GaN focused ion beam micromachining with gas-assisted etching

I. Chyr and A. J. Steckl[a)]
Nanoelectronics Laboratory, University of Cincinnati, Cincinnati, Ohio 45221-0030

(Received 20 July 2001; accepted 17 September 2001)

Halide gases, such as Cl_2, IBr, or ICl, are common etchant species for the etching of III nitrides and other compound semiconductor materials in plasma etching processes. We have investigated the Ga^+ focused ion beam milling of GaN in conjunction with gas-assisted etching (GAE) by halide gases I_2 and XeF_2. We have observed that I_2 and XeF_2 GAE with a 30 keV Ga^+ ion beam leads to significantly enhanced GaN etch rates. When these gases are utilized with appropriate ion beam scan strategies (such as ion beam current, beam dwell time, and beam overlap), we have measured GaN etch rate enhancements of 6× to 9× and 2× to 3× faster for I_2 and XeF_2, respectively. [DOI: 10.1116/1.1417550]

I. INTRODUCTION

Gas-assisted etching (GAE) or gas-assisted deposition (GAD) are very useful extensions of the focused ion beam (FIB) technique. With the introduction of reactive chemical precursors, a FIB is able to enhance the etching effect on selected regions, deposit metals or insulating materials on specific areas in order to repair masks, or modify integrated circuits. GAE-FIB has been utilized with various gases for different material systems. This includes the use of Cl_2 with GaAs, Si, and InP;[1,2] XeF_2 for SiO_2, W,[3] and diamond;[4,5] and H_2O on PMMA[6] and other photoresists.[7] Young *et al.*[2] and Harriott[3] summarized the advantages of gas-assisted etching with FIB: (1) enhancement of the FIB etch rate; (2) reduction of unwanted implantation from primary ion species into the substrate; (3) reduction of redeposition on the sidewall or surrounding structures; (4) selectivity on multiple layer structures. There are several major parameters which influence the results of the GAE process: frame time (the time elapsed between ion beam visits of the same pixel), ion beam dwell time at a single pixel, ion current density, and gas flux. With the proper scan strategies, by changing these parameters one can increase the etch rates on target materials or improve the milling results to the processing areas.

GaN and related nitride compounds have become a great success for visible and ultraviolet (UV) light emitting diodes (LEDs)[8] and laser diodes.[9] However, some fabrication issues concerning sample preparation and processing techniques still remain unresolved. In particular, high reflectivity mirror facets are hard to obtain by conventional processing procedures due to the large misalignment[10] between the sapphire substrate and GaN-based materials. Recently, several groups[11,12] have applied FIB micromachining to produce flat and smooth mirror facets for GaN laser diodes. We have previously reported[13] on the use of FIB milling to fabricate gratings for short cavity GaN lasers. To date, no quantitative analysis of FIB micromachining (FIBM) with GAE has been reported for GaN. In this article, we present results of GAE-FIBM of GaN.

II. EXPERIMENT

In this study, we have utilized the FEI FIB 200 focused ion beam system and Ga^+ liquid metal ion sources (LMIS). The experiments were done at 30 keV ion energy. The milled patterns are square regions of $20\times20\ \mu m^2$. The total ion dose is kept constant at approximately 3×10^{17} ions/cm^2. Therefore, the milling time becomes shorter as the ion current is increased. There are two gas injection systems, I_2 and XeF_2, installed in the chamber. These two materials are in solid form at room temperature and are contained within sealed crucibles. The melting points for I_2 and XeF_2 are ~114 and 129 °C, respectively. During operation the crucibles are heated slightly (to 25 °C for XeF_2 and to 35 °C for I_2), causing evaporation and gas injection into the FIB chamber through a needle whose tip is 75–100 μm above the sample surface. The diameter of the needle is ~100 μm. During gas-assisted etching, the chamber pressure increases from the normal value of $(1-5)\times10^{-7}$ mbar to more than 10^{-5} mbar. Through the user interface, we are able to change the milling parameters such as ion beam current, beam dwell time, beam overlap percentage, and gas species. However, due to the hardware setup of the gas injection system, we could not adjust the distance between the needle and the sample surface to control the gas flow rate and volume.

The primary GaN material used for milling experiments was grown by hydride vapor phase epitaxy (HVPE) on sapphire substrates.[14] The GaN film was relatively thick (~20 μm) which allowed the milling experiments to be performed without encountering any substrate effects. Profiles of the depth and surface roughness were measured by the Digital Instruments Dimension 3100 atomic force microscope (AFM) to obtain accurate sample information. Secondary electron images of milled patterns were obtained in the FIB system by scanning the Ga^+ ion beam on the GaN samples after the milling process had finished.

