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Fabrication of GaN mesa structures

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Abstract

We report on nickel based technology for the fabrication of GaN mesa structures. Ti/Ni ohmic contacts for n-doped GaN with contact resistivity $R_c \sim 2 \times 10^{-5} \Omega \times \text{cm}^2$ and Ni ohmic contacts for p-doped GaN with $R_c \sim 4 \times 10^{-2} \Omega \times \text{cm}^2$ were formed. Both types of contacts were used as masks for GaN reactive ion etching (RIE) in a $\text{CCl}_2\text{F}_2/\text{Ar}$ gas mixture. Maximum etch rates of ~ 40 nm/min were obtained. Mesa structures up to 3 μm in height were formed.

1. Introduction

GaN and related $\text{Al}(\text{In})_x\text{Ga}_{1-x}\text{N}$ solid solutions are wide band gap materials which are promising for a variety of applications such as laser diodes [1], light emitting diodes [2], microwave and high temperature devices [3] [4]. To realize the great potential of III-V nitrides and to follow the recent progress in their epitaxial growth, especially by MOCVD [5] and HVPE [6], it is necessary to develop the appropriate post-growth device fabrication technologies. Among other problems, the formation of ohmic contacts for n- and p-doped materials and RIE process are of the greatest importance. An additional requirement for these technological stages is their mutual compatibility in the process of device fabrication. The goal of this research was to find a metallization suitable for self-aligned technology so the ohmic contact could be used as a mask for GaN RIE. or so that the mask could be used as a contact. Various metals have already been used to form contacts to GaN. Ti, Ti/Al and TiN [7] [8] [9] [10] were utilized for n-GaN. TiN, Au, Au/Mg [8], Cr/Au [11] and Ni/Au [12] were used for p-GaN. We investigated Ni-based contacts, since Ni can be used as a mask for GaN plasma etching [13], and as a Schottky barrier to n-GaN [14]. In addition, Ni is usually used to form an ohmic contact to 6H-SiC, which is a highly suitable substrate material for the epitaxial growth of GaN [15] [16].

2. Ti/Ni ohmic contacts on n-doped GaN

2.1. Sample preparation and measurement techniques

1.5 μm thick GaN layers with a donor concentration of $\sim 1 \times 10^{18} \text{ cm}^{-3}$ were grown for this study on 6H-SiC substrates by MOCVD at Cree Research, Inc. To form the Ti/Ni contact, 50 nm thick Ti was deposited by e-beam sputtering on GaN at 350°C. After that, the Ti film was covered by a 250 nm thick Ni layer deposited at room temperature. After the contact pattern had been formed by photolithography, the samples were annealed in a nitrogen atmosphere at a pressure of 200 Torr and temperatures ranging from 300°C to 1150°C. The circular TLM (CTLM) technique was applied to measure the contact resistivity. The scheme of photomask used for CTLM is

shown in Figure 1. The diameter of the inner dot contact ($2r$) was $40\ \mu\text{m}$ and outer ring contact diameters ($2R$) varied from 100 to $500\ \mu\text{m}$. The composition of the metal both before and after annealing was measured by Auger electron spectroscopy (AES) and X-ray diffraction (XRD).

2.2. Experimental results

Annealing at temperatures lower than 950°C only resulted in Ti diffusion towards the surface of the sample (Figure 2). In addition to Ti diffusion, an interaction between Ni and GaN was observed after annealing at temperatures above 950°C . The formation of a Ga_2Ni_3 phase was observed by XRD. The content of Ga_2Ni_3 decreased sharply after annealing at temperatures above 1050°C . Linear current voltage (I-V) characteristics for the contact were observed after annealing at temperatures above 950°C . The contact with the lowest value of $R_c = 1.1 \times 10^{-5}\ \Omega\text{cm}^2$ was formed after annealing for 30 sec at 1040°C . No contact degradation was observed up to dc current densities of $30\ \text{kA/cm}^2$. Figure 1 presents the dependence of total resistance R_{tot} on $\ln(R/r)$ measured for this sample at room temperature. Although the formation of ohmic contacts at high temperature was caused mainly by the interaction of Ni with GaN, the Ti also played an important role in this process. The diffusion of Ti through the nickel cap layer was accompanied by the removal of oxygen from the metal-nitride interface as shown in Figure 2.

