Microstructure and Electrical Properties of Low Temperature Processed Ohmic Contacts to p-Type GaN

Mi-Ran Park, Young-Joo Song, and Wayne A. Anderson

With Ni/Au and Pd/Au metal schemes and low temperature processing, we formed low resistance stable Ohmic contacts to p-type GaN. Our investigation was preceded by conventional cleaning, followed by treatment in boiling HNO_3:HCl (1:3). Metallization was by thermally evaporating 30 nm Ni/15 nm Au or 25 nm Pd/15 nm Au. After heat treatment in $O_2 + N_2$ at various temperatures, the contacts were subsequently cooled in liquid nitrogen. Cryogenic cooling following heat treatment at 600 °C decreased the specific contact resistance from 9.84×10^{-4} Ωcm^2 to 2.65×10^{-4} Ωcm^2 for the Ni/Au contacts, while this increased it from 1.80×10^{-4} Ωcm^2 to 3.34×10^{-4} Ωcm^2 for the Pd/Au contacts. The Ni/Au contacts showed slightly higher specific contact resistance than the Pd/Au contacts, although they were more stable than the Pd contacts. X-ray photoelectron spectroscopy depth profiling showed the Ni contacts to be NiO followed by Au at the interface for the Ni/Au contacts, whereas the Pd/Au contacts exhibited a Pd:Au solid solution. The contacts quenched in liquid nitrogen following sintering were much more uniform under atomic force microscopy examination and gave a 3 times lower contact resistance with the Ni/Au design. Current-voltage-temperature analysis revealed that conduction was predominantly by thermionic field emission.

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I. INTRODUCTION

Great interest has existed in III-nitride semiconductors since the successful development of GaN-based materials and operation of electronic and optoelectronic devices, such as blue and green light emitting diodes (LEDs) and laser diodes (LDs) [1]-[4]. The formation of stable reliable low resistance ohmic contacts to p-type GaN has been an obstacle to achieving good performance of those devices. For devices with large contact areas, such as LEDs and LDs, a specific contact resistance (ρ_c) of between 10^{-4} and 10^{-6} Ωcm^2 is considered acceptable and for devices with smaller contact areas, values of ρ_c of between 10^{-5} and 10^{-7} Ωcm^2 are necessary [5]. Bilayer metal schemes, such as Ni/Au and Pd/Au, have been studied by many groups [6]-[10]. These have been studied because of their stable electrical and thermal properties and their high work function, which is one criterion for forming low resistance Ohmic contacts to p-type materials.

This paper presents our investigation of the effects of cryogenic cooling after heat treatment on the formation of Ni/Au and Pd/Au contacts. We compare these effects with the effects on forming Ni/Au and Pd/Au contacts annealed in a combined O_2/N_2 gas ambient. High temperature annealing may degrade homogeneity, possibly caused by spiking of the metals between themselves or between the metal and the semiconductor due to the differences in thermodynamic properties of the materials. Annealing was conducted in an oxygen and nitrogen mixed gas ambient as reported by Y. Koide et al. [6]. It is speculated that this process removes hydrogen atoms contained in Mg-doped GaN epilayers.

Removing hydrogen atoms may result in an increase in the hole concentration and a decrease in the contact resistance. Annealing in a nitrogen gas ambient is believed to remove nitrogen vacancies which act as donors. We expected that cryogenic treatment would improve Ohmic behavior by reducing the disadvantages, such as spiking or balling-up of the metals and porousness in the metal layers, that result from the discrepancy in the thermodynamic properties of the materials during cooling of the heated samples. To test our expectation, we examined the changes in electrical and structural properties after cryogenic treatment.

II. EXPERIMENTAL

The metal contacts were made on GaN films grown by metalorganic chemical vapor deposition (MOCVD) on (0001) sapphire substrates. The GaN films consisted of Mg doped p-type GaN with a thickness of 2 μm on a 30 nm thick GaN buffer layer. The hole concentration of the p-GaN layer was 1.41×10^{17} cm^{-3} and the resistivity was 3.5 Ωcm. The samples were sequentially ultrasonically cleaned in trichloroethylene, acetone, and methanol, then rinsed in 18 MΩ de-ionized (DI) water. The cleaned samples were chemically etched in boiling aqua regia of HNO_3:HCl = 1:3 for 10 minutes to remove the native oxide and the contamination of the GaN surface as suggested by J.K. Kim et al. [11]. We then used a photolithographic process to form the pattern for the circular transmission line method (c-TLM). In this pattern, the radius of the inner circular contact was 400 μm and the spacings between the inner and outer circles ranged from 25 μm to 75 μm. Prior to deposition of the metal, the sample was again etched in a warm solution of HNO_3 and HCl. We used a warm solution, that is, non-boiling aqua regia: the solution was boiled and then cooled for 2 to 3 minutes since very hot solution ruins the photoresist. The Ni (30nm)/Au (15 nm) and the Pd (25 nm)/Au (15 nm) contacts were deposited by thermal evaporation and patterns were formed by lift-off. The contacts were then annealed at temperatures ranging from 400 to 700 °C for 10 minutes in a conventional furnace in an oxygen and nitrogen mixed gas ambient. In order to study the effects of cryogenic treatment, some samples were subsequently cooled by dipping them in liquid nitrogen after the heat treatment and then brought to room temperature in air. Sister samples were not quenched in liquid nitrogen.

The current versus voltage (I-V) curves were measured as deposited and in each interval between heat treatments. After each measurement of I-V curves, the specific contact resistance (ρ_c) was determined by using the c-TLM technique. The samples, with and without cryogenic treatment, showing the lowest contact resistance were examined by scanning electron microscopy (SEM) images from a model Hitachi S-4000, atomic force microscopy (AFM) images using a model Quesant Res/stg, and electron spectroscopy for chemical analysis (ESCA) depth profiles using a model Surface Science SSX-100 with an ion energy of 4.5 keV, a raster size of 2×2 mm, a sputter time per step of 1 to 2 min, and a x-ray spot size of 1000 μm. Cross-sectional transmission electron microscopy (X-TEM) using a model JEM-2010 Electron Microscope was also employed to study the microstructure of the metal/metal and metal/semiconductor interfaces.

