THICKNESS DEPENDENCE OF ELECTRONIC PROPERTIES OF GaN EPI-LAYERS

W. GÖTZ*, J. WALKER, L.T. ROMANO, AND N.M. JOHNSON
Xerox Palo Alto Research Center, Palo Alto, California 94304, USA
R.J. MOLNAR
Massachusetts Institute of Technology, Lincoln Laboratory, 244 Wood Street, Lexington, Massachusetts 02173, USA

ABSTRACT

The electronic properties of heteroepitaxial GaN were investigated for unintentionally doped, n-type films grown by hydride vapor phase epitaxy on sapphire substrates. The GaN layers were characterized by variable temperature Hall-effect measurement, capacitance-voltage (C-V) measurements, and deep level transient spectroscopy (DLTS). The measurements were performed on as-grown, 13 μm thick films and repeated after thinning by mechanical polishing to 7 μm and 1.2 μm. The room temperature electron concentrations as determined by the Hall-effect measurements were found to increase from $\sim10^{17}$ cm^{-3} (13 μm) to $\sim10^{20}$ cm^{-3} (1.2 μm) with decreasing film thickness. However, the C-V and DLTS measurements revealed that the ionized, effective donor and deep level concentrations, respectively, remained unchanged in regions close to the top surface of the films. These findings are consistent with the presence of a thin, highly conductive near interface layer which acts as a parasitic, parallel conduction path. Possible sources of such a shunt near the GaN/sapphire interface include oxygen contamination from the sapphire substrate or a structurally highly defective, 300 nm thick interface layer.

INTRODUCTION

Hydride vapor phase epitaxy (HVPE) has been revitalized as a promising GaN growth technique to overcome the apparent lack of GaN substrates for the growth of electronic and light emitting III-V nitride devices by metalorganic chemical vapor deposition (MOCVD) or molecular beam epitaxy (MBE) [1,2]. It has been demonstrated that HVPE is capable of growing thick GaN films on sapphire substrates with greatly improved structural and electronic properties as compared to heteroepitaxial GaN films grown by MOCVD or MBE [3,4]. Dislocation densities as low as $\sim5\times10^{7}$ cm^{-2} [5] and room temperature electron mobilities of ~900 cm^2/Vs [6] have been accomplished for ~74 μm thick, unintentionally doped GaN films with an electron background concentration of $\sim8\times10^{16}$ cm^{-3} (300 K). Characterization of Schottky diodes formed on HVPE-grown GaN films by deep level transient spectroscopy (DLTS) revealed deep level concentrations below 10^{16} cm^{-3} [6]. However, in the same films the presence of deep levels with thermal ionization energies in the range from ~100 to ~200 meV and concentrations as high as $\sim5\times10^{18}$ cm^{-3} were derived from the temperature dependence of the electron concentration by Hall-effect measurements [5].

The above contradictory results motivated the present study. For the analysis of the Hall-effect data, uniformity of the electronic properties throughout the film thickness is assumed. To investigate the electronic properties as a function of film thickness (d) a 13 μm thick HVPE-grown GaN layer was characterized by variable-T Hall effect, C-V measurements and DLTS. Subsequently, the film thickness was reduced to 7 and to 1.2 μm by mechanical polishing and characterized by the same techniques at each of the thickness steps. Characterization of the HVPE film was complemented by secondary ion mass spectrometry (SIMS).

*Permanent address: Hewlett-Packard Company, 370 West Trimble Road, San Jose, CA 95131, USA

Mat. Res. Soc. Symp. Proc. Vol. 449

EXPERIMENTAL

The GaN material used in this study was grown in a vertical HVPE reactor which is described in Ref. [7]. The nucleation of the GaN on c-plane sapphire substrates was enhanced by a GaCl pretreatment [6,7]. The film was grown at 1050°C to a thickness of 13 μm with a growth rate of 13 μm/h. X-ray diffractometry of the as-grown film revealed a FWHM of the (002) rocking curves of ~5 min. The tilt component of the film was measured by the (002) reflection to be 4.5 arcmin and the twist component was measured by the (112) asymmetric reflection to be 11.5 arcmin [5].

