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The role of nitrogen-nitrogen pairs in the deviation of the GaAsN lattice parameter from Vegard's law

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We propose several physical mechanisms that may account for the difference between the nitrogen composition in GaAsN materials measured by secondary ion mass spectroscopy and x-ray diffraction. A simplified model proposed here proves that Vegard's law remains valid as long as all nitrogen atoms in the GaAsN alloys are located substitutionally at the arsenic sites. The theoretical results based on N-N pair defects are in good agreement with the experimental data, suggesting that the N-N pairs are the predominant nitrogen-related defects that cause deviation from the GaAsN lattice constant predicted by Vegard's law. © 2004 American Institute of Physics.
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I. INTRODUCTION

The attractiveness of employing InGaAsN materials grown on GaAs substrates for the fabrication of long-wavelength optoelectronic devices has generated a large amount of interest in GaAs-based nitride materials in the past 7 years.¹ Since the demonstration of an InGaAsN laser diode emitting at the wavelength of 1.1 μm in 1996,² devices for both 1.31 and 1.55 μm communication systems have been achieved by such types of laser diode.^{3,4} InGaAsN, which has been used to fabricate the long-wavelength laser diodes, benefits from the large energy gap bowing coefficient of GaAsN. GaAsN has been reported to have the largest energy gap bowing coefficient, up to 20 eV per mole percentage, as compared with other ternary semiconductor compounds.^{5,6} Thus, a small nitrogen composition can significantly reduce its energy gap. The high sensitivity of the energy gap to the nitrogen concentration necessitates precise determination of the nitrogen content in the GaAsN material. Furthermore, the material quality of GaAsN is also highly sensitive to the nitrogen concentration, as significant deterioration occurs with increasing nitrogen composition. A previous report⁷ has shown that a nitrogen composition of 5% or more has the effect of completely quenching the photoluminescence of the GaAsN materials. The accurate determination of nitrogen composition in this material is therefore important not only for the energy band tailoring but also for the material quality control.

X-ray diffraction (XRD) is probably the most convenient nondestructive method routinely used to examine the nitrogen composition in GaAsN and related materials. Alternatively, secondary ion mass spectroscopy (SIMS) is sometimes employed to calibrate and verify the nitrogen composition. However, the nitrogen composition results ob-

tained by these methods are not always in agreement with each other, especially when the nitrogen composition in GaAsN ternary exceeds 3%. Spruytte *et al.*⁸ have reported that the nitrogen composition results deduced from the SIMS measurements are higher than those obtained from the XRD measurements. The reverse has been reported by Li *et al.*⁹

By comparing the nitrogen contents deduced from the XRD and SIMS measurements, this paper provides a physical insight into the origins of the above contradiction between Spruytte's results and Li's data, using a batch of GaAsN samples specially grown by a solid source molecular beam epitaxy (SSMBE) system. A theoretical model is proposed to relate the nitrogen compositions obtained from the XRD and SIMS measurements. The analysis reveals that the nitrogen-related point defects have a significant influence on the determination of nitrogen composition of GaAsN material, and N-N pairs and present as predominant nitrogen-related defects in GaAsN, especially, with high nitrogen composition.

II. EXPERIMENTAL PROCEDURE

The GaAsN samples were grown in a SSMBE system, equipped with standard effusion cells for the group III elements and cracker cells for the group V elements. The purity of all the sources is 99.9999%. The nitrogen radicals are generated using a radio-frequency (RF) plasma-assisted nitrogen source, and supplied to the substrate in a dispersive mode.¹⁰ All samples were grown on the (001)-oriented semi-insulating GaAs substrates prepared using the standard preparation procedures. Prior to growth, the surface oxide desorption, was carried out under the As₄ overpressure. The beam equivalent pressure of Ga is 4.5×10^{-7} torr, resulting in a growth rate of $\sim 1.0 \mu\text{m/h}$ verified by the time resolved reflection high-energy electron diffraction measurements. The As/Ga flux ratio is fixed at 14. The nitrogen plasma source operates under a nitrogen background pressure of

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FIG. 1. A typical XRD (004) rocking curve of the GaAsN samples (solid), also presented is a simulation curve based on the dynamic theory (dashed) showing the nitrogen composition in GaAsN epilayer of 1.45%.

3.6×10^{-6} torr in the presence of As_4 flux and is activated by the RF power greater than 60 W to maintain the plasma in high brightness mode.