III. RESULTS AND DISCUSSION

1. Electrical Properties

The samples chemically etched in boiling aqua regia of HNO_3:HCl = 1:3 for 10 minutes prior to deposition of the metals showed a much improved I-V behavior and a decreased specific contact resistance for both the Ni/Au and Pd/Au contacts but showed nonlinear I-V characteristics. The current versus voltage curves for the Ni/Au and Pd/Au contacts to p-GaN as a function of annealing temperature are shown in Figs. 1 and 2. The annealed contacts shown in those two figures were treated in an O_2/N_2 ambient. For the as-deposited case, both Ni/Au and Pd/Au contacts showed similar characteristics of non linear I-V with a leaky Schottky behavior. However, the Pd/Au contact revealed a lower specific contact resistance of 7 to 10×10^{-4} Ωcm^2 compared to 4 to 6×10^{-2} Ωcm^2 for the Ni/Au contact in the linear region, although the work function of the Ni (5.15 eV) was slightly higher than the Pd (5.12 eV). These characteristics agree with previous studies [7], [8]. The non linear I-V behaviors for Ni/Au contacts continued to improve by heat treatment up to a temperature of 600 °C (Fig. 1). For the Pd/Au shown in Fig. 2, the effect of annealing temperature on I-V characteristics was only a little different from the Ni/Au. Note that annealing at a temperature of 300 °C for the Pd/Au contact rapidly degraded the I-V curve more than the as-deposited contact, and continuing heat treatment until 600 °C improved the I-V behavior and decreased the contact resistance. Annealing at a temperature of 700 °C gave a much improved linear I-V curve and the lowest contact resistance. The reduction of the specific contact resistances to 9.84×10^{-4} Ωcm^2 for Ni/Au and 1.80×10^{-4} Ωcm^2 for Pd/Au were obtained by heat treatment at 600 °C and 700 °C, respectively. The lowering of the contact resistance and improving of the linearity may have come from a more intimate contact of the metal with the semiconductor or any new phases having a higher work function. Intimate contact leads to more current flow across the interface by breaking up some of the interfacial contamination between the metal and semiconductor. Possible

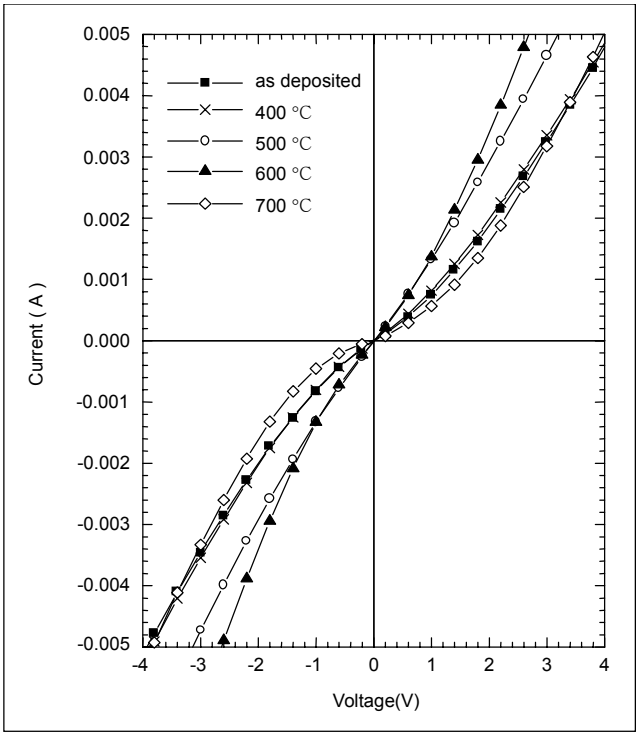


Fig. 1. Annealing temperature (in an O_2/N_2 ambient) dependence of I-V characteristics of Ni/Au contacts to p-GaN.

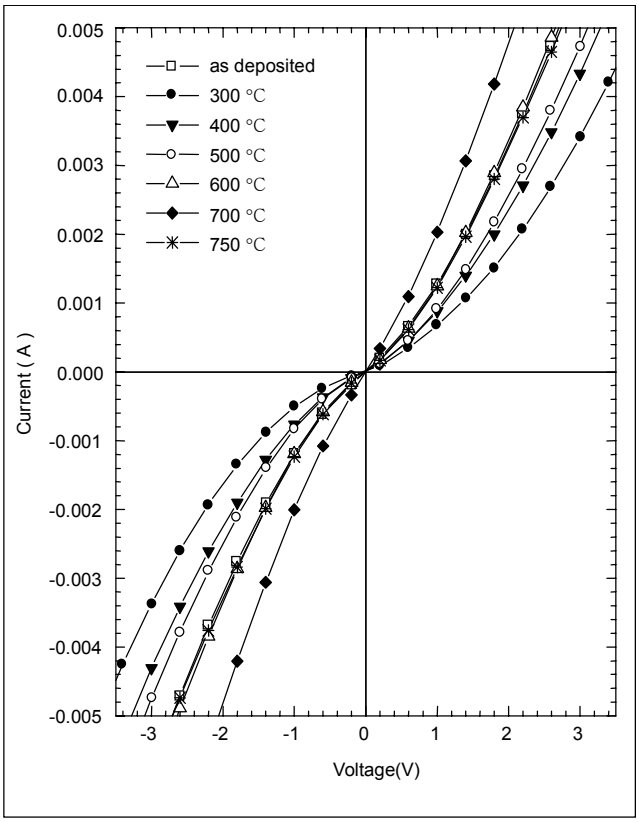


Fig. 2. Annealing temperature (in an O_2/N_2 ambient) dependence of I-V characteristics of Pd/Au contacts to p-GaN.

new compounds reduce the potential offset at the metal/semiconductor interface by forming a layer of the compound with a higher work function or causing a highly doped region. We believe that the lower contact resistance of the Pd/Au contact with the lower work function of Pd was due to a new phase with a higher work function, which will be discussed from the ESCA profiles.

In contrast, the effects of the cryogenic treatment on forming those two contacts were quite different (Fig. 3). With further treatment by cooling in liquid nitrogen after the annealing, the Ni/Au contact exhibited better linearity of I-V characteristics and a contact resistance of $2.65\times10^{-4}\ \Omega\ cm^2$ (Fig. 4). However, the Pd/Au contact showed behavior opposite to that of the Ni/Au contact in that the cryogenic treatment degraded the specific contact resistance value to $3.34\times10^{-4}\ \Omega cm^2$ (Fig. 4). The effect of cryogenic treatment on improving Ohmic behavior could have been caused by some of the semiconductor dissolving in the metal on heating and recrystallization with

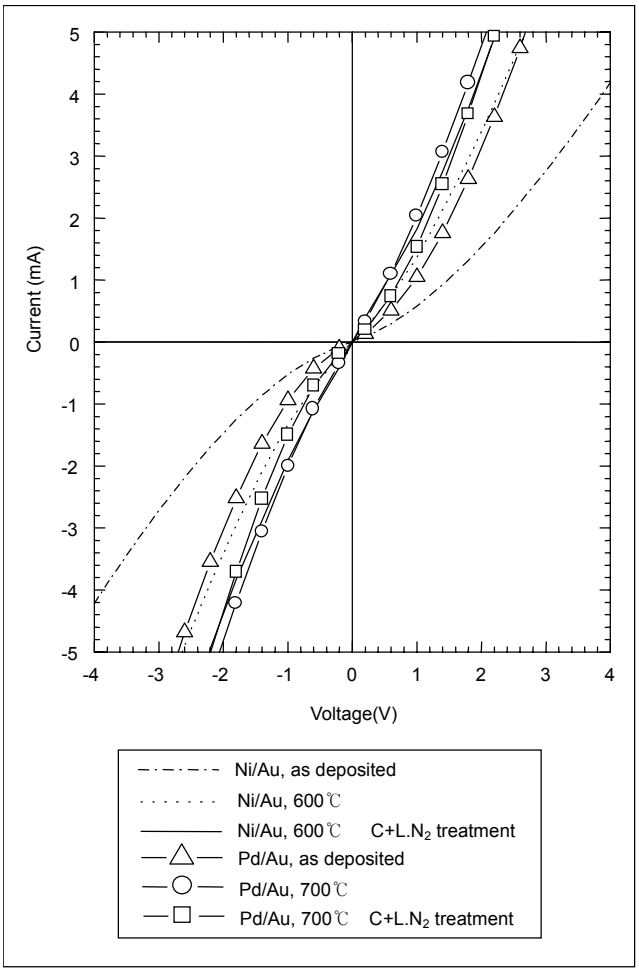


Fig. 3. Current versus voltage curves for the Ni/Au and the Pd/Au contacts to p-GaN as-deposited and annealed with and without cryogenic treatment in liquid nitrogen.

