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Citation for published version (APA):
Karouta, F., Jacobs, B., Vreugdewater, P., Melick, van, N. G. H., Schön, O., Protzmann, H., & Heuken, M. (1999). High etch rate and smooth morphology using a novel chemistry in reactive ion etching of GaN. *Electrochemical and Solid-State Letters*, *2*(5), 240-241. https://doi.org/10.1149/1.1390797

DOI:
10.1149/1.1390797

Document status and date:
Published: 01/01/1999

Document Version:
Publisher’s PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

• A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
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Electrochemical and Solid-State Letters, **2** (5) 240-241 (1999)
S1099-0062(98)11-049-0 CCC: $7.00 © The Electrochemical Society, Inc.

High Etch Rate and Smooth Morphology Using a Novel Chemistry in Reactive Ion Etching of GaN

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Reactive ion etching of GaN metallorganic vapor-phase epitaxy grown on a (0001) sapphire substrate has been investigated using various chemistries based on $SiCl_4$. The influence of gas combinations, gas flow, pressure, and radio-frequency (rf) power on etch rate and morphology was studied. Very high etch rates (160 nm/min) were obtained by adding SF_6 to $SiCl_4$:Ar. Smooth surfaces and high etch rates (±100 nm/min) were achieved at an rf power of 105 W (dc bias of -290 V).
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Manuscript submitted November 12, 1998; revised manuscript received January 15, 1999. Available electronically February 16, 1999.

GaN-based materials are commonly considered as potential candidates for light sources in the green and blue spectral range. The bandgap energy of AlGaInN varies between 1.95 and 6.2 eV. Significant research has been performed on high-brightness blue light emitting diodes (LEDs) and laser diodes (LDs). For example, the advantages of using LEDs over conventional electric incandescent lighting are faster response time, longer life, lower energy consumption, and higher luminescence efficiency. Also GaN, a wide-gap material, presents a potential use for high-temperature and high-power microelectronic applications. The high electron drift velocity, low thermal-generation rate, and high breakdown field make GaN and its related materials the most technologically interesting electronic materials for field-effect transistor applications. However, the intrinsic characteristics of this material system and, more specifically, its inertness, due to an atomic gallium-nitrogen bond energy of 8.92 eV/atom, make processing possible only with dry etching techniques.

Dry etching of GaN has been extensively investigated using a wide range of plasma-based machines and sources. Most authors use electron cyclotron resonance (ECR) plasma based on CH_4-H_2-Ar, Cl_2-H_2, or CH_4-H_2-Ar-Cl_2 [1-3] or chemically assisted ion beam etching (CAIBE).[4,5] Only a few reports describe dry etching of GaN using various chemistries in a more conventional reactive ion etching (RIE) and the number of papers dealing with a $SiCl_4$-based chemistry is even lower.[6]

Experimental

In this paper, we present the results of RIE etching of metallorganic vapor-phase epitaxy (MOVPE) grown GaN on a (0001) sapphire substrate. The experiments were performed in a conventional parallel plate and load-locked Oxford Plasmalab System 100. Various combinations have been investigated using $SiCl_4$:Ar, and eventually adding SF_6. The mask used in these experiments is a plasma-deposited SiN_x layer ~300 nm thick, annealed at 450°C for 10 min. Patterning the mask was carried out either by wet etching in a buffered HF solution or by using a dry etching process with SF_6:Ar [10:10 standard cubic centimeters per minute (sccm)] at 150 W and 40 mTorr. The etch rate of the SiN_x mask was ~200 nm/min.

The influence of gas flow, pressure, and radio-frequency (rf) power were investigated. The basic gas flow rate used was $SiCl_4$:Ar (10:10) sccm. Most gas combinations were investigated at three rf power values (70, 105, and 140 W) and at three pressure values (20, 40, and 60 mTorr). These parameters, combined together, determine the dc voltage which is related to the acceleration energy to the active species.

Results and Discussion

A few processes were run using the basic combination $SiCl_4$:Ar (10:10 sccm). Etch rates ranged from 11 to 35 nm/min with dc bias voltages of -225 to -376 V, respectively. These results are similar to those of Adesida et al.[6] Figure 1 shows a typical scanning electron microscopy (SEM) photograph of a smooth etched sample. Patterning of the SiN_x mask with a buffered HF solution caused the corrugation in the wall.

