Molecular beam epitaxy growth of the dilute nitride $GaAs_{1-x}N_x$ with a helical resonator plasma source

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Dilute nitride semiconductors of composition $GaAs_{1-x}N_x$ $(0.0017<x<0.0115)$ are grown by plasma-assisted molecular beam epitaxy with a helical resonator plasma source for active nitrogen. The plasma source is self-starting at the operating pressure and can be operated at two different frequencies for which the emission spectrum is dominated by N_2 molecules or by N atoms. For the same power the molecular-rich mode is found to produce a higher flux of active nitrogen. After extended operation the plasma tube becomes contaminated with As which reduces the flux of active nitrogen and creates a below band gap emission band in the photoluminescence of the $GaAs_{1-x}N_x$. For the clean discharge tube no difference is observed in the photoluminescence for samples grown in the molecule-rich or atom-rich mode.
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I. INTRODUCTION

The dilute nitride alloy $In_yGa_{1-y}As_{1-x}N_x$ is a small band gap semiconductor which can be grown epitaxially on GaAs substrates. It has several promising advantages over InGaAsP alloys grown on InP substrates for 1.3 and 1.55 μm wavelength light sources.[1] However, the dilute nitride alloys such as $In_yGa_{1-y}As_{1-x}N_x$ and $GaAs_{1-x}N_x$ suffer from degradation in their electronic properties with increasing N content.[2] It is important for device applications to minimize this degradation. In molecular beam epitaxy (MBE) growth, the most popular method for incorporating N is through chemical activation of nitrogen gas in a 13.5 MHz radio frequency inductive plasma discharge. In this article we describe the results of dilute nitride growth experiments with a helical resonator plasma source. We explore the effects of the operating conditions of the plasma source on the incorporation of nitrogen in GaAs and on the electronic quality of the dilute nitride alloy.

The helical resonator source has several advantages relative to conventional inductively coupled plasma sources. For example, since it is a resonant device, the discharge starts when the rf power is turned on, under standard operating conditions, so that it is not necessary to use an inert carrier gas to keep the source operating. Also by changing the operating frequency we are able to operate it in two different modes in which different ratios of atoms to excited state molecules are produced in the discharge. An important unsolved question for dilute nitride semiconductor growth is whether the best electronic properties are obtained for films grown with excited state N_2 molecules or with N atoms, or whether the active nitrogen species makes any difference at all.

II. HELICAL RESONATOR NITROGEN PLASMA SOURCE

The helical resonator plasma source, shown in Fig. 1, is located in a standard effusion cell port in an elemental source molecular beam epitaxy system. Its first two unloaded resonances are at 63 and 180 MHz, in the absence of a plasma discharge. The helix itself consists of a 16-turn gold-plated, self-supporting oxygen-free high-conductivity copper coil grounded at one end and open at the other end, inside a coaxial Ta shield tube with a cap on the open end. A 10 mm diameter pyrolytic boron nitride (PBN) discharge tube is lo-

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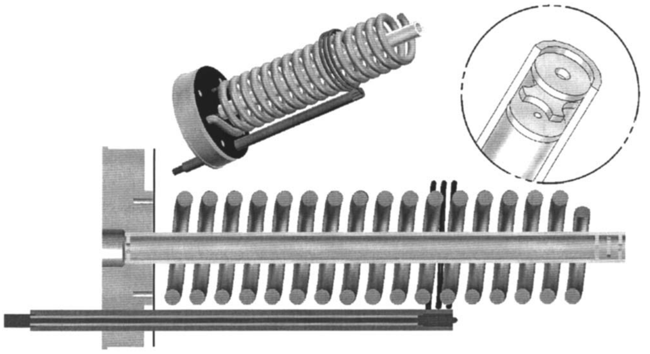

FIG. 1. Drawing of plasma source showing helical resonator, coupling loop, and pyrolytic boron nitride (PBN) discharge tube. The discharge tube is 1 cm in diameter. A constriction made from PBN at the upstream end of the discharge tube prevents the plasma from expanding back into the stainless steel gas feed line. A three-element baffle (inset) at the open end of the discharge tube blocks the line of sight to the discharge, suppressing the ion flux. The cylindrical Ta ground shield that surrounds the helix is not shown.