III. RESULTS AND DISCUSSION

Before we explain our experimental results of the enhanced etch with FIB micromachining, let us define the terminology used in the following discussion. The definition of beam dwell time is the time that the ion beam stays in one

[a)]Electronic mail: a.steckl@uc.edu

 1071-1023/2001/19(6)/2547/4/$18.00

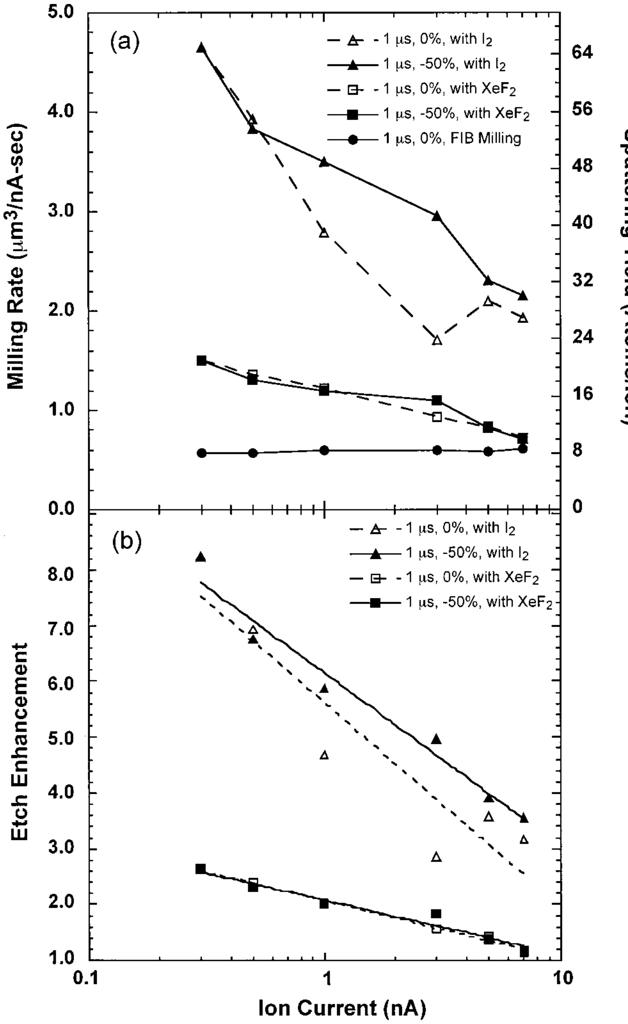

FIG. 1. Effect of the Ga^+ FIB current on (a) FIBM and GAE-FIBM milling rates of GaN and (b) GAE etching enhancement. The beam dwell time is 1 μs and the beam overlaps are 0% and -50%.

