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Raman scattering study of GaN nanostructures obtained by bottom-up and top-down approaches

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Abstract

GaN nanocolumnar structures were grown by plasma-assisted molecular beam epitaxy (PAMBE) and also fabricated by electron cyclotron resonance reactive ion etching (ECR-RIE) of a compact GaN film parallel to the [111] direction of the Si(111) substrates. Scanning electron microscopy shows that the nanocolumns fabricated by PAMBE have a length of about 300–500 nm with diameters ranging from 20 to 150 nm while nanowhiskers formed by RIE have diameters of 40–80 nm and a height between 1.4 and 1.7 μm . A comparative study of the vibrational spectrum (including optical and interface phonons) of the nanostructures using conventional macro-Raman and micro-Raman scattering as well as surface-enhanced Raman scattering is presented.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Intense research concentrating on the fabrication, physical properties and applications of one-dimensional materials has been developed in recent years. The fabrication of a wide variety of nanocolumns and nanowhiskers by means of bottom-up as well as top-down approaches has been achieved in several semiconductor material systems (Si [1], III–V [2, 3], III–nitride [4, 5]). Currently physical properties [6, 7] as well as sophisticated applications, such as single electron transistors (SETs) and field-effect transistors (FETs) [8, 9], are being studied intensively. GaN nanowires with improved electronic and optical properties in comparison to bulk single GaN crystals are considered also as promising materials for the fabrication of optoelectronic devices with superior performance [10].

Many fundamental questions dealing with the internal electronic and vibrational structure, the effect of the large surface in comparison to the bulk material and size-dependent transport

phenomena are starting to be addressed. In this context our previous investigation of PAMBE-grown GaN nanowires demonstrates the effect of surface Fermi-level pinning and its interplay with the nanowire dimensions on the recombination behaviour of electron–hole pairs in photoconductivity through these nanocolumns [6].

Understanding phonon properties is equally important from a fundamental point of view and for potential applications of the nanostructures. Optical phonons of nanocolumnar structures which can be utilized for analysis of their structural parameters (diameter, shape of nanoobjects and built-in mechanical strain, etc) have already been studied by several groups [11–14].

As mentioned above, a subject of special interest is the extremely large surface with respect to the bulk. In this regard GaN nanocolumns are especially suitable for the purpose of studying surface (or interface) related vibrational modes. It is already well established theoretically that in heterogeneous nanocolumnar material structures composed of at least one polar semiconductor a set of interface (or surface) phonon modes occurs in the region between the transverse optical (TO) and the longitudinal optical (LO) modes [15]. Experimentally, first-order interface modes (modes with quantum number $l = 1$) known as Fröhlich modes have been observed recently in the Raman spectra of GaN columnar structures [15]. It is expected that the Fröhlich mode frequency as well as other interface phonon mode frequencies depend on the dielectric constant of the surrounding material, the filling factor (the fraction of the total volume occupied by particles) and the shape of the nanostructures [11, 15]. However, the intensity of Raman scattering by the interface modes of nanostructures with a low filling factor can be rather weak, preventing a frequency analysis. It can be increased by several orders of magnitude due to surface-enhanced Raman scattering (SERS) by using a deposition of metal clusters on the sample surface.

SERS is based on the enhancement of the Raman line intensity through two effects: the electromagnetic mechanism [16] when the electric field felt by the Raman dipole is enhanced due to the excitation of the plasmon resonance in surrounding Ag particles and the chemical mechanism [17, 18] is based on increased polarizability of the molecules that are adsorbed on a rough metallic surface under the influence of incident radiation. For our study the first mechanism is especially important. Since the first discovery of SERS [19], this technique has attracted substantial interest because of its potential applications in nanoscience [20–22], for the detection of nanoobjects, such as nanotubes (see for a review [23, 24]) and single molecules [25–27] as well as for biological science [28, 29]. Surface-enhanced Raman spectroscopy has been used for the study of interfacial phenomena and ultrathin films [30–32] as well as vibrational modes in various inorganic materials (see [33–35] and references therein). Nevertheless, to the best of our knowledge no observation of SERS by nanocolumnar structures has been reported yet.

In our study we present a comparative Raman study including macro-Raman, micro-Raman and surface-enhanced Raman scattering by the GaN nanocolumnar structures fabricated via etching of a compact epilayer and those obtained by a self-assembling growth process.