A 300 nm-thick undoped GaAs buffer layer was first grown at 590°C . The substrate temperature was then reduced to 460°C for the growth of the GaAsN epilayer. The GaAsN sample was 100 nm thick with a 20 nm-thick GaAs cap layer. To avoid lattice relaxation, the thickness of the GaAsN epilayer and GaAs cap layer was reduced to 50 and 10 nm, respectively, in the samples with the nitrogen composition exceeding 3%. The XRD rocking curve data was collected in the symmetrical (004) reflection direction. The SIMS measurements were performed with the 10 keV Cs^+ ions as the primary ions. The SIMS profiles were recorded after performing a calibration using a GaAsN standard. The nitrogen compositions in the GaAsN samples were then extracted by comparing the intensity ratio of $^{202}\text{CsN}^+$ to $^{208}\text{CsGa}^+$.

III. RESULTS AND DISCUSSION

A. Vegard's law

Figure 1 shows a (004) rocking curve of a typical GaAsN sample (solid), together with a simulation curve obtained by using the dynamic theory (dashed).¹¹ The structure of this sample is schemed in the inset of Fig. 1. A distinct GaAsN diffraction peak is present in the right-hand side of the GaAs substrate peak, indicating the GaAsN samples are of high macroscopic structural quality. However, the high macroscopic structure quality does not imply that these samples are free from microscopic defects such as substitutes, antisites, and interstitials, as will be discussed in the following sections. Strong Pendellosung fringes are observed in the XRD spectrum, indicating the presence of a smooth and abrupt GaAsN/GaAs interface. A close fitting of the simulation result to the experimental spectrum shows the sample has the nitrogen composition of 1.45%.

The accuracy of determining the nitrogen composition is clearly affected by the accuracy of the XRD measurement

itself. More fundamental factors affecting the accuracy of nitrogen composition are the validity of Bragg's equation and Vegard's law, upon which the nitrogen composition deduction is based. Bragg's equation is a well-known relationship in optics, which relates the dependence of the x-ray diffraction angle on the spacing between two reflection surfaces. Some uncertainties in the validity of Vegard's law have however been reported.^{8,9} This has raised questions on the theoretical basis of using XRD measurement as a routine method for alloy composition determination. Hence, it is considerably significant to verify the validity of Vegard's law. In an ideal GaAs crystal, the distance $R_{\text{Ga-As}}$ between two neighboring gallium and arsenic atoms can be written as

$$R_{\text{Ga-As}} = r_{\text{Ga}} + r_{\text{As}}, \quad (1)$$

where r_{Ga} and r_{As} are Pauling covalent radii of gallium atom and arsenic atom, respectively. $R_{\text{Ga-As}}$ is oriented along the $\langle 111 \rangle$ direction of the crystal unit cell.

Assuming a variation in $R_{\text{Ga-As}}$ due to some causes, the elastic strain ε along $\langle 111 \rangle$ direction will be

$$\varepsilon = \frac{\Delta R_{\text{Ga-As}}}{R_{\text{Ga-As}}}. \quad (2)$$

When projected along the three axis directions $\langle 100 \rangle$, $\langle 010 \rangle$, and $\langle 001 \rangle$, the components ε_x , ε_y , and ε_z along the three axes are the same as ε

$$\varepsilon_x = \varepsilon_y = \varepsilon_z = \varepsilon. \quad (3)$$

Therefore, the stress along the $\langle 001 \rangle$ direction (i.e., z axis) due to the variation in $R_{\text{Ga-As}}$ will be

$$f_z = C_{11}\varepsilon_z + C_{12}(\varepsilon_x + \varepsilon_y) = (C_{11} + 2C_{12})\varepsilon. \quad (4)$$

The strain along the $\langle 001 \rangle$ direction $\Delta d_{\perp \text{GaAs}}/d_{\perp \text{GaAs}}$ will then take the following form:

$$\frac{\Delta d_{\perp \text{GaAs}}}{d_{\perp \text{GaAs}}} = \frac{f_z}{C_{11}} = \left(\frac{C_{11} + 2C_{12}}{C_{11}} \right) \varepsilon. \quad (5)$$

In an ideal GaAsN solid solution, it is reasonable to assume that every incorporated nitrogen atom occupies a substitutional arsenic site. Therefore, based on Eq. (1) and (2), the strain ε along the $\langle 111 \rangle$ direction due to the nitrogen atom incorporation becomes

$$\varepsilon = \frac{\Delta R_{\text{Ga-As}}}{R_{\text{Ga-As}}} = \left(\frac{r_{\text{N}} - r_{\text{As}}}{r_{\text{Ga}} + r_{\text{As}}} \right) x. \quad (6)$$

