Vibrational properties of $GaAs_{0.915}N_{0.085}$ under hydrostatic pressures up to 20 GPa

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We investigated a $GaAs_{0.915}N_{0.085}$ single crystal under hydrostatic pressures up to 20 GPa by Raman spectroscopy. The zinc blende optical phonons show great similarities to those of binary GaAs under the same conditions demonstrating that nitrogen incorporation has no major influence on the GaAs-I to GaAs-II phase transition and its partial reversibility upon decompression. Frequency shifts of the nitrogen local vibrational mode under hydrostatic pressure are very different from those in binary GaN because of the different compressibilities of the two materials and the overstretched character of the Ga—N bond in Ga(As,N). This is also reflected by the anharmonicity of the Ga—N bond potential in Ga(As,N).

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I. INTRODUCTION

In GaAs the substitution of a few percents of As by N (Refs. 1 and 2) introduces significant changes to the band structure and therefore to the optical properties.[3] The large difference in electronegativity and atomic radius between As and N, despite their isoelectronicity, leads to the formation of an N-induced E_+ band from an N concentration of $x=0.2\%$ on.[3,4] This E_+ band is related to the GaAs-like E_- band via a level-anticrossing behavior which invokes a strong redshift of the fundamental band gap with increasing x.[5]

Investigations concerning the vibrational properties of Ga(As,N) can give further insight into the influence of nitrogen incorporation on the crystalline structure and the local nitrogen environment. Regarding lattice vibrations, the Ga(As,N) system shows a two-mode behavior which is typical of alloys where the atoms occupying equivalent lattice sites strongly differ with respect to their masses.[6,7] There is only weak interaction between the GaAs-like phonons, on the one hand, and the local vibrational mode (LVM) of the N atoms on the other hand. This makes the LVM a sensitive probe of the local environment of the N atoms. Raman spectroscopy has turned out to be a powerful tool to make use of this probe.

Much work has already been done to study the effects of epitaxial strain and alloying on the lattice vibrations of Ga(As,N).[6,8] Information about crystalline disorder and the symmetry of the E_+ N-induced band, respectively, has been gained from second-order as well as symmetry-forbidden Raman scattering.[9,10] All these investigations were carried out at ambient pressure.

The behavior of lattice vibrations under variable high hydrostatic pressure can give insight into the mechanical properties of the Ga—N bonds in the ternary material and into the effects of the impurity on the host crystal as there is already considerable information available about binary GaAs and GaN under such conditions. A major aspect of the pressure dependence of lattice vibrations is the frequency shifts of the modes, providing information about the force constants of the chemical bonds (see, e.g., Ref. 11 for a review on tetrahedral semiconductors).

Another interesting aspect is studying pressure-induced structural phase transitions which are characterized in vibrational spectra by the disappearance of the signals belonging to one structure and formation of signals belonging to the other structure. GaAs undergoes a first phase transition around 17 GPa from the zinc blende towards an orthorhombic structure[12] indicated by the complete disappearance of the zinc blende optical phonons.[13] A high pressure phase diagram of GaAs using data from several experimental methods was given by Besson *et al.*[14] McMahon and Nelmes give an overview of the systematics of the structural phase transitions of tetrahedral semiconductors under hydrostatic pressure.[15] Here, we present a Raman study of a $GaAs_{0.915}N_{0.085}$ sample and corresponding binary reference samples of GaAs and GaN under hydrostatic pressures up to approximately 20 GPa.

II. SAMPLE PREPARATION AND EXPERIMENTAL DETAILS

The $GaAs_{0.915}N_{0.085}$ sample studied was grown on a (100)-GaAs substrate by metal-organic vapor-phase epitaxy at 500 °C. Its layer thickness is 1 μm. Triethylgallium, 1,1-dimethylhydrazine, and tertiarybutyl arsine were used as the source materials. The nitrogen concentration was determined by x-ray diffraction assuming the absence of interstitial N atoms. The layer was fully relaxed and was easily cleaved off the substrate. Thus, it was easy to remove a small grain from the substrate by a steel cannula and to transfer it into the pressure cell. A diamond-anvil cell (Diacell Products, type B-05) of the Mao-Bell type[16] was used to apply hydrostatic pressure ranging from 1 to 20 GPa. We chose silicon oil as the liquid pressure medium because of its few own Raman signals and its weak photoluminescence in the investigated spectral range. The measurements were carried out using a Renishaw Raman microscope system with 514.5 nm excitation light. The spectra were acquired using a CCD detector with thermoelectric cooling. The hydrostatic pressure was determined by the pressure-induced shift of the R_1 fluorescence line of ruby powder[18] which had been loaded into the

