

Annealing effects on the nanoscale indium and nitrogen distribution in Ga(NAs) and (GaIn)(NAs) quantum wells

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(Received 25 April 2007; accepted 21 August 2007; published online 16 October 2007)

III/V semiconductors containing dilute amounts of nitrogen are metastable and need to be thermally treated after growth to optimize optoelectronic properties. The influence of thermal annealing on the nitrogen depth profile in metal organic vapor phase epitaxy-grown Ga(NAs)/GaAs as well as (GaIn)(NAs)/GaAs heterostructures is examined on a nanometer scale by combining several high resolution transmission electron microscopy techniques, also with Rutherford backscattering spectrometry. Annealing conditions, which are optimized for quaternary alloys with respect to photoluminescence intensity, do not result in element redistribution for the In containing material. Contrary to the quaternary material, the result of annealing the ternary Ga(NAs) is a pronounced pileup of the nitrogen profile without any out diffusion of nitrogen. These findings have important influence on device structures, which often contain Ga(NAs) barriers for strain-compensation purposes together with (GaIn)(NAs) active regions. In the light of metastability considerations for the ternary and quaternary alloy, one can conclude that the In contained in the quaternary material stabilizes the material and suppresses phase separation. Consequently (GaIn)(NAs) is more stable than its ternary counterpart Ga(NAs). © 2007 American Institute of Physics.

[DOI: [10.1063/1.2794739](https://doi.org/10.1063/1.2794739)]

INTRODUCTION

Mixed III-V nitride semiconductor alloys have a theoretically predicted^{1,2} and experimentally observed large bandgap bowing, which would allow 1.3–1.55 μm wavelength emission based on GaAs substrates. Wavelengths between 1.3 and 1.6 μm are of importance for applications in optical local area networks, metro area networks, and long haul communications due to the minimum losses in optical fibers in these wavelength regions. Further applications of this quaternary material include a 1 eV bandgap material, which can be grown lattice matched to GaAs or Ge to serve as one of the absorbing materials in terrestrial as well as space multijunction solar cells.³

Because of the large miscibility gap of (GaIn)(NAs), the growth of high-quality structures such as multiple quantum well devices requires a detailed understanding of the composition of the alloy as well as of phase separation effects. It has been shown that N concentrations above 3%–4% (Ref. 4) cannot be achieved in the quaternary material system without significant deterioration of the crystal structure. This seems to be an intrinsic material property and is found in both molecular beam epitaxy and metal organic vapor phase epi-

taxy (MOVPE) grown materials.⁵ On the one hand, the material crosses the border from the metastable to the unstable regime, and on the other hand, strain limits the maximum accessible In content in the quantum wells.

Another characteristic property of the material system is that it has to be thermally annealed before it can be used for device applications, as photoluminescence (PL) intensity drastically improves as well as localization can be minimized after an appropriate thermal treatment.^{5,6}

Moreover, devices employing (GaIn)(NAs) as an active region are often characterized by the insertion of Ga(NAs) barriers instead of GaAs barriers, mainly to realize strain compensation for the strongly compressively strained active material. The barrier material is consequently treated with the same annealing procedure as the active region of the devices.

The topic of the present paper is to gain quantitative information on the composition of the quaternary and ternary alloys (GaIn)(NAs) and Ga(NAs) before and after annealing with nanometer resolution as both material systems are metastable and, hence, element redistribution as well as phase separation might result if specific annealing conditions are applied.^{7,8} These annealing conditions depend strongly on growth conditions and are mainly optimized for the PL efficiency of the (GaIn)(NAs) and little attention has been paid so far to the effect of these annealing conditions on the Ga(NAs) barrier material.

