Characteristics of Ni/Au/Ni/Au ohmic contact in a p-AlGaN
/GaN semiconductor

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Abstract. The low-resistance NiAu/Ni/Au ohmic contact in p-type AlGaN/GaN semiconductor was carefully investigated by electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS) after two-step annealing at 550 and 750°C. It is shown that complicate double-direction diffusion and reaction occurred in the metal layer and underlying GaN layer. The four metal stacks of Ni/Au/Ni/Au turned into almost one layer and an intimate relationship established at NiAu/GaN boundary, which should play a primary role in ohmic contact to reduce the contact barrier. At the intimate interface, the metal layer close to the contact was enriched with Ga and Au, and the GaN upper layer was metalized by Au and Ni. Numerous Ni atoms were oxidized and formed dispersive NiO nanoclusters in the metal layer, which might have a hindering effect on upward migration of Ga atoms. Dislocations connected with the contact boundary absorbed interstitial atoms of Au or Ni may serve as channels for current carrier transportation. Thus, a low-resistance p-GaN ohmic contact can be obtained by the above combination of these microstructural characteristics.

1. Introduction

GaN-based III-nitride semiconductors, are increasingly attractive in the use of high performance optoelectronic devices such as LEDs and LDs, and also high frequency high power microelectronic applications such as HEMTs and MESFETs etc. However, the high work function and activation energy of AlGaN film always has a low carrier concentration, high resistivity and poor crystal quality, and results in a severe difficulty in ohmic contact formation [1]. Due to the wide bang gap of GaN (3.4 eV) and the low work function of the metals (4–5 eV), it is difficult to obtain a low metal/p-GaN barrier height. Another difficulty is related to the low free holes concentration in p-type doped GaN [2].

High work function metals such as Ni, Au, Pt and Pd have been used to form a variety of metallization schemes on p-GaN semiconductor, including Ni/Au [3], Ti/Pt/Au [4], Pt/Pd/Au [5], Cr/Ni/Au [6], Pd/Au [7], and Pd/Ni/Au [8]. The reported contact resistance is in the range of 10^-2–10^-6Ω·cm². Generally the metal electrode is fabricated by deposition, then a high temperature heat treatment in a specific atmosphere is performed to form alloyed metal layer and to improve the adhesion strength of metal-semiconductor, which contributes to the low contact resistivity. According reports by other researchers [9,10], Ni/Au/Ni/Au contact becomes ohmic at the temperature about 500°C or more.

In this study, a low resistance contact of Ni/Au layer stacks on p-type AlGaN/GaN is characterized after annealing by different methods. The intermixing of the metals in the alloyed contact layer together
with the diffusion of Ga, N and Al from the AlGaN/GaN semiconductor are characterized by X-ray photoelectron spectroscopy (XPS) depth profiles. The microstructure of the contact was observed with a high resolution transmission electron microscopy (HRTEM). The results show that an intimate relationship established between NiAu/GaN boundary which plays a primary role in the ohmic contact, and double diffusion enhance this effect.

2. Experimental details
The examined sample is a GaN multilayer of p-i-n heterostructure usually used as UV detector. Multilayer films of gallium nitride were grown on (0001)-oriented sapphire substrate in low-pressure metal-organic chemical vapor deposition (MOCVD) system. From the bottom up, the heterostructure consists of an AlN buffer layer, an 800 nm Si-doped n-Al0.65Ga0.35N, a 200 nm undoped Al0.3Ga0.7N, a 150 nm Mg-doped p-Al0.5Ga0.5N, a 20 nm p-AlGaN buffer layer and a 30 nm p-GaN. Subsequently, the grown sample was annealed at 750°C for 10 mins in N2 to activate Mg atoms. After cleaning process, standard technological processes are adopted to prepare the devices. Ni (20 nm)/ Au (20 nm)/ Ni (20 nm)/ Au (20 nm) contact as p-electrode and Ti (50 nm)/ Au (50 nm)/ Ti (30 nm)/ Au (30 nm) as n-electrode were deposited through E-beam evaporation, then annealed in the air at 550°C for 3 min and in high purity N2 ambient at 750°C for 30s respectively. More detail on the device fabrication process is given elsewhere [11]. Figure 1 shows the cross-sectional schematic of the examined sample.