The Hall-effect measurements were conducted in the temperature range between 80 and 500 K with a magnetic field of 17.4 kG. For the measurements, samples of 5 × 5 mm^2 size were cut from the wafers and Ohmic metal contacts were deposited in the Van der Pauw geometry. For the analysis a temperature independent Hall scattering factor of unity value was assumed.

Conventional capacitance-voltage (C-V) measurements were conducted to investigate the depth profile of the dopants below the surface of the GaN films. Schottky diodes were fabricated by evaporating Au through a shadow mask onto the surface of the GaN samples. Large area Ohmic contacts were also deposited on the surface. The measurements were conducted with a 1 MHz, 10 mV test signal up to a reverse bias of ~20 V.

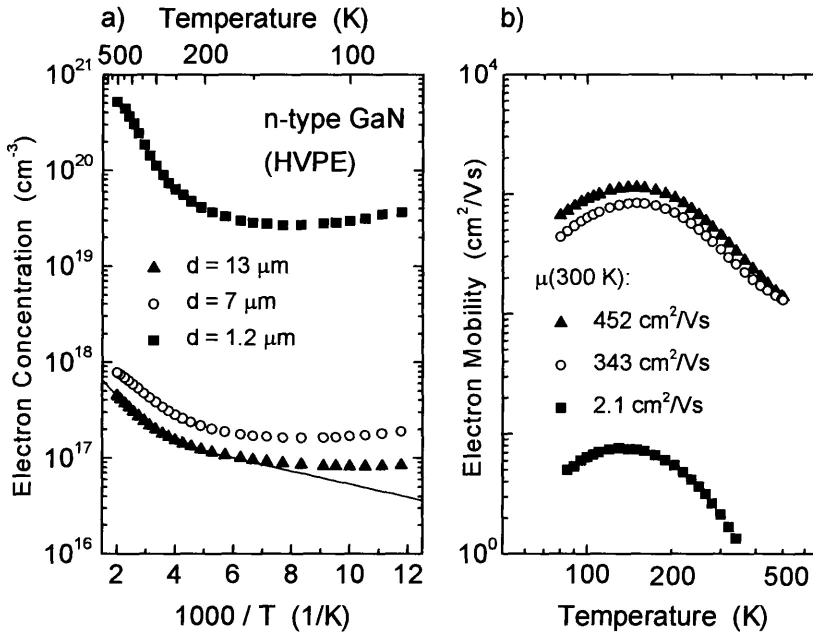

Fig. 1. Electron concentration vs reciprocal temperature (a) and electron mobility vs temperature (b) for three different film thicknesses as determined from variable-T Hall-effect measurements.
The solid line in Fig. 1a results from a fit of the charge neutrality condition to the high temperature branch of the experimental Hall data. The fit yields parameters for two independent donors which are given in the text.

The Schottky diodes were also utilized to perform DLTS measurements in the temperature range between 75 and 475 K. The DLTS system employed in this study is described in Ref. [8].

The film thinning was accomplished by mechanical polishing. The final polish achieved an rms surface roughness of <10 Å.

SIMS depth profiles for Si and O were measured using a Cs^+ primary ion beam in a CAMECA IMS 4F system with GaN implantation standards.

HALL-EFFECT RESULTS

Results from variable-T Hall-effect measurements are shown in Fig. 1 (symbols). Figure 1a displays electron concentrations as a function of temperature for the as-grown film (d = 13 μm) and after thinning by mechanical polishing to thicknesses of 7 μm and 1.2 μm. The electron concentrations significantly increase with decreasing film thickness. Figure 1b shows the electron mobility as a function of temperature for the three different film thicknesses. The electron mobility decreases with decreasing film thickness.