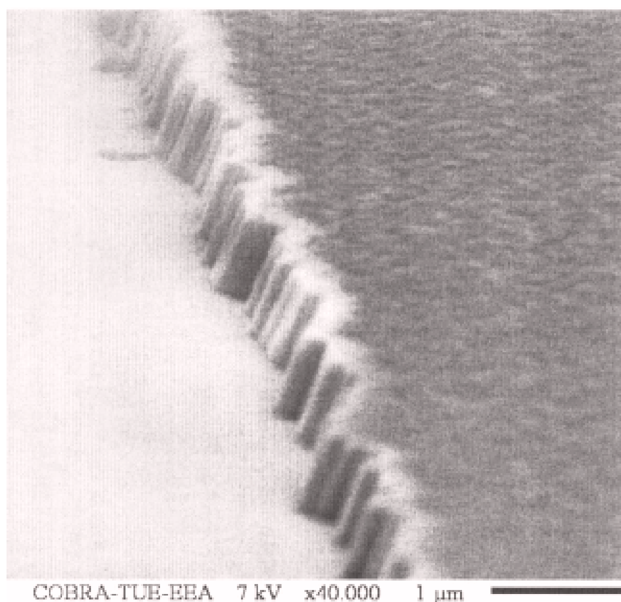

Figure 1. A SEM photograph of the etching process using $SiCl_4$:Ar (10:10 sccm) at 105 W, 20 mTorr, and a dc bias of 390 V.

Surprisingly, higher etch rates have been obtained when adding 2 sccm of SF_6. The presence of a fluorine component, in addition to the chlorine, appears to enhance the etching process drastically, making etch rates of more than 150 nm/min possible. A large number of processes based on this chemistry were carried out at the aforementioned rf powers and pressures. A general observation is that etch rates increase with increasing bias voltage or decreasing pressure. The highest etch rate, of 154 nm/min, was obtained using an rf power of 140 W at 40 mTorr resulting in a dc bias of about -360 V. Such a high etch rate is comparable to etch rates obtained in ECR plasmas using very corrosive gases such as Cl_2, HBr, or IBr. This etch rate is among the largest etch rate ever reported using RIE including those using corrosive gas mixtures. Increasing the SF_6 flow from 2 to 4 sccm did not produce any significant increase in the etch rate. At the same time, the dc bias was slightly increased. Figure 2 shows a steady increase of the etch rate as a function of the dc bias for the three gas combinations used.

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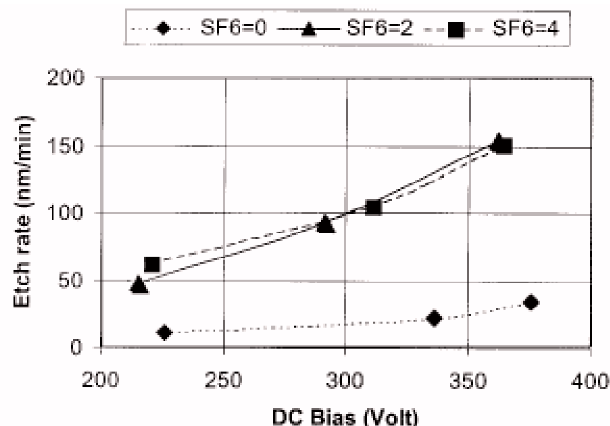

Figure 2. Etch rates vs. dc bias using various gas flows at 40 mTorr.

A systematic SEM investigation reveals that the etched samples at an rf power of 140 W have pillars of GaN distributed over the etched surface. Figure 3 shows a SEM photograph of such a sample where patterning the SiN_x mask was also performed in a buffered HF solution. However, the surface morphology appears to be very smooth when the etch processes were performed at 70 or 105 W. For instance, Fig. 4 shows a SEM photograph of an etched sample at 105 W and 40 mTorr using $SiCl_4$:Ar:SF_6 (10:10:2 sccm) resulting in an etch rate of 95 nm/min. Here, the SiN_x mask was dry etched. The latter process is actually used as a postgrowth polishing process to reduce surface roughness. The same process at 20 mTorr also results in a smooth surface, with a dc bias of 350 V and an etch rate of 100 nm/min. The gas combination of $SiCl_4$:Ar:SF_6 (10:10:2 sccm) etches the SiN_x mask at a rate of 20 nm/min at an rf power of 105 W. This means that the etch selectivity of GaN toward SiN_x is 5 to 1.

