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# Comparison of ICl- and IBr-Based Plasma Chemistries for Inductively Coupled Plasma Etching of GaN, InN and AlN

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#### **ABSTRACT**

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A parametric study of the etch characteristics of GaN, AlN and InN has been carried out with ICl/Ar and IBr/Ar chemistries in an Inductively Coupled Plasma discharge. The etch rates of InN and AlN were relatively independent of plasma composition, while GaN showed increased etch rates with interhalogen concentration. Etch rates for all materials increased with increasing rf chuck power, indicating that higher ion bombardment energies are more efficient in enhancing sputter desorption of etch products. The etch rates increased for source powers up to 500 W and remained relatively thereafter for all materials, while GaN and InN showed maximum etch rates with increasing pressure. The etched GaN showed extremely smooth surfaces, which were somewhat better with IBr/Ar than with ICl/Ar. Maximum selectivities of ~ 14 for InN over GaN and >25 for InN over AlN were obtained with both chemistries.

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#### INTRODUCTION

Plasma etching techniques have been predominantly used in the patterning of III-nitrides for photonic devices such as laser diodes and light-emitting diodes (LEDs). 1-12 Currently, all of the LEDs and a majority of the lasers are ridge wave guide structures in which the mesas are formed by dry etching. 13 Therefore, most of the previous etching studies have been focused on obtaining the relatively large etch depths (2-4 µm) typical of ridge or facet heights, where the final surface morphology on the field is less important. Moreover, an n-type ohmic contact is deposited on the etched surface, and loss of nitrogen from this surface actually improves contact resistance. Attention is now turning to the development of GaN-based high power/high temperature electronics for power switching and transmission applications. 14-18 In these devices, the etch depth is much shallower, but smooth morphologies and high selectivities for InN over the other nitrides are required because layers based on InN will probably be used to obtain low ohmic contact resistance. In some cases rectifying contact will be deposited on the etched surface, so its electrical quality is most important.

Shul et al.<sup>1,10</sup> reported Inductively Coupled Plasma (ICP) etching of GaN, AlN, InN, InAlN and InGaN at low dc biases (≤ -100 V) with Cl<sub>2</sub>, CH<sub>4</sub>/H<sub>2</sub>, Cl<sub>2</sub>/Ar, Cl<sub>2</sub>/N<sub>2</sub> and Cl<sub>2</sub>/H<sub>2</sub> plasma chemistries. They controlled the etch rates in the range of 500 ~ 1500 Å/min for electronic device structures, and obtained maximum etch selectivities of ~ 6 at higher ICP source powers (850 W) for InN over the other nitrides. Alternative chemistries have included IBr<sup>19</sup> and ICl,<sup>20</sup> operated under Electron Cyclotron Resonance conditions. These interhalogens appear to be readily dissociated, producing high

done on the ICP etching of III-nitrides with ICl- and IBr-based plasma chemistries. The ICP configuration is the preferred one for high density etching because of its superior uniformity, control and lower cost of ownership.

In this paper, a parametric study of ICP etching of GaN, AlN and InN with ICl-and IBr-based plasma chemistries is reported. The effects of plasma composition, rf chuck power and ICP source power on etch rates, etch yield, selectivity, dc biases and ion flux at the sheath edge have been investigated. There is no clear advantage in terms of etch rates or selectivity for either chemistry. The ICP discharges are well suited for achieving controllable etch rates (500 – 1500 Å/min) and high selectivities (up to 30) for InN over AlN and GaN.

#### **EXPERIMENTAL**

The AlN and InN samples were grown by Metal Organic Molecular Beam Epitaxy (MOMBE) on  $Al_2O_3$  substrates at 800 °C and 575 °C, respectively in an Intevac Gen II system. <sup>21,22</sup> The GaN was grown at 1040 °C on  $Al_2O_3$  substrates by Metal Organic Chemical Vapor Deposition (MOCVD). Total layer thicknesses were ~ 1 $\mu$ m for the AlN and InN, and 2-3  $\mu$ m for the GaN.