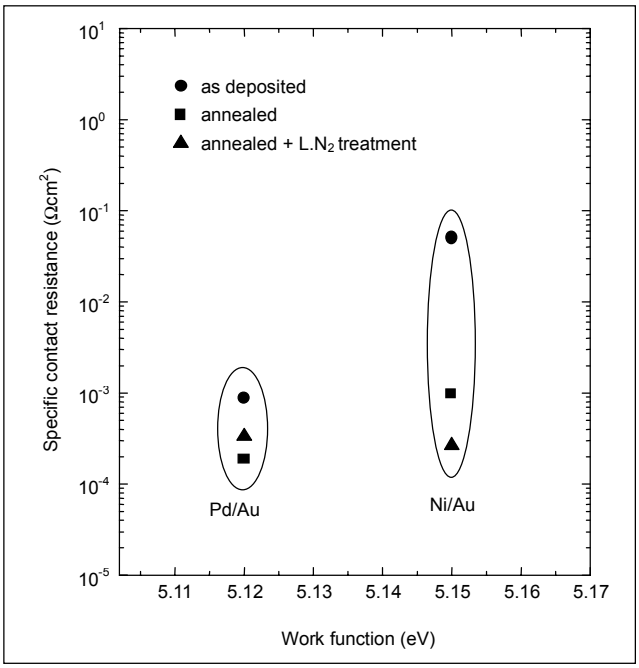


Fig. 4. Specific contact resistance for Ni/Au and Pd/Au contacts to p-GaN as a function of the work function of metal.

a high concentration of the electrically active element in solid solution on subsequent cooling [12]. It could also have been due to improved morphology as will be explained later. Therefore, the mechanisms and the reactions at the interface between the metal and the semiconductor for the Ni/Au contact and the Pd/Au contact would be different. Table 1 summarizes the results of contacts to p-type GaN.

Temperature dependent I-V curves were observed on the Ni/Au contact annealed at 600 °C for 10 min plus cryogenic treatment and Pd/Au contact annealed at 700 °C for 10 min plus cryogenic treatment as in Figs. 5 and 6, respectively. Those figures show log(I) vs. V and log(I) vs. V curves and they have similar behavior. The current of the Ni/Au contact is more stable over the whole voltage range than that of Pd/Au, although its increase with temperature shows more dependence on temperature than the Pd/Au contact (Figs. 5(a) and 6(a)). The slopes of the log(I) vs. log(V) curve (Figs. 5(b) and 6(b)) range from 1.005 to 1.124 for the Ni/Au contact and from 1.222 to 1.625 for the Pd/Au contact. This indicates that the Ni/Au contact has better Ohmic behavior and more thermal current than the Pd/Au contact. As a result, we would speculate that Ni, with a work function of 5.15 eV, formed a little lower barrier height with p-GaN than did Pd with a work function of 5.12 eV. Increasing the temperature of the sample may increase the carrier concentration and thin the barrier width, resulting in a thermionic emission dominated current.

2. Structural Properties

Figure 7 shows the ESCA depth profiles of Ni/Au contacts heated in O_2/N_2. The ESCA depth profile for the as-deposited sample of the Ni/Au contact shows abrupt sharp signals corresponding to the interfaces of the Au/Ni/GaN and all of the species (Au, Ni, Ga and N). The ESCA depth profile shows no evidence of interdiffusion from deposition alone. However, the ESCA profiles change in behavior with annealing and cryogenic treatment. In the ESCA profile of an annealed sample (Fig. 7(b)), Ni diffused to the surface of the contact and into the GaN. The O signal followed the Ni signal, which indicates an NiO phase. This is evident from chemical analyses, which showed core levels of Ni $2p^3/_2$ and NiO at the binding energies of 852.6 eV and 854.7 eV, respectively. On the other hand, the Au exchanged positions with the Niand formed an intimate contact with the GaN layer. These results agree with previous reports in which Ni diffused through the Au capping layer to the surface of the contact where it oxidized [7]. A similar behavior in ESCA depth profiles for the annealed plus

Table 1. Properties of the contacts to p-type GaN.

Semiconductor		Deposited Metal	Chemical Treatment	Heat Treatment In an O_2/N_2	Cryogenic Treatment (Liquid N_2)	Specific Contact Resistance (Ω cm^2)	Stability & Reproducibility
Type	Hole (cm^{-3}) Concentration						
p	1.41×10^{17}	Ni/Au	No	as deposited		8.92×10^{-1}	Good
			Boiling HNO_3:HCl 1:3	as deposited		3 to 9.5×10^{-2}	
				600 °C, 10 min		9.84×10^{-4}	
				600 °C, 10 min	Y	2.65×10^{-4}	
		Pd/Au	Boiling HNO_3:HCl 1:3	as deposited		5 to 8.5×10^{-4}	Moderate
				700 °C, 10 min		1.80×10^{-4}	
				700 °C, 10 min	Y	3.34×10^{-4}	