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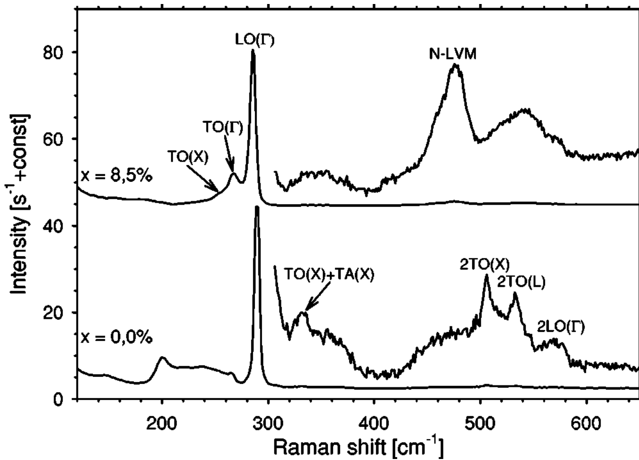

FIG. 1. Typical Raman spectra of epitaxial GaAs (lower curves) and $GaAs_{0.915}N_{0.085}$ (upper curves) single crystals taken at room temperature and ambient pressure in LO-allowed backscattering geometry $x(y',y')\bar{x}$ with 514.5 nm excitation wavelength. The spectral range higher than 300 cm^{-1} has been enlarged by a factor of 30 with respect to intensity to show the relatively weak nitrogen LVM and second-order features. Peak assignments are given according to Refs. 8 and 22.

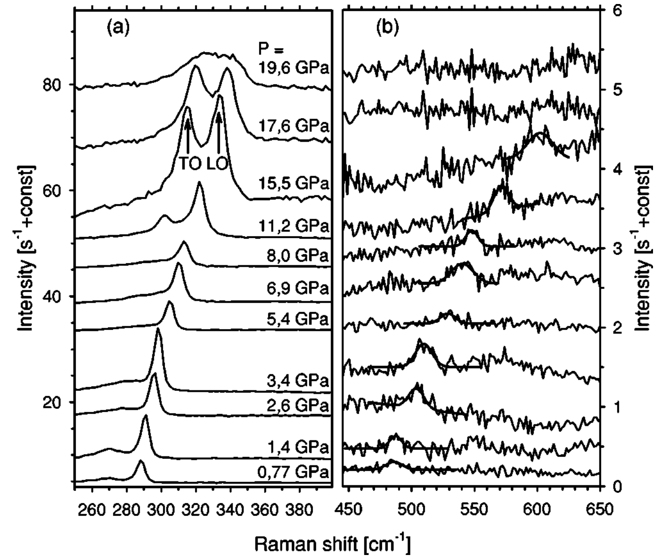

FIG. 2. Room-temperature Raman spectra of $GaAs_{0.915}N_{0.085}$ under increasing hydrostatic pressure in the spectral ranges of the GaAs-like phonons (a) and of the GaN-like LVM (b). In (b) the fits of the LVM to a Gaussian line shape are also shown for pressures between 0.77 and 15.5 GPa.

cell together with the sample. As the pressure medium used solidifies under high pressure the uniformity of the pressure was checked by taking fluorescence spectra of different grains of ruby. Epitaxial layers of the corresponding binaries GaAs and wurtzite GaN were measured for comparison.