The samples were patterned with Apiezon wax and etched in a Plasma-Therm ICP 790 system. The temperature of the back-side cooled chuck was held at 23 °C. The rf chuck power was varied between 50 and 350 W, and ICP source power between 300 and 1000 W. The process pressure was held constant at 5 mTorr, while the total flow rate of

IBr/Ar or ICl/Ar gas was 15 sccm. Etch rates were calculated from stylus profilometry measurements of the etched samples after the removal of the mask material. The error of these measurements is approximately  $\pm 5$  %. Surface morphology was examined by atomic force microscopy (AFM) system operating in tapping mode with Si tip. The selectivity was calculated for InN over AlN and GaN.

#### RESULTS AND DISCUSSION

Figure 1 shows the effect of plasma composition on etch rates of InN, AlN and GaN in IBr/Ar and ICl/Ar discharges at 5 mTorr, 750 W source power and 250 W rf chuck power. The etch rates of InN and AlN are relatively independent of plasma composition in IBr (Fig. 1, top) and ICl (middle) plasmas over a broad composition range, indicating the etch mechanism is dominated by physical sputtering. The dc bias voltages increased with increasing etch gas concentrations, resulting in decrease in ion flux entering the sheath layer (Fig.1, bottom). The ion flux at the sheath edge was calculated using a global self-consistent model developed for the ICP etching system.<sup>23</sup> The increase in dc biases or decrease in ion flux can be explained by the fact that compared to pure Ar discharges, additional collisional energy losses (which include excitation of vibrational and rotational energy levels, molecular dissociation and negative ion formation<sup>24</sup>) are present with IBr and ICl. The decrease in ion flux also implies an increase in concentrations of neutral species such as Cl, Br and I. The etch rate of GaN increased up to 66.7% IBr and remained almost constant at higher concentration (Fig. 1, top). By contrast the etch rate steadily increased with increasing ICl concentration in the ICl/Ar plasma (middle). This result indicates that etching of GaN in both chemistries is more attributed to chemical etching by increased concentrations of reactive neutrals than ion-assisted sputtering.

The effect of ICP source power on etch rates, dc bias voltages, and ion fluxes at the sheath edge are shown in Fig 2 for IBr/Ar (top) and ICl/Ar discharges (middle) at 5 mTorr. Flow rates of etch gases were 2 sccm IBr or ICl and 13 sccm Ar. During these runs the rf chuck power was held constant at 250 W, which results in a decrease in dc bias as the ICP power increased. Lower dc biases were attributed mainly to increased ion density at higher ICP powers (bottom). InN again showed higher etch rates than AlN and GaN. The high etch rates for InN are similar to the previously reported results observed for InP where efficient ion-assisted desorption of InCl<sub>x</sub> occurs under ICP conditions.<sup>25</sup> The etch rates of InN, AlN and GaN increased up to 500 W, and remained relatively constant at higher source power. The increase in etch rate with increasing the source power is due to the higher concentration of reactive species in the plasma, suggesting a reactant-limited regime, and to higher ion flux to the substrate surface. The relatively constant etch rate with further increase of the ICP power is attributed to the competition between ion-assisted etch reaction and ion-assisted desorption of the reactive species at the substrate surface prior to etch reactions.

Figure 3 shows the effect of rf chuck power on the etch rates, dc bias, and ion flux at the sheath edge. Etch rates for all materials increased in both IBr (top) and ICl (middle) discharges as the rf power or the ion-bombarding energy increased. InN showed higher etch rates again than GaN and AlN: maximum etch rate of InN is  $\sim 6,000$  Å/min. The increase in etch rate with the chuck power can be attributed to enhanced sputter

desorption of etch products as well as physical sputtering of the InN surface. The dc bias voltage increased monotonically with increasing rf chuck power from 50 to 350 W, but the ion flux at the sheath edge increased slightly (Fig.3, bottom). This is because the main role of the chuck power is to increase the ion-bombarding energy. The effect of the rf power on etch rate (or etch yield) and ion flux at the sheath edge in the ICP system is described in detail elsewhere. <sup>23,26</sup> It is also interesting to see that the magnitude of etch rate is in the order of bond energies, InN (7.72 eV) < GaN (8.92 eV) < AlN (11.52 eV). <sup>27</sup>

