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LETTER TO THE EDITOR

Dry etching of thin-film InN, AlN and GaN

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Abstract. Smooth, anisotropic dry etching of InN, AlN and GaN layers is demonstrated using low-pressure (1–30 mTorr) $CH_4/H_2/Ar$ or Cl_2/H_2 ECR discharges with additional DC biasing of the sample. The etch rates are in the range 100–400 Å min^{-1} at 1 mTorr and -150 V DC for Cl_2/H_2, while higher biases are needed to initiate etching in $CH_4/H_2/H$ discharges. The presence of hydrogen in the gas chemistries is necessary to facilitate equi-rate removal of the group III and nitrogen etch products, leading to smooth surface morphologies.

Interest has recently been revived in the use of group III nitrides (InN, GaN and AlN) and their ternary counterparts for optical devices, operating in the red to ultraviolet [1]. Excellent progress has been made in obtaining reproducible thin-film properties in material grown by organometallic vapour phase epitaxy and plasma-enhanced molecular beam epitaxy, leading to the realization of p–n junction GaN light-emitting diodes (LEDs) with room temperature emission in the UV [2] and double-heterojunction AlGaN/GaN LEDs [3]. The achievement of better device yields is currently limited by a lack of two technologies — a reliable ohmic contact for the nitrides and established etching procedures, particularly dry etching. In this letter we report on electron cyclotron resonance (ECR) plasma etching of high-quality InN, GaN and AlN films grown by ECR-assisted metal-organic molecular beam epitaxy. Anisotropic pattern transfer is achieved with low-pressure BCl_3/Ar, $CH_4/H_2/Ar$, CCl_2F_2/Ar or Cl_2/H_2 discharges.

Nitride layers were grown at a rate of 50–75 Å min^{-1} on semi-insulating GaAs substrates using group III metalorganics (trimethylindium, triethylgallium and trimethylamine alane respectively for InN, GaN and AlN) and atomic nitrogen generated by a 2.45 GHz ECR wavement MPDR source. The deposition temperature was ~ 500 °C, and the films were polycrystalline-columnar. Layers up to ~ 0.4 μm thick were patterned with Hunt 1182 photoresist and subsequent etch rates were measured by stylus profilometry after removal of this photoresist in acetone.

ECR discharges of BCl_3/Ar, CCl_2F_2/Ar and $CH_4/H_2/Ar$ were initially examined for etching the nitride films. These are our standard mixtures for etching AlGaAs, GaAs and InP respectively. Process pressures in the range 1–30 mTorr with a forward microwave power of 200 W and additional DC biases of -25 to -300 V at the sample position derived from 13.56 MHz power application were employed. Total gas flow rates were in the range 15–32 SCCM.

Figure 1 shows the etch rates of the three nitride materials $10BCl_3/5Ar$ discharges as a function of DC bias (top) at constant pressure (1 mTorr) or as a function of pressure (bottom) at constant DC bias (-250 V). The near-linear increase in etch rates with increasing bias indicates that sputter-enhanced removal of the etch products is still a limiting factor under these conditions. Increasing the process pressure enhances the AlN and GaN etch rates as more active chlorine species are supplied to the surface, but in the case of InN, the $InCl_3$ etch product is relatively involatile at room temperature and the rates are not strongly dependent on pressure under our conditions.

CCl_2F_2/Ar discharges yielded similar etching characteristics to those of BCl_3/Ar for InN and GaN, but in the case of AlN higher biases were required to initiate the etching. This result is expected on the basis of the formation of the involatile species AlF_3, which inhibits the etch rate [4].

The nitride etch rates as a function of DC bias (top) and pressure (bottom) in $7CH_4/17H_2/8Ar$ discharges are shown in figure 2. The rates are again nearly linearly dependent on bias, and decrease slightly at high pressures for InN and GaN, most likely as a result of a competition between polymer deposition and etching. The $CH_4/H_2/Ar$ discharges yielded the smoothest etched-surface morphologies, which is an indication that the group III and N etch products are being removed at near-equal rates. The latter are presumably NH_x species