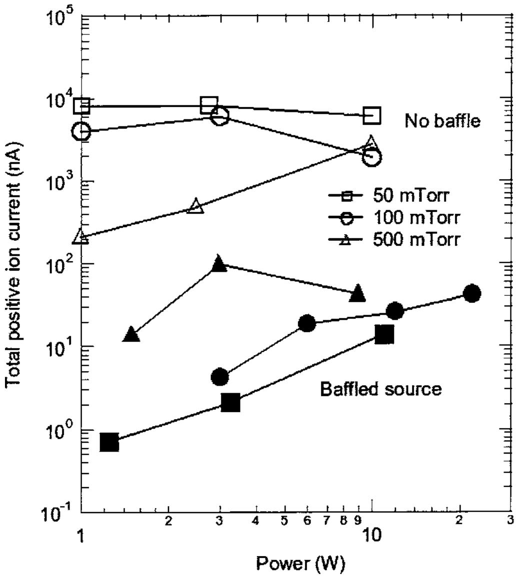

FIG. 2. Ion current from the plasma source, measured with a Faraday cup in a test chamber. The ion current is measured as a function of N_2 gas pressure in the discharge tube and the net forward rf power supplied to the discharge.

cated in the center of the helix. The relatively small diameter for the discharge tube was selected following experiments with different diameter tubes, in order to maximize the intensity of the atomic emission lines in the discharge at resonance. The resonator is driven by a wideband oscillator and power amplifier through a three-turn coupling loop as shown in Fig. 1, with 3.9 m of coaxial line between the power amplifier and the source. Helical resonators have the property that at resonance the input impedance at an intermediate tap position is purely real and can be adjusted to any value between zero and a maximum value determined by the losses in the resonator, by adjusting the position of the tap. Neglecting parasitic impedances, in principle one can match the helix resonance to the output impedance of the rf generator by adjusting a single tuning parameter. Furthermore since the source is a resonant device, high fields are present in the unloaded resonator, which means that the plasma discharge will start spontaneously under a wide range of operating pressures. This means that quantum well structures, which require rapid cycling of the active nitrogen flux, can be grown simply by turning the plasma source on and off, eliminating the need for shutters, an inert carrier gas, or growth interruptions.

High purity N_2 gas is introduced through a leak valve into the PBN discharge tube inside the resonator. The 5 9's grade N_2 gas is passed through an in-line purifier (SGT Super-Clean® filters, triple filter part No. F0301). A PBN plug with a small hole was placed at the back end of the discharge tube. This constriction prevents the plasma from expanding back into the stainless steel gas feed tube. As bombardment with energetic ions is known to degrade the electronic properties of semiconductors,[3,4] the exit of the discharge tube is equipped with a three-stage PBN baffle so that there is no line of sight to the plasma and all gas species exiting the discharge zone experience several wall collisions before hitting the substrate. The baffle is found to reduce the ion current by two to four orders of magnitude compared to the source without the baffle installed, as shown in Fig. 2.

The ion currents in Fig. 2 were measured in a separate test chamber, with and without the ion baffle on the plasma source, using a Faraday cup. The Faraday cup had a 9 mm diameter opening and was placed about 80 mm in front of the source. A set of three retarding grids allowed us to separate the electron and positive ion contributions to the current. The total current from the source was obtained by multiplying the measured current by a geometric factor estimated from the geometry of the apparatus, assuming a cosine distribution of ions from the plasma tube exit. The electron current increased approximately as the square of the source power and at high operating power, the electron current dominates. At high powers we were unable to measure the ion current due to the large electron current. At 500 mTorr operating pressure and 10 W of net forward power the maximum ion energy was about 100 eV, as determined by the voltage required to suppress 95% of the ion current.