The change of GaN bulk electronic properties caused by the temperature treatments was monitored by measuring the sheet resistivity R_s . R_s did not increase substantially after annealing at temperatures below 1050°C . Further increase in the temperature led to a simultaneous increase in R_c and R_s ($R_c = 2 \times 10^{-5}\ \Omega\text{cm}^2$; $R_s = 85\ \Omega/\text{sq}$ after annealing for 140 sec at 1050°C and $R_c = 2 \times 10^{-4}\ \Omega\text{cm}^2$; $R_s = 140\ \Omega/\text{sq}$ after annealing for 30 sec at 1150°C).

3. RIE of GaN

Dry etching of III-V nitrides in chloride based plasmas by the RIE technique [17][18][19], the electron cyclotron resonance discharge technique [20][21][22], and chemically assisted ion beam etching [23][24] has been reported.

We examined three RIE masking materials for GaN using a gas mixture containing CCl_2F_2 [13]. They were: (1) $0.1\ \mu\text{m}$ -thick Ni deposited by e-beam sputtering on the substrate heated to 350°C ; (2) $0.2\ \mu\text{m}$ -thick Ni deposited by thermal evaporation on the substrate heated to 550°C ; and (3) the ohmic contact discussed above.

The plasma was generated by a radio frequency ($13.56\ \text{MHz}$) discharge into a stainless steel/quartz reactor using induction plasma excitation (H-mode) and a maximum rf power of $5\ \text{kW}$. The reactor has a diameter of $17\ \text{cm}$ and height $15\ \text{cm}$. A grounded electrode served as a substrate holder. The electrode was not cooled and its temperature during the etch was about 200°C . To avoid the deposition of a CF_x -like film, we used Ar as a carrier gas. Single crystal GaN and AlGaIn epitaxial layers up to $8\ \mu\text{m}$ in thickness grown on the (0001)_{Si} face of 6H-SiC [5][25] were used to study the etch process.

The etch rates of both Ni masks were very low ($<10\ \text{\AA}/\text{min}$) under the RIE conditions used. Ti/Ni contact also demonstrated good chemical stability sufficient for the formation of $2\ \mu\text{m}$ height mesa structures, in spite of the fact that the Ni layer was mixed with Ga through annealing. Furthermore, the as-deposited Ti/Ni metalization lifted off during the exposure in $\text{CCl}_2\text{F}_2/\text{Ar}$ plasma, since Ti was etched through the edge of pattern.

Etch rates of GaN ranging from $60\ \text{\AA}/\text{min}$ to $400\ \text{\AA}/\text{min}$ were obtained depending on the pressure and gas flow rate. The etch rates for n-GaN and p-GaN were similar and did not depend on the Al content in $\text{Al}_x\text{Ga}_{1-x}\text{N}$ up to $x = 0.15$. The maximum GaN etch rate was about $400\ \text{\AA}/\text{min}$ at a pressure of $10\ \text{mTorr}$ for a CCl_2F_2

$2:\text{Ar}$ ($12\ \text{sccm} : 12\ \text{sccm}$) gas mixture. The SEM micrograph shown in Figure 3 demonstrates a cleaved GaN/SiC sample with a strip-like mesa structure formed by RIE. The height of mesa structure is $3.2\ \mu\text{m}$. The Ni mask deposited by e-beam sputtering is not removed. The surface of the GaN layer is smooth and free from contamination after etching. Both walls of the mesa structure are inclined at an angle about 60 degrees with respect to the surface. Two kinds of microrelief are clearly seen on mesa-structure side wall: (1) vertical peculiarities caused by the mask edge effect, and (2) uniformly spread hollows caused by the nature of RIE process.

4. Ni mask as a contact to p-GaN

It was observed that the thermally evaporated Ni revealed almost linear I-V characteristic when was used as a masking material for RIE of p-GaN. To prove this fact, Ni was deposited on 0.5 μm thick p-GaN layer with N_a-N_d concentration of about 10^{18}cm^{-3} as was measured by Hg probe. After deposition, Ni was patterned for CTLM measurements. The contacts demonstrated symmetrical I-V characteristics with offset voltages less than 100 mV and linear I-V dependencies at higher voltages. Contacts were investigated at dc current densities up to 330 A/cm^2 . Contact resistance was estimated to be $R_c = 4 \times 10^{-2}\ \Omega\text{ cm}^2$. Figure 4 presents the dependence of R_{tot} on $\ln(R/r)$ measured at current density of 33 A/cm^2 and room temperature. In contrast, Ni deposited by e-beam sputtering did not reveal such a near linear I-V characteristic, even when annealed at temperature 550°C .