2. Experimental details

The GaN nanocolumns were grown on Si(111) substrates by PAMBE. Details of the growth process are reported elsewhere [36].

The GaN compact film (sample 111-O) for further fabrication of nanocolumns was grown under metal-rich conditions by PAMBE. It was nucleated using an 8 s deposition of Al followed by multiple AlN/GaN buffer layers. The growth temperature was 770 °C and the deposition rate amounted to 4.3 nm min⁻¹ for the AlN layers and 8.2 nm min⁻¹ for the GaN layers, resulting

in a total sample thickness of about 2270 nm. Despite the buffer layers, tensile strain imposed by the Si substrate resulted in the occurrence of cracks in the compact layer.

All the samples discussed in this paper are listed in table 1, in which we report the growth parameters. In our experiments we slightly changed the nominal III–V ratio and the growth temperature, resulting in different stoichiometry and deposition rates. Sample NC-23 is composed of a layer grown with constant III–V ratio followed by a buffer with a gradual increase of the Ga supply [36].

Sample 111-O was etched to fabricate nanocolumns (then called sample 111-A). For the etching process a Ti mask was used, even though it is well known that it is not suitable for electron cyclotron resonance reactive ion etching (ECR-RIE) of GaN. Using a thin Ti mask for etching with Ar/CH₄/H₂/Cl₂, the etch profile becomes inhomogeneous, resulting in spikes. A thin Ti layer of 350 nm thickness was patterned by means of optical lithography and resist lift-off. We expected that the Ti mask erosion should take approximately 6–7 min with a rate of 50–60 nm min⁻¹. The Ti micro-masking effect was amplified by removing CH₄ and adjusting the microwave power (to about 300 W). The gas relation between Ar and Cl₂ was 3 sccm/16 sccm. Hydrogen was used to start the plasma but it was not present during the etching process. The ECR cavity was formed with an upper magnet current of 185 A and collimated with a lower magnet current of 25 A. Once the plasma was stable under a constant pressure of 2.5 μbar, the etching process began by applying an RF power of 60 W (bias–196 V). For this process we estimated a GaN etch rate of about 150–160 nm min⁻¹. Reference values under similar conditions are 170–190 nm min⁻¹ (Cl₂/Ar, RF = 50–60 W [37]) and 90–110 nm min⁻¹ (Cl₂/H₂/Ar, RF = 50–60 W [38]). A cycle of two minutes with an RF power set to 60 W followed by a 30 s break without DC was repeated eight times. The 30 s break should decrease heating and keep the sample at the nominal temperature of 2 °C. The influence of the 30 s break on the nanocolumn formation is not yet understood in detail. During this time Cl radicals might further act as a preferential chemical etchant as compared to the more physical RIE process.

Even if we cannot explain completely the interplay between the process parameters and the nanocolumn formation, we observe a good reproducibility of the columns.

Raman spectra of nanocolumnar structures were taken from planar and cleaved sample surfaces using a Dilor XY800 triple spectrometer supplied with a microscope which allows the light to be focused to a 1 μm spot. In order to avoid heating of the sample the laser power measured in front of the microscope was kept at 5 mW. The scattering geometries applied were $z(x-)\bar{z}$, $y(x-)\bar{y}$ and $y(z-)\bar{y}$, where z is parallel to and x and y are perpendicular to the growth direction.

After growth, part of the samples were transferred *ex situ* to an ultra-high vacuum chamber for the deposition of Ag clusters and Raman spectra were taken during the process in a macro-Raman geometry. The nanostructure was mounted on a holder of a manipulator which allows to adjust the sample with respect to optical axes of the Raman set-up. Ag was evaporated at about 900 °C from a Knudsen cell which was thoroughly degassed. The Ag evaporation rate was 1.6 nm min⁻¹. The amount of metal deposited onto the sample was evaluated from the change in resonance frequency of the quartz crystal microbalance. A preliminary AFM study of Ag-covered semiconductor surfaces revealed the appearance of Ag clusters of an elongated shape with a size of 5–10 nm [39] which have the plasma resonance in the blue–green spectral region [40]. The 488 and 514.5 nm lines of an Ar⁺ laser with a power of 300 mW were used for excitation. Raman spectra from planar GaN films were measured for comparison. The spectral resolution was 2 cm⁻¹ over the entire spectral range.