In some commercial plasma sources the exit aperture consists of an array of small holes oriented so as to maximize the uniformity of the flux at the substrate. The small holes provide a line of sight to the plasma. Nevertheless the ion currents measured for the baffled helical resonator source in Fig. 2 are similar to the ion currents reported for a commercial source with the small diameter holes.[5]

Electrically biased deflection plates at the exit of the plasma source were found to be only partially effective at suppressing the ions, presumably due to production of secondary ions on the deflection plates. From the conductance of the baffle on the end of the discharge tube we estimate the pressure in the discharge tube to be about 0.03 mbars under typical growth conditions, with a background nitrogen pressure of 2×10^{-6} mbar in the growth chamber.

The forward and reflected rf power, P_F and P_R, are monitored with a directional watt meter and the difference is the

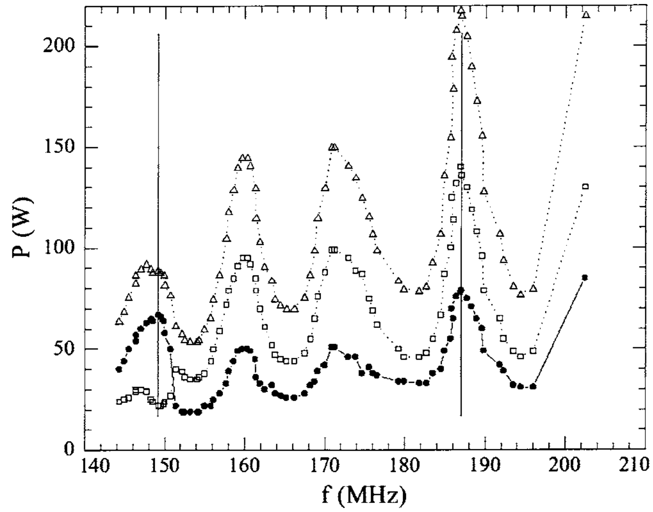

FIG. 3. P_f (△) and P_r (□) measured with the watt meter and P_{load} (●), calculated as P_f-P_r, as a function of frequency. The two vertical lines indicate the frequencies of the two operating modes of the plasma source discussed in this article: on resonance (left) and off resonance (right).

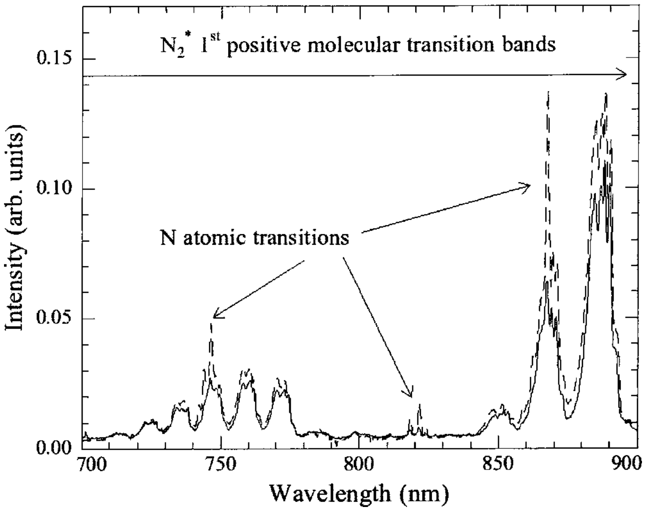

FIG. 4. Emission spectra for on-resonance operation at $P_{load}=100$ W (dashed line) and off-resonance operation at $P_{load}=80$ W (solid line).

net power to the load, $P_L=P_F-P_R$. Figure 3 shows the forward and reflected power and the net power to the load P_L as a function of the rf with the plasma ignited. Once ignited the plasma discharge remains ignited throughout the frequency range in Fig. 3. The large evenly spaced oscillations in the reflected power as a function of frequency are caused by standing waves in the coaxial line between the generator and the resonator due to impedance mismatch. From measurements of the frequency and power required to start and maintain the plasma, the helix resonance is believed to be at 149 MHz with the plasma on, indicated by a vertical line in Fig. 3. We denote this mode of operation as "on resonance." At the helix resonance we expect a large longitudinal field inside the discharge tube.[6] Away from the helix resonance, the highest net power to the load P_L occurs at 187 MHz. This defines another high power operating mode, which we refer to as "off resonance." The resonances in the transmission line coupling the generator to the plasma source make it possible to optimize coupling to the plasma source at a frequency that does not match the helix resonance so that the helix can also be driven efficiently at a nonresonant frequency. We speculate that the electric field distribution in the discharge for the nonresonant operation is different than at the helix resonance. For example, the electric field may be primarily a circulating (transverse) electric field in the off-resonance condition whereas on resonance the field is primarily longitudinal.[7]