5. Conclusion

In summary, a Ni based technology suitable for the fabrication of GaN mesa structures was developed. The main achievements are: (1) Ti/Ni ohmic contacts with $R_c \sim 2 \times 10^{-5}\ \Omega\text{ cm}^2$ to n-GaN, (2) Ni contacts with $R_c \sim 4 \times 10^{-2}\ \Omega\text{ cm}^2$ to p-GaN, and (3) GaN RIE using gas mixture containing dichlor-difluoromethane; the above contacts serving as masks.

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References

- [1] S Nakamura, M Senoh, S Nagahama, N Iwasa, T Yamada, T Matsushita, H Kiyoku, Y Sugimoto, *Jpn. J. Appl. Phys.* **35**, L74-L76 (1996).
- [2] KS Kong, M Leonard, G Bulman, J Edmond, unpublished (1996).
- [3] H. Morkoc, S. Strite, G. B. Gao, M. E. Lin, B. Sverdlov, M. Burns, *J. Appl. Phys.* **76**, 1363-1398 (1994).
- [4] VE Chelnokov, KV Vassilevski, VA Dmitriev, in *Wide Bandgap Electronic Materials, NATO ASI series, 3. High Technology, vol.1*, Edited by: MA Prelas, P Gielisse, G Popovici, BV Spitsyn, T Stacy, (Kluwer Academic Publishers, Dordrecht, 1995) 453-461.
- [5] VA Dmitriev, KG Irvine, JA Edmond, et al., *Inst. Phys. Conf. Ser.* **141**, 497-502 (1995).
- [6] YuV Melnik, IP Nikitina, AS Zubrilov, AA Sitnikova, YuG Musikhin, VA Dmitriev, *Inst. Phys. Conf. Ser.* **142**, 863-866 (1996).
- [7] J Brown, J Ramer, M Banas, LF Lester, KJ Malloy, SD Hersee, unpublished (1996).
- [8] LL Smith, MD Bremser, EP Carlson, TW Weeks, Y Huang, MJ Kim, RW Carpenter, RF Davis, unpublished (1996).
- [9] Y Wu, W Jiang, B Keller, S Keller, D Kapolnek, S Denbaars, U Mishra, unpublished (1996).
- [10] M. E. Lin, Z. Ma, F. Y. Huang, Z. F. Fan, L. H. Allen, H. Morkoc, *Appl. Phys. Lett.* **64**, 1003-1005 (1994).
- [11] MC Yoo, KH Shim, JM Myoung, AT Ping, K Kim, I Adesida, unpublished (1996).
- [12] S Nakamura, in *Proc. of Int. Symposium on Blue Laser and Light Emitting Diodes*, Edited by: A Yoshikawa, K Kishino, M Kobayashi, T Yasuda, (Chiba University, Chiba, 1996) 119-123.
- [13] KV Vassilevski, VE Sizov, AI Babanin, YuV Melnik, AS Zubrilov, *Inst. Phys. Conf. Ser.* **142**, 1027-1030 (1996).
- [14] EV Kalinina, NI Kuznetsov, VA Dmitriev, KG Irvine, CH Carter, *J. Electron. Mater.* **25**, 831-834 (1996).

- [15] J Crofton, PG McMullin, JR Williams, MJ Bozack, *J. Appl. Phys.* **77**, 1317-1319 (1995).
- [16] MG Rastegaeva, AN Andreev, VE Chelnokov, KV Vassilevski, AI Babanin, IP Nikitina, AA Lavrent'ev, in *Int. Seminar Semiconductor Silicon Carbide and SiC-Based Devices, Book of Abstracts*, Edited by: VE Udaltsov, (Novgorod State University, Novgorod, 1995) 50-52.
- [17] M. E. Lin, Z. F. Fan, Z. Ma, L. H. Allen, H. Morkoc, *Appl. Phys. Lett.* **64**, 887-888 (1994).
- [18] AT Ping, I Adesida, MA Khan, et al., *Electron. Lett.* **30**, 1895-1897 (1994).
- [19] I. Adesida, A. Mahajan, E. Andideh, M. Asif Khan, D. T. Olsen, J. N. Kuznia, *Appl. Phys. Lett.* **63**, 2777-2779 (1993).
- [20] S. J. Pearton, C. R. Abernathy, F. Ren, J. R. Lothian, P. W. Wisk, A. Katz, *J. Vac. Sci. Technol. A* **11**, 1772-1775 (1993).
- [21] S. J. Pearton, C. R. Abernathy, F. Ren, *Appl. Phys. Lett.* **64**, 2294-2296 (1994).
- [22] S. J. Pearton, C. R. Abernathy, F. Ren, *Appl. Phys. Lett.* **64**, 3643-3645 (1994).
- [23] AT Ping, C Youtsey, I Adesida, et al., *J. Electron. Mater.* **24**, 229-234 (1995).
- [24] A. T. Ping, I. Adesida, M. Asif Khan, *Appl. Phys. Lett.* **67**, 1250-1252 (1995).
- [25] V. A. Dmitriev, K. Irvine, C. H. Carter, Jr., A. S. Zubrilov, D. V. Tsvetkov, *Appl. Phys. Lett.* **67**, 115-117 (1995).

