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ABSTRACT

Semiconducting nanowires (NWs) fabricated from III–V materials have gained significant attention for their application in advanced optoelectronic devices. Here, the growth of GaAs/GaInNAs/GaAs core-multishell NWs with a triple quantum-well structure, having about 2% N and 20% In, is reported. The NWs are grown via selective area plasma-assisted molecular beam epitaxy on patterned Si(111) substrates with SiO₂ mask holes. The nucleation and growth of the GaAs nanowires' core are carried out by Ga-induced vapor–liquid–solid growth at the open holes. Finely controlled, vertically aligned, regular core-multishell NWs with uniform wire length and diameter are obtained with a 96% yield and targeted nitrogen concentrations of 0%, 2%, and 3%. The GaInNAs NWs exhibit a spectral red shift relative to the GaAs NWs' peak. Their emission wavelength increases with the N content reaching up to 1.26 μm , which makes them a promising tool in telecommunication light sources.

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III–V semiconductor nanowires (NWs), elaborated with III–V group materials, have been gaining particular attention for their application in a variety of optoelectronic devices, including solar cells, photodetectors, and light-emitting devices.^{1–7} They are known to provide electrically and optically active media and well-shaped cavity for one-dimensional nanomaterial architectures. The growth mechanism of nanowires allows for sensitive control of their dimensions, crystal structures, and material compositions with possible control of the doping levels for the design of complex heterostructures.⁸ Advanced epitaxial techniques with III–V semiconductors and Si enable such controls using the heteroepitaxial growth approach.⁹ Therefore, the use of III–V nanowires on versatile Si substrates offers a realistic prospect for large-scale integrated systems with superior electronic and optical functionalities.^{2,3,10–17} GaInNAs alloys, which have nitrogen

composition typically up to several percent in the dilute level, are the materials of interest, owing to their tunable bandgap, band offset, and lattice constants exhibiting optical functions at a near-infrared regime.^{18–21} GaInNAs can achieve a bandgap and light emission in the wavelength range between 1.3 and 1.55 μm , which are deemed applicable in telecommunications.^{22–24}

Numerous works have been reported on the selective area growth of III–V NWs using patterned substrates. This is particularly popular since it enables the fabrication of well-ordered NWs' arrangement and the suppression of the parasitic cluster deposition (i.e., inter-nanowire planar growth).^{17,25,26} In the case of GaAs NWs, the self-catalyzed growth method using constituent Ga droplets to seed one-dimensional nanowire growth has been commonly applied. This method helps avoiding the contamination associated with foreign metal catalytic

droplets, including Au.^{27,28} In previous works, we have demonstrated the fabrication of high-quality GaNAs nanowires with 2% of N, enabling lasing at the wavelength of $1\ \mu\text{m}$ up to 100 K.^{29,30} The improvement in the optical efficiency and the extension of the emission for longer wavelengths of the NWs are especially desired for their application in telecommunications. In this study, we explored the preferable growth conditions of GaInNAs NWs using the self-catalyzed selective area molecular beam epitaxy (MBE).³¹ As per our findings, it was determined that the growth of NWs can be carried out at a wider growth window concerning As pressure, V/III ratio, and growth rate. We demonstrated the fabrication of vertically aligned arrays of high-quality GaAs/GaInNAs core-multishell NWs showing clear room temperature (RT) photoluminescence (PL) at $1.26\ \mu\text{m}$.

