Combinatorial optimization of Ti/Al/Ti/Au ohmic contacts to n-GaN

A.V. Davydov[*1], **A. Motayed**[3], **W.J. Boettinger**[1], **R.S. Gates**[2], **Q. Z. Xue**[4], **H. C. Lee**[4], and **Y. K. Yoo**[4]

[1] Metallurgy Division, MSEL, NIST, 100 Bureau Drive, Gaithersburg, MD, USA
[2] Ceramic Division, MSEL, NIST, 100 Bureau Drive, Gaithersburg, MD, USA
[3] Electrical Engineering Dept., Howard University, 2300 6th St., Washington, DC, USA
[4] Intematix Corp., 351 Rheem Blvd., Moraga, CA, USA

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A combinatorial library of Ti/Al/Ti/Au metal contacts to n-type GaN thin films was characterized electrically and microstructurally. Various Ti/Al/Ti/Au thicknesses were deposited by combinatorial ion-beam sputtering (CIBS) on an n-GaN/sapphire substrate followed by rapid-thermal annealing (RTA) at 600°C to 900°C in argon for 30 s. The most Al-rich metallization in the library, Ti(20nm)/Al(170nm)/Ti(5nm)/Au(50nm), was found to have the smoothest surface morphology (rms roughness = 20 nm), while possessing an acceptably low contact resistivity (2.2×10^{-5} Ω·cm^2) after RTA at 750 °C. XRD analysis of this composition showed that, regardless of RTA temperature, the same two compounds, Al_3Ti and Al_2Au, were formed in the contact layer. For all other library elements, the interfacial phases in the metal layers were subject to continuous transformations as a function of RTA temperature. We surmise that these temperature-dependent transformations inflicted the excessive surface roughness in the contacts.

1 Introduction

The performance of GaN-based devices is often limited by the difficulty in making low-resistive, morphologically smooth and thermally stable ohmic contacts to both n- and p-type layers. The optimization of the metal contact scheme and the processing schedule involves extensive experimentation and is conducted mostly on a trial-and-error basis. In commonly used Ti/Al/Ti/Au metallization to both n-GaN and n-AlGaN layers, the overall composition, i.e., layer thickness ratio, is not yet optimised and varies from Al-rich [1–3] to Ti-rich [4] and to Au-rich [5, 6]. The thermal processing limits for enabling ohmic behaviour in the contacts also vary. Therefore, the methods of high-throughput experimental research appear suitable for optimizing electrical contacts in a multivariable space of metal compositions and processing temperatures. This paper develops a strategy to improve electrical and morphological characteristics of Ti/Al/Ti/Au ohmic contacts by optimizing metal layer thicknesses and rapid-thermal-annealing (RTA) temperatures using a combinatorial approach. To restrict the combinatorial space to be studied, we designed the optimum number of library compositions by first plotting the $Ti_xAl_yAu_z$ compositions of previously researched Ti/Al/Ti/Au contacts on the ternary Ti-Al-Au composition triangle (not shown here). We then chose the library matrix that both introduced new compositions and reproduced most previously reported metallizations. The combinatorial contact library was deposited and annealed incre-

[*] Corresponding author: e-mail: davydov@nist.gov, Phone: +01 301 975 4916, Fax: +01 301 975 4553

mentally in the 600 °C to 900 °C temperature interval, followed by electrical and microstructural characterization to identify the most promising ohmic contacts.

2 Experimental

A 35 mm x 35 mm square substrate for the metallization study was cut out from commercial GaN/c-sapphire wafer. The 7 μm thick Si-doped n-type GaN layer was grown by hydride-vapor-phase epitaxy at TDI, Inc**. Transport properties of the GaN were assessed by Hall measurements prior to metallization in several locations on the wafer. The GaN parameters were as follows: sheet resistance R_{sh} = 17 ± 1 Ω/square, carrier concentration n=(2.3 ± 0.5)×10^{18} cm^{-3} and mobility μ_n= 250 ± 50 $cm^2V^{-1}s^{-1}$.

