

# Measurement of the Al mole fraction of bulk AlGa<sub>N</sub> and AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructure by photoconductance method

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A photoconductance method was used to determine the band-gap energy and, therefore, the Al mole fraction of bulk Al<sub>x</sub>Ga<sub>1-x</sub>N and Al<sub>x</sub>Ga<sub>1-x</sub>N/GaN heterostructures. The results are compared with those obtained by a more elaborate photoluminescence method. © 1999 American Institute of Physics. [S0021-8979(99)09017-9]

## I. INTRODUCTION

AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructure field effect transistors (HFET) have been actively investigated for high temperature, high power, and high speed electronic devices in recent years.<sup>1,2</sup> The Al mole fraction of the AlGa<sub>N</sub> layer is an important parameter for the design and performances of the devices. The band-gap energy,  $E_g$ , of AlGa<sub>N</sub>, which is directly related to the Al mole fraction, is conventionally measured using photoluminescence (PL) and/or the optical absorption method. In the case of PL, a He–Cd laser ( $E_\lambda = 3.81$  eV) can be used as an excitation light source for the measurement of Al<sub>x</sub>Ga<sub>1-x</sub>N with  $x$  up to ~15%. However, for Al<sub>x</sub>Ga<sub>1-x</sub>N layers with  $x > 0.15$ , the wavelength of these lasers are too long to be useful. Special lasers with large photon energies are needed. In the case of the absorption method applied to AlGa<sub>N</sub> layers grown on SiC substrate or AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures, the band-gap energy of the AlGa<sub>N</sub> layer is larger than that of SiC and Ga<sub>N</sub>, thus preventing the use of conventional optical absorption method for the measurement. To circumvent these difficulties, we have developed a photoconductance method to measure the band-gap energy of AlGa<sub>N</sub> in these structures. This method is based on the change of electrical conductivity when light with photon energy larger than the band-gap energy is absorbed by the semiconductor. Since the conductivity,  $\sigma$ , is given by

$$\sigma = ne\mu, \quad (1)$$

where  $n$  is the carrier concentration,  $e$  the electron charge, and  $\mu$  the mobility. When light is illuminated onto the surface of a semiconductor, photons are absorbed, resulting in additional carriers,  $\Delta n$ . The conductivity increases to

$$\sigma + \Delta\sigma = ne\mu + (\Delta n)e\mu. \quad (2)$$

When the photon energy of the monochromatic incident light increases from low photon energies to the band-gap energy

of AlGa<sub>N</sub>, the photoconductance<sup>3</sup> markedly increases with  $\Delta n$  proportional to the absorption coefficient at  $E_g$ . The change in photoconductance as a function of incident photon energy can be used to determine the band-gap energy of the semiconductor. In this article, we report the experimentally determined band-gap energy and the Al mole fraction using the photoconductance method on both bulk AlGa<sub>N</sub> and AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructures. The results are compared with those obtained by photoluminescence.

## II. EXPERIMENT

The samples were grown by metalorganic vapor phase epitaxy (MOVPE). The structure of bulk samples consisted of an Al<sub>x</sub>Ga<sub>1-x</sub>N layer, about 1  $\mu\text{m}$  in thickness, grown on SiC (0001) substrate with a 0.1  $\mu\text{m}$  thick AlN buffer layer [see Fig. 1(a)]. The structure of the AlGa<sub>N</sub>/Ga<sub>N</sub> heterostructure consisted of a 300–500  $\text{\AA}$  thick Al<sub>x</sub>Ga<sub>1-x</sub>N layer on top of a ~3  $\mu\text{m}$  thick undoped Ga<sub>N</sub> layer grown on sapphire (0001) substrate with an 0.04  $\mu\text{m}$  AlN buffer layer [see Fig. 1(b)]. The samples were first cleaned with organic solvents followed by etching in a HCl:HF:H<sub>2</sub>O solution, and loaded in an e-beam evaporator equipped with dry pumps. Square pads 150×150  $\mu\text{m}^2$ , of a bilayer structure of Al(710  $\text{\AA}$ )/Ti(300  $\text{\AA}$ ) were patterned on the AlGa<sub>N</sub> layer as ohmic contacts by the lift-off technique. The spacing between two adjacent pads was about 100  $\mu\text{m}$ . The ohmic contact was formed at 850 °C for 40 s in a N<sub>2</sub> ambient with a resultant resistance between two adjacent pads of about 300  $\Omega$ . After the formation of the ohmic contact, a Au layer (~1200  $\text{\AA}$  thick) was deposited on top of the ohmic contact pads to facilitate Au wire bonding for the photoconductance measurements. During measurement, the incident light was focused on an area between the contact pads as shown in Figs. 1(a) and 1(b).

