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## MBE growth of high quality GaAsN bulk layers

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**Abstract.** In the present work we have carefully optimized the operation of plasma source and the growth parameters of GaAsN layers. We have demonstrated the possibility of incorporation about 1.5% of N into GaAs without decreasing the photoluminescence intensity.

### 1. Introduction

Presently one of the main directions of III–V compound epitaxy is the development of long-wavelength GaAs-based structures. 1.3  $\mu\text{m}$  VCSEL pseudomorphically grown on GaAs substrate can realize the low-cost light source required for optical local area network. First demonstrations of the 1.3  $\mu\text{m}$  GaAs-VCSELs based on InAs QDs and InGaAsN QWs were presented in [1] and [2], respectively. The most outstanding results for edge emitting lasers were achieved by MBE grown InGaAsN QWs using RF (radio-frequency) plasma nitrogen source [3]. So far the same team has presented the best characteristics of 1.3  $\mu\text{m}$  VCSEL [4].

However, in spite of extensive investigation of the growth condition and post-growth annealing, there is a wide dispersion of the published characteristics of InGaAsN QW lasers. This variation indicates that the technology of InGaAsN-based lasers is still immature compared with the InGaAs-based counterparts. Presumably this situation can be attributed to different nitrogen sources and plasma conditions, which are used to grow (In)GaAsN. Nevertheless, little attention is paid to the study of the effect of plasma condition on (In)GaAsN quality. Defects from ion damage, impurities coming from the plasma source, and non-radiative recombination fundamental to the band structure are some of the accepted reasons to explain the decreasing efficiency of the radiative recombination even in the case when N content is less than 1–2%. However, in the present work we have achieved highly efficient radiative recombination in GaAsN layers by optimizing the RF plasma nitrogen source operation, growth regimes, and post-growth annealing.

### 2. Experiment

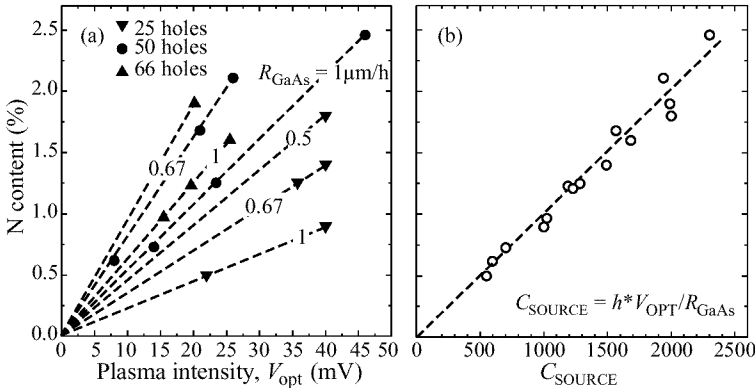
In our experiments we used Riber Epineat apparatus equipped with conventional effusion cells for group III elements and arsenic. UNI Bulb RF Plasma Source produced by Applied EPI was used to generate active species of N. Samples under investigation were grown on  $n^+$ -(001) GaAs substrate. A special structure design with the layer to be studied sandwiched between short period AlGaAs/GaAs superlattice (SL) was used for PL studies. We varied the growth temperature and growth rate of the core layer whereas substrate temperature of the rest of the structure was set at 600 °C.

The crystal quality of the samples was evaluated by x-ray diffraction measured with incident beam in the [004] and [511] azimuths. PL measurements were carried out using a doubled frequency YAG:Nd solid state laser ( $\sim 40 \text{ W/cm}^2$ ) and CCD a (Charge Couple Device) spectrometer.

To clarify the effect of plasma source operation we chose the simpler GaAsN composition rather than the highly strained InGaAsN QW. Effects related to 2D–3D growth mode transition and the presence of high strain could complicate our study.