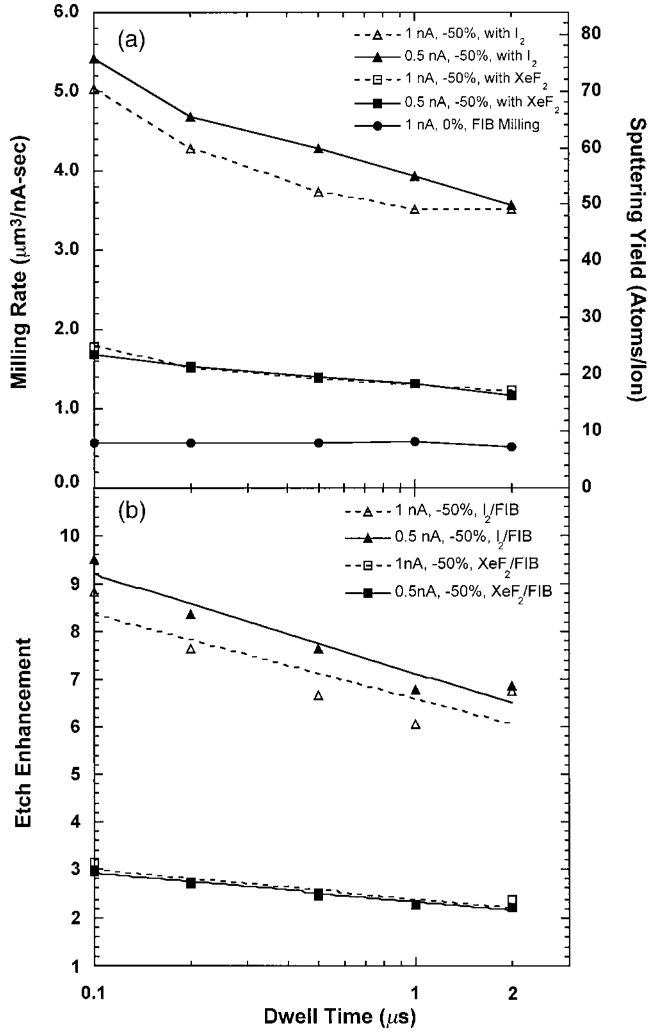

FIG. 2. Effect of the Ga^+ FIB dwell time on (a) FIBM and GAE-FIBM milling rates of GaN and (b) GAE etching enhancement. The beam overlaps are 0% and -50% and the ion beam currents are 0.5 and 1 nA.

pixel and mills that specific pixel. The beam overlap is the ratio of the overlap of two pixels divided by the diameter of the ion beam. If the beam overlap is "+50%," it means that there is an overlap equal to the beam radius between two adjacent beam locations (center to center). A "-50%" beam overlap indicates that the gap between two pixels is equal to one half of the beam diameter.

Figure 1 shows the milling rates and I_2 and XeF_2 etching enhancements as a function of the ion beam current and of different beam overlap percentages. In Fig. 1(a), as the ion beam current is increased from 0.3 to 7 nA, the milling rate per unit of ion current with FIB micromachining only is essentially constant at approximately 0.6 $\mu m^3/nA\,s$, which corresponds to a sputtering yield of ~8 atoms/ion. In other words, the volumetric milling rate ($\mu m^3/s$) for FIBM increases linearly with the ion beam current. The milling rate for FIBM was essentially identical for beam overlaps of -50%, 0%, and +50%. When I_2 gas was added to the FIBM, the GaN milling rate increased 3× to 2.0–2.4 $\mu m^3/nA\,s$ for large beam currents and up to 8× to 4.7 $\mu m^3/nA\,s$ for a beam current of 300 pA. For GAE with XeF_2, an increase of up to 2.5× (i.e., 1.2–1.6 $\mu m^3/nA\,s$) is measured for beam currents less than 1 nA. The etching enhancement for GAE with these two gases is shown in Fig. 1(b). The enhancement in the milling rate is seen to decrease monotonically with an increase in ion beam current for both gases. When we utilized smaller ion currents with these two halide gases, redeposition on the target surface is reduced and the etching effect is greatly enhanced. Therefore, the dominant etching mechanism appears to be the chemical reaction with the materials sputtered from the surface by the focused ion beam. In contrast, the ion bombardment at larger ion currents produces copious material by redeposition, overwhelming the chemical reaction on the surface. Therefore, the etching enhancement is significantly less at the largest beam currents (7 nA) we have utilized compared to the smallest ion currents (300 pA). However, it is important to keep in mind that the volumetric milling rate increases with ion current for both FIBM and GAE-FIBM.

The effect of FIB dwell time on the GaN milling rate is shown in Fig. 2. As expected, the FIBM rate was found to be