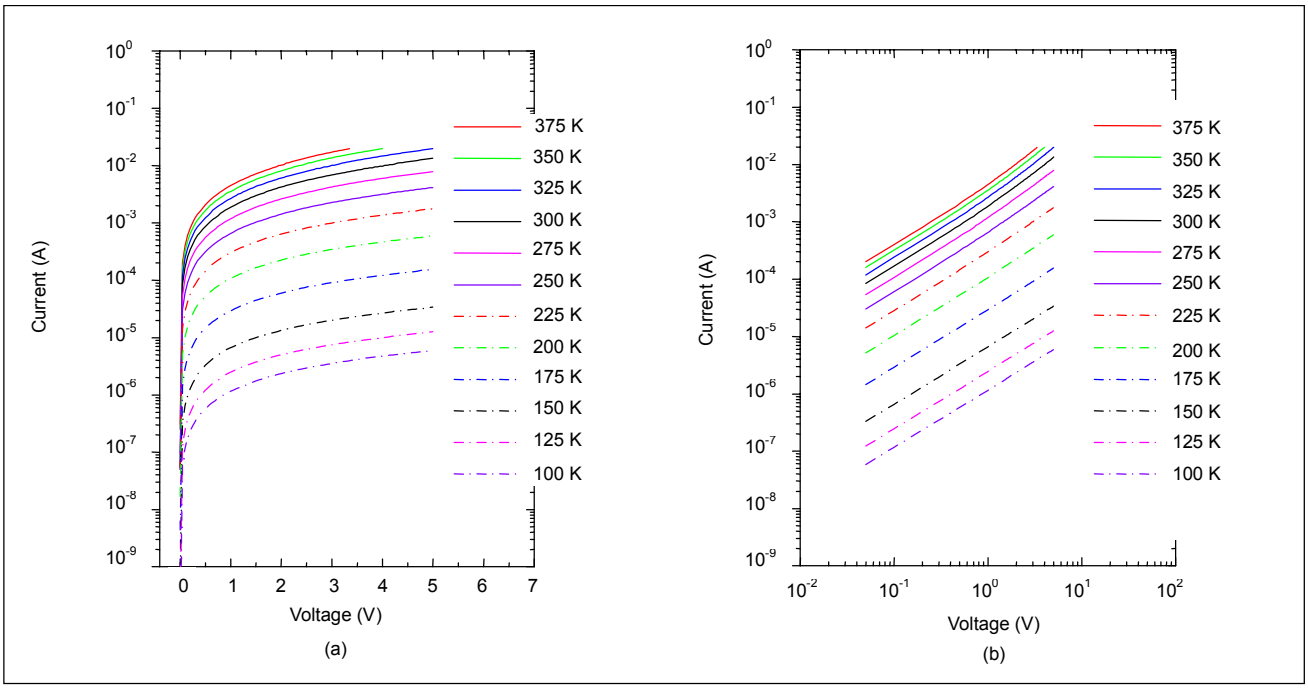


Fig. 5. Ni/Au contact to p-GaN annealed at 600 °C plus cryogenic treatment: (a) log(I) vs. V plot and (b) log(I) vs. log(V) plot at various temperatures.

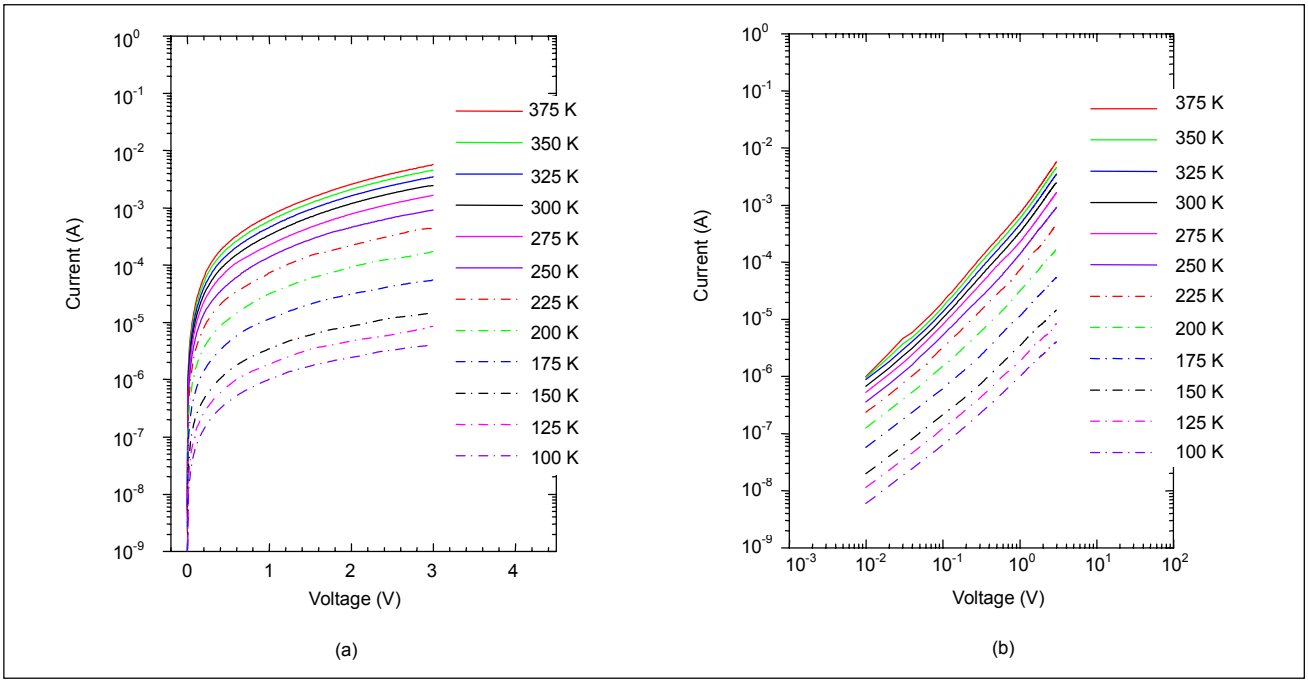


Fig. 6. Pd/Au contact to p-GaN annealed at 700 °C plus cryogenic treatment: (a) log(I) vs. V plot and (b) log(I) vs. log(V) plot at various temperatures.

cryogenically treated sample is shown in Fig. 7(c). Our study indicates that a direct contact to GaN would be a combination of NiO and Au or Au alone. Y. Koide et al., using x-ray diffraction (XRD), reported an NiO layer and no evidence of interaction between the metals and GaN for Ni/Au contacts annealed in an O_2/N_2 ambient [6]. On the other hand, H. S. Venugopalan et al. [13] found an NiGa phase for the annealed Ni/n-GaN scheme and J. T. Trexler et

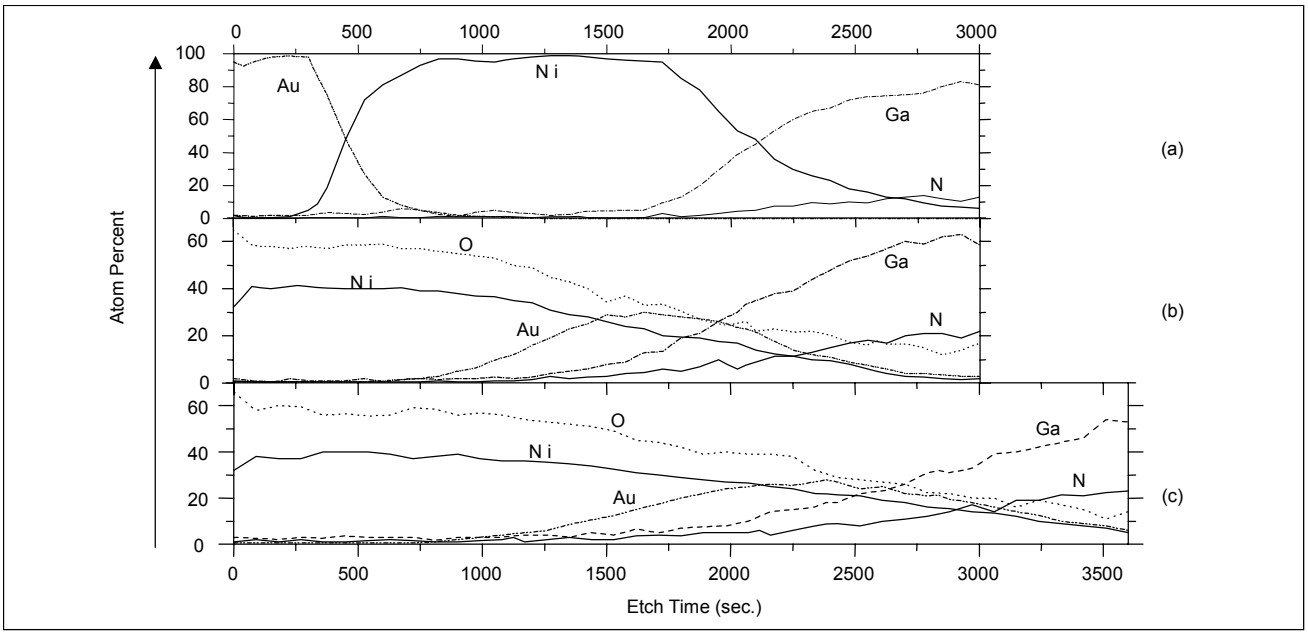


Fig. 7. ESCA depth profiles for the Au/Ni/p-GaN: (a) as-deposited, (b) annealed at 600 °C for 10 min in O_2/N_2, and (c) annealed and cryogenically treated.