III. RESULTS AND DISCUSSION

A. GaAs-like phonons

Figure 1 shows the Raman spectrum of the $GaAs_{0.915}N_{0.085}$ layer in comparison with the spectrum of the GaAs reference sample. These spectra were obtained at ambient pressure with the samples mounted under the microscope's objective in an $x(y',y')\bar{x}$ backscattering geometry from the (100)-oriented surface. Both spectra are dominated by the LO phonon peak of the GaAs matrix in concordance with the selection rules for zinc blende crystals in the applied scattering geometry.[19] The LO signal of the $GaAs_{0.915}N_{0.085}$ (at about 288 cm^{-1}) is at slightly lower wave numbers than that of the GaAs reference (at 291 cm^{-1}). This difference in LO frequency between the binary and the ternary crystal originates from alloying effects.[6] A weaker signal at around 270 cm^{-1} belongs to the symmetry-forbidden TO phonon. This signal is detectable even in the binary crystal because the backscattering geometry is not perfectly realized due to the nonzero aperture angle of the objective. In case of the ternary crystal the TO intensity is significantly stronger and even a weak signal of the zone-boundary TO(X) phonon arises indicating the alloy disorder due to the incorporation of the nitrogen atoms. Additional confirmation of the disorder increase with N incorporation is given by the deterioration of the second-order Raman signals of Ga(As,N) compared to those of GaAs. The frequency shifts of the spectral features of the LO and TO phonons, respectively, in $GaAs_{0.915}N_{0.085}$ obey the predictions made by Prokofyeva *et al.* for relaxed layers.[6] This is expected as the sample under study is fully relaxed. Ga(As,N) shows an additional broad peak at around 475 cm^{-1} which does not exist in the GaAs spectrum. It can be assigned to the local vibrational mode of the impurity nitrogen atoms each of which is tetrahedrally coordinated by four gallium atoms[17,20] and will be discussed in detail in the next subsection.

All the pressure-dependent spectra shown in this paper were taken with linearly polarized excitation light and no polarizers on the detection side. One series of spectra was taken on the pressure upstroke only. To confirm its results and to gain additional information about the phase transition a second series was taken on the upstroke and on the downstroke successively. Figure 2(a) shows the low-frequency range of the first series of spectra where the GaAs-like phonon signals are located. As was expected from a crystal in a pressure range far from any phase transition, the frequencies of both LO and TO phonon modes increase on the pressure upstroke due to the anharmonicity of the bond potentials.

In Fig. 3(a) the TO and LO frequencies determined by curve fit procedures are plotted versus pressure for both series of spectra. The frequency shifts of both phonon signals were fitted with quadratic functions of the form

$$\nu(P) = \nu_0 + \alpha \cdot P + \beta \cdot P^2. \qquad (1)$$

The corresponding fitted curves are shown in the figure as black lines. The constant, linear, and quadratic parameters ν_0, α, and β used to plot the black curves were taken as the averages between the respective parameters from the two measurement series [open and closed circles in Fig. 3(a)]. For comparison, experimental data (crosses) and the corresponding quadratic fits (grey lines) obtained for the binary GaAs reference are shown in the same figure. Table I gives

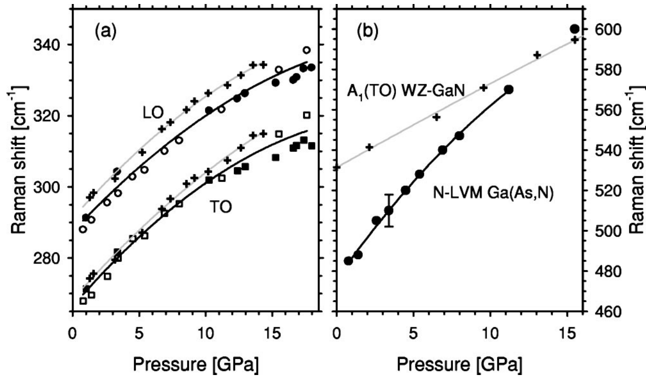

FIG. 3. Pressure dependences of the mode frequencies on the pressure upstroke in $GaAs_{0.915}N_{0.085}$ obtained from Raman spectra. (a) the GaAs-like LO and TO frequencies are represented by circles and squares, respectively. Open and closed symbols correspond to two measurement series. The two black solid lines show the quadratic fits to the experimental frequency shifts of both phonons as an average between the two series. For comparison, the experimental phonon frequencies (crosshairs) and the respective quadratic curve fits (grey lines) of GaAs are shown. (b) The shift of the GaN-like LVM in Ga(As,N) and a quadratic fit to the data points (black line) are plotted where the point at 15.5 GPa has been neglected in the curve fit. Exemplarily an error bar is given. As an example for a GaN mode, the pressure dependence of the A_1(TO) phonon in wurtzite-GaN is plotted (crosshairs). The grey line corresponds to a quadratic fit to these data.