The experimental data for the original film thickness was analyzed using the charge neutrality condition [9] assuming two independent donors and acceptor compensation (solid line in Fig. 1a). The analysis yields a shallow donor with an activation energy (thermal ionization energy) of ~18 meV and a concentration of $\sim 2\times10^{17}$ cm^{-3}. The presence of a second donor is required to explain the high-temperature portion of the experimental Hall-effect data. The parameters for the second donor are ~180 meV and $\sim 5\times10^{17}$ cm^{-3} for the activation energy and concentration,

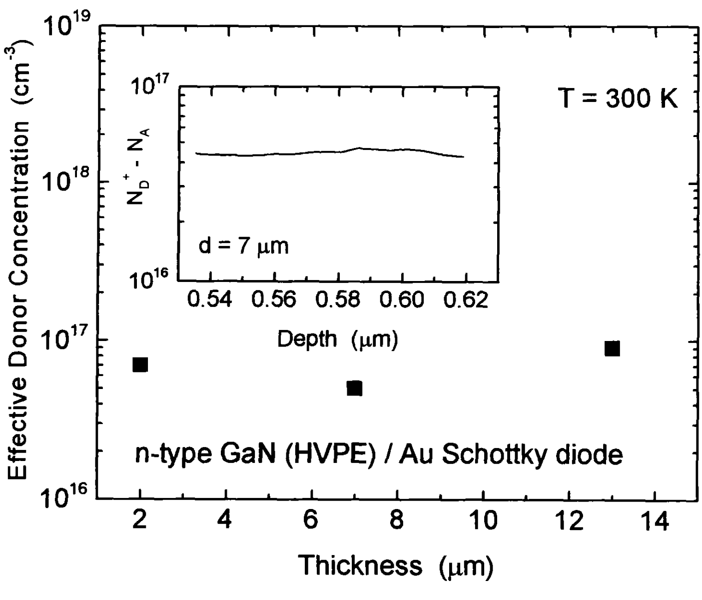

Fig. 2: Net donor concentration (N_D^+-N_A) for HVPE-grown GaN as determined by C-V measurements on Schottky diodes for three different film thicknesses. The inset demonstrates the net donor depth profile for a film thickness of 7 μm.

respectively. The analysis assumes that these donors are uniformly distributed throughout the thickness of the HVPE film.

C-V MEASUREMENTS, DLTS AND SIMS

Results from C-V measurements as a function of film thickness are shown in Fig. 2. The effective donor (N_D^+-N_A) concentrations depicted in Fig. 2 are average concentrations derived from the depth profiles. As an example, the depth profile of N_D^+-N_A determined for a film thickness of 7 μm is shown in the inset of Fig. 2. The C-V results indicate that N_D^+-N_A stays approximately constant from the original film thickness to a depth of ~ 1 μm away from the GaN / sapphire interface.

Results from DLTS are shown in Fig. 3. Displayed is a DLTS spectrum measured for our HVPE-grown GaN material at the original film thickness (13 μm). The spectrum which was recorded for an instrumental emission rate of 46.2 s^{-1} reveals the presence of four discrete deep levels. They are labeled DLN_1, DLN_2, DLN_3, and DLN_4. For this particular sample, the deep level DLN_2 appears only as a shoulder and, therefore, was not considered for analysis. The measurement was repeated after each polishing step; however, the spectra are not shown in Fig. 3. The DLTS spectra were analyzed assuming a temperature independent capture cross section. The defect parameters for DLN_1, DLN_3, and DLN_4 are depicted in Fig. 3 as functions of film thickness. Activation energies for electron emission to the conduction band for DLN_1,