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EXPERIMENT

The samples used for this study have been grown by MOVPE in a commercial reactor system (AIX200) at a reduced pressure of 50 hPa in hydrogen carrier gas. Owing to the metastability of the material systems under investigations, comparatively low growth temperature of 525 °C has been used. The deposition temperature for all samples was calibrated to the Al/Si eutectic formation at 577 °C. As a consequence of the low growth temperatures, metal organic sources were used, which show a significant decomposition already at these temperatures. For the group V elements tertiary butyl arsine (TBAs) and 1,1-dimethyl hydrazine (UDMHy) were employed rather than the more common hydride gases. The group III MO sources were triethyl gallium (TEGa) and trimethyl indium (TMIn). The growth rate for all samples under investigation here was set to 1 $\mu\text{m}/\text{h}$. To achieve the desired nitrogen fraction in the crystal, group V pressure conditions have been adjusted in the reactor accordingly. Details of the growth and elemental incorporation characteristics of the dilute nitride material system grown by MOVPE can be found in a separate paper.⁹

For comparison of the annealing behaviour of quaternary (GaIn)(NAs) and ternary Ga(NAs), different superlattice layer structures have been grown containing the respective materials as thin (10–20 nm) layers separated from each other by at least 100 nm thick GaAs regions. Those samples were analyzed as grown and after annealing. The annealing conditions were chosen as to optimize laser properties. Annealing takes place *in situ* and consists of a 5 min long TBAs stabilized step at 700 °C, followed by a further annealing step at 625 °C, 25 min long, and unstabilized in hydrogen atmosphere. After this annealing procedure laser material has shown to have maximum PL efficiency increase as well as the blueshift of the bandgap, which is a further intrinsic property of the (GaIn)(NAs).¹⁰ The blueshift has been shown to saturate at a certain level.¹¹

To have the possibility to assess the In depth profile in the quaternary alloy separately from the N depth profile, Rutherford backscattering spectrometric (RBS) measurements have been undertaken at the (GaIn)(NAs) layer before and after the annealing procedure. A 3.5 MeV He ion beam from a van de Graff accelerator has been impinged on the sample at an angle of 10° off the surface normal to enhance depth resolution and minimize channelling effects. The backscattered particles have been detected energy sensitively using a time of flight detector set at 170°. Depth profiles have been obtained from the RBS spectra by applying the computer code RUMP,¹² assuming the atomic density of GaAs (4.4×10^{22} at./cm³).

If no resonance reactions are used, nitrogen in GaAs based materials is not easily accessible in RBS analysis. Therefore, a different approach has been undertaken to access the N depth profile. Quantitative N depth profiles from the structures before and after annealing have been retrieved with nanometer resolution by quantitatively analysing cross sectional transmission electron microscopic (TEM) specimens.

TEM sample preparation has been done conventionally,

lapping down the cross sections with diamond polishing paste to a final thickness of 50 μm and, subsequently, Ar ion sputtering the sample until electron transparency. As the incorporation of N significantly hardens III/V semiconductors, special care has to be applied in this sputtering step, not to yield samples having thickness fluctuations in the electron transparent regions, especially across the quantum well. This can be avoided¹³ if the ion incident angle is set to below 3° at an acceleration voltage of 3 keV. A final ion polishing step at 1.2 keV ion energy is applied.

To gain the desired information, the composition evaluation by lattice fringe analysis (CELFA) technique¹⁴ has been utilized, viewing the cross sectional samples along the [010] zone axis. A Philips CM200/FEG at an energy of 200 kV has been used for TEM analysis. To obtain the lattice fringe images at conditions least sensitive to, e.g., sample thickness fluctuations, the samples have been tilted away from exact zone axis orientation by several degrees so that the center of Laue circle was at (0,20,1.5). The lattice fringe image is acquired by selection of the chemically sensitive (002) beam together with the undiffracted (000) beam, shifting the (002) beam to the optical axis of the microscope.