![Figure 1. Cross-sectional schematic of Ni/Au/Ni/Au metal stacks on p-AlGaN/GaN.](image)

Analytical XPS investigation was carried out using a Thermo Scientific ESCALAB 250Xi Microprobe fitted with an Ar+ ion gun for rapid, high-resolution depth profiling. Monochromatic Al Kα X-rays were used for excitation. Sputter depth profiling using Ar+ ion gun has an energy of 3 keV and the scan size of the ion beam has a diameter of 200 μm. using a JEOL Transmission Electron Microscope JEM 3010 equipped with a Gatan Energy Dispersive X-ray Spectroscopy (EDS), operating at 200 kV. Cross-section TEM specimen was prepared by dimpling and Ar+ ion milling, until perforation of the specific area of interest has occurred. The composition of the metal layers as well as layers adjacent regions was determined by EDS.
3. Results and discussion

Figure 2 depicts the I-V behavior of the examined contact after two-step annealing. It is clear that the ohmic contact is obtained for the linear I-V characteristic.

![Figure 2. I-V characteristic of the contact after two-step annealing.](image)

Figure 3. The XPS chemical depth profile of annealed GaN/Ni/Au/Ni/Au contact.

The XPS chemical depth profiles of the Ni/Au/Ni/Au contact and partial GaN layer obtained from the examined sample are shown in figure 3. On the surface of the contact (etch level 0), only O, Ni and Au elements exist, which indicates that Ni reacted with O\textsubscript{2} in the air, and Ni must have penetrated Au film through defects to the surface of the Ni/Au layer structure and combined with oxygen. This phenomenon is similar to the report by Chen et al.\cite{12}. They suggested that Ni atoms tend to diffuse out to the upper surface through the grain boundaries of the as-deposited Au film. In fact, the as-deposited AuNi layers with an imperfect crystal structure, there are much more defects than grain boundaries, such as vacancies, dislocations and tiny voids. All these defects can serve as diffusion routes at elevated
temperature during annealing treatment. Meanwhile, the number of these defects would decrease with annealing temperature rise and time prolong, because diffusion is the process of atom rearranging. Generally, heat treatment at higher temperature for longer time, the diffusion speed is much faster and the diffusion flow is much higher and distance much longer. So the AuNi metal layers crystal becomes more perfect as an integrated alloy layer after two-step annealing. As to the Ni diffusion to metallic layer surface, Ho et al [3] believed that the driving force of the Ni atoms diffused through Au film is that Ni has a stronger affinity with O\textsubscript{2} than Au. XPS result in figure 4 shows that O decreased dramatically with etch level increasing, but it also indicates that O is a composition always in the metal layer, which means that some of the Ni atoms in metal layer are oxidized. The shape of O 1s peaks provides some interesting information, as shown in figure 4(a). The O 1s peak at 529.4–530.2 eV for NiO atoms and the O 1s peak at either 531.2 or 531.7eV for Ni(OH)\textsubscript{2}. It can be seen that the surface is almost covered by fine NiO grains and small amount of Ni(OH)\textsubscript{2} also appears on the surface. The presence of OH\textsuperscript{-} is because the sample was annealed in air that contained a little water vapor, and water vapor diffusing into NiO and Ni(OH)\textsubscript{2} formed [13]. Figure 4(b) shows the Ni 2p core-level XPS spectra, the 851.9 eV peak for the NiO structure and the Ni 2p peak at 854.1 eV for Ni(OH)\textsubscript{2}. It indicates that Ni(OH)\textsubscript{2} only appears on the metal surface. The Ni 2p stays steady in early 3 or 4 etch levels, it means a great many Ni atoms in AuNi metal layer were oxidized. Similar result is also reported by others and believed that the NiO formed close to the interface are among the critical mechanisms for the formation of low-resistance ohmic contact with Au/Ni/p-GaN contact [14]. Figure 4(c) depicts Au 4f core-level XPS spectra with binding energies from 82 eV to 89 eV. The binding energies of 83.86eV and 87.56eV correspond to Au 4f\textsubscript{7} and Au 4f\textsubscript{5} respectively [15,16]. The peak spectra of Au are stable, this indicates that the deposited Au indiffused through the Ni layer and contacted with p-GaN surface without reacting with Ni, O and GaN. XPS spectra in figure 4(c) also reveal that a small amount of Au remained in the upper layer and mixed with NiO and some Au atoms diffused into top GaN layers. Figure 4(d) depicts the Ga 2p core-level XPS spectra, and it shows part of Ga diffused into metal layer and alloyed underlying metal.
Figure 4. XPS core-level spectra for different elements, (a) O1s; (b) Ni2p; (c) Au 4f; (d) Ga 2p.