These results raise the question about the role of fluorine in this etching process. As the GaN layer is grown on the (0001) sapphire substrate, planes of Ga atoms and N atoms alternate along the [0001] axis. Any etch process should present the capacity of removing the two different atomic layers. Therefore, we think that fluorine plays an important role in the chemical process by reacting with the nitrogen of GaN and producing a very volatile component (NF_3), which has a boiling point of -129°C, while that of NCl_3 is given as <71°C.[7] Moreover, the fact that an increase in the SF_6 flow from 2 to 4 sccm had no influence on the etch rate would mean that a kind of saturation effect had been reached. Further investigations are undergoing to elucidate this issue and also to exclude the possibility of a catalytic role of fluorine. Note that Adesida et al.[6] have investigated two gas combinations: $SiCl_4$:Ar (10:10 sccm) and $SiCl_4$:SiF_4 (10:10 sccm) without noticing any change in the etch rate. In their experiments, the fluorine originating from SiF_4 did not increase the etch rate, while in our experiments, the fluorine from SF_6 resulted in an important increase of the etch rate. This issue can be explained using thermodynamic data[8,9] and, more explicitly, the dissociation enthalpies. The dissociation enthalpies are 145.4 and 93.6 kcal/mol for $SiF_4 \rightarrow SiF_3 + F$ and $SF_6 \rightarrow SF_5 + F$, respectively. This means that more energy is needed to dissociate an F atom from SiF_4 compared to what is needed to do the same with SF_6.

A few experiments were carried out to determine the amount of physical sputtering during the etching process. To this end, $SiCl_4$ was omitted from the gases, and, for instance, with Ar:SF_6 (10:2 sccm) and at 105 W and 40 mTorr, the GaN was etched at 11 nm/min. This is less than 12% of the etch rate in GaN when adding to the abovementioned gases 10 sccm of $SiCl_4$.

Conclusion

High etch rates (about 100 nm/min) and very smooth surfaces were achieved in the RIE of MOVPE-grown GaN on sapphire substrates using $SiCl_4$ based chemistry. The key result resides in adding SF_6 to the $SiCl_4$:Ar mixture.

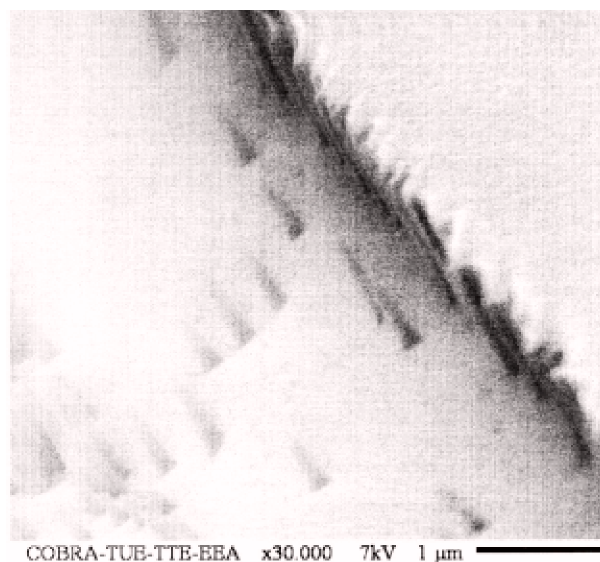

Figure 3. Residual pillars after etching at an rf power of 140 W using $SiCl_4$:Ar:SF_6 (10:10:2 sccm) at 40 mTorr.

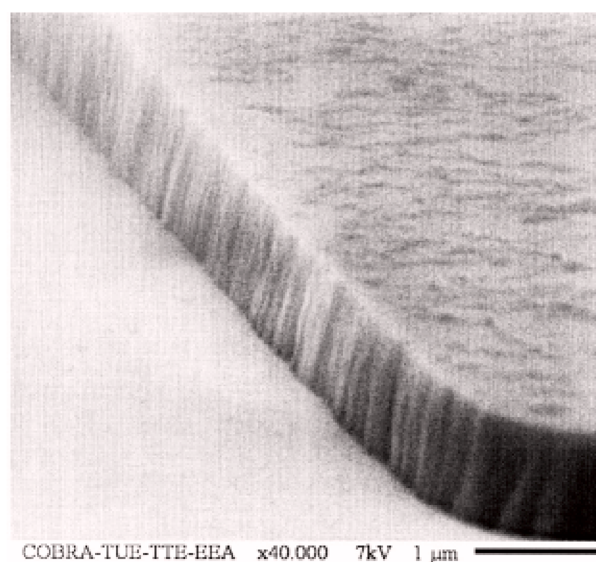

Figure 4. A very smooth surface of etched GaN using $SiCl_4$:Ar:SF_6 (10:10:2 sccm) at an rf power of 105 W and a pressure of 40 mTorr.

Acknowledgments

We would like to thank M. C. A. Donkersloot for fruitful discussions about thermodynamic data, T. G. v.d. Roer, L. M. F. Kaufmann for general discussions, and B. H. v. Roy for technical assistance.

Eindhoven University assisted in meeting the publication costs of this article.

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