Role of Graphene Interlayers in Mitigating Degradation of Ni/Au Ohmic Contact Morphology on p-type GaN

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ABSTRACT

We report an investigation of the effect of graphene interlayers in maintaining good surface morphology in low resistance Ni/Au Ohmic contacts to *p*-GaN ($\sim 10^{18} \text{ cm}^{-3}$) annealed at 600°C. Two different thin-film contact metallizations were compared, namely 20nm Ni/200nm Au with and without a graphene layer diffusion barrier placed between the metals. Raman spectroscopy measured at several spots indicated single layer graphene, but subsequent transmission electron microscopy indicated that more generally the graphene was several nm thick. The unannealed contacts showed specific contact resistances of $2.1 \times 10^{-5} \Omega\text{-cm}^2$ and $3.1 \times 10^{-4} \Omega\text{-cm}^2$ for Ni/Au and Ni/Graphene/Au, respectively. After rapid thermal annealing at 600 °C for 60 seconds in a flowing N₂ ambient, X-Ray Photoelectron Spectroscopy and cross-sectional Transmission Electron Microscopy showed the usual Ni/Au interchange occurred in samples

without graphene. By sharp contrast, insertion of graphene interlayers prevented the nickel and gold interchange up to 600°C and dramatically improved the morphological stability of the metallization stack. The results may be beneficial to optoelectronic devices in which reflectivity is critical.

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Introduction

GaN-based light emitting diodes (LEDs) have been extensively used for full color displays, light sources for traffic-light lamps, and headlamps for vehicles. The performance and reliability of GaN LEDs and laser diodes depends strongly on the quality of the p-Ohmic contact¹⁻¹⁰. For low hole concentration materials like p-GaN, high work function metals such as Ni, Pd, Cr and Pt with an overlayer of Au are required for low resistivity contacts^{4-7,11-20}. Ni-Au contacts are transparent on GaN, which is advantageous for optoelectronic applications^{1-3,6-8}. However, these contacts are often characterized by a specific contact resistance of the order of $10^{-3} \Omega \text{ cm}^{-2}$ when annealed near 500°C under N₂, which leads to a large voltage drop across the metal/GaN LED interface¹. A number of approaches to decreasing contact resistance have been reported, including annealing in air or O₂ ambients^{1,3,8}, use of Ni-based solid solution contacts⁷, various types of surface treatments involving etching, plasma exposure or cleaning steps^{1,8,9,14-16}, superlattice or tunnel junction layers¹, and catalytic metals that extract hydrogen from the p-GaN and increase near-surface hole concentration¹⁻³. Conventional Ni/Au contacts suffer from thermal degradation above 500 °C due to the outdiffusion and oxidation of Ni, and this leads to significant roughening of the morphology and a strong decrease in reflectivity of the contacts^{1,8}. For Ni/Au contacts on p-GaN, the Ni and Au layers trade locations upon annealing¹⁷. The Au layer becomes the interface to p-GaN and Ni becomes a high resistance incoherent top layer once

oxidized. Nickel is necessary in p-GaN contacts bilayer since Au depositions directly on GaN cause a non-uniform incoherent layer^{1,4}. Thus, Ni behaves as an adhesion layer between Au and p-GaN. The inter-diffusion mechanism, described by Ponce¹⁷, is due to two effects. Firstly, Ni-Au has a miscibility gap in the phase diagram for all compositions below 810°C. The second reason is due to the electronegativity difference of Au, Ni and Ga which are 2.4, 1.8 and 1.6 respectively. The largest difference in electronegativity is between Au and Ga, permitting Au to preferentially attach to Ga. Diffusion barriers have been utilized previous for Ni-Au on GaN devices to prevent the Au-Ni transformation, including TaN, TiN, ZrN, TiBr₂, Ir and W₂B¹⁸⁻²⁰. Methods to improve the thermal stability of the Ni/Au contacts to p-GaN are of significant interest.