III. EMISSION SPECTROSCOPY

We measured the emission spectrum of the plasma by collecting the light from a small window at the back of the plasma source and passing it through a monochromator. The spectra measured with the source operated on resonance and off resonance are shown in Fig. 4. Both spectra show the broad vibrational-rotational bands from the first positive series molecular $B\,^3\Pi_g-A\,^3\Sigma_u$ transition. The metastable $A\,^3\Sigma_u$ state (denoted N_2^*) is 6 eV above the N_2 ground state and is believed to be the main active species in the growth of GaN.[7] In the on-resonance mode, the emission spectrum also shows sharp atomic N lines that increase in intensity with P_L. The line at 868 nm in Fig. 4 is an unresolved eight-line multiplet from the $3s^4P-3p^4D^0$ transition in N atoms and is used to infer the relative amount of atomic N in the plasma. Similarly the intensity of the molecular 888 nm line is taken as a measure of the relative abundance of N_2^*. Although there are obvious difficulties with making quantitative inferences about the abundances of the different chemically active species from the plasma emission nevertheless the relative intensities of the atomic and molecular emission spectra should provide a useful indication of the relative abundances of atomic and molecular species.

Figure 5 shows how the atomic and molecular emission changes with net absorbed power. The off-resonance mode shows weak atomic emission and increasing molecular emission over the whole range of power (27–85 W). For on-resonance operation the atomic emission is much stronger and approximately proportional to the power whereas the molecular emission is weakly dependent on power and trending down. We interpret the decrease in the molecular emission at high power in the resonant operation as an indication that the molecular emission is being depleted by the dissociation of molecules into atoms. If we assume that the molecular and atomic emissions are an indication of the relative abundances of the chemically reactive molecular (N_2^*) and atomic species, respectively, then the two operating modes of the plasma source allow us to explore the effect of the two different species on the incorporation of nitrogen into the dilute nitride alloys and to find out whether one species is preferable to the other from the perspective of the electronic properties.

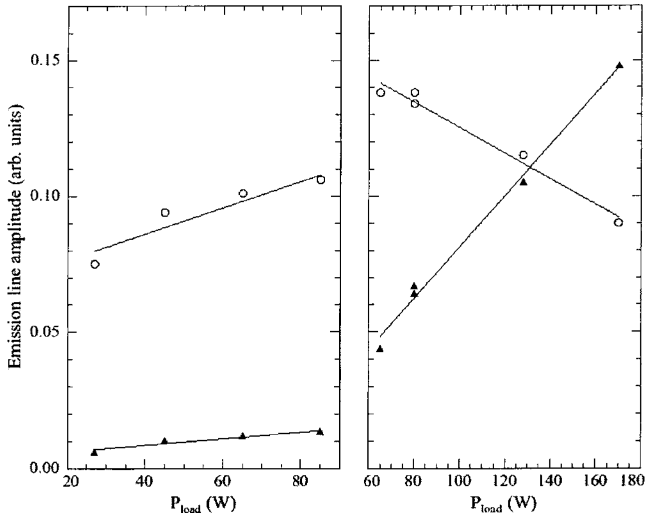

FIG. 5. Emission intensities for atomic N (868 nm line with background from molecular emission subtracted) (▲) and N_2^* (888 nm line) (○). Left plot is measured with the plasma operating in the off-resonance mode; the right plot in the on-resonance mode.