the parameters ν_0, α, and β of the curve fits, and the corresponding Grüneisen parameters γ including the standard deviation of each parameter derived for the TO and LO phonons of Ga(As,N) in comparison to those obtained for GaAs. The linear (parameter α) as well as the quadratic (parameter β) terms of the frequency shifts of both phonons in Ga(As,N) show only minor differences to the ones for the binary material. In fact, the deviations of the values are within the error of the experiment and the fitting procedure. Furthermore, for GaAs as well as for Ga(As,N) the α values of the TO phonons are close to those of the LO phonons so that their expected experimental error bars overlap. Therefore it is not possible to draw any conclusion from these measurements concerning the pressure behavior of the LO-TO splitting or the influence of nitrogen incorporation on it. In both measurement series all Raman signals of Ga(As,N) disappeared at a pressure of approximately 19 GPa. Considering the experimental inaccuracies, this value for the phase transition pressure agrees well with the 17.4 GPa found by Raman spectroscopy for GaAs by Venkateswaran *et al.*[13]

The absolute peak intensities show an irregular behavior with increasing pressure up to 8 GPa. This is due to slight variations of the sample orientation in the pressure cell as well as of the probe spot on the sample which occur when the pressure is changed. However, the strong increase of LO intensity from 10 GPa onwards must be considered a genuine effect. A detailed analysis of the resonance conditions is unfortunately rather difficult for the sample under study as neither its fundamental (E_-) band gap nor the pressure-dependent shift of the latter are accurately known. Optical measurements apart from micro-Raman spectroscopy were not feasible because of cracks in the sample surface due to relaxation. Empirical formulas for the N-induced band-gap reduction determined up to now are only valid for $x < 3\%$. The maximum intensity at resonance with the band gap under 514.5 nm excitation for binary GaAs has been found to occur at about $P = 10$ GPa.[14] We observe the corresponding resonant behavior for Ga(As,N) at approximately 15 GPa. However, one has to keep in mind that eventually the ongoing phase transition suppresses the scattering intensity in the upward stroke before the actual resonance is reached. The shift of the resonance towards higher pressure compared to

TABLE I. Pressure dependence of the phonon modes of zinc blende (ZB) $GaN_{0.915}As_{0.085}$, ZB GaAs, and wurtzite (W) GaN. The parameters for W and ZB GaN are taken from Refs. 24 and 25, repectively.

semiconductor	mode	ν_0/cm^{-1}	$\alpha/\frac{\mathrm{cm}^{-1}}{\mathrm{GPa}}$	$\beta/\frac{\mathrm{cm}^{-1}}{\mathrm{GPa}^2}$	γ
ZB GaN_xAs_{1-x}	LO	287.4±1.0	3.97±0.25	−0.072±0.013	1.22
$x=8.5\%$	TO	266.3±1.1	4.33±0.29	−0.087±0.015	1.44
	LVM	476.1±1.5	10.7±0.63	−0.211±0.052	1.99
ZB GaAs	LO	291.2±0.9	4.25±0.25	−0.083±0.016	1.09
	TO	268.4±1.0	4.22±0.30	−0.065±0.019	1.17
W GaN	$A1$ (LO)	736.8±1.3	4.54±0.27	−0.043±0.012	1.46
(Ref. 24)	$A1$ (TO)	531.4±1.2	4.30±0.29	−0.014±0.014	1.92
	$E1$ (TO)	559.3±0.7	3.94±0.17	−0.019±0.008	1.67
	$E2$	566.5±1.4	4.24±0.29	−0.011±0.013	1.77
ZB GaN	LO	743±2	4.5±0.2		1.44
(Ref. 25)	TO	553±2	4.0±0.2		1.71