Several studies have demonstrated the use of graphene as a solid state diffusion barriers^{21,22}. Nicolet²³ summarized the necessary materials properties needed for effective barriers, including thermodynamic stability between layers, strong adhesion, low contact resistance and resistance to mechanical and thermal stresses²³. For semiconductor applications, the barrier should be also be thermally stable and electrically conducting. Diffusion barriers in general should either be amorphous or have large grain sizes to reduce the densities of fast diffusion paths along grain boundaries and defects. Although graphene theoretically has physical properties that exceed many materials for diffusion barriers, it is a two surface material with no bulk²⁴⁻²⁸. The interfacial properties of graphene in contact with a diffusing species as well as the interfacial contact with the host substrate are the key parameters in determining whether pristine graphene can prevent diffusion across its basal plane. There are many reports of the lack of adhesion between gold and graphene. However, in some instances, graphene sheathed with different metals will actually increase the bonding energy of both interfaces. Gong *et al.*²⁷ showed an

Au/graphene/Ag stack increased the bonding energy by 26%. A Pt/graphene/Cu stack had a binding energy increase of 60% for both films. Calculations for bonding energies of Ni/graphene/Au are not available, but Kim *et al.*²⁹ reported that an Au/graphene/Ni stack on silicon did not delaminate after annealing for 1 minute at 600°C.

In this study we report an investigation of the use of graphene as a diffusion barrier in Ni/Au Ohmic contacts on p-GaN. The graphene is shown to significantly improve the morphology of annealed contacts.

Experimental

The GaN samples consisted of 1.0 μm Mg doped GaN layers grown by Metal Organic Chemical Vapor Deposition on sapphire substrates. The total Mg doping concentration obtained from Secondary Ion Mass Spectrometry measurements was $5 \times 10^{18} \text{ cm}^{-3}$, leading to a room temperature hole concentration of $\sim 4 \times 10^{17} \text{ cm}^{-3}$ obtained from Hall measurements. Prior to metal deposition, the GaN substrate was cleaned with acetone followed by IPA and a DI water rinse. 20nm of Ni followed by 200nm of Au were deposited with an e-beam system at deposition rates of 0.2nm/sec and 0.5 nm/sec respectively. The films were deposited without breaking vacuum. The samples were annealed in a Solaris 150 rapid thermal processing system at temperatures from 600-700°C for one minute in an N_2 atmosphere. X-Ray Photoelectron Spectroscopy (XPS) was performed to look for Ni on the surface samples. A Physical Electronics PHI 5701 LSci XPS with a monochromatic aluminum x-ray source (energy 1486.6 eV) with source power 300 W was used, with an analysis area of 2 mm x 0.8 mm and exit angle of 50°. The electron pass energy was 35.75 eV. The approximate escape depth ($3\lambda \sin \theta$) of the electrons was 80 Å. Charge compensation was performed using an electron flood gun on the floating stage. The charge compensation flood gun is often not sufficient at eliminating all

surface charge, and additional corrections must be performed. Using the known position of a small adventitious carbon (C-C) line in the C 1s spectra at 284.8 eV, charge correction was performed. A simple peak model involving a single C-C peak at 284.8eV was insufficient to fit the spectrum and additional peaks were added and the first was constrained to 1.5eV above the main peak and of equal FWHM. This higher binding energy peak is ascribed to alcohol (C-OH) and/or ester (C-O-C) functionality. A further high binding energy peak, attributed to O-C=O, was added with a position constraint of 3.7eV above the main peak. All peaks were constrained to a peak area ratio of 2:1:1. A Ni peak would imply that the Ni-Au layers had swapped positions. Cross-sectional Transmission Electron Microscopy (TEM) was used to confirm the layer reactions as a result of annealing.

For testing of the specific contact resistance we utilized the circular transmission line model (CLTM). For this model the total resistance between two contacts separated by a circular gap is given by¹

$$R_t = \frac{R_{sh}}{2\pi} \left[\ln \left(\frac{R}{r} \right) + L_t \left(\frac{1}{R} + \frac{1}{r} \right) \right]$$

