at 30 keV Ga^+ micromilling of Si match our results nearly identically.

To understand the relationship between the FIB milling rate and the fundamental properties of the materials being milled, we show in Fig. 5 the sputtering yield as a function of the strength of the chemical bond in each material.[15] A clear relation is observed, with the milling rate uniformly

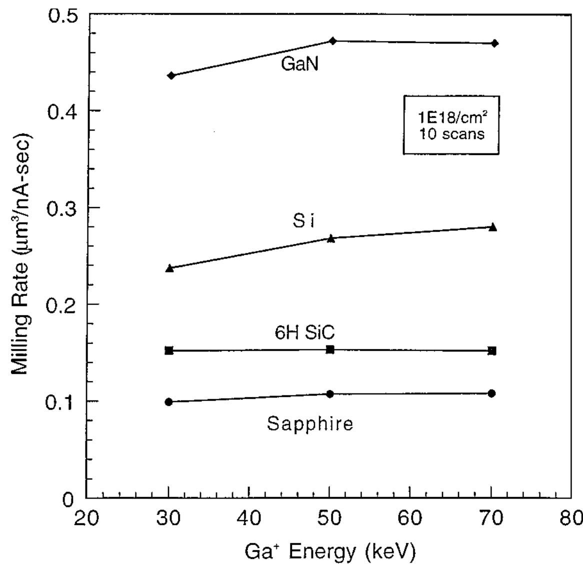


FIG. 3. Comparison of Ga^+ milling rate of GaN and that of potential substrate materials (Al_2O_3, SiC, Si) as a function of ion energy (dose: $1 \times 10^{18}\,cm^{-2}$, normal incidence, 10 scans).

decreasing as the chemical bond strength of the milled material increases. The bond strengths of several materials which have not yet been investigated are also indicated in Fig. 5, allowing prediction of their milling rate under the same FIB conditions.

In addition to the fabrication of end-facet mirrors for conventional semiconductor laser structures, another interesting application of FIB micromilling is in the fabrication of short cavity lasers which utilize air/semiconductor distributed Bragg reflection (DBR)[16,17] grating mirrors. With this type of DBR structure, the high index difference between the air spacing and the semiconductor mirror in the gratings can offer better reflectivity than conventional cleaved mirrors. In

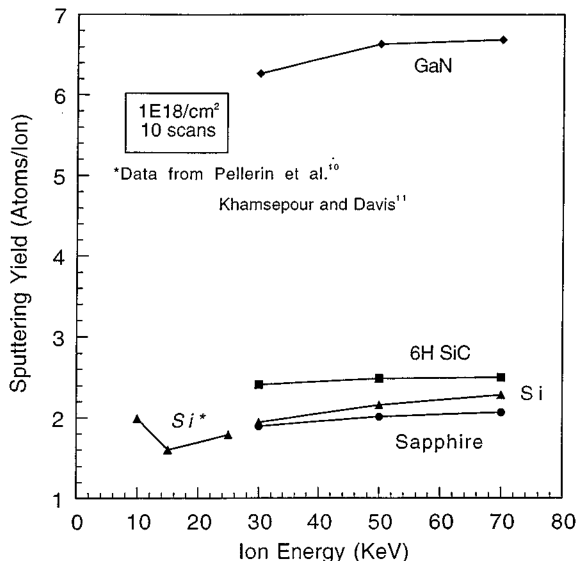
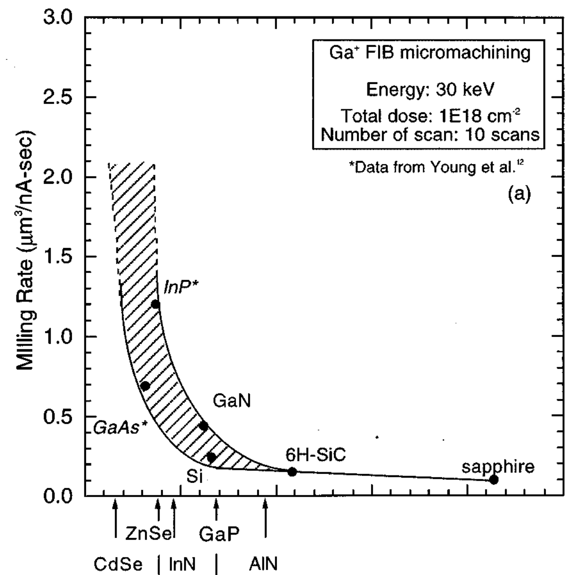


FIG. 4. Comparison of the sputtering yield of GaN, Al_2O_3, SiC, and Si (dose: $1 \times 10^{18}\,cm^{-2}$, normal incidence, 10 scans).