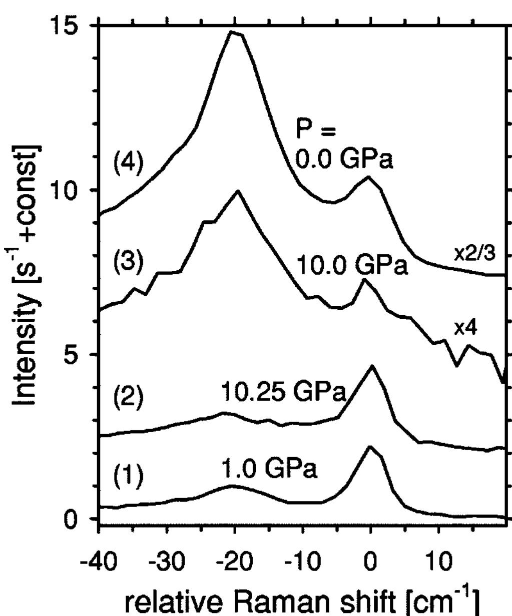

FIG. 4. Selected Raman spectra of $GaAs_{0.915}N_{0.085}$ from the second series taken on pressure upstroke (1 and 2) followed by pressure downstroke (3 and 4) after pressurization up to approximately 20 GPa. Spectrum (4) was taken at ambient pressure outside the pressure cell in $x(y',y')\bar{x}$ backscattering geometry. Raman shifts are given relatively to the one of the LO phonon peak. Enlarging factors for spectra (3) and (4) are denoted on the right side of the respective curves.

GaAs is in concordance with the facts that the band gap of $GaAs_{1-x}N_x$ redshifts and its rate of the pressure shift decreases with increasing x. Before the disappearance of the phonon signals in Fig. 2 a significant intensity increase of the TO phonon signal with respect to the LO phonon takes place from 10 GPa onwards. Again, this result resembles that found by Venkateswaran *et al.* for GaAs. The increase of the relative TO intensity in GaAs has been explained by reflection effects once the gap energy becomes larger than the laser energy causing TO scattering not to be strictly forbidden anymore. As $GaAs_{0.915}N_{0.085}$ shows the TO intensity increase at roughly the same pressure as GaAs despite the different gap energies of both materials, we rather tend to the interpretation that a relaxation of Raman selection rules arising from the onset of the phase transition is the main contribution to the relative TO intensity increase. However, both effects, though to different extents, may contribute to the observed TO enhancement effect.

Figure 4 shows four of the spectra from the second measurement series. In this series the pressure was taken up to the disappearance of all Raman features at approximately 19 GPa and then released in several steps. All the Raman shifts in Fig. 4 are given relatively to the LO phonon position. The two zinc blende phonons reappear upon decompression to just over 10 GPa and become stronger when the pressure is released further. Their intensity ratio is reversed relatively to that known from zinc blende crystals in this scattering geometry where the TO signal is forbidden and therefore should be much weaker than the allowed LO signal. Spectrum (4) which was taken of the fully recovered and cleaned sample in a well defined backscattering geometry outside the pressure cell shows that this unusual intensity ratio is not due to pressure-induced changes of the scattering geometry. Such a strong relaxation of selection rules remaining after decompression has been shown for binary GaAs as well and has been interpreted in terms of an only partial reversibility of the phase transition from GaAs-I towards GaAs-II. The hysteresis between the forward and reverse transition boundaries as well as the considerable remaining disturbance of the zinc blende lattice resemble the behavior of GaAs.[13] In conclusion, the zinc blende phonons and the pressure-induced structural phase transition of GaAs seem to be almost unaffected by nitrogen incorporation, despite 8.5% of nitrogen.

B. Nitrogen local vibrational mode

Another interesting aspect of Ga(As,N) under pressure is the behavior of the LVM which can be seen at 480 cm^{-1} in Fig. 1. Reference 6 gives an empirical formula for the dependence of the LVM's frequency in Ga(As,N) on the nitrogen concentration x. Obviously, the mode frequency in the sample under study is significantly lower than expected from this linear function. The reason is probably that the samples used in Ref. 6 to obtain this dependence only covered the compositional range up to $x \approx 3\%$. Mascarenhas and Seong pointed out that for $x > 3\%$ the mode frequency deviates from a linear dependence and tends towards lower frequencies (Ref. 8) being qualitatively in concordance with our finding. To our knowledge, a detailed analysis concerning the dependence of the mode frequency on the nitrogen concentration at higher x has not been performed yet and would exceed the scope of this work. It is expected, however, that the compositional dependence of the LVM's frequency will be different at higher x where nitrogen pairs and clusters are formed from the situation at lower x where virtually all the N atoms are isolated. Therefore, a simple linear dependence valid in the whole compositional range from $x=0\%$ up to $x=8.5\%$ would be rather surprising. However, as the LVM is a sensitive probe of the local environment of the nitrogen atoms, the frequency of this mode is directly related to the force constant of the covalent bond between gallium and nitrogen atoms.