As control measurement, the tetragonal distortion of the quaternary quantum wells was mapped under high resolution conditions. The strain value obtained can be compared to the composition of the material derived from the previous analysis. Hence, two independent techniques are presented to derive quantitatively the In and N contents separately. The first one relies on deriving the In content independently of the N content by RBS and using this In content as input for the CELFA analysis. The second one uses the knowledge of the tetragonal distortion of single lattice planes in combination with local image intensity from CELFA. This technique is the most convenient if the alloy under investigation is nearly lattice matched to the substrate, as effects of thin TEM foil relaxation then do not need to be taken into account. If this is not the case, strain mapping from high resolution images has to be accompanied by finite element simulation of the sample relaxation geometry. This requires the knowledge of the exact TEM sample thickness at the position of the measurement. Hence, in this case, the method combining RBS and TEM is more straightforward.

We have shown that the structure factors of N containing materials have to be recalculated¹⁵ from their theoretical values, if the intensity from the so-called chemical sensitive reflections, such as, e.g., the (002), is used to derive the composition and as N introduces significant static atomic displacements¹⁶ to the average cubic zinc blende lattice. Where quantitative nitrogen concentrations are given in the following, the corrected structure factors have been used for the evaluation of the CELFA images.

RESULTS AND DISCUSSION

In the following, the N depth distributions across as grown as well as annealed Ga(NAs) and (GaIn)(NAs) quantum wells will be determined quantitatively. The effect of

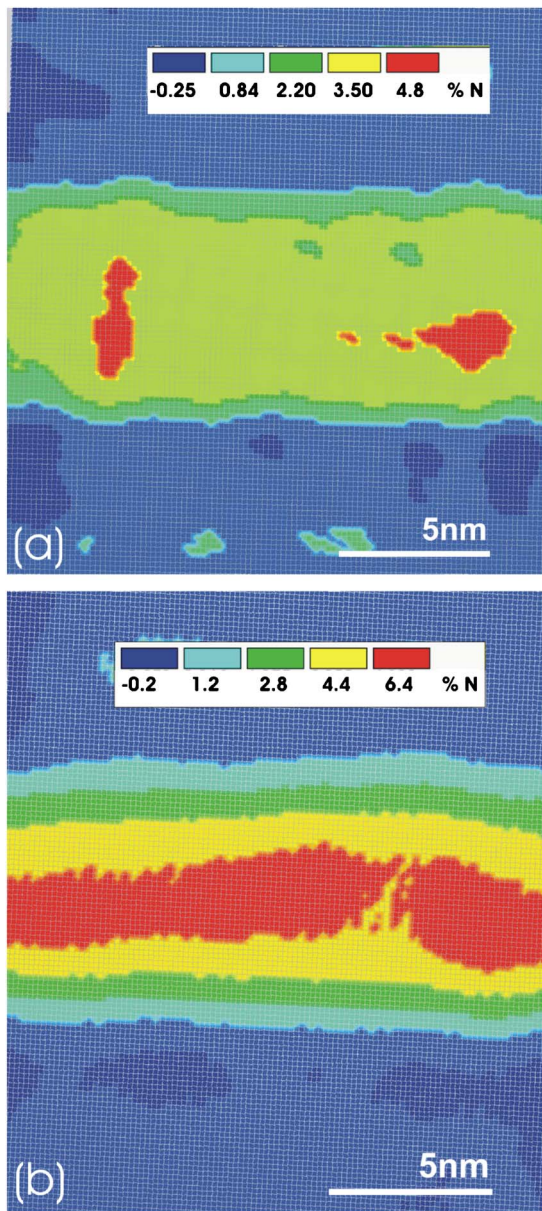


FIG. 1. (Color online) Nanoanalytical nitrogen map throughout a Ga(NAs) quantum well before (a) and after (b) annealing.

annealing on the composition of the ternary as well as quaternary materials on a nanometer scale will be presented and discussed.