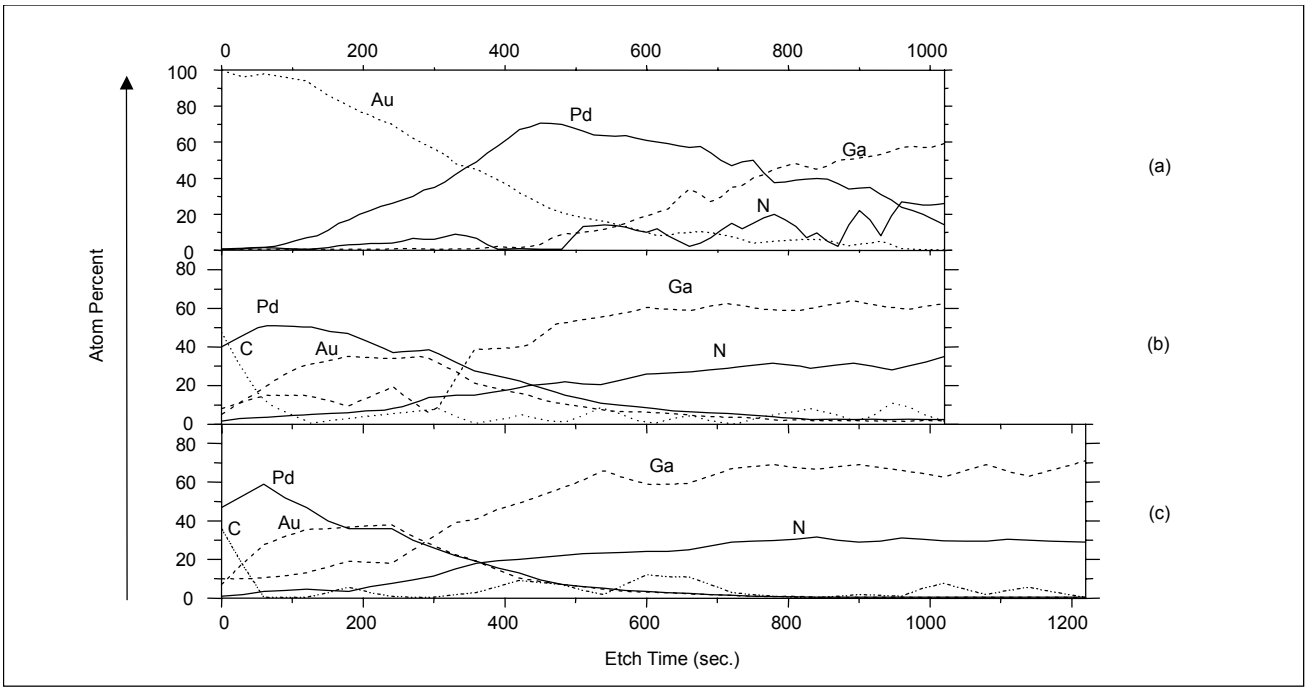


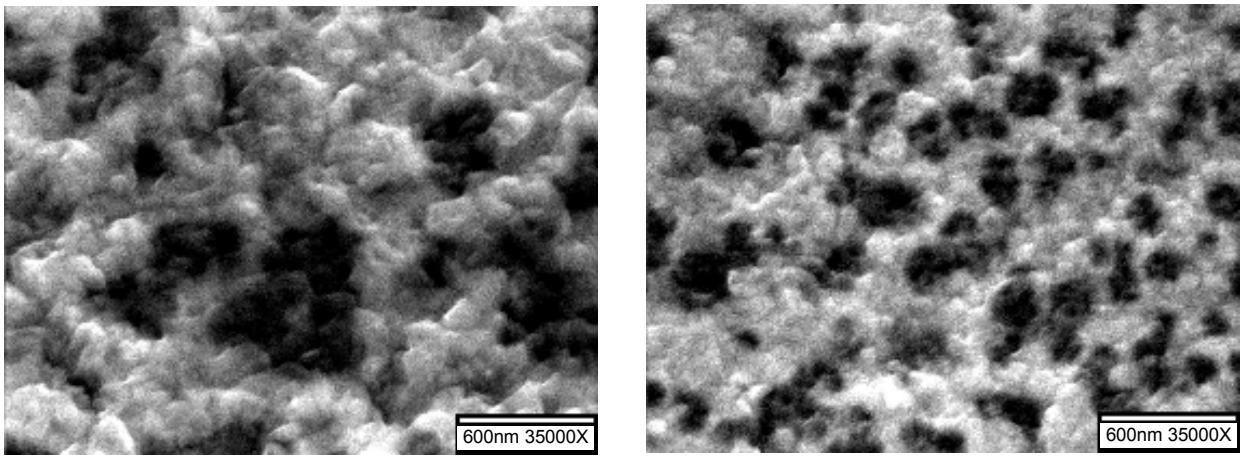
Fig. 8. ESCA depth profiles for the Au/Pd/p-GaN: (a) as-deposited, (b) annealed at 700 °C for 10 min in O_2/N_2, and (c) annealed and cryogenically treated.

al. [14] reported the possibility of an Ni-N solid solution for the annealed Ni/Au scheme.

The ESCA depth profiles of Pd/Au contacts are shown in Fig. 8. For both contacts, the sample that was annealed only and the sample that was cryogenically treated in addition to annealing, there is no evidence of formation of PdO but there is evidence of an Au:Pd solid solution, which is indicated by the Au signal followed by the Pd signal. This new phase (Au:Pd solid solution) was reported by other groups [6], [7]. The Ga and N signals tail out and extend out to the surface, which might indicate the decomposition of the GaN matrix. No significant difference was seen when we compared the samples only