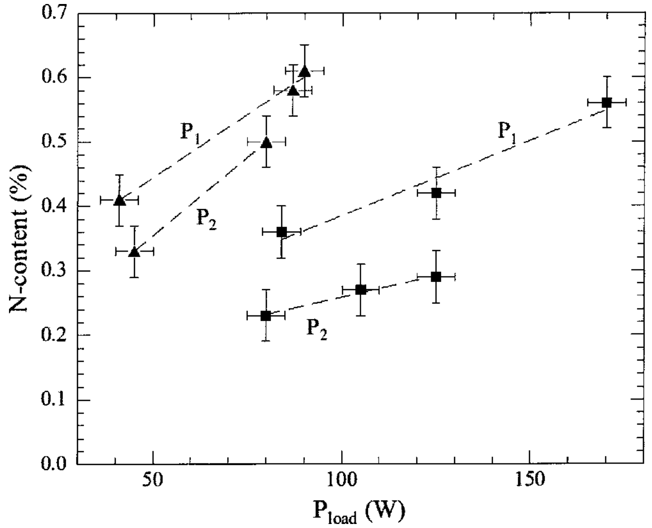

FIG. 6. N content of on-resonance (■) and off-resonance (▲) grown samples as determined by XRD for varying power levels (P_{load}) and pressures. $P_1 = 5 \times 10^{-6}$ mbar and $P_2 = 4 \times 10^{-6}$ mbar.

IV. SAMPLES

In order to explore the possibility that growth with atomic N or excited state molecular nitrogen might affect the electronic quality of the semiconductor, a series of $GaAs_{1-x}N_x$ dilute nitride samples was grown with the two different operating modes of the helical resonator (on and off resonances) on GaAs (001) substrates. A GaAs buffer layer approximately 350 nm thick was deposited on a thermally deoxidized substrate at a rate of 1 μm/h, followed by a 200 nm $GaAs_{1-x}N_x$ top layer grown at a rate of ~0.5 μm/h with a N_2 background pressure of $(4-5) \times 10^{-6}$ mbar corresponding to a N_2-flow rate of 0.10–0.14 SCCM (SCCM denotes cubic centimeter per minute at STP). An As_2 to Ga flux ratio of 8:1 was maintained throughout the growth, and the substrate temperature was held at 450 °C during the dilute nitride growth. The N content and thickness of the $GaAs_{1-x}N_x$ layer were determined using high resolution x-ray diffraction (XRD). The nitrogen concentrations were sufficiently low (<1.4%) so that all the films were pseudomorphically strained to the substrate.

V. RESULTS AND DISCUSSION

The N concentration as a function of the net rf power to the source is shown in Fig. 6 for pressures of 4×10^{-6} and 5×10^{-6} mbar. Increasing the pressure gives more N incorporation in both modes of operation consistent with the observed increase in emission intensity from both N_2^* and N atoms with increasing pressure. The nitrogen content also increases with rf power for both operating modes but does not extrapolate to zero at zero power over the measured power range. In this respect the nitrogen concentration more closely tracks the molecular emission, which also does not extrapolate to zero at zero power, unlike the atomic emission. Films grown at the same power in the off-resonance mode (N_2^*-rich mode) have a N content approximately a factor of 2 higher than films grown in the on-resonance mode (N atoms). The power dependence of the nitrogen content and the fact that the nitrogen content is higher in the molecule-rich mode both suggest that the excited molecular species N_2^* is more important than atoms for the incorporation of nitrogen. Carrère *et al.*[8] also conclude that N_2^* is the active species but only at flow rates above 0.1 SCCM whereas atomic N dominates the growth at low pressures. Our flow rates are close to this threshold (0.1–0.14 SCCM).

A gradual decrease in the amount of nitrogen incorporation was observed over a period of several years of operation of the plasma source. When the source was disassembled a gray deposit was observed inside the discharge tube and in the baffle. This deposit is assumed to be arsenic, which we would expect to poison the discharge tube. During the dilute nitride growth there is a background pressure of $\sim 10^{-7}$ mbar of As_2 and As_4, which normally have a low sticking coefficient on the walls of the vacuum chamber. We speculate that when the plasma source is operating the As_2/As_4 diffuses through the baffle into the plasma discharge where it can be dissociated or otherwise chemically activated so that it will stick to the wall. A related effect is observed on windows in the growth chamber that are exposed to UV light: over a period of time an As coating develops on the part of the window that is exposed to UV light.