Figure 5. Cross-sectional TEM image of Ni/Au/Ni/Au contact.

TEM image in figure 5 shows a full view of the contact, which consists of two different features by contract. According to the XPS results in-depth, the layer on top of the contact is labeled as “NiO”, which consists of fine NiO particles with irregular thicknesses and shapes. The layer being in contact with GaN is much darker than the adjacent layers and it is denoted as “AuNi”. As shown in figure 5, the actual thickness for different layers in the GaN heterostructure, the measured data are slightly different
to the nominal thickness showing in figure 1.

A higher magnification image of the annealed contact is shown in figure 6, it reveals no layered structure can be seen in the metal layer. It means that during two-step annealing, heavy diffusion of Ni and Au atoms occurred, and the diffusion reconstructed the Ni/Au/Ni/Au layer stacks on GaN base. Furthermore, the lattice image of the metal layer in figure 7(a) clearly shows distortion and the oxidized Ni in the form of nanoclusters as chains. The lattice image distortion should result in the mismatch strain between the NiO nanoclusters and the surrounding NiAu matrix.

Figure 6. Magnified cross-sectional TEM image of Ni/Au/Ni/Au contact

Table 1 gives the EDS results of the main alloy elements across AuNi metallic contact, and the sites of EDS taken are marked in figure 6. It is obvious that Ga atoms diffused out into metal layer as a kind of alloy elements, and Au and Ni diffused into the top p-GaN layer close to the contact. According to table 1, point 2, 3, 4 and 5 are along the “AuNi” layer stacks from top to bottom. The content of Ni in metal layer decreases obviously in this direction, but the Au concentration in the metal layer is much uniform. The content of Ga increases rapidly with depth, which means some Ga atoms escaped from AlGaN/GaN and enriched at the bottom of the metal layer. So a uniform AuNi metal layer contact is formed with the intermixed of the NiAu layer stacks metal together with the out diffusion of Ga, N and Al from the AlGaN/GaN semiconductor. It also indicates that Au and Ni also present in GaN upper layer. Compared with Ni, much more Au atoms penetrated into the upper GaN layer. The interdiffusion behavior further alloying across the contact by the elements exchange, which is obviously conductive to form intimate contact, and might narrow the barrier height of metal/GaN to help the low ohmic contact formation either.

Figure 6 also shows the Au/GaN interfaces is sharp, with no intermediate phase present. It is clear that NiAu metal layer and GaN have an intimate relationship or well coherent boundary. In fact, as shown in figure 7(b), lattice distortion and dislocations can be seen at the boundary between NiAu and GaN. Figure 7(b) shows the atomic arrangement at the NiAu/GaN interface by HRTEM. Both crystal structures of NiAu and GaN show well intact in appearance. However, lattice distortion and edge dislocations can be seen in the metal layer contact close to the boundary, which should be mismatching dislocations caused by the mismatch plane spacings between them and this characteristic would reduce mismatching strain obviously at the boundary, so a slight undulation interface can be seen. Some edge dislocations in metal layer should generate during alloying process when different deposited fine
particles mixed together with different crystal orientations. A stacking fault and a threading dislocation can be seen in GaN nitride. It is obviously that the stacking fault. The threading dislocation, dislocation line outlined with dashed line, is roughly vertical to the planar of GaN layers, which usually run through the entire Gallium nitride film to the surface [17]. Such dislocations as well as vacancies have been suggested to promote the diffusion process during heat treatments [18, 19]. By the lattice distance measured in figure 8, The crystallographic orientation relationship is: (0001)$_{\text{GaN}}$ // (111)$_{\text{NiAu}}$, [110]$_{\text{GaN}}$ // [110]$_{\text{NiAu}}$. It is believed that this relationship is favorable for forming the ohmic contact [20, 21].