The photoconductance measurement setup for bulk AlGa<sub>N</sub> samples is shown in Fig. 2(a). A halogen lamp was used as the light source. The light was monochromized using a monochromator (ISA, model HR-320), chopped with a fre-

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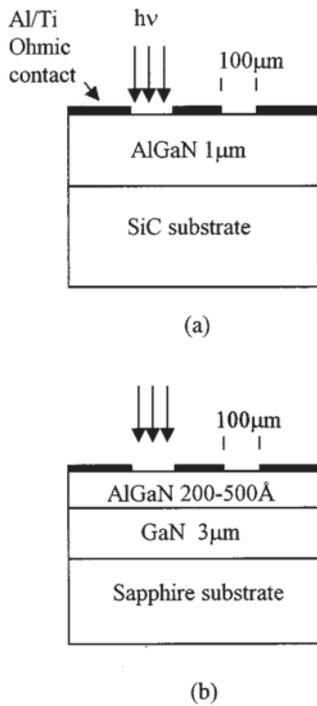


FIG. 1. Schematic diagrams of the structures of (a) bulk AlGaIn on SiC, (b) AlGaIn/GaN heterostructures.

quency of 338 Hz, and focused on the sample using a UV lens. The incident light intensity spectrum was calibrated using a UV sensitive photosensor (Hamamatsu H6780-04) with a known sensitivity,  $S(\lambda)$ . The incident light intensity spectrum  $I_0(\lambda)$  was given by:  $I_0(\lambda) = I(\lambda)/S(\lambda)$ , where  $I(\lambda)$  is the photoresponse of the photosensor. The photon spectrum of the incident light was given by  $I_0(\lambda)/h\nu$ . In our experiments, the photocurrent,  $\Delta i$ , which is proportional to  $\Delta n$  was measured with a lock-in amplifier (EG&G, model 5209), and with an applied voltage ranging between 0.1 and 0.3 V dc. The normalized photocurrent (current measured by the lock-in amplifier divided by the photon spectrum of the incident light) was then plotted against the wavelength of the incident light. The photon energy at which where the photocurrent rose rapidly was taken to be the bandgap energy of the semiconductor.

For samples with an AlGaIn/GaN heterostructure, optically generated electrons in AlGaIn may transfer to the quantum well located at the AlGaIn/GaN interface, thus increasing the two-dimensional electron gas (2DEG) concentration. These electrons would not recombine immediately after the illumination was turned off because of the spatial separation between the 2DEG and the holes in AlGaIn. Due to this persistent photoconductivity,<sup>4,5</sup> the experimental setup shown in Fig. 1(a), which measures the ac signals caused by the chopped light, could not be used on AlGaIn/GaN heterostructures since the persistent photoconductance decay time was much larger than that of the chopping period. A dc experimental setup was developed to measure the photoconductance of AlGaIn/GaN heterostructures. In this setup, the wavelength of the incident light was step scanned slowly

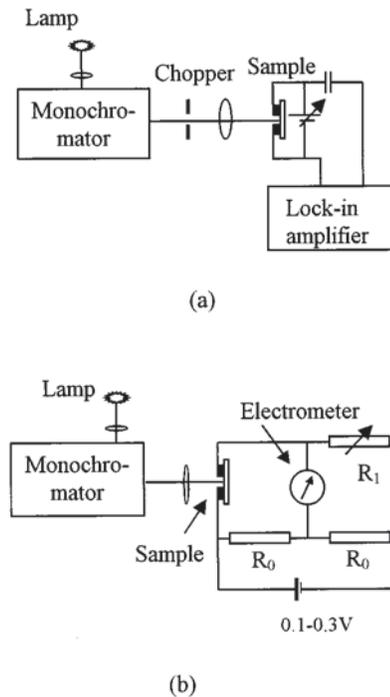


FIG. 2. Schematic diagrams of the measurement setup (a) for bulk AlGaIn, chopping frequency is 338 Hz, (b) for AlGaIn/GaN heterostructures,  $R_0$  is a resistor of about 200  $\Omega$  and  $R_1$  is variable resistor, ranging from zero to 1 k $\Omega$ .