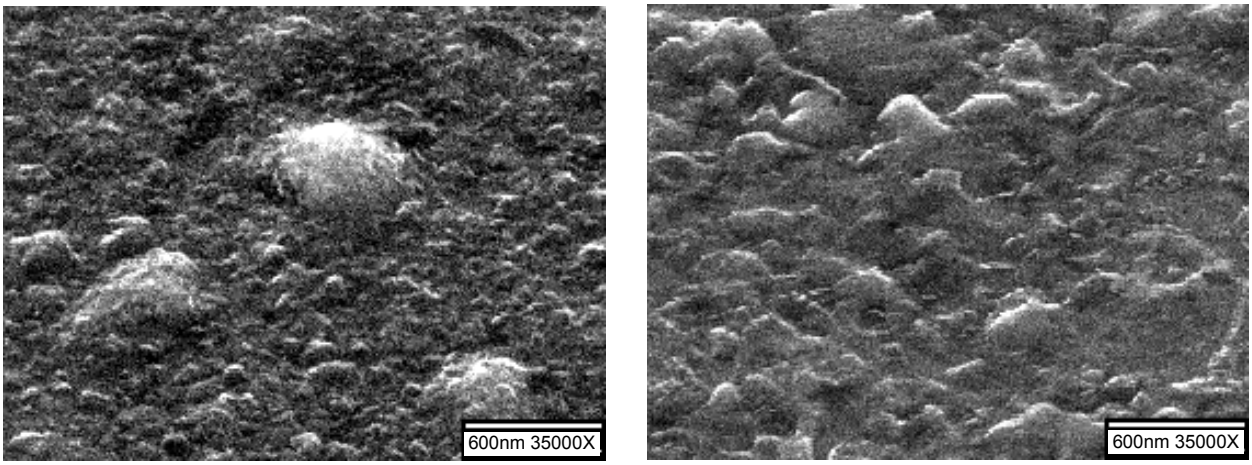
annealed with those annealed plus cryogenically treated.

We also examined the surface morphology of those contacts using SEM. The SEM images of the as-deposited samples for Ni/Au and Pd/Au contacts showed a smooth metal surface. However, Fig. 9 for the Ni/Au contact shows changes in the surface morphology of samples annealed (Fig. 9(a)) and cryogenically treated (Fig. 9(b)). The figures clearly show bright gray particles within a deep dark area. From a comparison with composition vs. depth in the ESCA profiles, we conclude that the bright gray particles should be NiO and the black areas should be pores in the Au layer or the mixing of Ni and the transition metal oxide (NiO). Figure 9(b) shows that the cryogenically treated sample has smaller dark areas (black) and a smoother surface of NiO (bright gray) than the sample in Fig. 9(a). For the Pd/Au contacts in Fig. 10, we can see a difference in particle size but not in roughness. The samples not cryogenically treated (Fig. 10(a)) have non-uniform larger particles. Contrary to this feature, the sample with liquid nitrogen cooling shows almost uniform particles. We believe that the "balling-up" of the NiO layer occurs during the heating process as a result of the compressive stress due to the difference of the thermal expansion coefficients between Ni (13.3×10^{-6} K^{-1}) [15], Au (14.1×10^{-6} K^{-1}) [15], Pd (11×10^{-6} K^{-1}), and GaN (6×10^{-6} K^{-1}) [16]. The differences in surface features for the contacts with and without cryogenic treatment are in the size and thickness of the different colored regions. In addition to the effect of compressive stress, strain was also induced during cooling because of the difference of the



(a) (b)

Fig. 9. SEM images for the Ni/Au contacts to p-GaN: (a) annealed at 600 °C for 10 min in an O_2/N_2 ambient and (b) cryogenically treated.



(a) (b)

Fig. 10. SEM images for the Pd/Au contacts to p-GaN: (a) annealed at 700 °C for 10 min in an O_2/N_2 ambient and (b) cryogenically

thermal expansion coefficients of the materials. Therefore, we suggest that subsequent fast cooling in liquid nitrogen minimizes the effect of compressive stress and strain, then results in a laterally more uniform surface, which is significant for the electrical properties. This is supported by the AFM data shown in Figs. 11 and 12, which show the surface roughness of the Ni/Au contacts. The average values of the surface roughness are 64.78 nm for the annealed only contacts and 37.38 nm for those treated in liquid nitrogen after annealing. There is little difference in the AFM images in the values of the surface roughness, 54.46 nm for annealed and 68.2 nm for annealed plus cryogenically treated, for the Pd/Au contacts.

The microstructure for the Ni/Au contacts was observed using XTEM (Figs. 13 through 17). Figure 13 shows a bright-field XTEM micrograph of the contact annealed at a temperature of 600 °C in O_2/N_2 ambient without cryogenic treatment. Figure 13 (lower view) shows that the metal/GaN interface line is clear and sharp and does not form new interdiffused compounds, and the GaN surface is covered by two thin layers. By a comparison with the ESCA depth profiles we found that the top layer should be NiO formed by outdiffusion of Ni and reaction of Ni with oxygen during high temperature annealing and cooling in the O_2/N_2 ambient. This

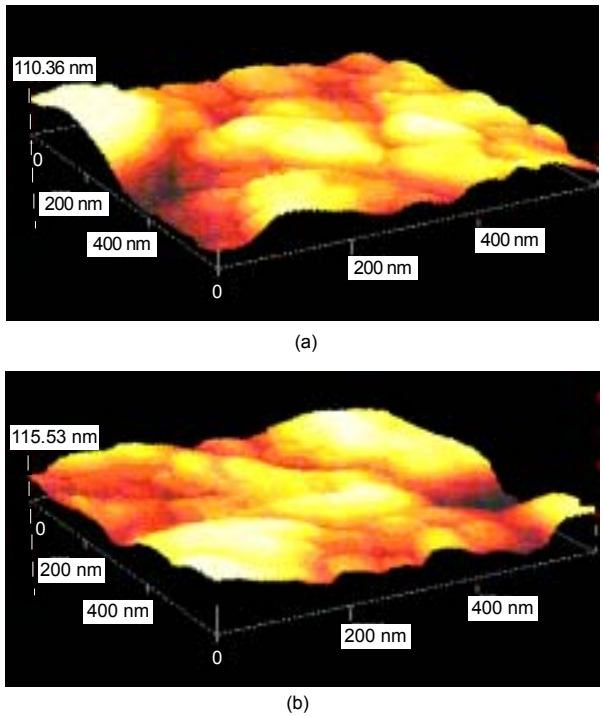


Fig. 12. AFM images for the Pd/Au contacts to p-GaN: (a) annealed at 700 °C for 10 min in an O_2/N_2 ambient and (b) cryogenically treated.

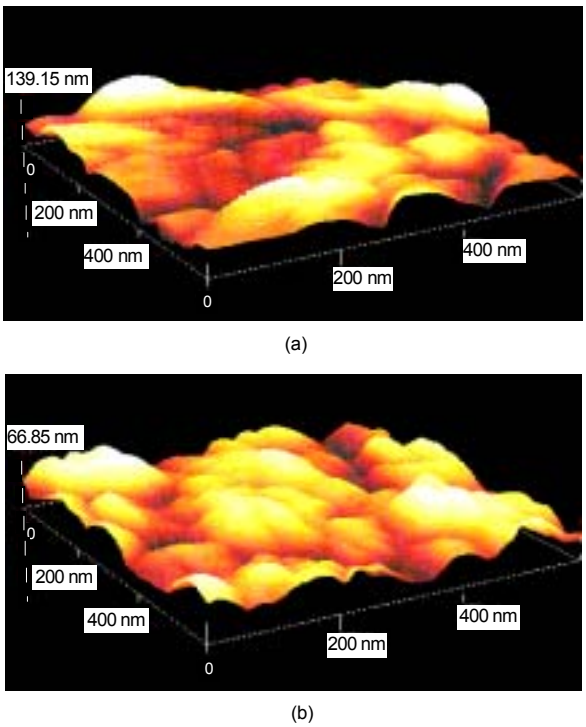


Fig. 11. AFM images for the Ni/Au contacts to p-GaN: (a) annealed at 600 °C for 10 min in an O_2/N_2 ambient and (b) cryogenically treated.