The GaN surface was prepared for metallization by degreasing in boiling organic solvents followed by sequential etching in boiling $NH_4OH:H_2O_2:H_2O$ (1:1:5) mixture and in $HCl:H_2O_2:H_2O$=1:1:5 mixture for 5 min, and rinsing with de-ionized water after each step. After photo-lithographic processing that defined the circular transfer length method (c-TLM) pattern for measuring contact resistance, the substrate was dipped in $HF:HCl:H_2O$=1:1:10 solution for 10 s, then rinsed, blown dry and loaded into the combinatorial ion-beam sputtering system [7] for metal deposition. Ti, Al, Ti and Au layers were deposited sequentially at 0.08±0.02 nm/s onto the GaN surface at room temperature. A shutter system was used to deposit an array of six rectangular elements with approximate 5 mm x 35 mm dimensions separated from each other by 0.3±0.1 mm gaps. Metal layer deposition sequence and thicknesses in each of the six strips, "A" to "F", are summarized in Table 1.

Table 1 Metal layer thicknesses (nm) in the Ti/Al/Ti/Au contact library

Metal/Series	A	B	C	D	E	F
Ti	20	20	20	20	20	20
Al	70	120	145	170	85	25
Ti	115	60	30	5	15	75
Au	50	50	50	50	150	150

To limit the number of composition variables, the Ti layer adjacent to the GaN was 20 nm thick in all samples. The Au layer was 50 nm thick in the "A"–"D" structures and 150 nm thick in the "E" and "F" structures. Only the Al layer and middle Ti layer thicknesses varied in the test samples.

After metal deposition and a photo-resist lift-off that developed c-TLM test structures on a substrate, the sample was cut into seven 5 mm-wide strips in the direction orthogonal to the metal deposition direction. Six of seven strips were annealed at 600 °C, 650 °C, 700 °C, 750 °C, 800 °C and 900 °C in an RTA

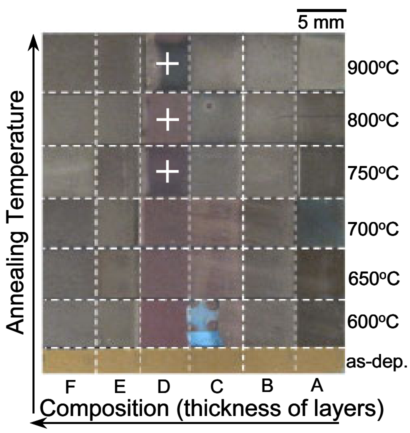

Fig. 1 Combinatorial library of Ti/Al/Ti/Au contacts with annealing temperatures indicated. The best library elements that satisfied both the "low contact resistivity" (ρ_c< 3×10^{-5} Ω·cm^2) and "smooth surface" (rms<40 nm) criteria are marked with "+". Notes: i) grid (white dashed lines) separates the library elements and is for an eye-guidance only; ii) left half of the "C-600°C" element was etched and used for Hall measurements.

** Certain commercial equipment, instruments, or material supplier are identified in this paper in order to specify the experimental procedure adequately. This does not imply endorsement by NIST furnace for 30 s in argon. The processed combinatorial array was composed of thirty-six 5 mm x 5 mm contact regions (plus one un-annealed reference strip) with each region having different metallization and thermal processing history as shown in the optical image in Fig. 1.

Contact resistance was measured with a 4-point probe method using the standard procedure for extracting specific contact resistivity as described in [8]. Gap spacing in the c-TLM test structures ranged from 2 µm to 30 µm. Comprehensive microstructural analysis was performed using a suite of characterization techniques: a) x-ray diffraction (XRD) in θ-2θ geometry to identify phases in the metal layers; b) optical microscopy and field-emission scanning electron microscopy to assess surface morphology; c) white-light interferomery to determine rms surface roughness (rms values for each sample were assessed over 100 µm x 100 µm scan area).