where R_{sh} is the sheet resistance of the p-GaN substrate, R and r are the radius of the outer and inner circular contact and L_t is the transfer length. The circular transmission line pattern we used consisted of a radius of 40 microns with 5, 10 and 15 micron spacings. Samples with graphene were prepared following the same technique as mentioned above. A twenty nm initial layer of Ni was deposited by e-beam evaporation. The graphene used in these experiments was Trivial Transfer GrapheneTM obtained from ACS Materials. The Trivial Transfer GrapheneTM was transferred from a PMMA/Graphene/polymer stack by using water soluble interface between graphene and polymer. The water soluble tape releases the PMMA/graphene and is left floating on the water surface³⁰. With tweezers, the graphene is maneuvered to the center of the Ni coated

p-GaN sample. The sample was blow dried with N₂, ensuring no water is under the graphene. The graphene was then baked at 200°C for five minutes on a hot plate under N₂ in a glovebox and the PMMA removed in an acetone bath for one hour. To ensure the PMMA was completely removed, the sample was baked in the e-beam system at 250°C at <10⁻⁶ Torr for three hours and allowed to cool. We know from sheet resistance samples and separate XPS measurements on calibration samples that this process completely removed all of the PMMA. Without breaking vacuum, 200nm of Au was then deposited. Raman Spectroscopy measured at several locations on the Ni/graphene structure showed a 2D to G peak ratio of approximately 2, indicating single layer graphene. However, as will be seen below, subsequent TEM results over large areas showed that more generally the graphene is a couple of nm thick and not single layer in all areas of the contact structure.

Results and Discussion

Figure 1 shows current-voltage (I-V) characteristics as a function of contact spacing for CTLM samples of Ni/Au and Ni/Graphene/Au contacts annealed at 600 °C. While this is higher than the usual annealing temperature range for Ni/Au/p-GaN, we use it to test the effectiveness of graphene in preventing metal intermixing to these temperatures. The calculated sheet resistances of Ni/Au and Ni/Graphene/Au were 11.06 Ω/□ and 27.62 Ω/□, respectively, and the specific contact resistances were calculated to be 2.06 x 10⁻⁵ Ω-cm² and 3.07 x 10⁻⁴ Ω-cm² for Ni/Au and Ni/Graphene/Au, respectively. The sheet resistance of the p-GaN was 460±260 kΩ/□ as-deposited and ~350± 170 kΩ/□ after annealing at 600°C for both contact structures. The lower value after annealing may result from continued outdiffusion of hydrogen that is passivating Mg acceptors. The large spread in sheet resistance values is typical of much p-GaN¹. This shows

Ni/Au with the metal intermixing has larger conductivity than Ni/Graphene/Au. Both film structures still produce high quality Ohmic contacts to the p-GaN.

Figure 2 shows SEM images of the contacts after 600°C annealing. For samples with graphene, the morphology remains smooth and residue-free. For Ni/Au on p-GaN, the film morphology has significantly degraded relative to the Ni/graphene/Au contact. The benefit of using a transparent sapphire substrate in our experiments is that backside microscope analysis of the frontside films is obtainable. It was clear in visually analyzing the p-GaN from the top down view that the surface appeared grayer in color as the annealing temperature was increased. It was also clear from visually analyzing the wafer backside that the bottom metal layer is becoming more bright and yellow in color. This indicates that Ni-Au intermixing was occurring. We used atomic force microscopy to measure root-mean-square (RMS) values of surface roughness over $3 \times 3 \mu\text{m}^2$ areas, with as-deposited values of 0.45 nm and values after 600 °C annealing of 0.64 nm for Ni/graphene/Au and 17.8 nm for Ni/Au.

We used XPS analysis to verify these results. As shown in Figure 3 with the spectra of Ni/Au (top) or Ni/graphene/Au (bottom) on p-GaN annealed at 600°C, a Ni peak at 852 eV appears in the contact structure without graphene interlayers. Figure 4 shows an expanded view of this data, showing that for a 600°C anneal with graphene (ie. Ni/graphene/Au), XPS did not show any trace of Ni on the substrate surfaces. This shows that the usual layer interchange between Ni and Au did not occur when graphene was present as a diffusion barrier. Note also that the binding energy of Ni_{2p1} is around 869.7 eV to 874.7 eV and since it does not appear at that location, this would suggest that the Ni/Au contact has at least a significant component of NiO/Au contact.