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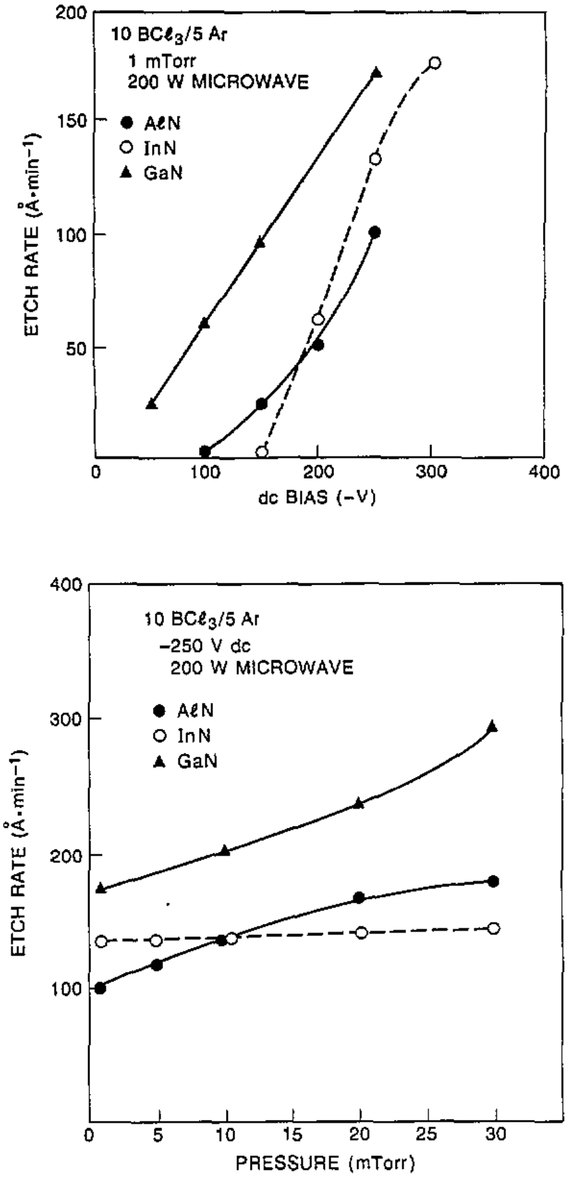

Figure 1. Etch rates of the nitrides in BCl_3/Ar discharges (1 mTorr, 200 W microwave power) as a function of applied DC bias (top) and as a function of pressure at fixed bias (bottom).

while the former are metalorganics or adducts involving methyl species and the group III metals. By contrast, while $GaCl_x$ and $AlCl_x$ species are volatile near room temperature we have noted earlier that $InCl_x$ species are not particularly volatile and must be heated to ≥ 130 °C to facilitate their removal. Similarly, nitrogen chlorides are not particularly easy to form. These results suggest that addition of hydrogen to chlorine-based discharges would enhance removal of the nitrogen, enabling the achievement of smooth morphologies. In addition, the use of elevated sample temperatures would most likely produce fast, smooth etching of InN as reported previously for InP in Cl_2/H_2 discharges [5].

Figure 3 shows the etch rates of the nitrides in Cl_2/H_2 discharges near room temperature, as a function of both H_2 flow rate at fixed pressure (top) and as a function of DC basis at fixed H_2 flow (bottom). The etch rates do increase with H'_2 addition and the morphologies become featureless, indicating that there is more efficient removal of the N etch product. Note also that the rates are significantly faster than for the other mixtures investigated. Examples of the etched surface morphologies are shown in the SEM micrographs of figure 4. At the top are features formed by $CH_4/H_2/Ar$ dry etching of ~ 300 Å of InN followed by ~ 7000 Å of the underlying GaAs at 1 mTorr pressure and −250 V DC bias. Both materials are etched in a smooth, anisotropic fashion under these conditions. At the bottom is a feature formed by etching ~ 2000 Å of AlN in a $10Cl_2/15H_2$, 1 mTorr, 200 W (microwave), −150 V DC discharge. The nitride etching is also smooth in this instance.

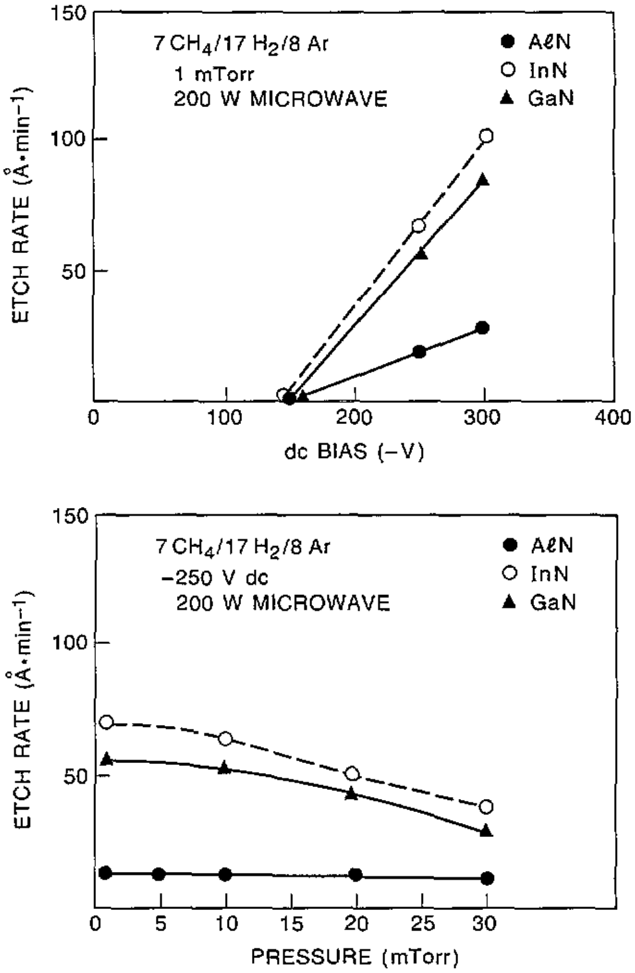

Figure 2. Etch rates of the nitrides in CH_4/H_2/Ar discharges as a function of applied DC bias (top) or as a function of pressure (bottom).