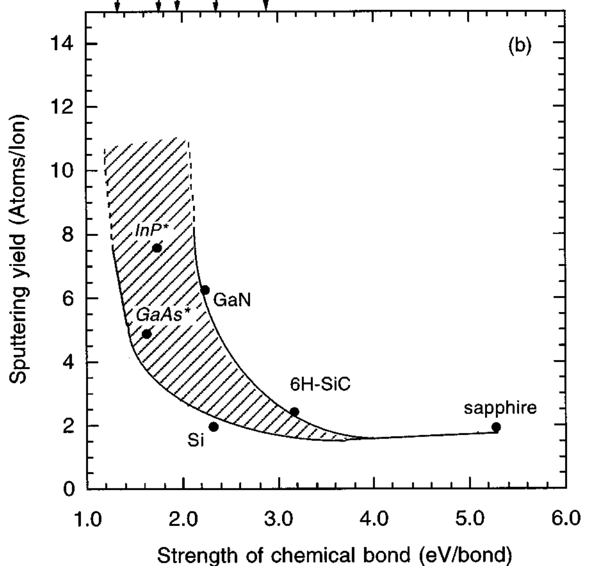


FIG. 5. Milling rate and sputtering yield of selected materials as a function of bond strength. The experimental data are for 30 keV Ga^+, 10 scans from this work (GaN, SiC, Si, Al_2O_3) and from Young *et al.* (Ref. 14) (GaAs and InP). Also shown are the bond strengths of other semiconductors of interest.

Fig. 6 we show a SEM photograph of a prototype for this type of laser fabricated in GaN using FIB micromilling. For this structure, the length of each GaN grating element is designed to be 1.0 μm and the spacing of each grating is 1.5 μm. The fabricated length of the grating elements is narrower ($\sim$0.5 μm) than the design value because the "tail" distribution of the ion beam is sufficient to remove a significant amount of material at the dose utilized. The width of the mirror gratings and the laser waveguide are 8 and 4 μm, respectively. The Ga^+ FIB energy was increased to 70 keV in order to obtain a smaller beam diameter. The total ion dose utilized was $3 \times 10^{18}\,cm^{-2}$. To produce the pattern shown in Fig. 6, a series of 10 FIB scans was employed. The first 5 scans were parallel to the long axis of the rectangles which make up the milled pattern. The final 5 scans were parallel to the short axis of each rectangle. Using this scan

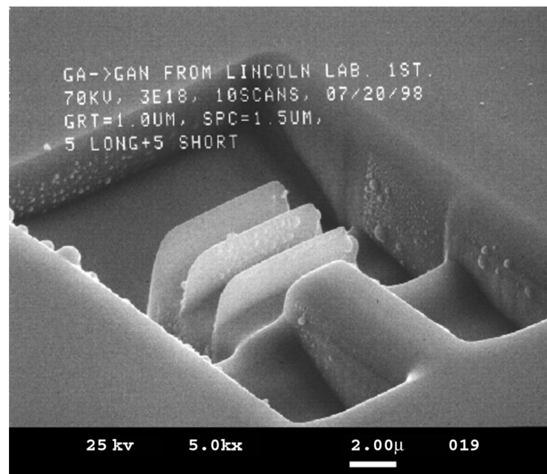


FIG. 6. SEM photograph of the GaN short cavity laser with DBR gratings fabricated by FIB micromilling: 70 keV Ga^+, $3\times10^{18}\,cm^{-2}$ dose, normal incidence. The center-to-center spacing of the individual mirrors is 2.5 μm.

strategy we have reduced the level of material redeposition during FIB micromilling of the pattern. However, as seen in Fig. 6, we did not completely prevent the redeposition from occurring along the walls of the cavity. The redeposition can be further decreased by increasing the number of scans. A smoother surface was obtained when fabricating the structure with 30 FIB scans, but at the expense of a shallower cavity.

IV. SUMMARY AND CONCLUSION

In summary, we have reported the Ga^+ FIB micromilling characteristics of GaN. We have compared the milling rate and sputtering yield of GaN with that of various materials used as substrates for epitaxial growth. The high FIB milling rate of GaN (significantly higher than that of common substrate materials) has positive implications for future fabrication of GaN devices. The FIB sputtering yield of several materials is found to be inversely proportional to the chemical bond strength of the material. This relation may be extendable to other unexplored materials. We also demonstrated that FIB micromilling has the capability to fabricate DBR gratings in order to obtain better reflectivity for laser diodes. In conclusion, we believe that FIB micromilling is a very promising technique for GaN laser fabrication.

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