The effect of reactor pressure on etch rate, etch yield (defined as number of atoms etched per incident ion<sup>23</sup>), dc bias and ion flux in ICl/Ar plasmas is shown in Fig. 4. During these experiments the source and chuck powers were held constant at 750 W and 250 W, respectively. InN showed a maximum etch rate at 15 mTorr and decreased with further increasing pressure, while the etch rate of GaN increased up to 10 mTorr and decreased thereafter (top). It is also seen that the etch rate of AlN was almost independent of the pressure. The increased etch rates of InN and GaN with pressure indicates that etching is limited by mass transfer of reactive gas species at the lower pressures. However, as the pressure increases further, the etch rate decreases due to either lower ion flux to the substrate surface or to redeposition of etch products. Etch yield data are shown in the lower part of the figure. The higher dc voltages or lower ion fluxes at higher pressures were attributed to increased collisional recombination which decreased the plasma ion density.

Etch morphology was examined using AFM for GaN samples etched at 750 W ICP power, 250 W rf chuck power and 5 mTorr in 2 sccm ICl/13 sccm Ar and 2 sccm IBr/13 sccm Ar discharges, respectively and the results are shown in Fig. 5 with the rms

roughness. It is seen that IBr/Ar chemistry (bottom) shows somewhat better morphology than ICl/Ar (top), resulting in overall mirrorlike smoothness in both chemistries.

In order to reduce the currently high contact resistance in GaN-based heterostructure field transistors, <sup>28</sup> and eventually heterojunction bipolar transistors, it is expected that InN-based contact layers will be necessary, <sup>29-30</sup> in analogy to InGaAs on GaAs. In such a case, the ability to selectively etch InN relative to the other nitrides will be crucial. Figures 6 to 8 show some selectivity data as functions of plasma composition, rf power, and ICP power in IBr- and ICl-based plasmas, respectively. The effect of plasma composition showed an overall trend of decrease in selectivities for InN over AlN and over GaN as the concentrations of IBr and ICl increase (Fig. 5). However, the selectivity of InN over AlN showed maximum values depending on ICP source power (Fig. 6) and rf chuck power (Fig. 7), while that of InN over GaN increased overall as the source and chuck powers increased. The maximum values of selectivity obtained in this work were ~ 30 for InN/AlN and ~ 14 for InN/GaN. It is also clearly seen that the selectivities of InN over AlN in both of IBr/Ar and ICl/Ar discharges are greater than those of InN over GaN.

#### **SUMMARY AND DISCUSSION**

Etching of GaN, AlN and InN has been carried out with ICl/Ar and IBr/Ar chemistries in an Inductively Coupled Plasma discharge. The effects of plasma composition, ICP source power, rf chuck power and pressure on etch rate, etch yield, dc bias and ion flux at the sheath edge were examined. The etch rates of InN and AlN are

relatively independent of plasma composition, while GaN showed increased etch rates with etch gas concentrations. Etch rates for all materials in the ICl- and IBr-based discharges increased with increasing the rf chuck power, indicating that higher bombarding energies are more efficient in enhancing sputter desorption of etch products. The etch rates increased up to 500 W ICP power and saturated for higher powers, while GaN and InN showed maximum etch rates with increasing pressure. The maximum selectivities obtained in this work were ~ 30 for InN/AlN and ~ 14 for InN/GaN, respectively. The selectivities of InN over AlN in both of IBr/Ar and ICl/Ar discharges are greater than those of InN over GaN.