from long wavelength ( $\sim 3800$  Å) to short wavelength ( $\sim 3000$  Å). Each scan step was about 20 Å with a dwell time of 5 min at each step. The photocurrent  $\Delta i$  was a very small amount compared to the dark electrical current,  $i$ , across the sample ( $\Delta i/i$  was about  $10^{-4}$ ). The conventional dc measurement was not sensitive enough to measure a small variation on a large background. A Wheatstone bridge was used to increase the sensitivity of the measurement. The sample was used as an arm of the Wheatstone bridge as shown in Fig. 2(b). A small dc voltage, ranging between 0.1 and 0.3 V, was applied to the bridge with the sample in the dark. The bridge was balanced by a variable resistor,  $R_1$ , such that the dark current was balanced to zero across the electrometer. The monochromatic light beam was then turned on incident to the sample surface. A current  $\Delta i_e$  as a function of incident photon wavelength was detected by the electrometer. This current  $\Delta i_e$  was proportional to the photocurrent  $\Delta i$  across the sample.

### III. RESULTS AND DISCUSSION

Figure 3 shows the photocurrent of four AlGaIn bulk samples with various Al mole fractions as a function of the wavelength. The bandgap energies of the AlGaIn samples were taken to be at the wavelength where the photocurrent increased with a maximum slope of  $dI/d\lambda$  (derivative of the photocurrent with respect to the incident light wavelength). The decrease of the photocurrent at higher photon energies

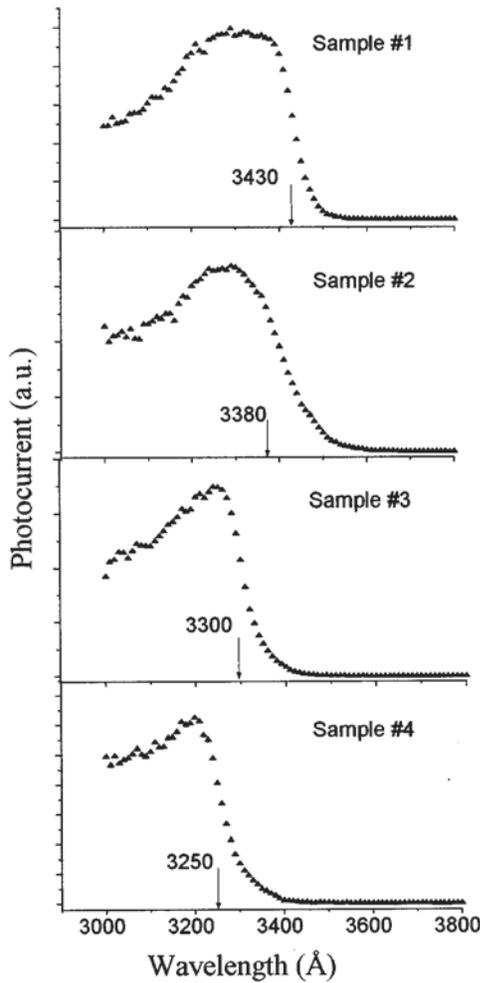


FIG. 3. Normalized photocurrent of bulk AlGaIn samples as a function of the wavelength of the incident light. The arrows indicate the wavelength responding to the band-gap energy of AlGaIn obtained by the maximum slope of  $dI/d\lambda$ . The results are listed in Table I.

was caused by surface recombination of the generated carriers as the absorption occurred closer to the surface with increasing photon energy.