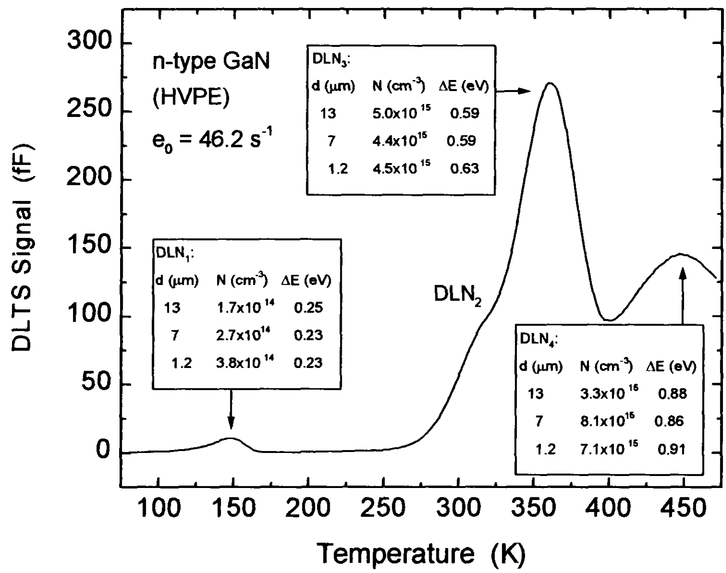

Fig. 3: DLTS spectrum for HVPE-grown GaN (original film thickness). Peaks in the spectrum indicate the presence of discrete deep levels. Parameters (concentration, N, and activation energy for electron emission to the conduction band, ΔE) for three deep levels are depicted for three different film thicknesses (d).

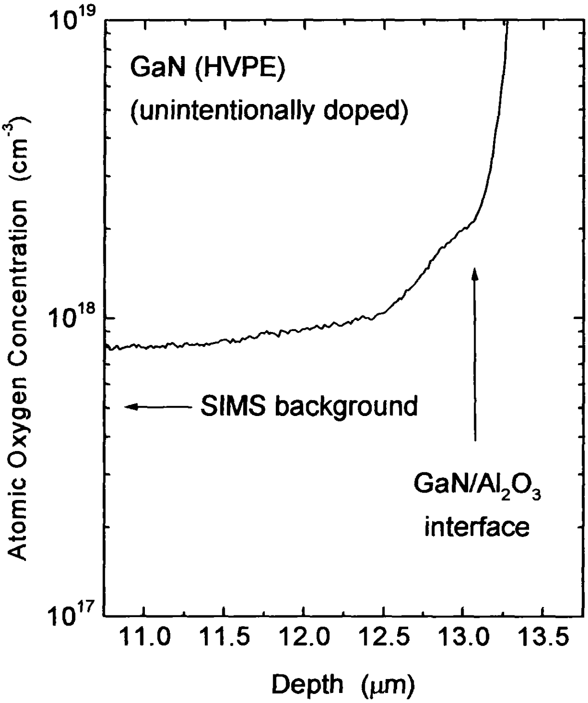

Fig. 4: Depth profile of the oxygen concentration near the GaN / sapphire interface. The oxygen background of the SIMS measurement is also indicated.

DLN_2, and DLN_4 were determined to range from 0.23 to 0.25 eV, 0.59 to 0.63 eV and from 0.86 to 0.91 eV, respectively. The activation energies for the three deep levels only vary within the experimental uncertainties for the measurements at different sample thicknesses indicating that each DLTS measurement detected the same deep levels. The concentrations of DLN_1 and DLN_3 also vary only within experimental uncertainties showing that these deep levels are almost uniformly distributed within the investigated thickness range. An exception is DLN_4, the concentration of which is about three times higher at a film thickness of 1.2 μm than at the original film thickness.

The oxygen concentration profile near the GaN / sapphire interface as determined by SIMS is shown in Fig. 4. The profile from the sample surface to a depth of ~11.5 μm is almost flat at an oxygen level of $\sim 8\times 10^{17}$ cm^{-3} (not shown in Fig. 4). At a depth of 11.5 μm the oxygen concentration slightly increases and rises between 12.5 μm and the GaN/sapphire interface from $\sim 9\times 10^{17}$ cm^{-3} to $\sim 2\times 10^{18}$ cm^{-3}. The Si concentration was also monitored but found not to exceed the background level of the SIMS measurement ($\sim 1\times 10^{17}$ cm^{-3}) throughout the film thickness (not shown).