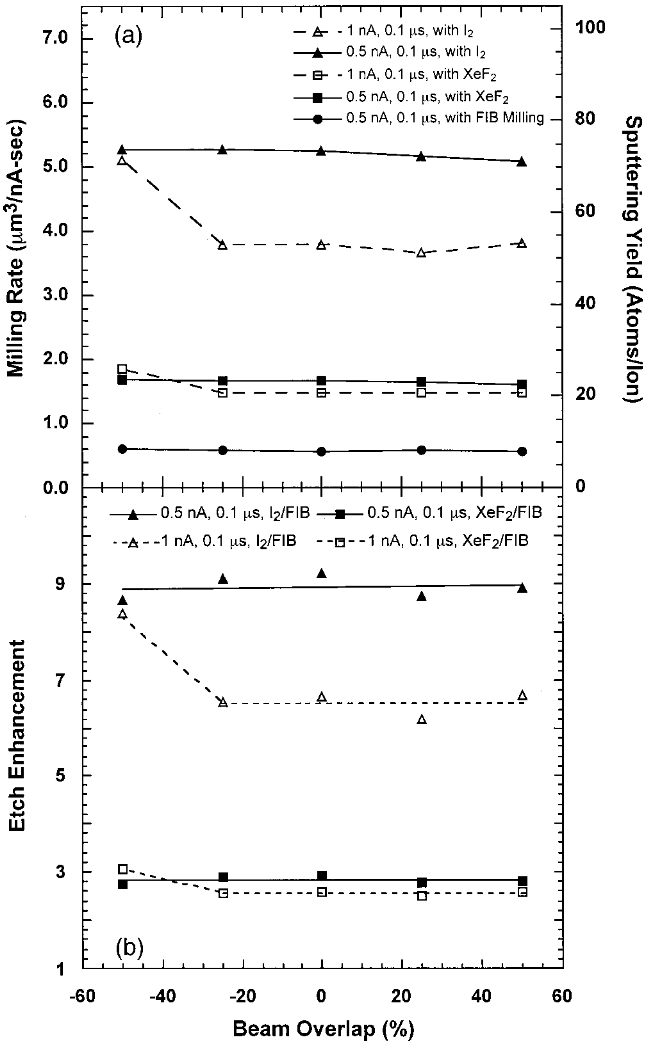

FIG. 3. Effect of the Ga^+ FIB beam overlap on (a) FIBM and GAE-FIBM milling rates of GaN and (b) GAE etching enhancement. The beam dwell time is 1 μs and the ion beam currents are 0.5 and 1 nA.

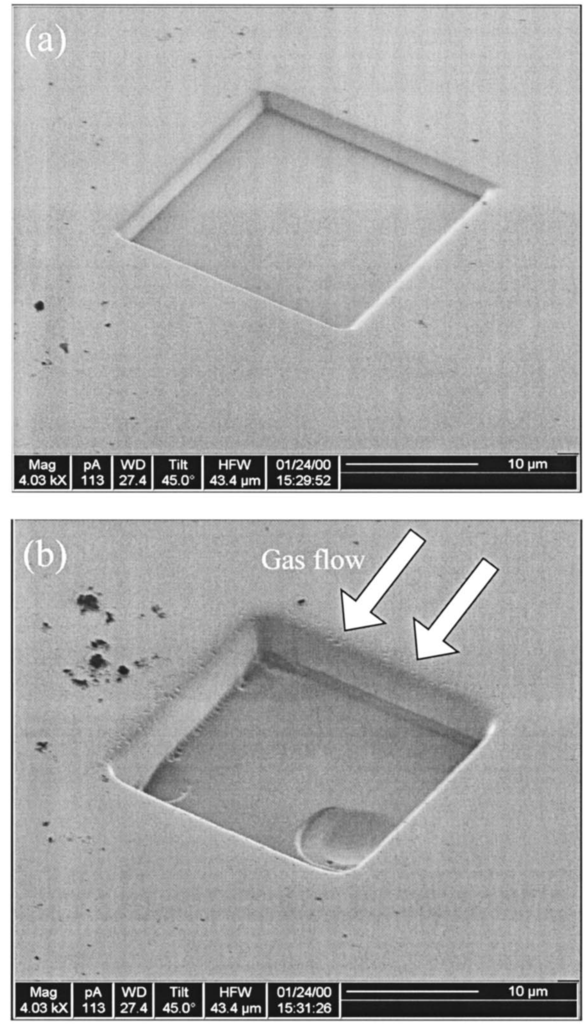

FIG. 4. SEM micrographs of (a) FIBM patterns milled in GaN and (b) GAE-FIBM with I_2 GAE. The milled area is 20×20 μm^2.

constant for dwell times from 0.1 to 2 μs, as shown in Fig. 2(a). Since the GAE gas flow rates were fixed by the FIB column design, we also investigated the effect of reducing the ion currents from 1 to 0.5 nA. From Fig. 2(b), we see that GAE-FIBM with I_2 and XeF_2 gases enhances the milling rates by factors of 6× to 8.5× and 2× to 3×, respectively. As the beam dwell time is reduced, the ion dose of a single scan at each pixel on the pattern is reduced. To maintain the fixed total dose, the number of scans is correspondingly increased. Thus, the etching enhancement is stronger at short beam dwell times because the chemical reaction governs the milling process. As the beam dwell time becomes longer, the dose of each single scan increases, enhancing the effect of physical spattering. This confirms the well-known conclusion that to obtain maximum improvement from the GAE process one needs to select a smaller ion beam current with a shorter beam dwell time.