Figure 7. HRTEM images of atomic arrangement (a) in the metal layer and (b) at Au/GaN interface.

Figure 7 also indicates no boundary between the 20 nm p-GaN and 20nm p-AlGaN buffer layers. It means that during the two-step annealing, with the in diffusion of Au, Ni and diffusion of Ga, N and Al, the GaN is metalized and extended to the underlying layer AlGaN, so the both layers intermixed as one layer. A great amount Ga and N diffusion, high dense vacancies left at the region close to the contact, which might be partly taken by Au and Ni atoms penetrated into.

Table 1. The composition (at%) of the spots in figure 6 measured by EDS.

| Atom % | Ni | Au | Ga |
|--------|----|----|----|
| 1      | 50.8 | 46.3 | 2.9 |
| 2      | 46.0 | 52.6 | 1.4 |
| 3      | 39.2 | 54.7 | 6.1 |
| 4      | 29.4 | 55.1 | 15.5 |
| 5      | 19.4 | 53.3 | 27.3 |
| 6      | 11.9 | 32.7 | 55.5 |
| 7      | 0.0  | 0.0  | 100.0 |
Figure 8. HRTEM image of the NiAu/ GaN contact after two-step annealing.

In the past, many techniques were employed to obtain ohmic contact, such as intentional chemical cleaning to obtain a clear surface, plasma treatment to create surface defects [22,23]. However, annealing treatment after metal layers deposited is widely accepted as an effective method to achieve p-GaN ohmic contact. From above investigation, the low-resistant contact is obtained after two-step annealing with a series of interface reaction took place. Microstructure observation clear shows a well intimate relationship formed between AuNi metal with GaN layer after annealing. A periodic atom arrangement at the boundary is undoubted in favor for carrier transportation, so the well coherent relationship at the contact is the primary factor to create low-resistant contact. Diffusion is another marked phenomenon. Out diffusion Ga atoms as solid solution element in the bottom of the metal layer or even forming AuGa intermetallic compound [24], so high content of Au in metal layer close to the contact has an effect to solute Ga atom escaped from GaN layer [25]. Partial oxidize Ni atoms forming reticulate NiO in metal layer, which shows obviously effect to enrich Ga in the bottom metal layer. In other words, the dispersive or reticulate NiO nanoclusters in metal layer well hinder Ga diffusion further upwards. This maybe why the formation NiO is positive to establish an ohmic contact [15,26]. Ga diffusion left dense vacancies in the upper GaN layer and increased hole concentration in p-type GaN. It is assumed that a low-resistance ohmic contact to p-type GaN achieved with high-hole concentration in p-GaN can enhance the tunneling effect and reduce the contact resistance of metal-semiconductor interface [27]. Furthermore, Au and Ni diffusion inward to GaN as interstitial impurity dislocations connected with the contact boundary would attractive interstitial atoms and impurity elements, which may serve as channels for current carrier transportation [28]. These structural characteristics maybe obviously lower the barrier or serve as intermediate band gap, and result in low-resistance contact.

4. Conclusion
Generally, after two-step annealing at 550°C and 750°C, a good ohmic contact to p-type AlGaN/GaN contact is achieved. Microstructure observation shows that the four metal layer stacks of Ni/Au/Ni/Au turned into almost one layer and an intimate relationship established at NiAu/GaN boundary. The contact composition depth-profile investigated by XPS shows that high content of Au emerged in the intermediate and lower NiAu metal layers. Ni existed in the whole metal layer, but its amount reduced with depth and it was oxidized obviously. The dispersive nanoclusters of oxide NiO in metal layer show distinct hindering effect to Ga further diffusion into upper layer, which keeps the out diffusion Ga atoms
from GaN metalized with the bottom metal layer. In conclusion, well coherent relationship at NiAu/GaN boundary plays a primary role in ohmic contact achieved. The high-concentration Ga in the metal layer close to the contact, and Au and Ni that metalized the GaN upper layer may further reduce the contact barrier. Dislocations connected with the contact boundary absorbed interstitial atoms of Au and Ni may serve as channels for current carrier transportation. The combination of these characteristics leads to a low-resistance contact.

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