Note that for the laser power densities we used we did not observe any significant changes of either the Raman peak positions or the Raman line shape due to sample heating. The

Table 1. Growth conditions for GaN nanocolumns.

Sample	T_{sub} (°C)	N in AlN layer			N in GaN layer		Ga in GaN layer	Ga in GaN graded layer		Growth time (min)
		N_{flux} (sccm)	P_{forward} (W)	Al_{flux} (mbar)	N_{flux} (sccm)	P_{for} (W)	Ga_{flux} (mbar)	$\text{Ga}_{\text{flux,min}}$ (mbar)	$\text{Ga}_{\text{flux,max}}$ (mbar)	
NC-10	790	—	—	—	4.0	500	2.7×10^{-8}	—	—	240
NC-11	800	—	—	—	4.0	500	2.5×10^{-8}	—	—	240
NC-13	770	—	—	—	4.0	500	2.7×10^{-8}	—	—	240
NC-16	795	—	—	—	4.0	500	2.1×10^{-8}	—	—	360
NC-23	800	—	—	—	4.0	500	3.3×10^{-8}	3.3×10^{-8}	5.5×10^{-8}	180
111-O	770	2.5	450	1.1×10^{-7}	4.0	500	3.4×10^{-7}	—	—	311

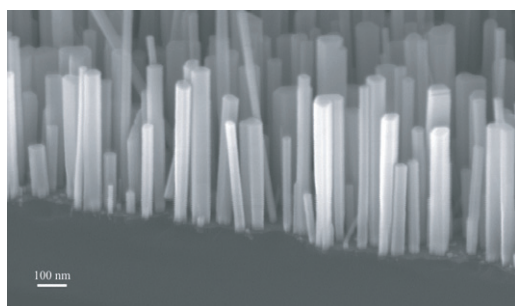


Figure 1. Cross-sectional SEM micrograph of GaN nanocolumns grown on a Si(111) substrate, sample NC-10.

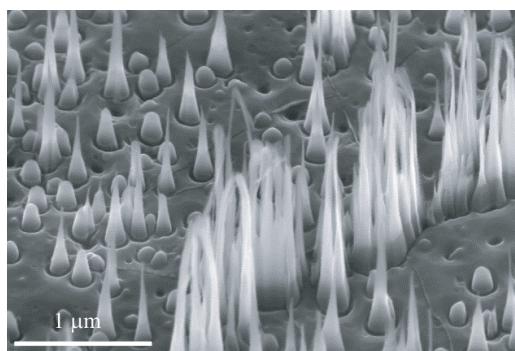


Figure 2. Cross-sectional SEM micrograph of GaN nanowhiskers fabricated by ECR-RIE etching, sample 111-A.

deviation of the Raman signal from Si phonons in the micro and macro configurations did not exceed a value of 1.5 cm^{-1} . Therefore, the spectra measured in the micro and macro configurations were normalized with respect to Si phonon frequency (521.5 cm^{-1}). It is worth mentioning that a local temperature increase is expected in the micro-Raman configuration especially at higher laser intensities (above 10 mW) which can even cause sample melting. However, the Raman signal originates mostly in the wings of the beam profile, where the temperature is substantially lower than at the centre. Therefore, the maximal detected shift of the Si phonon does not exceed 2 cm^{-1} . For a more detailed discussion we refer the reader to [41].

3. Results and discussion

The morphology and layer thickness of the GaN nanocolumnar samples were studied by cross-sectional scanning electron microscopy (SEM) using a Leo 1550 microscope.

A typical morphology of the GaN nanocolumns (sample NC-10) grown by MBE is shown in an oblique cross-sectional SEM image (figure 1). Highly dense isolated hexagonal nanocolumns vertically aligned with respect to the sample surface are homogeneously grown on the whole substrate. The majority of the nanocolumns have a length of about 300–500 nm with diameters ranging from 20 to 150 nm.