For nitride layers deposited on GaAs substrates one can obtained etch selectivities of ~ 3-5 for these films over the substrate using $CH_4/H_2/Ar$ discharges. For Cl_2-based discharges there is no selectivity since the GaAs will etch at faster rates. However, selectivities would be high for etching of nitrides deposited on Al_2O_3,

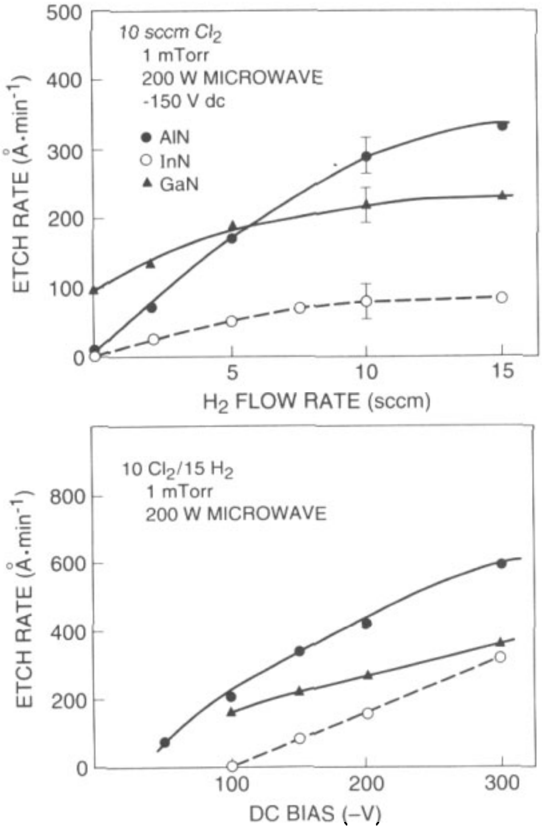

Figure 3. Etch rates of the nitrides in Cl_2/H_2 discharges as a function of H_2 flow rate (top) or as a function of applied DC bias (bottom).

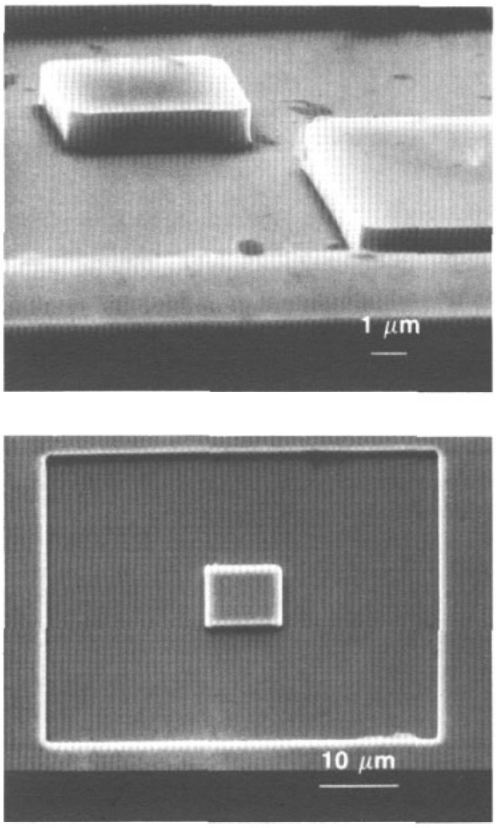

Figure 4. SEM micrographs of features etched into an InN/GaAs structure using a $CH_4/H_2/Ar$ discharge (top) and into an AlN film using a Cl_2/H_2 discharge (bottom).

SiC or ZnO substrates using any of the gas mixtures used here.

In conclusion, we have demonstrated that chlorine- and methane/hydrogen-based discharges, in conjunction with simple photoresist masking, can be used for small, anisotropic pattern transfer into InN, AlN and GaN thin films. This facilitates fabrication of wide-gap photonic devices.

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References

[1] See for example, Strite S and Morkoc H 1992 GaN, AlN and InN: a review *J Vac. Sci. Technol.* **B 10** 1237–66 and references therein

[2] Nakamura S, Senoh M and Mukai T 1991 Highly p-type, Mg-doped GaN films grown with GaN buffers *Japan. J. Appl. Phys.* **30** L1708–9

[3] Akasaki I, Amano H, Kito M and Kiramatsu K 1991 High quality double heterojunction AlGaN/GaN LEDS *J. Lumin.* **48/49** 666–9

[4] Cooper C B III, Salimian S and MacMillan H F 1987 Use of thin AlGaAs and InGaAs stop-etch layers for RIE processing of III–V compound semiconductor devices *Appl. Phys. Lett.* **51** 2225–7

[5] Constantine C, Barratt C, Pearton S J, Ren F and Lothian J R 1992 Microwave Cl_2/H_2 discharges for high rate etching of InP *Electron. Lett.* **28** 1749–50