The investigated samples were grown on phosphorus-doped n-type patterned Si(111) substrates in a plasma-assisted MBE system.^{7,29–31} A SiO_2 patterned substrate, with holes having $500 \times 500\ \text{nm}^2$ square openings with their periodic pitch $1.5\ \mu\text{m}$ template, was prepared by SiO_2 sputtering, electron beam lithography (F7000S-VD02, Advantest, Japan),³² and inductively coupled plasma reactive ion etching (CE-300I, Ulvac, Japan). A conventional solid-source effusion cell was used for supplying Ga, and an As-valved cracker cell was operated in the As_4 condition. Nitrogen was supplied by a radio frequency plasma source. The GaAs NWs core was then formed by vapor-liquid-solid (VLS) growth assisted by constituent Ga seed particles when Ga and As fluxes were supplied on the patterned substrate.^{33–38} We then grew three series of GaAs/GaInNAs/GaAs core-multishell samples by varying the flux of N atoms using the following procedure. The V/III ratio would have a critical role on the structural and optical properties of the nanowire sample.³⁹ The selective area growth by the patterned substrate enabled the nucleation of the wires by a wider range of growth parameters.⁴⁰ We employed 3–10 times larger V/III ratio in this study as compared to our previous reports.^{29,30} The high V/III ratio during the growth should promote the preferential zinc blende phase formation, suppressing the formation of twin defects,⁴⁰ and uniform nitrogen distribution at the growth of the GaNAs shell under vapor-solid growth as in the case of thin films.⁴¹

The beam equivalent pressures of As were adjusted to $6 \times 10^{-4}\ \text{Pa}$ and that of Ga to $8 \times 10^{-6}\ \text{Pa}$ before the growth of the nanowire core. The As and Ga supplies corresponded to planar growth rates of 1.3 and 0.1 ML/s on GaAs(001), respectively.⁴² The atomic V/III ratio was, thus, 13 at these conditions. The GaAs core growth was initiated by opening the Ga shutter under an As overpressure at 560°C . Notably, to investigate the initial growth stage of the GaAs core on the patterned substrate, we prepared a test sample where the GaAs core was grown for just 5 min. Thereafter, the GaAs core was grown for 30 min at 560°C for the GaAs/GaInNAs/GaAs core-shell nanowire samples. By introducing a growth interruption, the catalyst Ga became crystallized. Subsequently, the lateral growth became dominant, which was expected to form the wire shells. During the interruption, the Ga and As fluxes were increased to 0.3 and 2.6 ML/s on GaAs(001), respectively, during the crystallization to obtain an adequate growth rate for the following shell layers.³⁴ The BEP and the flux of In were set to be $8 \times 10^{-6}\ \text{Pa}$ and 0.1 ML/s, respectively. The concentration group-III constituents in the GaInNAs layer were then designed to be 75% Ga and 25% In based on the fluxes' ratio. The first GaAs shell was grown for 30 min, followed by the second growth

interruption. During the interruption, the nitrogen plasma was ignited. We then grew the GaAs shell for 7.5 min. Afterward, we grew a three-period GaInNAs/GaAs quantum-well structure by growing each layer for 4.5/7 min and repeated the sequence three times. The outermost GaAs shell was continuously grown for 62.5 min. The nanowire, consequently, formed a GaAs/GaInNAs/GaAs core-multishell structure. Three series of structures were fabricated by varying the flux of N, which was then controlled by adjusting the microwave power of the plasma source between 0 and 130 W at a fixed N_2 gas flow rate of 0.05 sccm. The GaInNAs shell nominally contained 0%, 2%, and 3% of nitrogen and 20% of indium, which were estimated by high-resolution x-ray diffraction with a fitting based on the dynamical diffraction theory for bulk GaInNAs grown on a GaAs(001) substrate.⁴³

The structural characteristics of the NWs were investigated using scanning electron microscopy (SEM) and cross-sectional scanning transmission electron microscopy (STEM). Axially sliced single NW samples for STEM investigation were prepared by focused ion beam processing (Helios660, FEI, USA). STEM was conducted on a single NW using a JEM-ARM200F Dual-X TEM microscope (JEOL, Japan) operating at 200 kV with energy dispersive x-ray spectroscopy (EDS) (JED-2300, JEOL). STEM images were obtained in both bright-field (BF) and high-angle annular dark field (HAADF) modes.⁴⁴ PL measurements were carried out using a PL system at RT, where excitation was performed using a femtosecond pulsed laser emitting at 780 nm (at a power of 1.7 mW), and the PL signal was detected using an InGaAs charge-coupled device.