Figure 2 shows the SEM micrograph of a GaN structure (sample 111-A) fabricated by ECR-RIE etching. The nanoobjects have the shape of nanowhiskers with a diameter in the

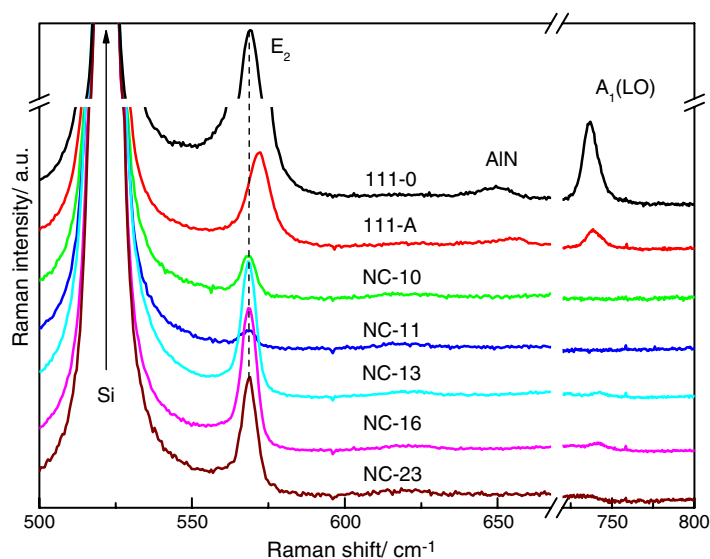


Figure 3. Raman spectra of samples under investigation taken in the $z(x-\bar{z})$ scattering geometry with the 488 nm laser line.

range 40–80 nm and a height between 1.4 and 1.7 μm . The etching is clearly anisotropic and the sidewalls are notably smooth. The nanowhiskers get thinner with increasing height. They are inhomogeneously distributed over the wafer but follow the Ti patterns deposited before etching. Note that the areal density of the nanowhiskers in sample 111-A is at least one order of magnitude lower compared to the nanocolumn density in sample NC-10.

Raman spectra measured in the $z(x-\bar{z})$ scattering geometry from a planar surface of the samples under investigation are shown in figure 3. Besides the strong Raman line at 521.5 cm^{-1} due to Si phonons, the Raman spectra reveal the symmetry-allowed E_2 and A_1 (LO) modes of GaN [42]. The first one is observed at about 569 cm^{-1} for all samples except for sample 111-A, where this line is blueshifted to 572 cm^{-1} . The latter appears prominently in the Raman spectra of the compact GaN film (sample 111-O) and sample 111-A at 736 and 738 cm^{-1} , respectively.

The asymmetric shape of the Raman peak can be due to a contribution of the GaN E_1 (LO) mode which is also observed for epitaxially grown GaN [43]. For other samples this line has a rather low intensity or is even absent in the Raman spectra, and therefore its quantitative analysis is hardly possible. A feature located near 650 cm^{-1} is due to a phonon contribution from the AlN buffer layer.

The increased frequencies of the E_2 and A_1 (LO) phonon modes in sample 111-A correspond to an increased biaxial compressive strain in the nanowhiskers, which might be due to defect formation caused by ECR-RIE. The exact origin of this shift is not clear, but we want to note that a similar shift was observed for GaN annealed in an oxygen atmosphere [44]. The phonon frequency positions for the planar GaN film and the nanocolumnar structures are similar and therefore they manifest comparable crystalline quality.

Raman spectra of the GaN film (sample 111-O) and the nanowhiskers (sample 111-A) measured in different scattering geometries are shown in figures 4(a) and (b), respectively. Raman scattering from cleaved surfaces allows additional lines at 533 and 560 cm^{-1} due to A_1 (TO) and E_1 (TO) phonons, respectively, to be observed. Note that the A_1 (TO) mode is not seen in the Raman spectra of sample 111-A because it is smeared out by the strong scattering from