The color-coded N concentration map across an as grown Ga(NAs) quantum well is shown in Fig. 1(a). One observes a rather homogeneous plateau with constant N concentration with some regions with slightly higher N content. The width of the interface of the quantum well, where the N concentration drops from above 3% to 0% is about 4 ML (monolayers). The quantum well is characterized by a physically rough interface to the GaAs barriers with Ga(NAs) islands having a diameter of several nanometers. The diameter of the islands as obtained from these cross section TEM images is in good agreement with the diameter of islands observed on atomic force microscopic images of interior Ga(NAs) interfaces.¹⁷

In Fig. 1(b), the N map of the same quantum well after

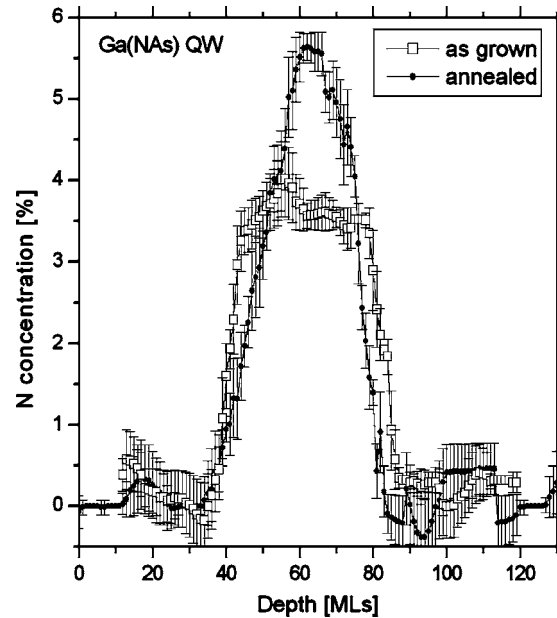


FIG. 2. Comparison of the nitrogen depth profile in a Ga(NAs) quantum well before and after annealing.

thermal treatment is shown. One observes a clear trend toward the pileup of the formerly flat N profile toward the center of the quantum well, where the N content is now significantly higher compared to the as grown sample. The interfaces of this quantum well are characterized by a chemical roughness rather than a physical one, i.e., a N concentration gradient. We find an increasing trend toward the pileup of the N profile with increasing N content in the structure for the ternary material. Information on the overall nitrogen content in the quantum well before and after annealing can be obtained from the nitrogen depth profile (Fig. 2) derived from these N maps. Averaging the N content took place across a 20 nm wide region of the quantum well. The error bars indicate some statistical fluctuations of the image intensity as well as minor TEM specimen thickness fluctuations within the averaged region. Integrating the N content across the N rich regions before and after annealing proves that the overall N content in the samples was not altered upon annealing and that, hence, neither N out diffusion nor activation of interstitial N to lattice sites¹⁸ has taken place for the MOVPE grown material. In summary, for the ternary Ga(NAs), we observe a homogeneous nitrogen distribution in the quantum wells after growth. The nitrogen profile piles up upon annealing. This finding cannot be explained by a classic diffusion behavior, rather the microscopic onset to N phase separation is observed.

Quantification of the composition from single TEM images using the chemically sensitive (002) reflection is complex for quaternary (GaIn)(NAs) if independent quantification of the N as well as In contents is aimed for on a nanometer scale. There is an infinite number of quaternary compositions for a single (002) beam intensity in TEM dark field. Hence, the problem is underdetermined, as one has solely measured one quantity for two unknown quantities. Therefore, either one, the In or N depth profile, has to be

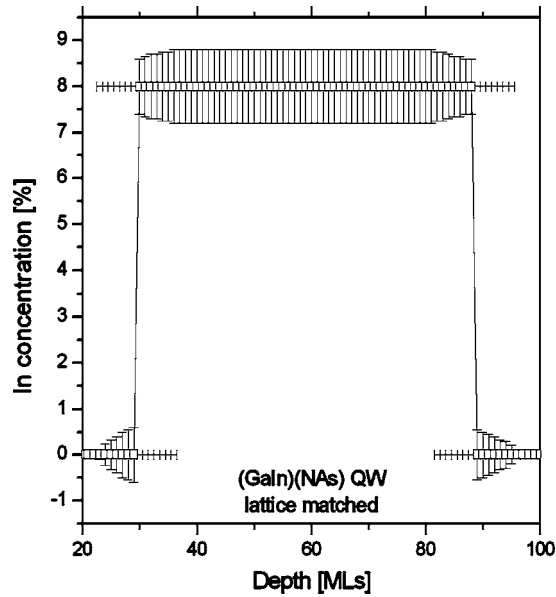


FIG. 3. Indium depth profile in a (GaIn)(NAs) quantum well before and after annealing (from RBS measurements and RUMP simulation to the RBS spectra).

used as an input parameter for the CELFA analysis. Then, the quantification of the remaining element is possible.