#### **ACKNOWLEDGEMENTS**

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#### REFERENCES

- R. J. Shul, G. B. McClellan, S. A. Casalnuova, D. J. Rieger, S. J. Pearton, C. Constantine, C. Barrat, R. F. Karlicek, Jr., C. Tran, and M. Schurman, Appl. Phys. Lett., 69, 1119 (1996).
- R. J. Shul, in GaN and Related Materials, ed. S. J. Pearton (Gordon and Breach, N. Y., 1997).
- 3. H. P. Gillis, D. A. Choutov, and K. P. Martin, JOM, 48, 50 (1996).
- 4. I. Adesida, A. Mahajan, E. Andideh, M. A. Khan, D. T. Olsen, and J. N. Kuznia, Appl. Phys. Lett., 63, 2777 (1993).
- M. E. Lin, Z. F. Fan, Z. Ma, L. H. Allen, and H. Morkoc, Appl. Phys. Lett., 64, 887 (1994).
- 6. H. Lee, D. B. Oberman, and J. S. Harris, Jr., Appl. Phys. Lett., 67, 1754 (1995).
- W. Pletschen, R. Niegurch, and K. H. Bachem, Proc. Symp. Wide Bandgap Semiconductors and Devices, Vol. 95-21 (Electorchemcal Society, Pennington, N. J., 1995), 241.
- 8. S. J. Pearton, C. R. Abernathy, and F. Ren, Appl. Phys. Lett., 64, 2294 (1994).
- L. Zhang, J. Ramer, J. Brown, K. Zhang, L.F. Lester, and S. D. Hersee, Appl. Phys. Lett., 68, 367 (1996).
- R. J. Shul, R. D. Briggs, S. J. Pearton, C. B. Vartuli, C. R. Abernathy, J. W. Lee, C.
   Constantine, and C. Barratt, Mat. Res. Soc. Symp. Proc., 449, 969 (1997).
- H. Cho, C. B. Vartuli, S. M. Donovan, C. R. Abernathy, S. J. Pearton, R. J. Shul, and
   C. Constantine, J. Vac. Sci. Technol. A, 16, 1631 (1998).

- 12. H. Cho, C. B. Vartuli, S. M. Donovan, K. D. Mackenzie, C. R. Abernathy, S. J. Pearton, R. J. Shul, and C. Constantine, J. Electron. Mat., 27, 166 (1998).
- S. Nakamura, in GaN and Related Materials, ed. S. J. Pearton (Gordon and Breach, N. Y. 1997).
- 14. O. Aktas, Z. Fan, S. N. Mohammad, A. Botcharev, and H. Morkoc, Appl. Phys. Lett., 69, 25 (1996).
- M. A. Khan, J. N. Kuznia, M. S. Shur, C. Eppens, J. Burm, and W. Schaff, Appl. Phys. Lett., 66, 1083 (1995).
- 16. Y. F. Wu, B. P. Keller, S. Keller, D. Kapolnek, S. D. Den Baars, and U. K. Mishra, IEEE Electron. Dev. Lett., 17, 455 (1996).
- 17. M. A. Khan, Q. Chen, M. S. Shur, B. T. McDermott, J. A. Higgins, J. Burm, W. Schaff, and L. F. Eastman, Electron. Lett., 32, 357 (1996).
- Y. T. Wu, S. Keller, P. Kozodoy, B. P. Keller, P. Parikh, D. Kapolnek, S. P. Denbaars, and U. K. Mishra, IEEE Electron. Dev. Lett., 18, 290 (1997).
- 19. C. B. Vartuli, S. J. Pearton, J. W. Lee, J. D. Mackenzie, C. R. Abernathy, and R. J. Shul, J. Vac. Sci. Technol. B, 15, 98 (1997).
- J. W. Lee, J. Hong, E. S. Lambers, and S. J. Pearton, J. Vac. Sci. Technol. B, 15,652
   (1997).
- 21. C. R. Abernathy, J. Vac. Sci. Technol. A, 11, 869 (1993).
- 22. C. R. Abernathy, Mat. Sci. Eng. Rep., R14, 203 (1995).
- 23. Y. B. Hahn and S. J. Pearton (to be published).
- 24. M. A. Lieberman and A. J. Lichtenberg, Principles of Plasma Discharges and Materials Processing, John Wiely & Sons Inc., N. Y. (1994).

- 25. J. W. Lee, J. Hong, and S. J. Pearton, Appl. Phys. Lett., 68, 847 (1996).
- Y. B. Hahn, D. C. Hays, S. M. Donovan, C. R. Abernathy, J. Han, R. J. Shul, H. Cho,
   K. B. Jung, and S. J. Pearton, J. Vac. Sci. Technol. A, submitted (1998).
- 27. CRC Handbook of Chemistry and Physics, 70<sup>th</sup> Ed., eds. R. C. Weast, D. R. Lide, M. J. Astle, and W. H. Beyer (CRC Press Inc., Boca Raron, FL, 1989).
- 28. J. Burm, K. Chu, W. J. Schaff, L. F. Eastman, M. A. Khan, Q. Chen, J. W. Yang, and M. S. Shur, IEEE Electron. Dev. Lett., 18, 141 (1997).
- 29. S. M. Donovan, K. D. MacKenzie, C. R. Abernathy, S. J. Pearton, F. Ren, K. Jones, and M. Cole, Appl. Phys. Lett., 70, 2592 (1997).
- 30. F. Ren, C. R. Abernathy, S. J. Pearton, and P. W. Wisk, Appl. Phys. Lett., 64, 1508 (1994).
- F. Ren, R. J. Shul, C. R. Abernathy, S. N. G. Chu, J. R. Lothian, and S. J. Pearton,
   Appl. Phys. Lett., 66, 1503 (1995).