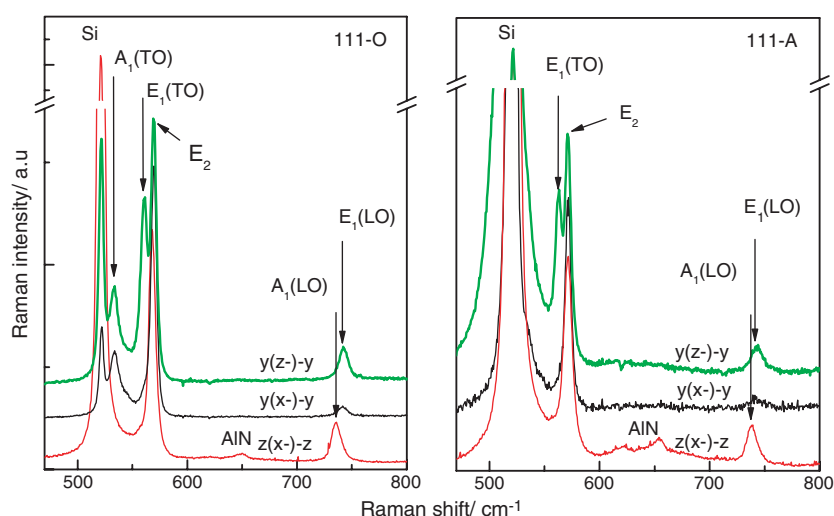


Figure 4. Raman spectra of samples 111-O (film) and 111-A (etched whiskers) taken at different scattering geometries with the 488 nm laser line.

Si. The E_1 (LO) mode at 742 cm^{-1} which is not allowed in the Raman spectra measured from planar surfaces becomes active in Raman spectra taken from cleaved surfaces of the samples. These results are consistent with the data obtained for hexagonal GaN epilayers grown on (111)-oriented MgAl_2O_4 substrates [45]. Raman spectra of the as-grown nanocolumns (not shown here) measured in different scattering geometries are very similar to those of the GaN film and nanowhiskers. This indicates that the Raman selection rules used for GaN films can also be applied to the nanostructures.

In order to investigate surface-related vibrational states, Raman monitoring of the GaN nanocolumnar structures during deposition of Ag was performed. Raman spectra of GaN nanocolumnar structures (samples NC-16 and 111-A) measured in the $z(x-\bar{z})$ scattering geometry with the 514 nm laser excitation line for various thicknesses of deposited Ag are shown in figures 5 and 6(a), respectively. One can see from the figures that the changes of the Raman spectra of the samples 111-A and NC-16 due to Ag deposition are similar. As the deposition of Ag clusters proceeds, the intensity of the Si phonon decreases due to absorption by Ag clusters, while the intensity of the E_2 phonon line increases. In addition, a new strong band occurs in the spectra between the frequency positions of the A_1 (TO) and A_1 (LO) phonons at about $680\text{--}730\text{ cm}^{-1}$. The increasing intensity of the mode by three orders of magnitude with increasing Ag nominal thickness (from 0.1 to 10 nm) manifests surface-enhanced Raman scattering. The intensity of the band is much more pronounced for the dense array of nanocolumns (sample NC-16, figure 5) than for the nanowhiskers (sample 111-A, figure 6(a)). For comparison, figure 6(b) shows the Raman spectra of the GaN compact film (sample 111-O) measured under the same conditions. The intensity of the Raman lines decreases with increasing Ag nominal thickness. As expected, no enhancement of Raman scattering was observed by Ag deposition on GaN planar structures. The asymmetric line shape of the band indicates that more than one vibrational mode contributes to the Raman scattering in this region. In our opinion, it is most probable that this band is due to interface modes localized in the vicinity of the GaN nanocolumn surface and/or tips [11]. Calculations of the frequency position of the modes were performed within the dielectric continuum model [11], supposing a cylindrical shape of the free-standing nanocolumns and nanowhiskers. An elliptical cross-

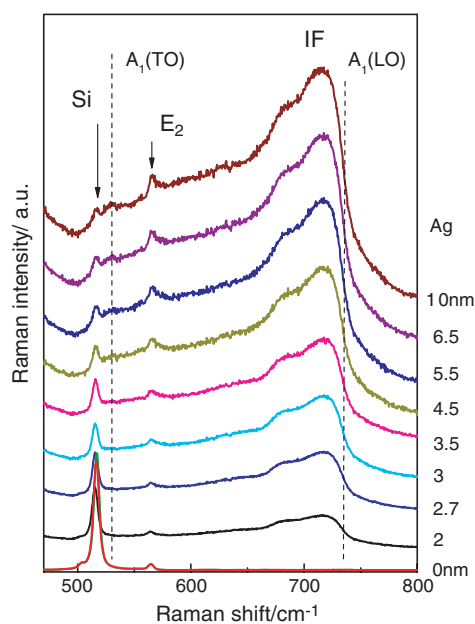


Figure 5. Raman spectra of sample NC-16 measured in the $z(x-\bar{z})$ scattering geometry with the 514 nm laser excitation line for various thicknesses of deposited Ag. The vertical lines indicate the frequency positions of A_1 (TO) and A_1 (LO) modes. The spectra of the samples with deposited Ag are shifted in the vertical direction equidistantly.