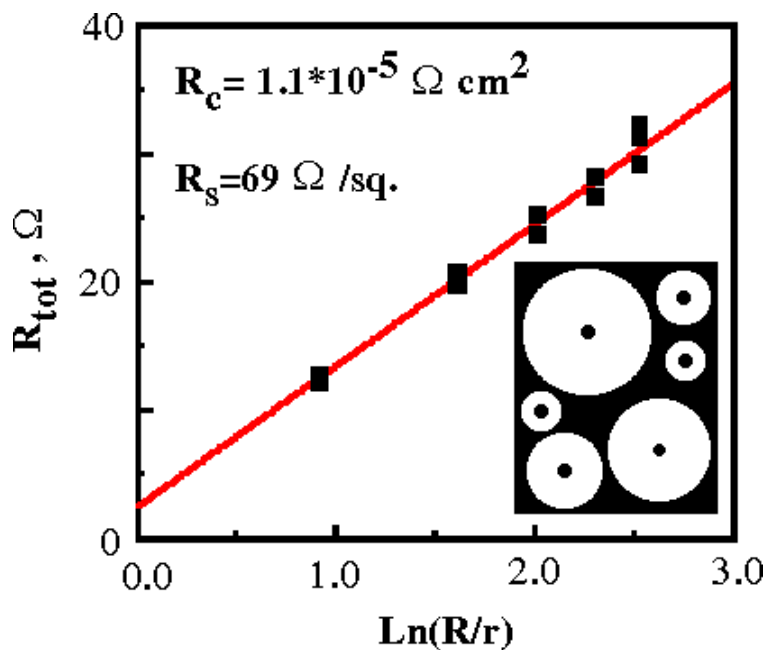


Figure 1. The R_{tot} dependence of $\ln(R/r)$ for Ti/Ni contact on n-GaN after annealing for 30 sec at 1040°C. The scheme of pattern used for contact resistivity measurements is shown in the inset. The black regions are covered by metal.

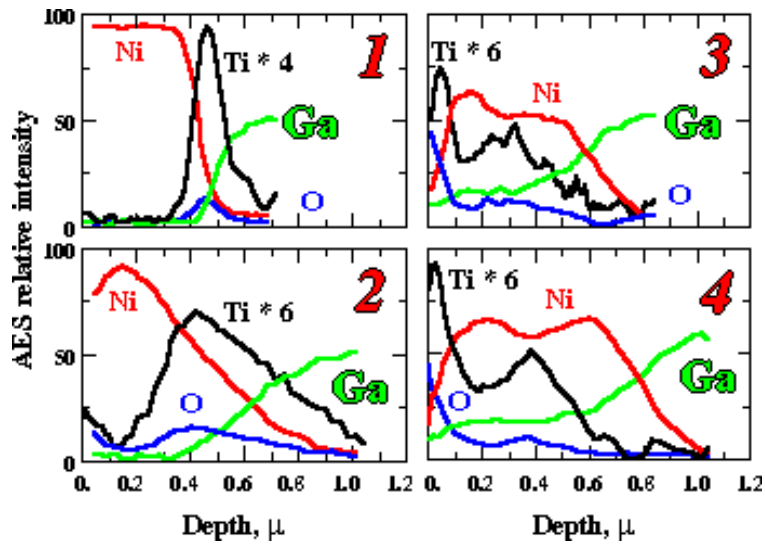


Figure 2. The AES profiles of Ti/Ni contacts: (1) as deposited; (2) annealed at 600°C for 300 sec; (3) annealed at 950°C for 140 sec; and (4) annealed at 1040°C for 140 sec.