CONCLUSIONS

The experimental data presented in this study demonstrate that shallow as well as deep levels are homogeneously distributed in the major portion of the 13 μm film. This is evident from C-V (Fig. 2) and DLTS measurements (Fig. 3), respectively, which were conducted for three different film thicknesses. The DLTS measurements also show that the concentration of detected deep levels do not exceed concentrations of $\sim 10^{16}$ cm^{-3}. However, Hall measurements indicate a significant increase in electron concentration with decreasing film thickness as well as the presence of a deep donor level in concentrations above $\sim 10^{18}$ cm^{-3}. This behavior may be explained by the presence of a thin, highly conductive GaN layer close to the GaN/sapphire interface. For depth inhomogeneities the Hall effect measurements yield an effective areal density of free electrons $n_{s,eff}$ and an effective Hall mobility μ_{eff} [10]. For a two-layer model the product $n_{s,eff}$ μ_{eff} is given by

$$n_{s,eff}\mu_{eff} = n_i\mu_i d_i + n\mu(d - d_i), \tag{1}$$

where d_i is the thickness of the interface layer and n, μ and n_i, μ_i are the electron concentrations, mobilities of the GaN film (without interface layer) and the interface layer, respectively. The presence of a ~300 nm thick, highly defective interface layer was detected from transmission electron microscopy for the HVPE film investigated in the present study [5]. Under the assumption that this interface layer is responsible for the observed electrical phenomena a lower limit ($\mu_i = \mu$) of the shallow donor and the deep level concentration in the interface layer can be estimated with Eq. (1). With $d_i \sim 300$ nm the shallow donor concentration in the interface layer becomes $>5\times10^{19}$ cm^{-3} and the deep level concentration becomes $>2\times10^{20}$ cm^{-3}. The atomic concentration of potential donors (O, Si) in the interface layer (Fig. 4) cannot account for the estimated shallow donor concentration. Thus for both shallow donors and deep levels the responsibility of native defects is implied.

Further study is needed to decide whether a two-layer model for the electrical conductivity in heteroepitaxialy-grown GaN films is generally applicable.

ACKNOWLEDGMENT

The work at Xerox was supported by DARPA, agreement # MDA972-95-3-008 and the work at Lincoln Laboratory was supported by the Department of the Air Force and by DARPA. Opinions, interpretations, conclusions, and recommendations are those of the authors and not necessarily endorsed by the United States Air Force.

REFERENCES

1 T. Detchprohm, K. Hiramatsu, N. Sawaki, and I. Akasaki, J. Cryst. Growth **137**, 170 (1994)
2 R.J. Molnar, W. Götz, L.T. Romano, and N.M. Johnson, Electrochem. Soc. Proc. **96-11**, 212 (1996)
3 S.N. Mohammad, A.A. Salvador, and H. Morkoç, Proc. IEEE **83**, 1306 (1995)
4 S.D. Lester, F.A. Ponce, M.G. Craford, and D.A. Steigerwald, Appl. Phys. Lett. **66**, 1249 (1995)
5 W. Götz, L.T. Romano, B.S. Krusor, and N.M. Johnson, Appl. Phys. Lett. **69**, 242 (1996)
6 R.J. Molnar, to be published in J. Cryst. Growth
7 R.J. Molnar, P. Maki, R. Aggarwal, Z.L. Liau, E.R. Brown, I. Melngailis, W. Götz, L.T. Romano, and N.M. Johnson, Mater. Res. Soc. Symp. Proc. **423**, 221 (1996)
8 W. Götz, Mater. Res. Soc. Symp. Proc. **378**, 491 (1995)
9 W. Götz, N.M. Johnson, D. P. Bour, C. Chen, H. Liu, C. Kuo, and W. Imler, Mater. Res. Soc. Symp. Proc. **395**, 443 (1996)
10 N.M. Johnson, in *Hydrogen in Semiconductors*, edited by J.L. Pankove and N.M. Johnson (Academic, San Diego, CA, 1991), p. 118