Figures 4(a) and 4(b) show the typical photocurrent spectra of the AlGaIn/GaN heterostructure for two samples (Nos. 9 and 5 in Table I). To minimize persistent photoconductivity, the samples were kept in a dark room before and during the measurement. The wavelength of the incident light was scanned from low photon energies to high photon energies as shown in curve 1 in Figs. 4(a) and 4(b). In these curves, the first jump in photocurrent corresponds to the band-gap absorption of GaN and the second jump corresponds to the bandgap absorption of AlGaIn. Curve 2 in Figs. 4(a) and 4(b) was obtained by scanning from high photon energies to low photon energies. It can be seen that the sudden changes in the photocurrent shown in curve 1 became much smaller due to the persistent photoconductivity in the heterostructure even though the interval between datum points was as long as 5 min. The band-gap energy of the

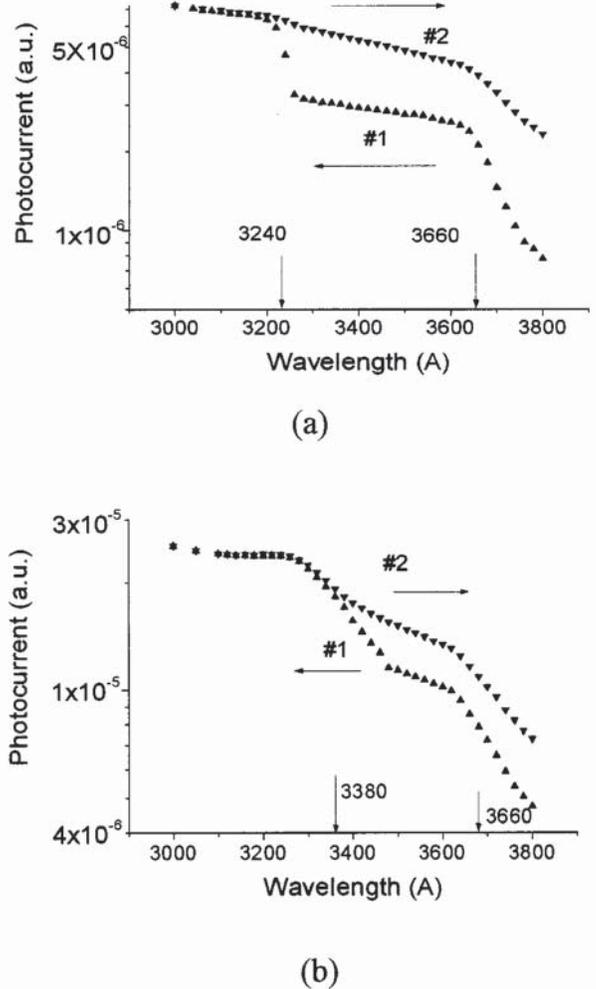


FIG. 4. Normalized photocurrent as a function of the wavelength of the incident light of an AlGaIn/GaN heterostructure. (a) for sample No. 9 and (b) for sample No. 5 in Table I. Curve 1 was obtained by scanning from low photon energy to high photon energy and curve 2 was obtained in the reverse direction, the horizontal arrows indicate the direction of scanning in wavelength. The vertical arrows indicate the band-gap wavelength of GaN and AlGaIn.

relevant layers was obtained again by differentiating the photocurrent with respect to the wavelength.

For comparison, the band-gap energies of the same samples were also measured by the PL method. For PL measurements, excitation pulses with pulse width of about 7 ps at

TABLE I. Band gaps and the Al mole fraction,  $x$  measured on bulk  $Al_xGa_{1-x}N$  and  $Al_xGa_{1-x}N/GaN$  heterostructures by photoconductance (PC) and photoluminescence measurement (PL).

Sample		Bulk AlGaIn				AlGaIn/GaN heterostructure					
		#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
PC	$E_g$ (eV)	3.62	3.67	3.75	3.82	3.67	3.73	3.68	3.71	3.83	4.05
	$x$	0.11	0.13	0.17	0.20	0.13	0.16	0.13	0.15	0.21	0.30
PL	$E_g$ (eV)	3.65	3.72	3.76	3.84	3.79	3.77	3.77	3.76	4.00	4.00
	$x$	0.12	0.16	0.17	0.23	0.19	0.18	0.18	0.17	0.28	0.28