Figure 2(b) shows spectra of the LVM mode obtained in the first measurement series under pressure. The LVM can be seen as a weak signal above the detector noise. The fits of the LVM to a Gaussian line shape are also shown in the figure for pressures up to 15.5 GPa. The pressure dependence of the LVM's frequency obtained from the spectra is plotted in Fig. 3(b). Additionally, this figure also depicts the pressure dependence of the $A1$(TO) phonon mode of wurtzite GaN. The pressure dependence of the LVM and $A1$(TO) mode of wurtzite GaN differ considerably. The pressure dependence of the former is much stronger. This is also reflected by the parameters obtained by fitting the experimental data using Eq. (1). The fitted curves are also shown in Fig. 3(b). The corresponding parameters are given in Table I for the LVM of $GaAs_{0.915}N_{0.085}$, zinc blende, and wurtzite GaN modes. All

GaN modes show a much weaker pressure dependence than the LVM of $GaAs_{0.915}N_{0.085}$. It is obvious that the LVM, often being referred to as the GaN-like mode, does not show a GaN-like behavior under pressure.

The knowledge of the pressure dependence of the mode frequencies ω for vibrations of the Ga—N bonds in Ga(As,N) and GaN, respectively, allows one to calculate the pressure dependence of the effective force constants D for these modes by the equation

$$D(P) = m\omega^2(P). \tag{2}$$

In case of the GaN LO phonon m is the reduced mass of the system consisting of a N atom and a Ga atom whereas in case of the Ga(As,N) LVM, neglecting interactions between the LVM and the lattice, it is simply the nitrogen atomic mass. It has been proven by infrared studies of GaAs implanted selectively with the different isotopes ^{14}N and ^{15}N that the interaction between the LVM and the neighboring atoms is sufficiently weak to regard the LVM as a simple oscillator.[17] When a crystal is compressed hydrostatically the interatomic distances decrease and the force constant increases due to the anharmonicity of the bond potential resulting in an increase of the vibrational frequency. From the bulk moduli B and their first derivatives B' with respect to pressure it is possible to approximately obtain the dependences of the bond lengths b on the pressure for both compounds using Murnaghan's equation of state[21]

$$b(P) = b(0)\left(1 + \frac{B'}{B}P\right)^{-1/3B'}. \tag{3}$$

This allows one to plot all the value pairs $[D(P), b(P)]$ into a graph providing data points showing how D depends on b. The numerical values of the bulk moduli show a broad variation in the literature. We assumed $B=74.66$ GPa for GaAs (Ref. 26) and $B=237$ GPa for both wurtzite and zinc blende GaN.[27] As the pressure derivatives of B we used $B'=4.67$ for GaAs (Ref. 26) and $B'=4.3$ for GaN.[27] We calculated B and B' for Ga(As,N) as the linear interpolations between the values of the two binaries. Zero-pressure lattice constants of the binary materials are $a_0=5.65325$ Å for GaAs and $a_0=4.5$ Å for zinc blende GaN from Ref. 23. The zinc blende GaN LO phonon frequency and its pressure-induced shift were taken from Ref. 25. The bond lengths at ambient pressure are given by $b(0)=\sqrt{3}/4a_0$. In Fig. 5 the results are shown for the LVM and for the LO phonon of wurtzite GaN. Obviously, the anharmonicity of the bond potentials, defined as the partial derivative of the force constant with respect to the bond length, is much higher for the Ga—N bond in GaN than for the one in Ga(As,N). Evaluated at ambient (zero) pressure we obtain the anharmonicities $(\partial D/\partial b)_{Ga(As,N)}=9.3\times10^{12}$ N/m^2 and $(\partial D/\partial b)_{GaN}=16.8\times10^{12}$ N/m^2. It is obvious that there is no simple interpolation between the two sets of data points in the figure. This highlights again that the LVM of N in GaAs is not just "GaN-like." A further theoretical analysis in terms of the change of the character of the mode (from localized to extended) as well as in terms of local strain and electronic effects is required to fully understand these results.