Let us consider next the influence of beam overlap (while keeping the total dose constant) from gas-assisted FIB milling on GaN. In the case of FIBM only, Fig. 3(a) shows that the milling rate is ~0.6 μm^3/nA s, for overlaps from +50% to −50% and for 0.5 nA beam current and 0.1 μs dwell time. For GAE-FIBM we have used the same FIB parameters as well as increasing the ion beam current to 1 nA, since these were conditions previously shown to result in larger milling rate enhancement. As shown in Figs. 3(a) and 3(b), GAE with I_2 results in a milling rate in the case of 0.5 nA of ~5 μm^3/nA s, independent of the overlap. This represents a 9× enhancement over the FIBM-only case. Utilizing GAE with XeF_2 produces a constant milling rate of ~1.8 μm^3/nA s for all conditions of beam overlap and for both 0.5 and 1 nA ion beam currents. This represents a 3× enhancement in the etching rate compared to FIBM only of GaN. This is in contrast to the I_2 GAE, where increasing the beam current from 0.5 to 1 nA results in lower etch enhancement for beam overlaps greater than −20%. Clearly, the iodine radicals are more reactant with GaN than fluorine radicals, and this similar to the situation in GaAs plasma-based etching. Furthermore, the GAE enhancement is reduced when the ion beam dose is increased either directly or by increasing the beam overlap.

Figure 4 contains scanning electron microscope (SEM) micrographs of GaN regions patterned by FIBM only and by GAE-FIB with I_2. An example of FIBM only is shown in Fig. 4(a). The sidewalls are nearly vertical and the etched surface is smooth. As shown in Fig. 4(b), GAE with I_2 results in a deeper milled area by a factor of 2× to 3×. However, the edges of the milled region are not as sharp as those for the FIBM only and the etched surface appears nonuniform, with a pronounced tilt toward the lower corner. The I_2 gas was introduced from the upper corner of the pattern, apparently resulting in increasing chemical reaction efficiency in the direction of the gas flow. Rounded imprints (bubbles) are occasionally found in the corners of the pattern. Several explanations for this phenomenon are possible: liquefied gas droplets which produce higher etch rates, Ga redeposition, and/or nonuniform gas flow inside the milled area.

IV. SUMMARY AND CONCLUSION

We have reported gas-assisted Ga^+ focused ion beam milling of GaN using I_2 and XeF_2. An enhancement in the milling rate of 6× to 9× is obtained for I_2 GAE and of 2× to 3× for XeF_2 GAE. We have investigated the effect of the ion beam current, beam dwell time, and beam overlap on the GaN milling rate and on the GAE enhancement. In general, the volumetric milling rate increases with the ion beam current while the GAE effect is strongest under a smaller ion beam current.

ACKNOWLEDGMENTS

This work was supported in part by the Department of Defense. The authors are grateful to S. Mogren for many helpful discussions.

[1] R. J. Young, J. R. Cleaver, and H. Ahmed, Microelectron. Eng. **11**, 409 (1990).
[2] R. J. Young, J. R. Cleaver, and H. Ahmed, J. Vac. Sci. Technol. B **11**, 234 (1993).
[3] L. R. Harriott, Jpn. J. Appl. Phys., Part 1 **33**, 7094 (1995).
[4] P. E. Russell, T. J. Stark, D. P. Griffis, J. R. Phillips, and K. K. Jarausch, J. Vac. Sci. Technol. B **16**, 2494 (1998).
[5] A. Dutta, Y. R. Wu, and Y. L. Wang, Appl. Phys. Lett. **75**, 2677 (1999).
[6] T. J. Stark, G. M. Shedd, J. Vitarelli, D. P. Griffis, and P. E. Russell, J. Vac. Sci. Technol. B **13**, 2565 (1995).
[7] T. J. Stark, D. P. Griffis, and P. E. Russell, J. Vac. Sci. Technol. B **14**, 3990 (1996).
[8] H. Amano, M. Kito, K. Hiramatsu, and I. Akasaki, Jpn. J. Appl. Phys., Part 2 **28**, L2112 (1989).
[9] S. Nakamura *et al.*, Jpn. J. Appl. Phys., Part 2 **36**, L1568 (1997).
[10] J. Edgar, J. Mater. Res. **7**, 235 (1992).
[11] T. Ito, H. Ishikawa, T. Egawa, T. Jimbo, and M. Umeno, Jpn. J. Appl. Phys., Part 1 **36**, 7710 (1997).
[12] H. Katoh *et al.*, Jpn. J. Appl. Phys., Part 2 **37**, L444 (1998).
[13] I. Chyr, B. K. Lee, L. C. Chao, and A. J. Steckl, J. Vac. Sci. Technol. B **17**, 3063 (1999).
[14] R. Molnar, W. Gotz, L. T. Romano, and N. M. Johnson, J. Cryst. Growth **178**, 147 (1997).