### 2.1. Effect of aperture design

The first set of GaAsN layers was grown at 470 °C using different aperture design. In a simple way, ignition of plasma in N<sub>2</sub> gas flow leads to formation of N atomic species as well as excited molecules and ions of N<sub>2</sub>. The former incorporate into growing film whereas the latter lead to non-radiative defect formation. Thus, the quality of the layer is expected to be dependent on plasma composition, which can be affected by the aperture layout. Three apertures with different conductance ( $\varnothing$  200  $\mu$ m, number of holes,  $h = 25, 50, 66$ ) were used. The intensity of plasma can be controlled by the RF power and the flow rate of N<sub>2</sub>. Figure 1(a) shows the dependence of nitrogen content,  $y$ , in GaAs<sub>1-y</sub>N<sub>y</sub> layers on plasma light intensity as measured by a photodetector voltage,  $V_{OPT}$ . In Figure 1(b) we showed N content versus parameter  $C_{SOURCE}$ , which is definite as  $h \times V_{OPT}/R_{GaAs}$ , where  $R_{GaAs}$  is the growth rate. It is clearly seen that the dependence of  $y$  on  $C_{SOURCE}$  is linear. Thus,  $C_{SOURCE}$  is an universal parameter responsible for N content in the growing film.

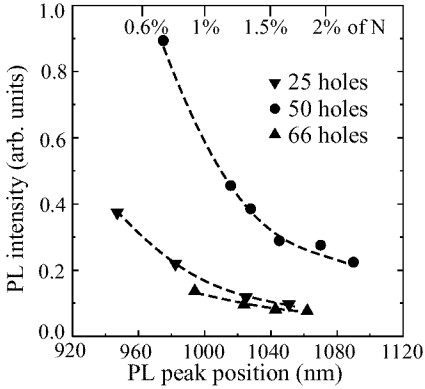


**Fig. 1.** Dependence of N composition of GaAsN layers on plasma conditions. (a) N content vs plasma light intensity at different GaAs growth rate and different aperture layouts, (b) N content vs parameter  $C_{SOURCE}$ .

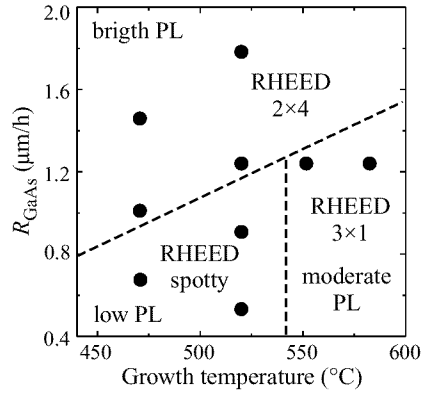
Figure 2 shows the dependence of PL intensity of 0.2  $\mu$ m-thick GaAsN layers on the PL peak position and nitrogen composition. All samples were grown under the same growth conditions. N<sub>2</sub> flow rate was tuned to get the lowest RF power at a given plasma intensity. One can see that we can obtain the required nitrogen composition for any aperture. However, the optical quality of the samples is quite different. Using the 50-hole aperture allowed us to improve several folds the PL intensity of GaAsN layers. All following growth experiments were done using the 50-hole aperture.

### 2.2. Effect of growth temperature and growth rate

We found that in our case the sticking coefficient of nitrogen is temperature independent from 430 to 550 °C and decreases at higher temperatures. GaAsN (1.3%) samples grown within the 520–540 °C temperature range demonstrated the brightest and narrowest PL spectra. At temperatures below 540 °C the  $2 \times 4$  reconstruction of RHEED pattern was similar to the case of the GaAs growth. However, we observed  $3 \times 1$  reconstruction when substrate temperature was higher than 550 °C. This fact is presumably due to N segregation



**Fig. 2.** Dependence of the PL intensity of GaAsN layers on wavelength for different aperture layouts.



**Fig. 3.** “Growth temperature/growth rate” diagram of RHEED pattern. Points indicates samples grown at given growth rate and temperature.

on the growing surface [5]. This is in good agreement with PL and XRD measurements, which showed the presence of N-rich regions in grown films.

We did not find strong dependence of GaAsN quality on V/III ratio within typical range.