3 Results and discussion

The six experimental library compositions, with relationships to the literature, are: 1) Ti-rich composition "A" corresponds to that in the Ti/Al/Ti/Au contact from [4]; 2) (Al, Ti)-rich composition "B" is an average of those from [1–3]; 3) composition "C" (which is slightly richer in Al than "B") is new with no corresponding metallization from the literature; 4) Al reach composition "D" is also new; 5) Au-rich "E" composition is new but relates to metallization from [5]; and 6) Au-rich composition "F" (which is slightly richer in Ti than "E") is new but close to that from [6].

The maps of contact resistivity and surface roughness obtained for the library members are shown in Fig. 2 and Fig. 3, respectively. To select the "best" ohmic metallization in the thirty-six-element library in Fig. 1, the following two criteria were imposed: the contacts had to possess a) low contact resistivity with $\rho_c < 3\times10^{-5}$ Ω·cm^2 and b) smooth surface morphology with rms roughness below 40 nm. The relatively high limit for the "cut-off" rms value (40 nm) was dictated by the original roughness of the GaN surface (20±5 nm), which is typical for thick HVPE-grown GaN films. In general, both ρ_c and rms criteria are relative quantities and depend on electrical and structural properties of the specific GaN substrate and as-deposited metal film.

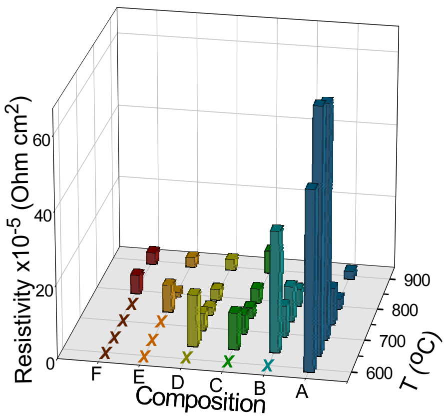

Fig. 2 Specific contact restivity as a function of metal composition and annealing temperature ("×" indicates non-ohmic behavior in the contacts).

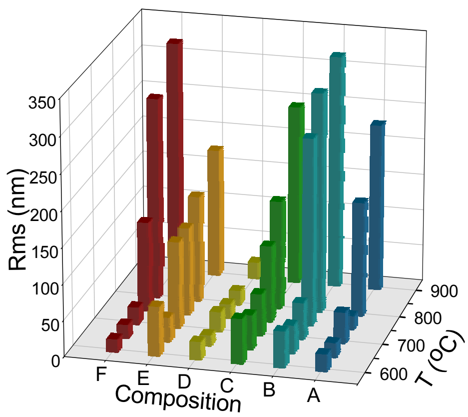

Fig. 3 Roughness (rms) as a function of metal composition and annealing temperature.

Following the above criteria, samples "D-750 °C", "D-800 °C", "D-900 °C" were singled out as optimal (marked with "+" in Fig. 1) with the "D-750 °C" metallization being the best (ρ_c=2.2×10^{-5} Ω·cm^2 and rms=20 nm). Overall, the "D"-series (highest Al-content) was the best in the library since its rms roughness was practically the same as on as-deposited contacts (rms values remained between 15 nm and 28 nm after RTA). The contact resistivities for this series were also low: the contacts became ohmic after RTA at 650 °C and remained below the ρ_c=3×10^{-5} Ω·cm^2 limit even after the 900 °C RTA. The other library series, "A", "B", "C", "E" and "F", were not acceptable primarily due to considerable surface roughening upon annealing above 700 °C–750 °C. The contact resistivities for these series were also relatively high, with both Al-low/Au-rich compositions, "E" and "F", being non-ohmic until high-temperature annealing (see Fig. 2).