It is rather important to derive the In content of the (GaIn)(NAs) quantum wells independently of the N content but for the same quantum well, which is used later on for the quantification of the second element, as under certain growth conditions, N incorporation influences the In content in the material and, hence, (GaIn)As reference layers cannot be used as calibration for the In for the later stage analysis of quaternary films.

One method to derive the In content in the quantum well independently of its N content is RBS. In Fig. 3, the In content from RUMP simulation to the RBS spectra of a quantum well containing nominally 8% In and 3% N is depicted before and after annealing. A rectangular In profile has been assumed. RBS in the setup used here allows very accurate measurement of the overall In composition of the thin structures with an error bar of less than 3% of the total In content measured. Hence, the In content in the (GaIn)(NAs) quantum well is determined to be $(8 \pm 0.3)\%$. It does not change upon annealing. It has been tested in how far deviations from this rectangular profile would be detectable from this kind of measurement by performing RUMP simulations to the same RBS spectrum and checking the influence of differently inhomogeneous layer compositions. We conclude from that study that deviations from the rectangular profile will be detectable if they are larger than $0.01 = \Delta \text{In} \Delta d$, with ΔIn indicating the variation of the In content (normalized to its nominal value) and Δd being the intermediate layer thickness, normalized to the complete quantum well thickness. As an example, for a 15 nm thick (GaIn)(NAs) quantum well having a nominal In content of 8%, we would be able to resolve deviations from the assumed rectangular profile if they are larger than 0.8% in absolute In content over a layer thickness of 1.5 nm. The fact that the assumption of no compositional fluctuation of the In profile is justified will also become clear

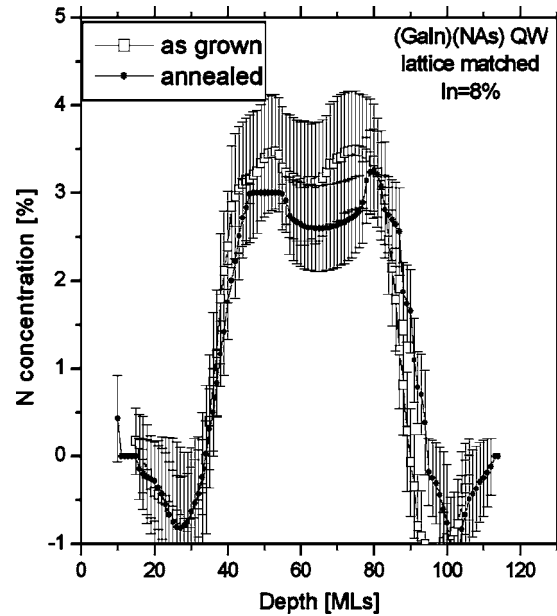


FIG. 4. Nitrogen depth profile in a (GaIn)(NAs) quantum well before and after annealing (from TEM analysis), assuming the In depth profile from RBS measurements.

after evaluation the N depth profile as well as the strain state of the quantum well (Figs. 4 and 5). The quantification of the In from RBS is possible with significantly reduced error bars, if layer thicknesses are known from other techniques, such as, e.g., TEM or high resolution x-ray diffraction.

Interestingly, if one now compares the N depth profiles obtained for the ternary Ga(NAs) (Fig. 2) to those for quaternary (GaIn)(NAs) of the same N content before and after annealing, one does not observe a significant change in the N distribution throughout the quaternary material upon annealing. The N concentration as a function of the position across a $(\text{GaIn}_{0.08})(\text{N}_{0.03}\text{As})$ quantum well, derived from CELFA analysis with the assumption of 8% In in the structure, is depicted in Fig. 4. Neither profile shape, width, nor maximum N concentration change for the quaternary material upon annealing.