First, we investigate an initial stage of the NW's growth on a patterned substrate by growing the GaAs core for 5 min, as shown in the scanning electron microscopy (SEM) image in Fig. 1. The nanowires' nucleation was observed on the open windows of the SiO_2 mask, where multiple NWs started growing to its large area size as compared to the individual nanowires.⁴⁵ The nucleation of the multiple wires should be induced by the crystallized Ga droplets impinging onto the windows and those diffused from the SiO_2 mask.^{45–48}

Figures 2(a) and 2(b) show SEM images of the GaAs/GaInNAs/GaAs core-shell NWs' arrays fabricated on the sample surface. The concentration of In is 20%, and that of nitrogen is nominally 2% in the GaInNAs shell. Figure 2(c) shows a cross-sectional scanning transmission electron microscopy (STEM) image of several individual

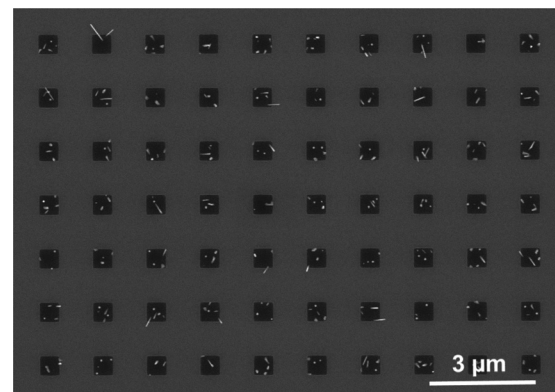


FIG. 1. SEM image of the initial nucleation of the GaAs core on the patterned substrate, which was captured after a 5-min growth duration.

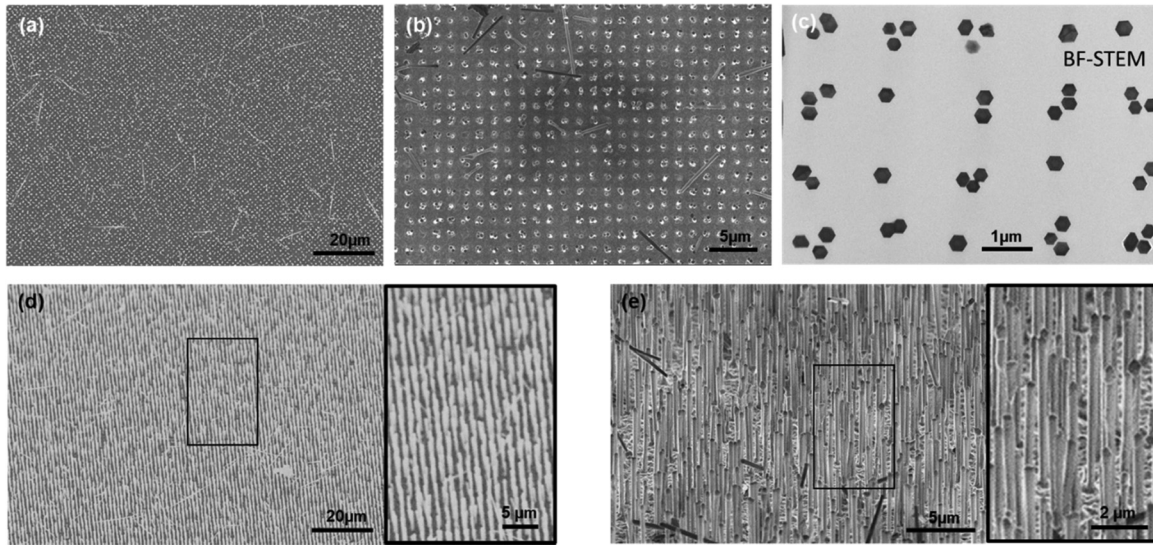


FIG. 2. (a) and (b) SEM images at different magnifications. (c) Bright-field (BF)-STEM image zoomed around few individual nanowires. (d) and (e) Tilted SEM images at 45° angle (d) and 52° angle (e) of the GaAs/GaInNAs/GaAs core-multishell nanowires, right panels are the enlarged images at the areas delimited with rectangles, respectively.