Hence, the lattice constant variation due to the nitrogen atom incorporation becomes

$$\Delta d_{\perp \text{GaAs}} = d_{\perp \text{GaAs}} \cdot \left(\frac{C_{11} + 2C_{12}}{C_{11}} \right) \left(\frac{r_{\text{N}} - r_{\text{As}}}{r_{\text{Ga}} + r_{\text{As}}} \right) x. \quad (7)$$

Figure 2 shows the calculated dependence (dashed line) of the lattice constant on the nitrogen composition in the GaAsN alloy using this simplified model. The calculated results indicate a linear relationship between the lattice constant of GaAsN and its nitrogen composition. This trend is similar to the prediction of Vegard's law, which is denoted by the solid line. The maximum error between the calculated results from this simplified model and Vegard's law is

FIG. 2. A comparison of the calculated lattice constants obtained from this model (dashed line) with that from the Vegard's law (solid line) and that from the first-principle total-energy calculations as well (solid circles) (see Ref. 13).

$\sim 2.6\%$. By taking into account the extremely large lattice mismatch of $\sim 20\%$ between GaAs and cubic GaN,¹² the data deduced from this simplified model is generally consistent with that from Vegard's law. Neugebauer *et al.*¹³ have reported that the atomic relaxation in such a large lattice-mismatch material system is of great significance to the lattice property. When the atomic relaxation is taken into consideration, the data obtained from the total-energy calculation is consistently in agreement with that from Vegard's law (dots on solid line). But the theoretical results from the total-energy calculations are in good agreement with our simplified model for the cases where the internal relaxation is not allowed (dots on dashed line). This suggests that the 2.6% discrepancy between our simplified model and Vegard's law comes from the atomic relaxation. Therefore, Vegard's law remains valid provided that the GaAsN material is an ideal alloy, and the XRD remains an effective method for the nitrogen composition determination in the ideal III-As-N nitrides.

B. Deviation from Vegard's law

Figure 3 shows a nitrogen concentration profile obtained from the SIMS measurement of a GaAsN sample with 1.65% nitrogen composition. Due to the fluctuation of the nitrogen composition in the GaAsN epilayer, the average nitrogen composition is indicated with the dashed line. The nitrogen composition within the first 5 nm from the sample surface is high, probably caused by the surface adsorption effect. If this is the case, a GaAs cap layer would be needed to minimize the nitrogen surface adsorption effect. A 10 nm-thick nitrogen diffused region is observed on both sides of the GaAsN layer, indicating that a 10 nm-thick GaAs blocking layer is required to minimize the nitrogen diffusion effect.

Unlike the XRD measurement, where only the nitrogen atoms that have effects on the GaAsN's lattice constant can be detected, the SIMS measurement can detect all the nitrogen atoms within the GaAsN alloy regardless of where their occupational sites are. Therefore, intuitively, the nitrogen

FIG. 3. A nitrogen composition profile of a typical GaAsN sample with nitrogen content of 1.65%, deduced from the SIMS measurement.

composition value deduced from the SIMS measurement should never be lower than that from the XRD measurement. Hence,

$$[N]_{\text{SIMS}} \geq [N]_{\text{XRD}}. \quad (8)$$

It is worthy to note that the condition $[N]_{\text{SIMS}} = [N]_{\text{XRD}}$ applies only for the above-mentioned ideal GaAsN alloy, i.e., every incorporated nitrogen atom occupies a substitutional site. Figure 4 shows a comparison of the nitrogen compositions obtained by the SIMS and XRD measurements. The horizontal axis denotes the XRD-measured nitrogen composition, while the vertical axis denotes the SIMS-measured nitrogen composition. The solid diagonal line indicates the equality of the XRD-measured nitrogen composition and the SIMS-measured nitrogen composition. Both methods give the same composition results for the GaAsN samples where the nitrogen composition is less than $\sim 3\%$. However, the nitrogen composition deduced from the SIMS measurement becomes larger than that from the XRD measurement when the nitrogen composition in the GaAsN sample is higher than 3%. Equation (8) is therefore approximately validated by these experimental results. Our experimental results are in

FIG. 4. A comparison of nitrogen compositions measured with the SIMS and XRD approaches.

TABLE I. Parameters of GaAs and GaN for the Vegard's law verification [Ref. 12].

	D (nm)	C ₁₁ (GPa)	C ₁₂ (GPa)	r (nm) ^a
GaAs	0.565 325	1221	566	Ga:0.126; As:0.12
GaN	0.45	293	159	N:0.075

^aData adopted from the periodical table compiled by Sargent-Welch scientific company.

agreement with those of Spruytte *et al.*,⁸ but different from those reported by Li *et al.*⁹ where their explanations were somewhat ambiguous and insufficient.