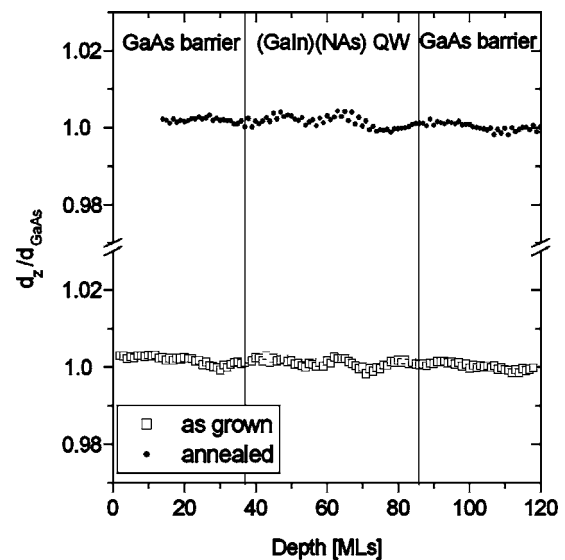


FIG. 5. Strain state analysis across the (GaIn)(NAs) quantum well also shown in Figs. 3 and 4.

To corroborate that both elemental profiles, the one of the nitrogen as well as the one of the Indium, are homogeneous and inhomogeneities are, e.g., not cancelling out their effect on the dark field intensity of the lattice fringe image, we performed strain state analysis of the (002) lattice planes for the as grown as well as annealed (GaIn)(NAs) quantum wells. The lattice constant in growth direction, measured from the lattice fringe images, has been normalized to the lattice constant of the GaAs barriers and is plotted for the as grown as well as annealed (GaIn)(NAs) quantum wells as a function of depth in Fig. 5. As we deal with quaternary material, which has been grown lattice matched to the GaAs substrate, one does not expect any relaxation of the thin TEM foil and, hence, also no influence of TEM sample thickness on the results. One clearly sees no deviation of the lattice constant in growth direction neither when crossing the barrier quantum well interface nor the quantum well barrier interface, indicating that the quaternary films before and after annealing are perfectly matched to the GaAs substrate. As the lattice constant throughout the quantum well also shows no fluctuation, one can—together with the homogeneous intensity distribution of the (002) reflection from the lattice fringe image—conclude that the assumption of a homogeneous In profile was indeed correct. Identical values of the (002) dark field intensity can only be achieved, if the In content decreases and the N content increases simultaneously or if the In content increases with the N content decreasing. The former situation would lead to a smaller, the latter situation to a larger lattice constant in growth direction, what has not been observed. As we know from Fig. 5 that the (GaIn)(NAs) quantum wells are completely lattice matched, we can calculate the theoretical nitrogen content, assuming Vegard's law and an In content of 8% in the structure. With the lattice constants of the binary counterparts from Ref. 19, one obtains a nitrogen content of 2.9% in the quaternary material. This is also in quantitative agreement with what we obtain from the evaluation of the lattice fringe images shown in Fig. 4 for the nitrogen content.

Hence, annealing conditions, which do not change neither the In nor N distribution in the quaternary (GaIn)(NAs) alloy and result in optimum PL efficiency, lead for the In-free Ga(NAs) material to microscopic phase separation and nitrogen pileup. This is an important result also regarding the stability of the two material systems compared. The fourth alloy constituent In, which is present in the quaternary material stabilizes the material against phase separation and decomposition. The reason possibly lies in the trend toward formation of In–N bonds upon annealing in the quaternary material in contrast to the ternary. We performed valence force field calculations on the strain energy of different substitutional $\text{Ga}_x\text{In}_y\text{N}$ configurations in cubic zinc blende crystals ($x+y=4$).²⁰ The results of these calculations are compiled in Table I. One clearly observes the trend that from the point of view of strain energy, it is favorable for the crystal, if the N moves into an In rich environment. Even taking the opposite trend of the binding energies into account—the In–N bond is weaker than the Ga–N bond—there is still an energy gain if we have an In rich environment of the N. Upon growth—on the free surface of the crystal—strain con-

TABLE I. Chemical binding and strain energies for different environments of nitrogen in cubic zinc blende lattices, referenced to a N surrounding of four Ga atoms.