XRD results revealed possible origin for the relative surface smoothness in the "D" series compared to the other series. In "D" contacts, only peaks from $Al_3Ti(m)$ and Al_2Au phases were observed after all annealing temperatures***, where $Al_3Ti(m)$ is a metastable $L1_2$ phase that is often observed in Al/Ti films [9] (importantly, the presence of Al_3Ti in the contact is considered to be critical for its ohmic behavior [10]). Unlike the "D" series, other library elements had different phases after different temperature anneals. Significant surface roughening correlated with certain phase changes in the metal contacts. For instance, in the "B" and "C" series, AlAuTi compound was formed after RTA at 600 °C. After RTA at T≥750 °C this phase disappeared and $Al_3Ti(m)$ phase appeared, and the rms values increased two-fold ("C-750 °C" vs. "C-700 °C", Fig. 3) and five-fold ("B-750 °C" vs. "B-700 °C", Fig. 3).

We also believe that the presence of liquid phase at RTA temperature in some samples caused significant surface roughening. For example, at 900 °C, the equilibrium composition "E" should consist of liquid and $AlAu_2Ti$ phases based on the estimated Al-Au-Ti phase diagram. Indeed, the surface of the "E-900°C" sample was covered with dendritic-like features upon cooling, which adversely affected the rms roughness of this sample (rms=177 nm).

4 Conclusions

A combinatorial approach enabled optimization of morphology and resistivity of commonly used Ti/Al/Ti/Au ohmic contacts to n-GaN. The most Al-rich metallization composition, Ti(20nm)/Al(170nm)/Ti(5nm)/Au(50nm), produced superior surface morphology with rms of 20 nm and low contact resistivity of 2.2×10^{-5} $\Omega\cdot cm^2$ after the 750 °C/30 s RTA anneal in argon. The temperature dependence of surface roughening correlated with phase transformations in the metal layers. Only D-series contacts remained fairly smooth after all RTA anneals; the other library elements suffered from increasing surface roughness with increasing annealing temperature. The superior morphology of contacts in the D-series is explained by the absence of phase transformations in the 600 °C to 900 °C temperature interval after the initial formation of Al_2Au and $Al_3Ti(m)$ phases.

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References

[1] D.F. Wang, S.W. Feng, C. Lu, A. Motayed, M. Jah, S.N. Mohammad, K.A. Jones, and L. Salamanca-Riba, J. Appl. Phys. **89**, 6214 (2001).
[2] L.F. Eastman, V. Tilak, J. Smart, B.M. Green, E.M. Chumbes, R. Dimitrov, H. Kim, O.S. Ambacher, N. Weimann, T. Prunty, M. Murphy, W.J. Schaff, and J.R. Shealy, IEEE Trans. Electron Dev. **48**, 479 (2001).
[3] J.A. Bardwell, G.I. Sproule, Y. Liu, H. Tang, J.B. Webb, J. Fraser, and P. Marshall, J. Vacuum Sci. Technol. B **20**, 1444 (2002).
[4] Y. Liu, J.A. Bardwell, S.P. McAlister, H. Tang, J.B. Webb, and T.W. MacElwee, phys. stat. sol. (a) **188**, 233 (2001).
[5] M.W. Fay, G. Moldovan, P.D. Brown, I. Harrison, J.C. Birbeck, B.T. Huhges, M.J. Uren, and T. Martin, J. Appl. Phys. **92**, 94 (2002).
[6] C.-Y. Hsu, W.-H. Lan, and Y.S. Wu, Appl. Phys. Lett. **83**, 2447 (2003).
[7] Y. K. Yoo and F. Tsui, MRS Bull. **27**, 316 (2002).
[8] G.S. Marlow and M.B. Das, Solid State Electron. **25**, 91 (1982).
[9] S. Wöhlert and R. Bormann, J. Appl. Phys. **85**, 825 (1999).
[10] C.M. Pelto, Y.A. Chang, Y. Chen, and R.S. Williams, Solid State Electron. **45**, 1597 (2001).

*** A very shallow and broad TiN 111 peak also appeared at ~37° 2θ after RTA at T≥750°C due to reaction with GaN.