C. Models for deviation from Vegard's law

In the ideal GaAs_{1-x₀}N_{x₀} alloy where every nitrogen atom occupies the substitutional arsenic site, the SIMS-measured nitrogen composition (x_{SIMS}) is equal to the XRD-measured nitrogen composition (x_{XRD})

$$x_{\text{SIMS}} = x_{\text{XRD}} = x_0. \quad (9)$$

However, Fig. 4 shows that the XRD-measured nitrogen composition values are smaller than the SIMS-measured nitrogen composition values, when the nitrogen composition in GaAsN is higher than 3%. This implies that some nitrogen atoms in GaAsN are not detectable by the XRD measurement. According to the Bragg's equation, this is true because only those nitrogen atoms that affect the lattice constant of GaAsN can vary the diffraction angle of the x ray, and therefore be detected. Nitrogen atoms that do not affect the lattice constant of GaAsN are not detected by the XRD measurements. For example, there are one and a half hollow tetrahedrons and six hollow octahedrons per unit cell as well in GaAs besides four arsenic atoms and four gallium atoms. While the nitrogen atom's covalent radius (0.075 nm) is much smaller than that of the gallium atom (0.126 nm) and the arsenic atom (0.12 nm) as is shown in Table I. This implies the presence of sufficient spaces for the interstitial nitrogen atoms. If there is a nitrogen atom filling in the center of such a hollow tetrahedron, the distance ($R_{\text{N}-\text{A}}$) between this interstitial nitrogen atom (N_i) and its nearest neighbors (A) is,

$$R_{\text{N}-\text{A}} = r_{\text{N}_i} + r_{\text{A}} \quad (10)$$

where, A denotes Ga for the gallium hollow tetrahedron, or As for the arsenic hollow tetrahedron. Because the GaAsN lattice constant is decided by Eq. (1), the lattice constant can be changed only when $R_{\text{N}-\text{A}}$ is larger than $R_{\text{Ga-As}}$. According to Ref. 14, if the following conditions as $r_{\text{N}_i} + r_{\text{A(A=Ga,As)}} \leq r_{\text{Ga}} + r_{\text{As}}$ for tetrahedron hollows and $r_{\text{N}_i} \leq (\sqrt{11/3} - 1)(r_{\text{Ga}} + r_{\text{As}})/2$ for octahedron hollows can be met, the effect of nitrogen interstitials in such hollows on the lattice constants will be negligible.¹⁴ Due to the very small volume of nitrogen atom, our calculations (not shown here) found that the above conditions can always be met, therefore the single nitrogen atoms in both types of hollows would not affect the lattice constant of GaAsN, and in turn the XRD-measured nitrogen composition remains unchanged. Suppose there are δx_0 single nitrogen atoms in such hollows, then the

SIMS-measured nitrogen composition (x_{SIMS}) increases by δx_0 . The possibility of finding a nitrogen atom in hollows is proportional to the amount of nitrogen present in the GaAsN lattice. Therefore, the number of single nitrogen atoms in hollows could be reasonably assumed to be proportional to the nitrogen composition in the ideal GaAs_{1-x₀}N_{x₀} alloy

$$\delta x_0 = k x_0 \quad (11a)$$

and

$$x_{\text{SIMS}} = x_0 + \delta x_0 \text{ for nitrogen interstitials.} \quad (11b)$$

Besides interstitial N atoms, other nitrogen-related factors such as As-N pairs, Ga-N pairs, and N-N pairs might occur in an actual GaAsN alloy that would increase x_{SIMS} and reduce x_{XRD} . Similar deduction can get the amount of nitrogen-arsenic (As-N) pair, nitrogen-gallium (Ga-N) pair, and nitrogen-nitrogen (N-N) pair as