Figure 5 (top) shows cross sectional TEM of Ni/Au on p-GaN after 600°C annealing that shows no sign of the Ni layer. There is no differentiation between Ni and Au films. This implies the Ni-Au intermixing process has occurred and verifies the XPS data. By contrast, the image in Figure 5 (bottom) of Ni/graphene/Au on p-GaN after 600°C annealing clearly shows the Ni layer is present and intact. The graphene prevents an interdiffusion between Ni and Au for anneal temperatures of 600°C. In as-deposited contacts, the Au layer usually exhibits large grains with while the Ni layer is polycrystalline with a grain size that is comparable to its thickness¹⁷. After thermal annealing, the Au layer is observed to transform to large single crystal platelets and the overlying Ni shows a reacted morphology¹⁷. The position of the Au and Ni layers is reversed during thermal annealing by solid-state interdiffusion¹⁷. An examination of the phase diagrams of the Ni/Au system shows there a miscibility gap at temperatures below 816°C³¹. In the temperature range 400–600°C, the solubility of Au in Ni is quite low, of order 5% and similarly for Ni in Au (roughly 12%). The Au layer after annealing remains continuous and incorporates Ni inclusions within its grain boundaries¹⁷. Note that the TEM shows that the graphene is several nm thick in most regions and this is certainly a more likely scenario than being able to use a uniform single atomic graphene layer on a device scale for diffusion barrier applications. For samples annealed at 700°C, the graphene interlayer did not prevent contact reaction and both samples with and without graphene showed switching of the Ni/Au layers and severely degraded morphology (RMS roughness > 15 nm in both cases).

Note that besides the present application of graphene as diffusion barrier for Ni/Au contacts on p-GaN, several other applications of graphene in connection with GaN have been proposed, e.g. as a transparent conductive electrode for GaN light emitting diodes³², and as a heat spreader for thermal management in high power AlGaIn/GaN transistors³³. Those authors showed that

thermal management of GaN transistors can be substantially improved via introduction of alternative heat-escaping channels implemented with graphene because of its high thermal conductivity³⁴. The graphene–graphite quilts were formed on top of AlGaIn/GaN transistors on SiC substrates and as a contact for AlGaIn/GaN heterostructures³⁵. These additional applications mean that continued study of integration of graphene in GaN-based devices is promising.

Summary and Conclusions

In conclusion, we have demonstrated that graphene can prevent the intermixing mechanism of Au/Ni on p-GaN substrates and therefore extend the thermal stability range of these contacts that are standard for GaN-based light-emitting diodes. We have verified through XPS measurements and STEM that the inter-diffusion of Ni and Au has been mitigated up to temperatures of 600°C. Circular Transmission Line Measurements show the Ni/graphene/Au contacts on p-GaN are Ohmic with a specific contact resistance measurements in the $10^{-4} \Omega\text{-cm}^2$ range and retain their smooth morphology to temperatures in excess of 600°C. The ability of the graphene interlayer to maintain the morphology of the Ni/Au contact is advantageous in applications where the reflectivity of the contact must be maintained.

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Figure Captions

Figure 1. I-V characteristics as a function of gap space for Ni/Au or Ni/graphene/Au contacts on p-GaN (top) and total resistance extracted from TLM analysis (bottom).

Figure 2. SEM image of Ni/Au contacts on p-GaN, showing surface morphology after 600°C annealing for 1 min. The region on the left does not have a graphene diffusion barrier layer between the Ni and Au and the contact morphology has degraded after this annealing cycle.

Figure 3. XPS spectrum of Ni/Au (top) or Ni/graphene/Au (bottom) on p-GaN annealed at 600°C. A Ni peak at 852 eV appears in the contact structure without graphene interlayers.

Figure 4. Expanded view of the spectra from Figure 3, showing the presence of Ni on the annealed Ni/Au contact (top) and absence of Ni on the surface of the Ni/graphene/Au (bottom).

Figure 5. (top) STEM image of Ni/Au on p-GaN after 600°C anneal that shows no sign of the Ni layer indicating the Ni-Au intermixing process has occurred. (bottom) STEM image of Ni/graphene/Au on p-GaN after 600°C anneal showing clearly the Ni layer is present and intact.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5