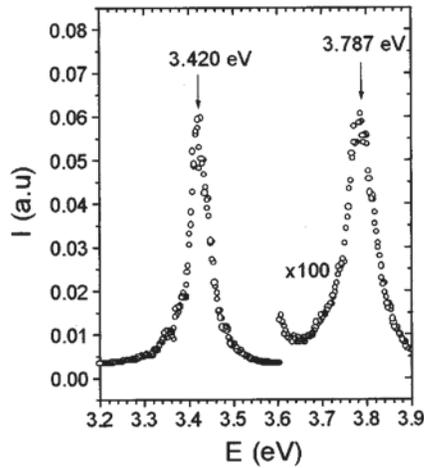


FIG. 5. Photoluminescence spectrum of an AlGaIn/GaN structure (sample No. 5 in Table I). The peak positions in the photoluminescence spectrum were taken as band-gap energies of the AlGaIn and GaN layers, respectively, as indicated by arrows.

a repetition rate of 9.5 MHz were provided by a picosecond UV laser system which consists of a yttrium-aluminum-garnet (YAG) laser (coherent Antares 76) with a frequency doubler which pumps a cavity-dumped dye laser (coherent 702-2CD) with Rhodamine 6G dye solution and a second frequency doubler after the dye laser to provide a tunable photon energy up to 4.5 eV. The laser output after the second doubler has an average power of about 20 mW. A single-photon counting detection system was used to record the photoluminescence spectra. A typical PL spectrum of an AlGaIn/GaN heterostructure is shown in Fig. 5. The peak positions of the photoluminescence spectrum were taken as the band-gap energies of AlGaIn and GaN layer, respectively, as indicated by arrows in Fig. 5. Since the AlGaIn layer is very thin, the PL intensity of the AlGaIn layer is about 100 times weaker than that of the GaN layer.

The Al mole fraction was calculated from  $E_g$  using the following equation:<sup>6</sup>

$$E_g^{\text{AlGaIn}} = E_g^{\text{GaN}}(1-x) + E_g^{\text{AlN}}x - bx(1-x), \quad (3)$$

where  $x$  is the Al mole fraction,  $E_g^{\text{AlGaIn}}$ ,  $E_g^{\text{GaN}}$ , and  $E_g^{\text{AlN}}$  the band-gap energies of  $\text{Al}_x\text{Ga}_{1-x}\text{N}$ , GaN, and AlN, respectively. Values of 3.42 and 6.2 eV were used for the band-gap energies of GaN and AlN in the calculation. The parameter  $b$  is the direct band-gap bowing factor. The most recently reported value of  $b$  was  $1.3 \pm 0.2$  eV.<sup>7</sup> The values of  $b = 1.0 \pm 0.3$  eV (Ref. 8) and  $b = 0$  eV (Ref. 9) have also been reported in the literature. We chose a value of 1 in our calculation. We found that this value of  $b$  gave the most consistent results of  $x$  when compared with the results obtained by other methods such as the energy dispersive x-ray (EDX) method. Table I shows the band-gap energies of AlGaIn measured by photoconductance and photoluminescence and the

deduced Al mole fractions for ten samples. It can be seen that the difference in  $E_g$  obtained by these two methods is within 1.5% for bulk samples and about 3% for AlGaIn/GaN heterostructure. This relatively large difference in  $E_g$  for AlGaIn/GaN heterostructures between the two methods may be caused by two reasons; (1) Since the AlGaIn layer in HFET was thin ( $\sim 300$  Å), and the PL signal was weak and broad, it was difficult to determine the accurate peak position for the bandgap. This is particularly true for samples with high Al mole fractions, i.e., sample 9 in Table I. The Al mole fractions of the samples 9 and 10 also measured using EDX.<sup>10</sup> They were 0.22–0.25 and 0.30, respectively. These values are closer to the results obtained by the photoconductance method; (2) sample inhomogeneity in Al mole fractions in the AlGaIn layer across the sample area may also contribute to the differences. The uncertainty of the deduced value of  $x$  (the Al mole fraction) is magnified by Eq. (3). A 3% error in  $E_g$  would produce an uncertainty of about 30% in  $x$  for  $x$  of around 15%. This uncertainty is rather independent of  $b$ .

#### IV. SUMMARY

The photoconductance method was used to determine the Al mole fraction in both bulk AlGaIn and AlGaIn/GaN heterostructure samples. The results were consistent with those obtained by a much more elaborate photoluminescence measurement. The photoconductance method is deemed a simple and convenient way to estimate the Al mole fraction in AlGaIn.

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