$$\delta x_0 = k x_0 (1 - x_0) \quad (12a)$$

and

$$x_{\text{SIMS}} = x_0 + \delta x_0 \text{ for As-N pairs,} \quad (12b)$$

$$\delta x_0 = k x_0 \quad (13a)$$

and

$$x_{\text{SIMS}} = x_0 + \delta x_0 \text{ for Ga-N pairs,} \quad (13b)$$

$$\delta x_0 = k x_0^2 \quad (14a)$$

and

$$x_{\text{SIMS}} = x_0 + 2 \delta x_0 \text{ for N-N pairs.} \quad (14b)$$

In Eq. (14), the amount of nitrogen-nitrogen (N-N) pair (δx_0) has a square dependence on the nitrogen content (x_0) in GaAs_{1-x₀}N_{x₀} alloy. This is because that the amount of nitrogen-nitrogen (N-N) pair (δx_0) is proportional to the possibility of finding a nitrogen atom and the possibility of finding another nitrogen in the GaAs_{1-x₀}N_{x₀} lattice. How x_{XRD} is affected by the presence of these nitrogen-related defect could be calculated using Eqs. (1)–(7). The theoretical relationship between the calculated SIMS nitrogen compositions and the calculated XRD nitrogen compositions is indicated in Fig. 4 by the dashed, dotted, dashed-dotted, and solid curves individually for each case. This theoretical consideration indicates that the presence of the nitrogen interstitials, As-N pairs and Ga-N pairs authentically causes the deviation of the GaAsN lattice constant from Vegard's law. However, when compared with the experimental data, these theoretical results show merely a linear dependence, which deviates from the nonlinear experimental behavior. This suggests that such nitrogen interstitials, As-N pairs and Ga-N pairs are not dominant defects in real GaAsN materials. As for nitrogen-nitrogen pairs, they not only cause deviation of the GaAsN lattice that Vegard's law predicts but also the theoretical result based on such defects shows good agreement with the experimental data when $k=1.5$, indicating that N-N pairs are the dominant defects in GaAsN materials grown by molecular beam epitaxy.

Spruytte *et al.*⁸ suggested that the N_2 molecules could be a type of nitrogen-related defect due to their small volume. Li *et al.*⁹ proposed the N-As and N-N pairs (even N-clusters) as energetically favored defects. In both cases, the theoretical models failed to match the experimental results. In one of our papers,¹⁵ we have assumed that the nitrogen-related defect concentration is proportional to the square of the substitutional nitrogen composition $[N]$, but no theoretical foundation of the square dependence was given. Here, we have proved that our simplified but effective model can explain the basis for the nonlinear relationship between the SIMS-measured nitrogen composition and the XRD-measured nitrogen composition, and verified that N-N pairs are the dominating defects in the GaAsN materials. This is analogous to the case of the low-temperature (for example, $\sim 250^\circ\text{C}$) grown GaAs, where the As-As pairs at the substitutional arsenic sites are the dominant defects.¹⁴ The GaAs growth requires the interaction between the gallium and arsenic species on the heated substrate surface ($\sim 250^\circ\text{C}$), where the As_4 or As_2 molecules ($\sim 500^\circ\text{C}$) and the gallium atoms ($\sim 900^\circ\text{C}$) are supplied. Since the substrate temperature ($\sim 250^\circ\text{C}$) is not high enough to ensure the complete interaction of the arsenic and gallium species, some arsenic dimers would remain in the GaAs epilayers. In a nitrogen plasma source,¹⁶ the nitrogen species include neutral nitrogen atom N, metastable activated nitrogen atom N^* , metastable activated nitrogen molecule N_2^* , nitrogen ion $N^{i+(i=1,2,\dots)}$, and nitrogen molecule ion $N_2^{i+(i=1,2,\dots)}$. The excited nitrogen molecule species, such as N_2^* and $N_2^{i+(i=1,2,\dots)}$, are effectively reactive species. Due to the difficulty in breaking the extremely strong N-N chemical bonds at the low substrate temperature ($\sim 460^\circ\text{C}$) in the MBE growth, these molecule species are readily incorporated into the GaAsN material to form the N-N pair defects. Therefore, there should be two nitrogen states in GaAsN, i.e., the nitrogen atom and the N-N pairs, both present in the substitutional arsenic sublattice sites. In accordance with the x-ray photoelectron spectroscopy results of $GaAs_{0.94}N_{0.06}$ in Ref. 8, the binding energy between the gallium and nitrogen atoms should be different from that between the gallium atom and the N-N pairs. The intensity of the Ga-NN bond is much lower than that of the Ga-N bond, because according to our model, the N-N pairs comprises only 9% of the substitutional nitrogen atoms.

IV. CONCLUSIONS

In summary, this paper reported the difference between the SIMS nitrogen composition and the XRD nitrogen composition of the GaAsN materials. A simplified model proved that Vegard's law remained valid so long as all the nitrogen atoms in the GaAsN alloys were located at the substitutional arsenic sites. Theoretical results based on the N-N pair defects were in good agreement with the experimental data, suggesting that the N-N pair was the dominant nitrogen-related defect causing deviation of the GaAsN lattice from that predicted by Vegard's law.

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