Configuration	ΔE_{chem} (eV)	ΔE_{strain} (eV)	ΔE_{tot} (eV)
Ga ₄ N	0	0	0
Ga ₃ In ₁ N	0.23	−0.55	−0.32
Ga ₂ In ₂ N	0.46	−1.05	−0.59
Ga ₁ In ₃ N	0.69	−1.50	−0.81
In ₄ N	0.92	−1.86	−0.94

siderations are less important: here, mainly chemical bond strengths play a role. Consequently, we find after growth, in our as grown samples, predominantly GaN rich bonding configurations. If one gives the crystal enough energy, upon annealing, to reach a more stable state, the N will hop to more In rich sites in the quaternary alloy. This stabilizes the homogeneous distribution of the N as it adopts the random In distribution in the quantum well.

This change in the nearest neighbor environment of the N is also responsible for the blueshift of the energy gap of (GaIn)(NAs) after annealing, which was observed for the samples used for this study. Unchanged macroscopic In and N depth distributions together with a change in local N environment lead to a semiconductor having different bandgaps.¹⁰

The stabilization of the N depth distribution by a further alloy element (in this case, In) is of course not possible for the ternary alloy, as the more stable configuration with In surroundings does not exist here. From the point of view of strain energy, one should in this case even expect a trend toward a more pronounced formation of $\langle 100 \rangle$ oriented N nearest neighbor pairs, which have a lower strain energy than two single nitrogen atoms in the cubic zinc blende lattice ($\Delta E_{\text{strain}} = -0.1$ eV).²⁰ This implies a trend toward N accumulation. We were also able to show this change in the nitrogen environment upon annealing by dark field TEM recently.²¹

As these results have important implications on the performance of devices which use Ga(NAs) as barrier material together with (GaIn)(NAs) as active material, we are currently investigating annealing conditions, which result in an improvement of material quality for the quaternary alloy without deteriorating the ternary Ga(NAs) barrier material.

SUMMARY

In summary, two methods were suggested to quantitatively depth profile nitrogen and indium in ternary Ga(NAs) as well as quaternary (GaIn)(NAs) separately from each other. The first technique combines Rutherford backscattering spectrometry to determine the In content in the alloy with quantitative high resolution transmission electron microscopy. The second technique exploits high resolution TEM images only and uses the information of the strain and image intensity together to derive the In and N separately. It should be emphasized that special attention has to be paid to depth profile the In and N in the quaternary alloy separately from each other but for the same quantum well. We find that

Ga(NAs) as well as (GaIn)(NAs) are characterized directly after MOVPE growth by a homogeneous plateau similar to depth distribution of the alloy constituents with several monolayer rough interfaces with the GaAs barriers. Upon annealing, which is optimized for improving the PL characteristic of the quaternary material, we do not find a macroscopic redistribution of elements for (GaIn)(NAs) in contrast to Ga(NAs), where we observe a trend toward pileup of the nitrogen depth profile and phase separation. These experimental findings can be explained by stability considerations, deduced from valence force field calculations. One finds that upon annealing the rearrangement of the N from a Ga rich environment into an In rich environment is favorable for (GaIn)(NAs), in which the material will be stabilized. Such stable configurations do not exist for Ga(NAs), which reacts with a N accumulation upon annealing.

ACKNOWLEDGMENTS

The work at the Philipps University Marburg has been supported in the framework of the DFG topical research group "Metastable Compound Semiconductors and Heterostructures."

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