After cleaning the discharge tube and baffle in aqua regia (HNO_3:HCl 1:3) the efficiency of N incorporation increased by a factor of ~3, as shown in Fig. 7. In this figure the N content in the samples grown with the clean tube has been scaled to eliminate the effects of different gas flows (additive corrections of 0.04%–0.09% N), different Ga fluxes (factor of 2.3), and a geometrical factor associated with moving the source closer to the substrate (factor of 0.44). The net effect of cleaning the tube was to increase the N incorporation by factors of 3.5 and 2.3 for on and off-resonance growths, respectively, when all other conditions were kept fixed. It is

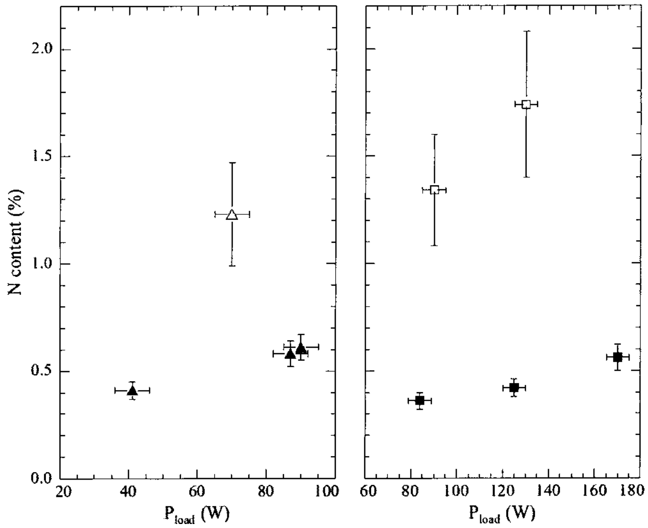

FIG. 7. Effect of As coating of the PBN discharge tube on the N concentration for on-resonance (■ and □) and off-resonance (▲ and △) operations. The samples represented by the solid markers are the samples in Fig. 4, grown at 5×10^{-6} mbar (P_1). The hollow symbols are samples grown after the removing the As coating. The N concentrations have been scaled to eliminate differences caused by (a) differences in growth rate, (b) changes in source to substrate distance, and (c) differences in gas flow for the growths after the discharge tube was cleaned.

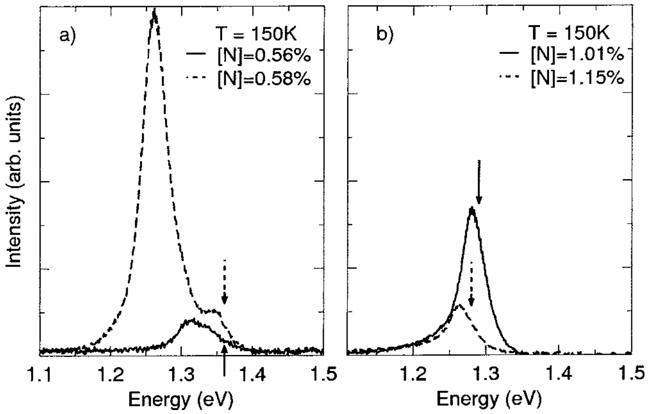

FIG. 8. Photoluminescence spectra measured at 150 K for GaNAs samples grown with an As coated pyrolytic boron nitride discharge tube (a) and with a clean discharge tube (b). Samples grown with the plasma source operating on resonance are indicated by solid lines and off resonance with dashed lines. The N concentrations determined by x-ray diffraction are indicated in the figures. The arrows indicate the band gaps estimated from the N concentrations (Ref. 9). All of the samples are as grown, with no postgrowth annealing.

possible that the As coating on the discharge tube changes the relative abundance of the atoms and excited state molecules. However, this is unlikely to be a large effect as the power dependence of the plasma emission spectrum was similar before and after cleaning. We conclude that arsenic deposits on the walls of the discharge tube and baffle poison the source by catalyzing recombination of atoms and/or deactivating N_2^*.