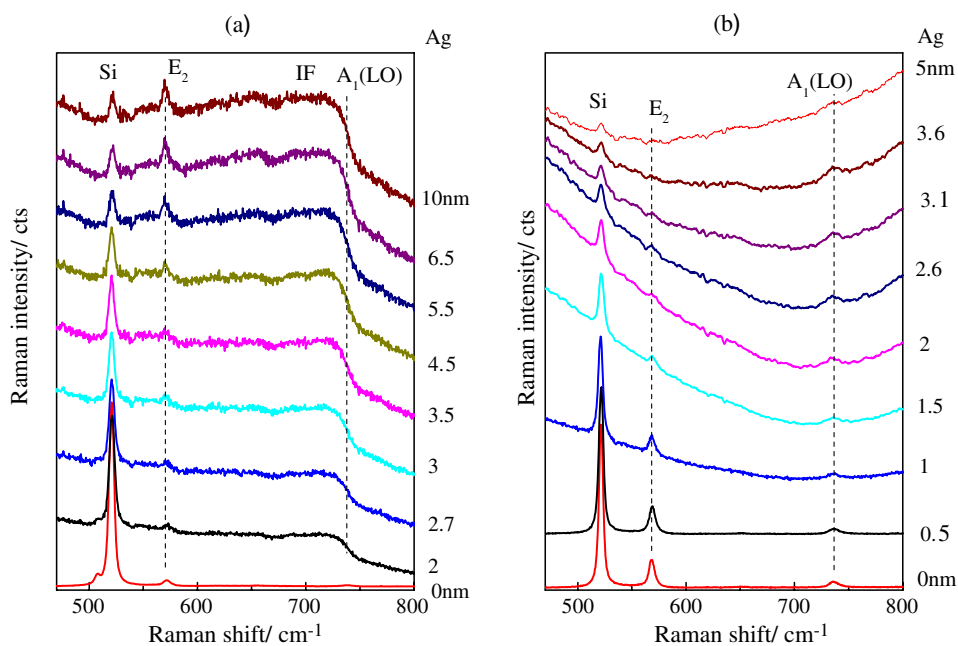


Figure 6. Raman spectra of samples 111-A (a) and 111-O (b) measured in the $z(x-\bar{z})$ scattering geometry with the 514 nm laser excitation line for various thicknesses of deposited Ag. The vertical lines indicate the frequency positions of A_1 (TO) and A_1 (LO) modes. The spectra of the samples with deposited Ag are shifted in the vertical direction equidistantly.

section with ratio of major to minor axis $R/r = 2$ was used in the calculation. In order to calculate the interface modes localized in the vicinity of the tips of nanowhiskers the approach proposed for calculation of the interface modes localized near the corners of rectangular wires was used [11]. The frequency positions of the interface modes localized in the vicinity of the nanocolumn surface and tips are either near $690\text{--}715\text{ cm}^{-1}$ or have an almost continuous spectrum in the range $670\text{--}725\text{ cm}^{-1}$, respectively. The experimental SERS spectra exhibit features located in the same spectral ranges. Note that no significant differences in the calculated frequency position of the interface modes were obtained taking into account an average diameter of the nanocolumns of samples NC-16 and 111-A obtained by SEM. Thus, the calculated values account well for the experimental observations.

4. Conclusions

GaN nanocolumns and nanowhiskers fabricated by PAMBE and ECR-RIE etching, respectively, were investigated by Raman spectroscopy. Raman spectra taken in different scattering configurations exhibit optical vibrational modes, which are similar to planar GaN films manifesting comparable crystalline quality. Comparison of the frequency positions of optical phonons in the GaN planar film and the etched nanostructures indicates that the nanowhiskers undergo biaxial compressive mechanical strain. Deposition of Ag clusters on GaN nanocolumnar structures allows surface-enhanced Raman scattering by interface vibrational modes in GaN nanocolumns. Calculated frequency positions of interface modes in GaN columns performed within the dielectric continuum model are in good agreement with the experimental data. The observed interface phonons might be of considerable interest in future carrier transport studies in nanocolumns, where surface scattering should play a major role.

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