## Figure Captions

Figure 1. Effect of plasma composition on etch rates in IBr/Ar (top) and ICl/Ar (middle) plasma chemistries, and dc bias and ion flux at the sheath (bottom).

Figure 2. Effect of ICP source power on etch rates in IBr/Ar (top) and ICl/Ar (middle) plasma chemistries, and dc bias and ion flux at the sheath (bottom).

Figure 3. Effect of rf chuck power on etch rates in IBr/Ar (top) and ICl/Ar (middle) plasma chemistries, and dc bias and ion flux at the sheath (bottom).

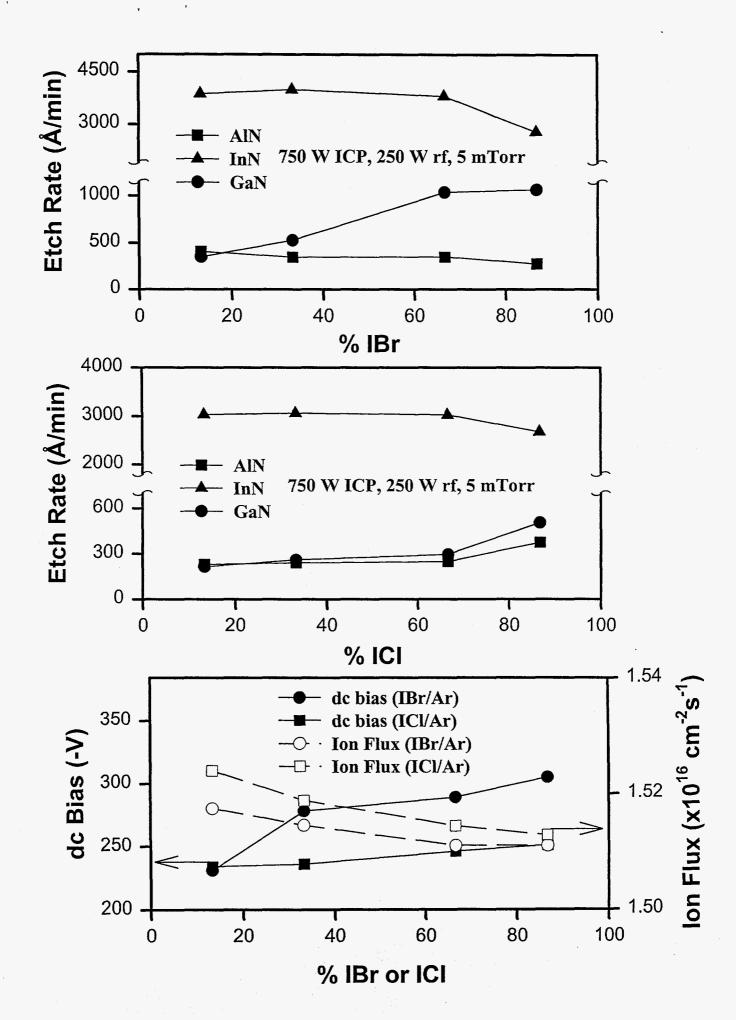
Figure 4. Effect of rf pressure on etch rates in ICl/Ar (top) plasma chemistry, and dc bias and ion flux at the sheath (bottom).