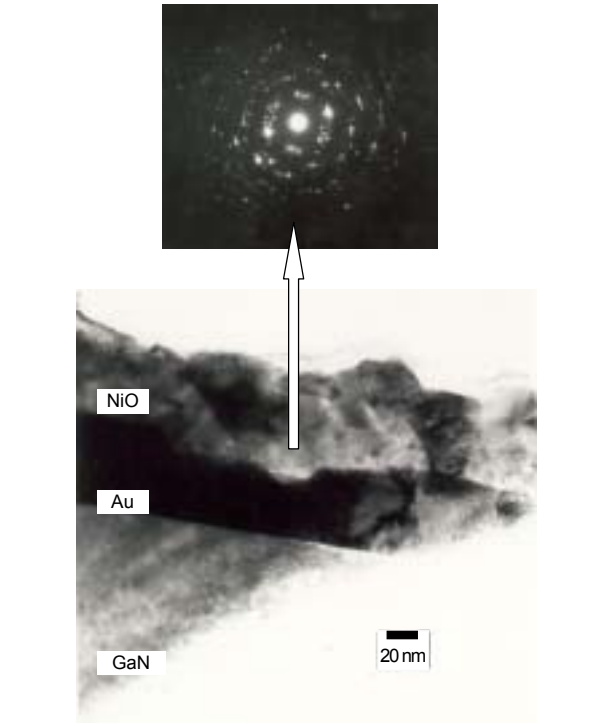


Fig. 13. XTEM image (lower view) and selective area diffraction pattern (upper view) for the Ni/Au contacts to p-GaN annealed at 600 °C for 10 min in O_2/N_2.

new phase showed a polycrystalline electron diffraction pattern in the upper view of Fig. 13. On the other hand, Au changes position with Ni and directly contacts the GaN surface as a result of the outdiffusion of Ni. Figure 14 shows segregation and porousness in the Au layers due to poor adhesion as a result of the balling-up and the spiking effects. These effects are well known properties of Au metal during high temperature annealing and the cooling process. However, for the contacts with cryogenic treatment (Fig. 15), the contact layer above the GaN surface includes three layers of different contrast: NiO, Ni, and Au layers from the top, respectively. This is confirmed by a comparison to energy dispersive x-ray (EDX) spectroscopy. Every EDX spectrum corresponding to probe points 1, 2, 3, and 4 depicted in Fig. 16 represents the different composition of each layer. EDX analysis of point 1 indicates an abundance of Ni and O and evidences the formation of an NiO phase in good agreement with previous results, while point 2 shows an increase in Au. An abundance of Au was detected in the cavity and sublayer at the metal/GaN interface, points 3 and 4. This suggests that Au is responsible for a discontinuous metal layer as well as the surface and interface roughness. We believe that the Ni did not completely react with the oxygen but remained as a thin layer due to the rapid quenching. As a result, the new NiO and outdiffused Ni layers should have protected the Au metal and minimized discontinuity and roughening of the surface, which increase the contact resistance.

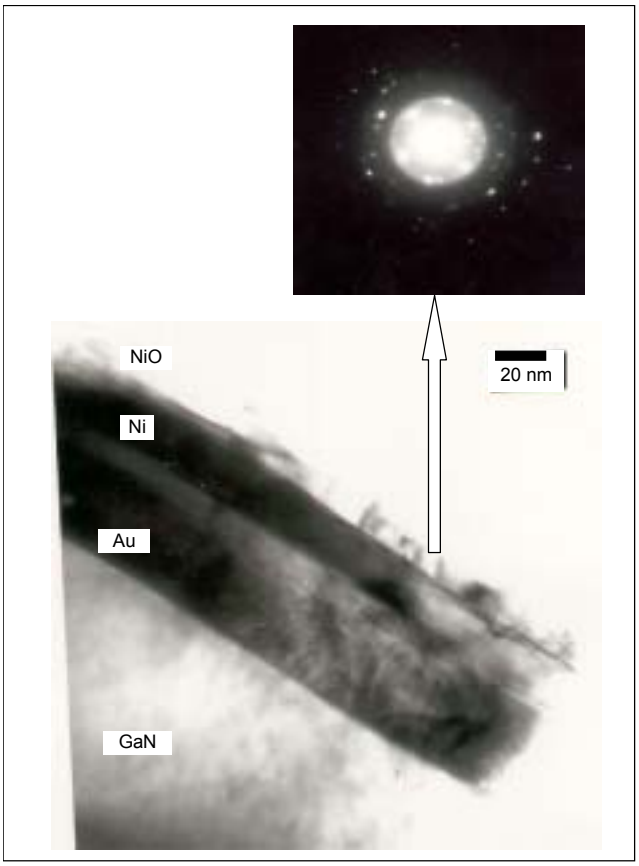


Fig. 15. XTEM image (lower view) and selective area diffraction pattern (upper view) for the Ni/Au contacts to p-GaN annealed at 600 °C for 10 min in O_2/N_2 and cryogenically treated.

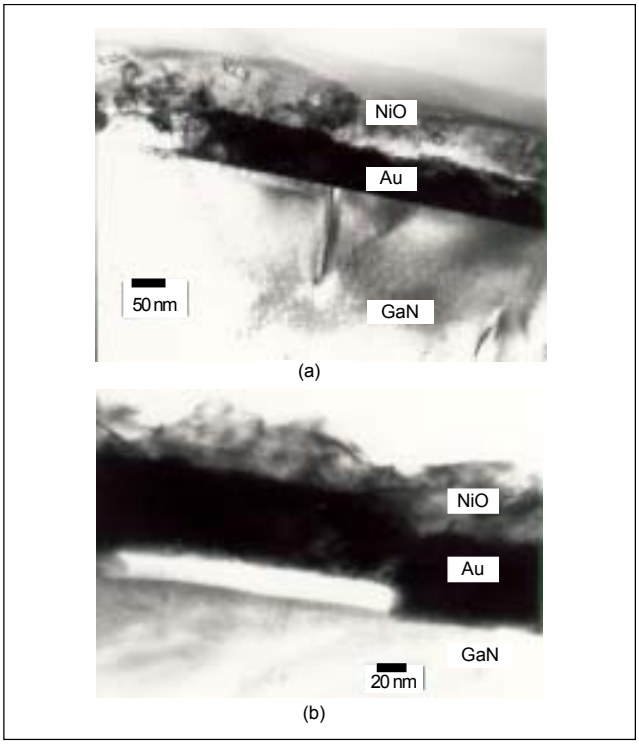


Fig. 14. XTEM image with cavities for the Ni/Au contacts to p-GaN annealed at 600 °C for 10 min in O_2/N_2: (a) segregation and (b) porousness in Au layer.

A compositional characteristic was also observed at the NiO/Ni/Au interface and the Au/p-GaN interface. There was no difference in the EDX analysis, which indicated an abundance of Au at each NiO/Ni/Au interface. No evidence was shown for new compounds of Ni and Au. Figure 17 shows an EDX spectrum of the Au/p-GaN interface. Each spectrum at points 1, 2, and 3 has a different composition of existing elements. A high resolution electron microscopy (HREM) image and EDX revealed the possibility of a Ga:Au transition metal in the Au/p-GaN interface layer (about 20 nm thick), but this was not clear.