Photoluminescence (PL) measurements were performed on the dilute nitride samples using a 523 nm pulsed (20 ns) frequency-doubled, diode-pumped YLF laser operated at a repetition rate of 400 Hz. This yielded an average power of 150 μW available to excite the GaNAs samples. Photoluminescence spectra measured at 150 K for unannealed samples grown on and off resonances with the As-contaminated discharge tube are shown in Fig. 8(a) and with the clean discharge tube in Fig. 8(b). All four spectra in Figs. 8(a) and 8(b) were measured with the same collection efficiency so that the relative intensities are accurately indicated. The sample with the strong in-gap emission in Fig. 8(a) has stronger PL in the case of the As-contaminated discharge tube and the sample with the lower nitrogen concentration has the stronger emission in Fig. 8(b) in the case of the clean discharge tube. Several samples were grown in the off-resonance mode and six in the on-resonance mode with the As coated discharge tube, with nitrogen concentrations ranging from 0.19% to 0.74%. Similarly we grew 18 samples on resonance and 10 off resonance with nitrogen concentrations ranging from 0.28% to 5.5% after cleaning of the discharge tube; PL spectra in Fig. 8 are representative of these two groups of samples. Figure 9 shows PL peak energies at 150 K for several samples from these groups, along with the band gap from Tisch *et al.*[9] adjusted for the 150 K measurement temperature, as a function of nitrogen content. Several of the samples in Fig. 9 with PL peak energies well below the band gap show a double peak structure similar to the spectrum in Fig. 8(a). The nitrogen concentrations for the samples in Fig. 9 were determined from the separation between the [004] diffraction peak of GaAs and the split-off peak from the pseudomorphically strained GaNAs epilayer, using 526 arc sec/1% N as a calibration factor.[8,10] This calibration factor is close to the value one would expect for a linear interpolation of the lattice constant between GaAs and GaN, following Vegard's law.[9] A typical high resolution

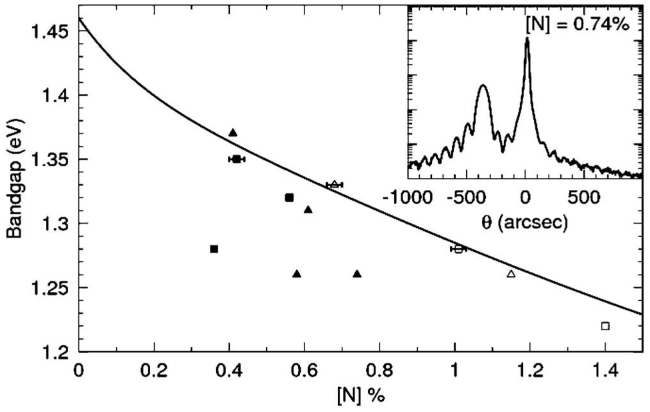

FIG. 9. Photoluminescence peak energies measured at 150 K as function of [N] for on resonance (■ and □) and off resonance (▲ and △); samples represented by solid markers were grown with an As-contaminated discharge tube, the open circles with a clean discharge tube. The solid line shows the expected band gap adjusted to the 150 K measurement temperature. Error bars are shown for three representative samples, [N]±0.02%, based on fits to high resolution x-ray diffraction scans. A representative XRD scan is shown in the inset for a sample with [N]=0.74%.

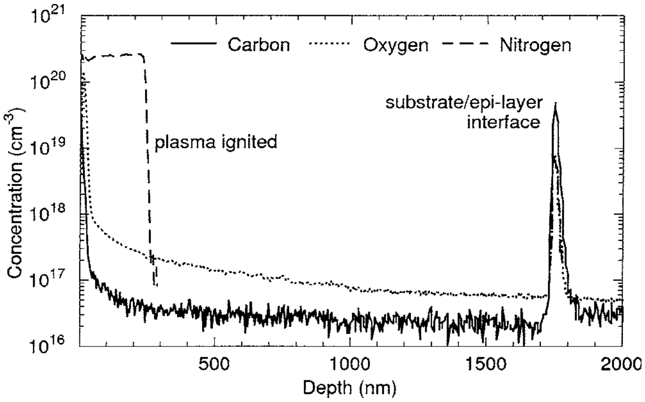

FIG. 10. SIMS depth profile of GaNAs sample. The slow decay of the carbon and oxygen concentrations with depth suggestions that these elements result from sample surface contamination after growth. For depths lower than the epilayer nitrogen is below the detection limit, 3×10^{16} cm^{-3}.