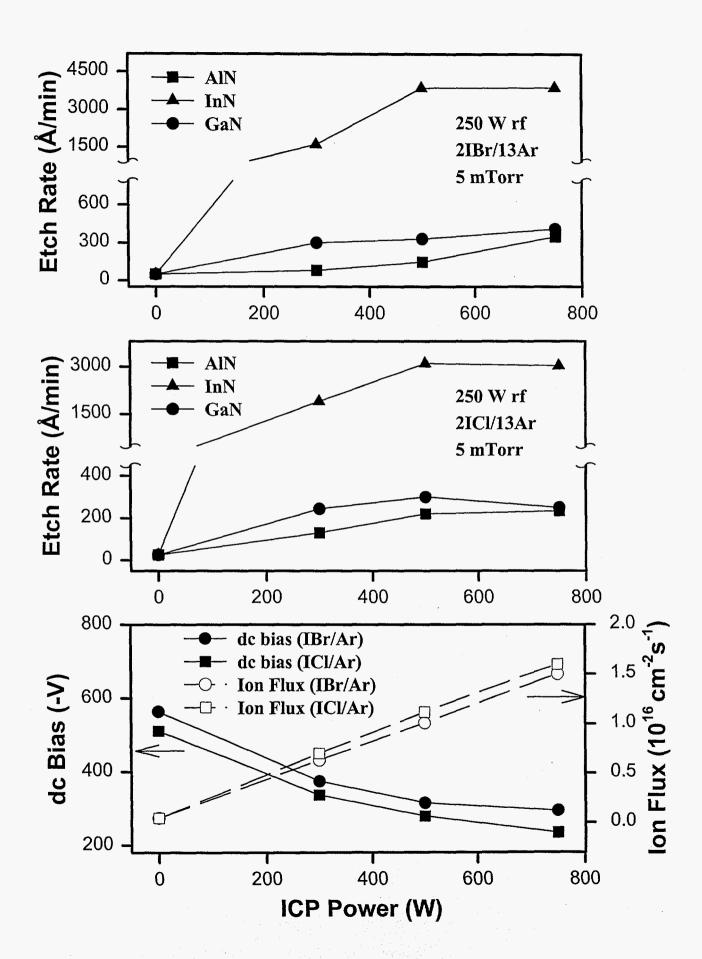
Figure 5. AFM scans for GaN etched in ICl/Ar (top) and IBr/Ar (bottom) plasmas.

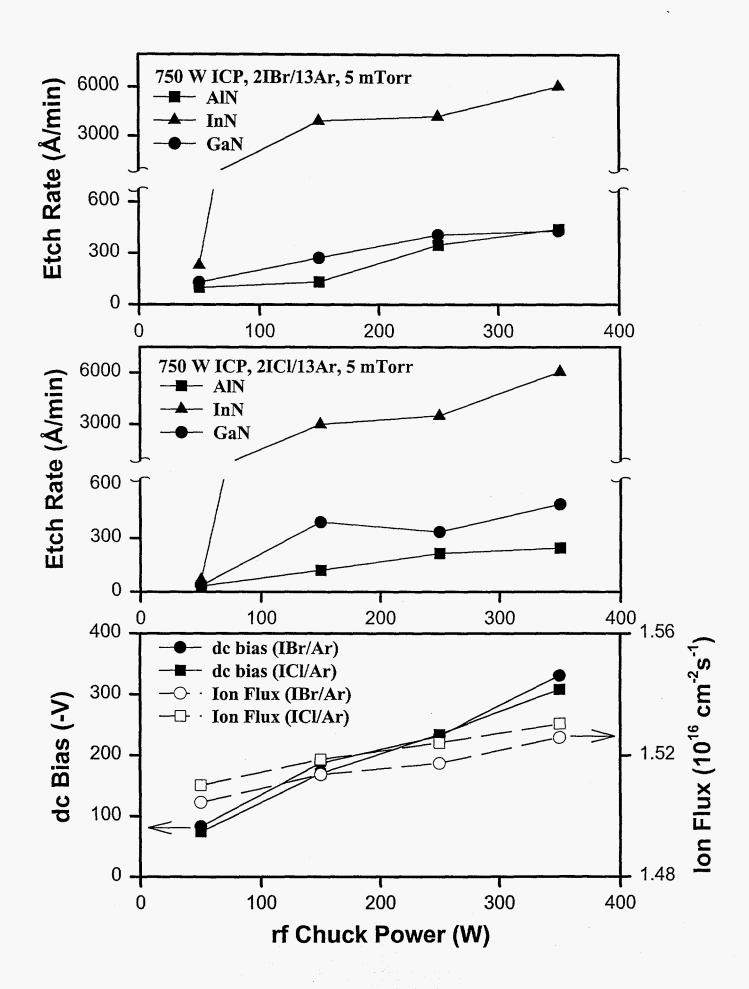
Figure 6. Effect of IBr (top) and ICl (bottom) concentrations on the selectivity for InN over GaN and AlN (750 W source power, 250 W rf chuck power, 5 mTorr).