We observed the strong dependence of GaAsN properties on growth rate. When growth rate became lower than a certain value, the growth mode changes from 2D to 3D and N-rich clusters are formed. This minimum growth rate depends on temperature. Figure 3 showed “temperature-rate” phase diagram of RHEED pattern during the growth of GaAsN layers with N content about 1.3%. The upper region corresponds to normal GaAs-like growth mode and bright PL intensity. Both of the bottom regions correspond to the growth modes leading to fluctuation of N content in layers and lower PL intensity. So, we can conclude that there is some region of high “growth rate/growth temperature” that is free of the effects related to phase separation during the growth of GaAsN.

2.3. *Effect of plasma operation condition*

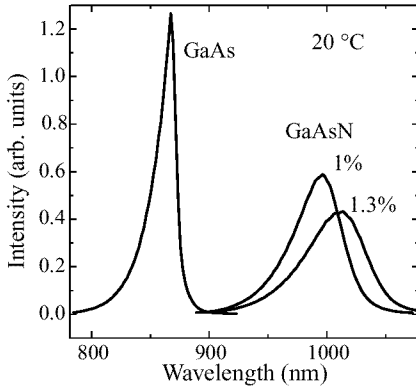
As we mentioned above one can tune the flow rate of nitrogen gas to get the lowest RF power at a required plasma intensity. However, we found that the plasma operation with lower nitrogen flow rate and therefore higher RF power is preferable to achieve brighter PL intensity. We also developed a “pre-operation” procedure to further enhance PL intensity. This procedure is the operation of plasma source at high flow rate and high power before growth run.

2.4. *Comparison of GaAsN with GaAs layers*

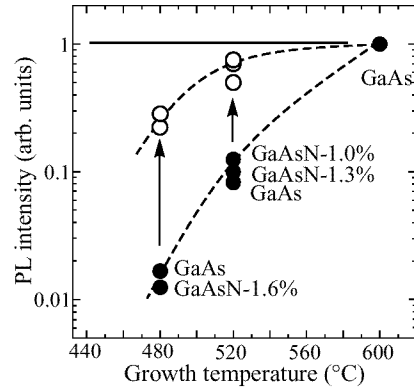
Figure 4 shows PL spectra of GaAsN(1%), and GaAsN(1.3%) grown using optimized conditions. The spectrum of GaAs grown at the same conditions is presented for comparison. Using AlGaAs/GaAs SLs allowed us to avoid leakage of non-equilibrium carriers into the substrate and surface and thereby obtain equal effective pumping of GaAs and GaAsN layers. The growth temperature was set at 520 °C and V/III ratio was about three. Adding N leads to strong red shift and broadening of PL line whereas integrated PL intensity remains almost the same.

2.5. *Effect of annealing*

We have also investigated the effect of in situ annealing on our GaAsN layers. We found that the maximum improvement in intensity for a given sample varied from 3 to 100 times



**Fig. 4.** PL spectra of GaAs and GaAsN layers.



**Fig. 5.** Growth temperature dependence of integrated PL intensity of GaAs and GaAsN layers as grown (solid circles) and after annealing (open circles).

depending on its initial quality. Figure 5 shows dependence of integrated PL intensity of the GaAsN and GaAs layers grown at different growth temperatures. The solid horizontal line shows intensity of GaAs grown at 600 °C, which is an optimal growth temperature for GaAs. One can see that PL intensity depends on growth temperature but not on the presence of N in the layers. The point defects related to low temperature growth can be removed by annealing. The PL intensity of samples after annealing is shown as open circles. For samples grown at 520 °C and annealed for 2 hours at 750 °C under arsenic overpressure we achieved almost the same level of PL intensity as GaAs grown at 600 °C.

### 3. Conclusion

The quality of GaAsN layers is very sensitive to the plasma source operation conditions and growth temperature and growth rate. Detailed optimization of these parameters and annealing regime allowed us to achieve very high optical quality GaAsN layers with N composition about 1.5%.

#### Acknowledgements

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