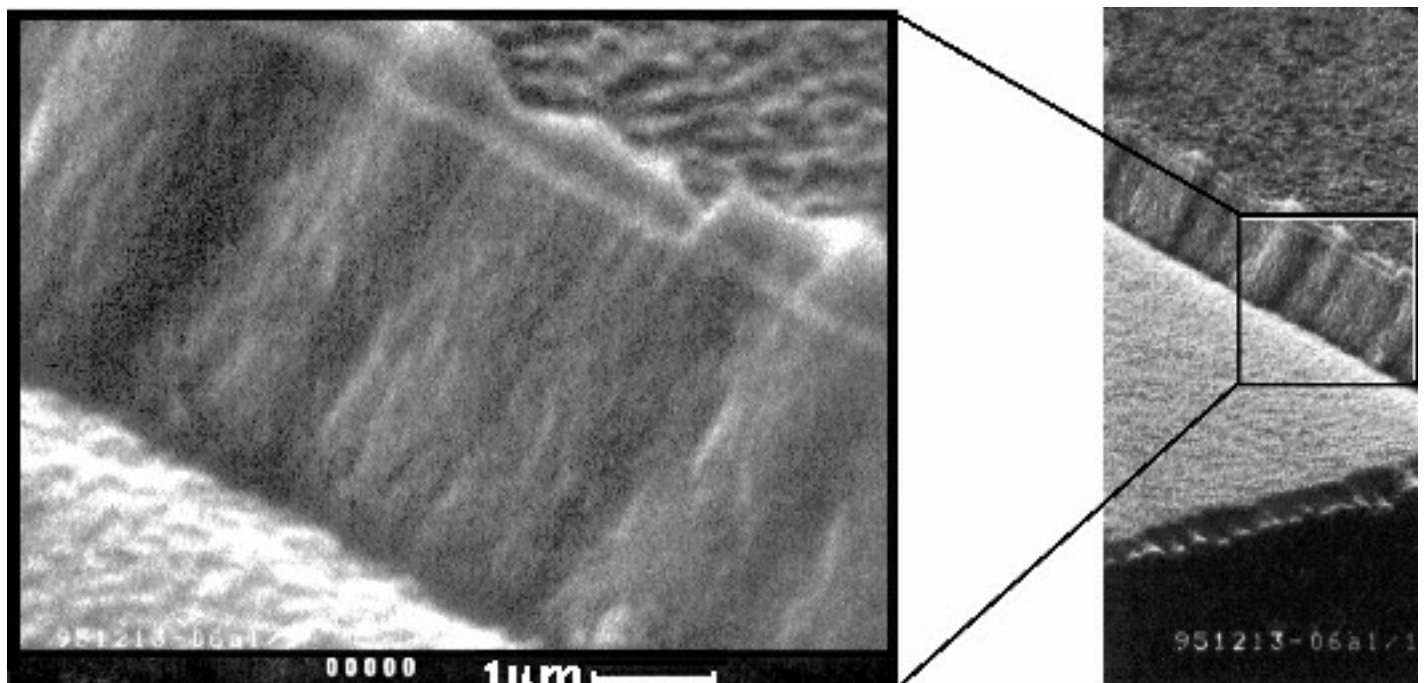


Figure 3. The SEM micrograph of a cleaved GaN/SiC sample with a stripe-like mesa structure formed by RIE in CCl_2F_2 mask, deposited by e-beam sputtering, is not removed.

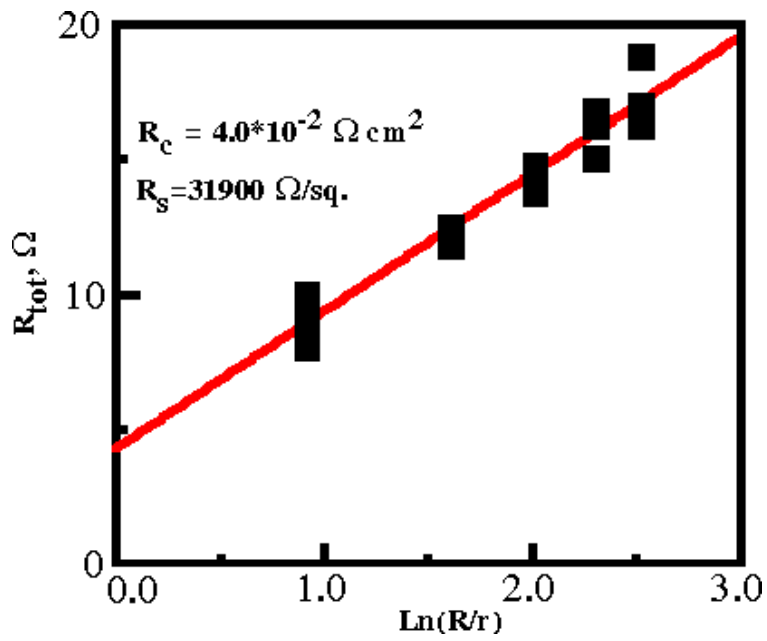


Figure 4. The R_{tot} dependence of $\ln(R/r)$ for Ni contact on p-GaN.

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