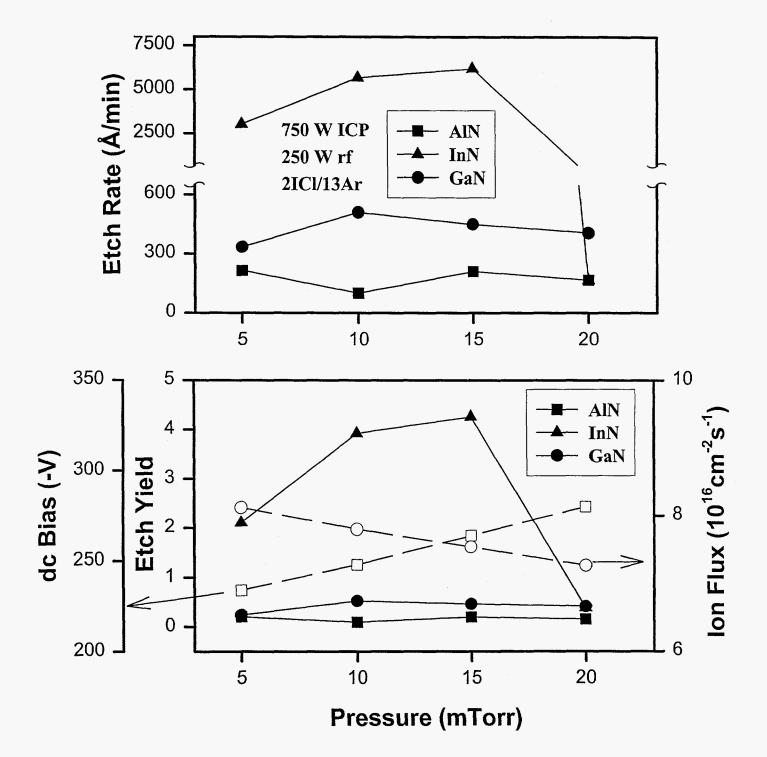
Figure 7. Effect of ICP source power on the selectivity for InN over GaN and AlN (750 W source power, 5 mTorr).

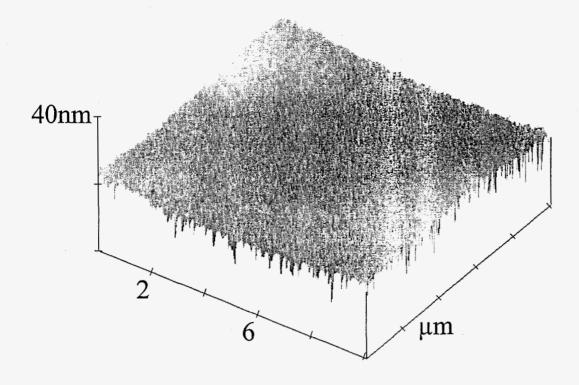
Figure 8. Effect of rf chuck power on the selectivity for InN over GaN and AlN (250 W rf chuck power, 5 mTorr).



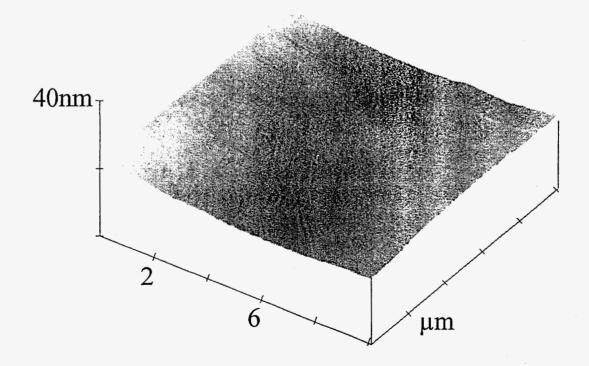








2ICl/13Ar RMS Roughness = 2.3nm



2IBr/13Ar RMS Roughness = 1.1nm