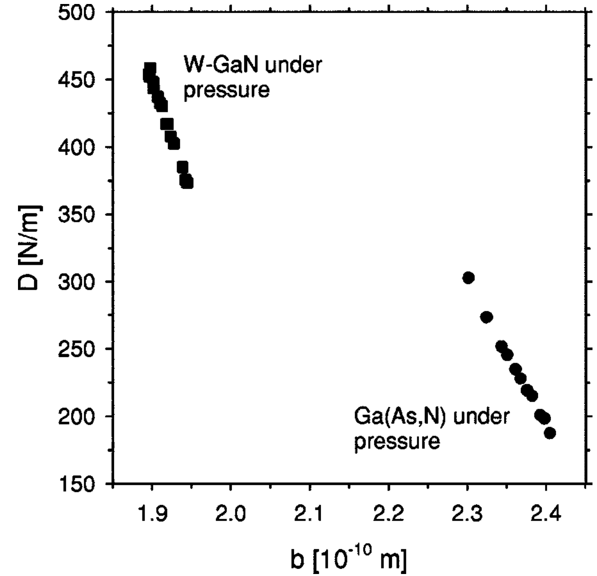

FIG. 5. Comparison of the force constant versus bond length calculated using Murnaghan's equation of state for the LO phonon of wurtzite GaN and the LVM of $GaAs_{0.915}N_{0.085}$.

However, already simple arguments can motivate why the calculated anharmonicity in Ga(As,N) is smaller than in GaN although the Ga(As,N) mode shifts much stronger than those of GaN. This apparent contradiction arises from the fact that Ga(As,N) is considerably softer than GaN, as indicated by the bulk moduli, and therefore the interatomic distances in the former material are changed more strongly by pressure than in the latter. It should be emphasized that the value for Ga(As,N) is just a rough approximation because the bulk modulus will not exactly behave as implied by the linear interpolation. Therefore, all the effects of a local lattice relaxation changing the interatomic distance with respect to its value in the ideal zinc blende structure and changing the local compressibility compared to the bulk compressibility were neglected. Nevertheless, as the two calculated values of $(\partial D/\partial b)$ differ significantly, the anharmonicity is certainly smaller in the ternary material than in zinc blende GaN.

This result can be qualitatively understood by comparing the lattice constants of the two compounds. In GaN the interatomic distance has a well-defined equilibrium value of 1.95 Å. In contrast, the Ga—N interatomic distance in Ga(As,N) is basically forced to the value of the Ga—As distance (2.41 Å) which is roughly 24% bigger. This means that the Ga—N bond in Ga(As,N) is overstretched by up to 24%, depending on local relaxation, with respect to its equilibrium length. As the bond potentials normally become flatter for increased interatomic distance also their derivatives with respect to interatomic distance are expected to decrease with overstretching and therefore the observed behavior matches the expectation.

IV. CONCLUSIONS

In summary, we investigated an epitaxial layer of $GaAs_{0.915}N_{0.085}$ under hydrostatic pressures up to 20 GPa by

laser Raman spectroscopy. The observed pressure-dependent frequency shifts of the GaAs-like lattice vibrations are to a first approximation equal to those found by other groups for binary GaAs. The first phase transition in Ga(As,N) which is indicated by the disappearance of the LO and TO phonon signals takes place around 19 GPa. Thus, the experiments shown here indicate that the transition pressure is not affected by the substitution of As atoms by N atoms. Both the frequency shifts and the phase transition show that the host matrix is rather undisturbed by the N incorporation and confirm the highly localized character of this isoelectronic impurity. The pressure-dependent shift of the GaN-like local vibrational mode was used to calculate the anharmonicity of the Ga—N bond potential using bulk moduli and lattice constants from the literature. A comparison of the calculated value with the one obtained from literature data for GaN qualitatively confirms the simple picture in which the Ga—N bonds are significantly overstretched in Ga(As,N) with respect to their equilibrium length realized in GaN.

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