[004] x-ray diffraction scan is shown in the inset in Fig. 9 for one of the samples. The clear Pendellosung fringes are indications that the epilayer has not relaxed.

In the PL spectra for the samples in Figs. 8(b) and 9 we see no double peak structure for any of the samples grown with the clean discharge tube. In the case of the As coated discharge tube there is a tendency towards more in-gap emission (and double peaks) for the samples grown in the off-resonance mode (molecular emission dominates in the plasma), although this trend is not definitive. The in-gap emission is an indication of shallow states in the band gap associated with defects or N clustering. The PL quantum efficiency is sensitive to the density of recombination centers. The quantum efficiency shows a trend to lower efficiency with increasing [N], but with considerable scatter. The samples with strong in-gap emission also showed higher efficiency but otherwise we observed no difference between the PL quantum efficiencies for samples grown with the atom or molecule-rich plasma conditions.

Several samples were analyzed with secondary ion mass spectroscopy (SIMS) to explore the possibility that the above mentioned changes in the PL spectra are due to oxygen or other contamination associated with the As coating on the discharge tube. A number of samples were depth profiled for gallium, arsenic, nitrogen, oxygen, carbon, boron, and silicon. Depth profiles for carbon, oxygen, and nitrogen are shown in Fig. 10 for one sample. There is no indication of an increase in the carbon or oxygen concentration when the plasma source is turned on, at the 10^{16} cm^{-3} level. In fact, no increase in any of the impurities was observed when the plasma source was turned on, except for N, for samples grown with the As coated discharge tube or with the clean tube. We conclude that the samples were not contaminated by the coating on the plasma discharge tube. The slow decay in the oxygen concentration with depth in Fig. 9 is believed to be due to contamination on the surface of the sample that occurred after growth. In all cases the boron concentration was below the detection limit (10^{17} cm^{-3}). The substrate epilayer interface can be clearly seen from the large amounts of oxygen and carbon at a depth of 1800 nm.

It is possible that the degradation of the PL spectra for the samples grown with the As coated discharge tube is caused by incorporation of reactive AsN species produced in the plasma source. In fact, AsN is observed with a mass spectrometer when the plasma source is operating, although we cannot tell whether it originates from the plasma source or from a surface reaction on the As coated walls of the growth chamber. Possibly this species contributes to the formation of N-related defects such as N interstitials, split interstitials, or As antisites.

VI. CONCLUSIONS

We have grown the dilute nitride semiconductor $GaAs_{1-x}N_x$ by plasma-assisted MBE using a helical resonator plasma source operating in the 140–200 MHz frequency band. The high fields present in the unloaded resonator mean that the plasma discharge will start at standard operating pressures, eliminating the need for an inert carrier gas or shutters to cycle the flux on and off in the growth of quantum wells. The plasma source can be operated at two different frequencies, with different relative abundances of nitrogen atoms and molecules as determined from the emission spectrum of the discharge. Operating at the helix resonance favors the production of N atoms, while operating away from the helix resonance favors the production of excited state N_2 molecules. For the same rf power delivered to the plasma, the operating mode that favors molecules shows twice as much nitrogen incorporation for the same rf power delivered to the discharge. This suggests that excited state molecules are incorporated more efficiently than atomic nitrogen.

Over several years of operation an As coating developed on the inside of the discharge tube, which reduced the flux of active nitrogen from the source. Films grown with the As-contaminated discharge tube showed strong below band gap photoluminescence associated with shallow gap states. This suggests that reactive AsN or other As-related species produced in the plasma create electronic states in the band gap. For the clean discharge tube the PL spectra were similar for films grown in both the atom-rich and molecule-rich growth modes and showed primarily emission at the band gap. We conclude that changing the ratio of atomic nitrogen to excited state molecular nitrogen in the discharge has no effect on the density of shallow defect states in the band gap according to the 150 K PL data, even though this measure of the density of shallow defect states is affected by As contamination of the discharge tube.

We are unable to draw any conclusions about the electronic quality of the dilute nitride material grown with the helical resonator source relative to material grown with an inductively coupled plasma source.

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