IV. CONCLUSIONS

Both Ni/Au and Pd/Au contacts to p-GaN show Ohmic behavior with and without cryogenic treatment. The cryogenic treatment on the Ni/Au contacts improves the I-V linearity and reduces the specific contact resistance from $9.84\times10^{-4}\ \Omega cm^2$ to $2.65\times10^{-4}\ \Omega cm^2$. We observed the phases NiO and Au:Pd for

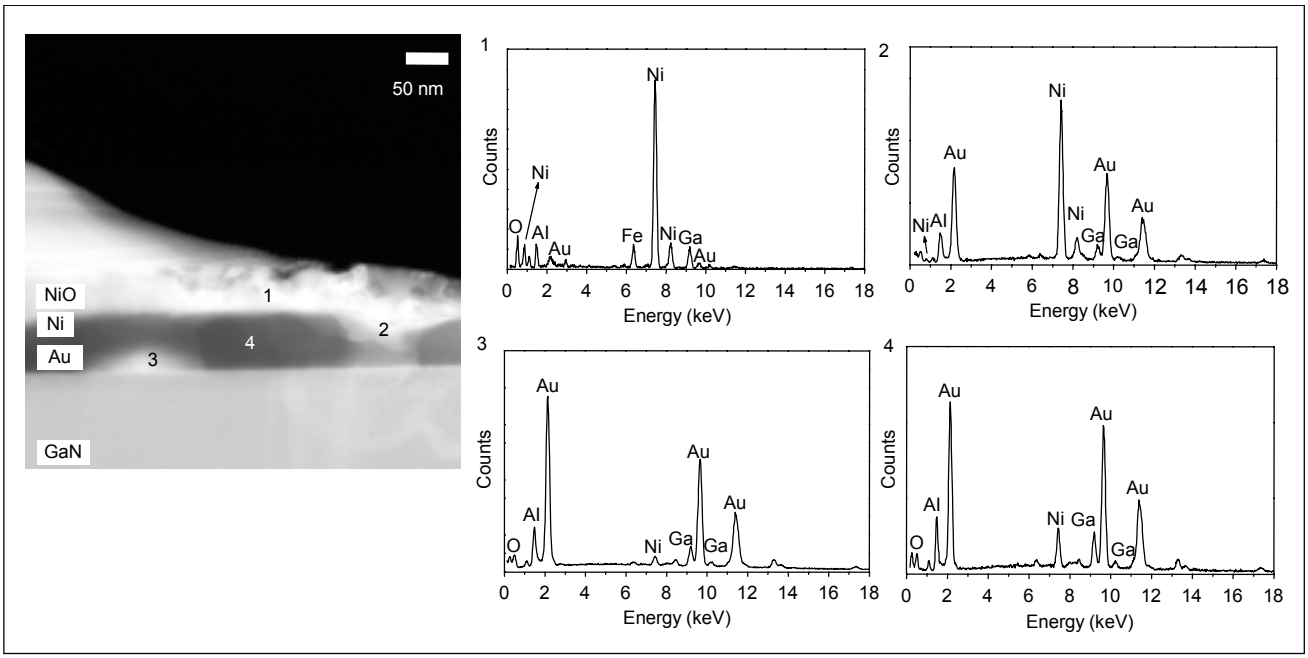


Fig. 16. XTEM image and EDX analysis for the Ni/Au contacts to p-GaN annealed at 600 °C for 10 min in O_2/N_2 and cryogenically treated.

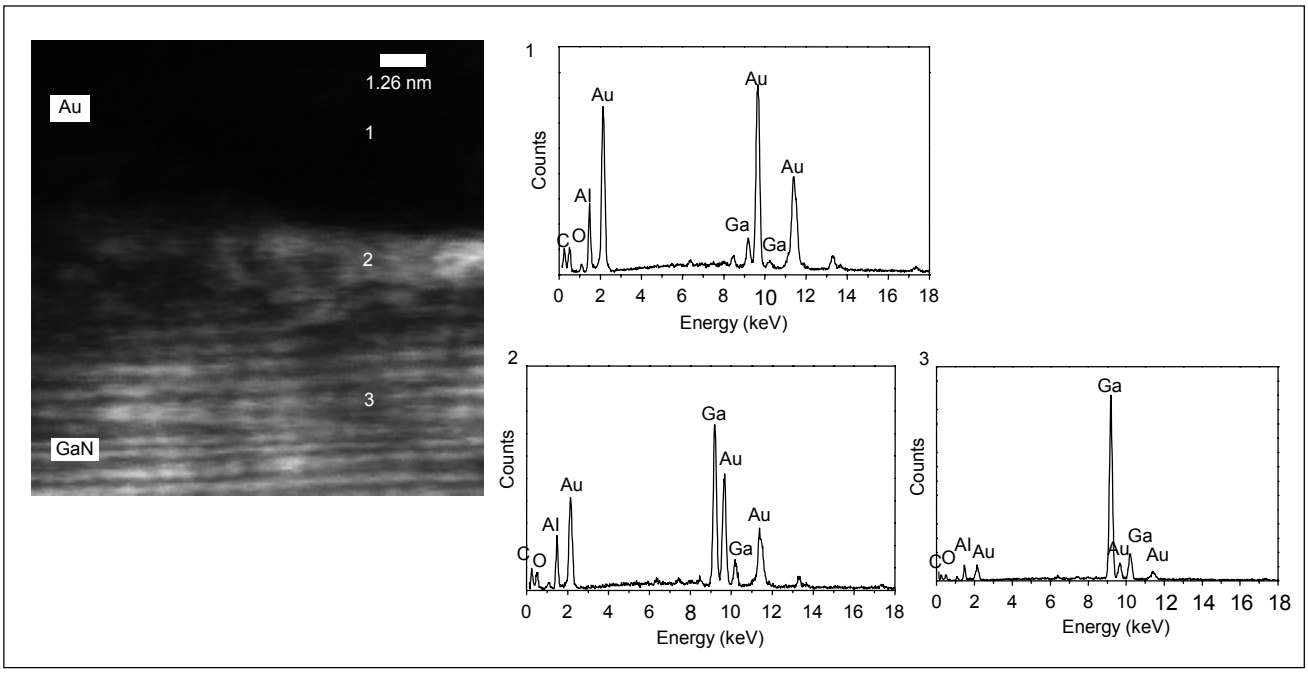


Fig. 17. HREM image and EDX analysis for the Ni/Au contacts to p-GaN annealed at 600 °C for 10 min in O_2/N_2 and cryogenically treated.

Ni/Au and Pd/Au contact systems, respectively. Subsequent fast cooling in liquid nitrogen after annealing affects the recrystallizing of NiO and Au:Pd solid solution produced from heat treatment, then improves their surface morphology. The contact resistance is mainly improved by: i) high temperature annealing which forms new phases at the metal/metal interface and the metal and the GaN surface compared to the as-deposited samples; and ii) cryogenic treatment, which affects both the recrystallization and surface morphology.

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