nanowires within the array. Figures 2(d) and 2(e) show 45° -tilted SEM images at different magnifications and angles of GaAs/GaInNAs/GaAs core-multishell nanowires nominally containing 20% In and 2% N in the GaInNAs shell. A clear formation of the nanowires was observed, reflecting the opening patterns of the SiO_2 mask. The wires were mainly vertically aligned over the area with a 96% yield. These observations suggest the adequate growth conditions of vertically aligned regular nanowire ensembles.⁴⁵

As shown in Figs. 2(c) and 2(e), the NWs exhibit straight side-walls with a hexagonal cross-sectional structure. The distributions of the wires' length and diameter were determined using Gaussian fits of the histograms extracted from the SEM images, as shown in Fig. 3. The mean value of the wires' diameter was found to be 335 nm with a standard deviation of 47 nm, whereas the mean length was determined as $7.9 \mu\text{m}$ with a standard deviation of $0.38 \mu\text{m}$. These findings indicate a more homogeneous length distribution as compared to the case of typical nanowires grown with no patterned substrate, which has shown a large standard deviation of the length of more than $0.6 \mu\text{m}$.^{33,49} This homogeneity has led to the regularly arranged nanowires, as seen in Figs. 2(d) and 2(e). Notably, the number of nanowires in Fig. 2(c) in each open window was fewer than in the sample fabricated for the investigation of the initial stage shown in Fig. 1, where the growth of the GaAs core was terminated at the initial stage after 5 min. This would be the phenomenon observed in all self-catalyzed VLS systems, where the low vapor flux of a group-III metal is compensated by the diffusion of surface adatoms so that the Ga droplet coalesces during the continuous 30-min growth of the GaAs core.⁵⁰ The resulting NW arrays showed a high uniformity in terms of their diameter and length with their occurrence reflecting the patterned Si substrate, which should be suitable for their integration in devices' applications. The variation in the wire diameter might illustrate the existence and effects of crystal defects such as twins and stacking faults.⁵¹ Thus far, we have observed phase changes in our dilute nitride GaNAs nanowire samples.⁵² Nevertheless, the observed variation in

the wire diameter is comparable to those observed in the samples used in our previous study without the patterned substrate, where we achieved good optical quality with lasing.^{29,33} Therefore, the preferable optical properties of the NWs' samples were investigated in this study.

Figures 4(a)–4(c) show the cross-sectional BF-STEM images of the GaAs/GaInNAs/GaAs nanowire sample nominally containing 20% In and 2% N in the GaInNAs shells. The nanowire is noted to have a regular hexagonal cross-sectional structure with an overall diameter of approximately 340 nm. The shell consists of three-periods

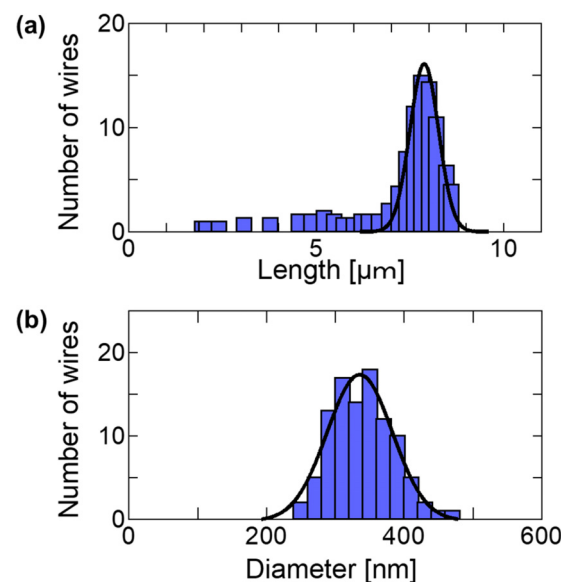


FIG. 3. Histograms extracted from the SEM images showing the distributions of the nanowires' (a) length and (b) diameter, which were determined using Gaussian fits.

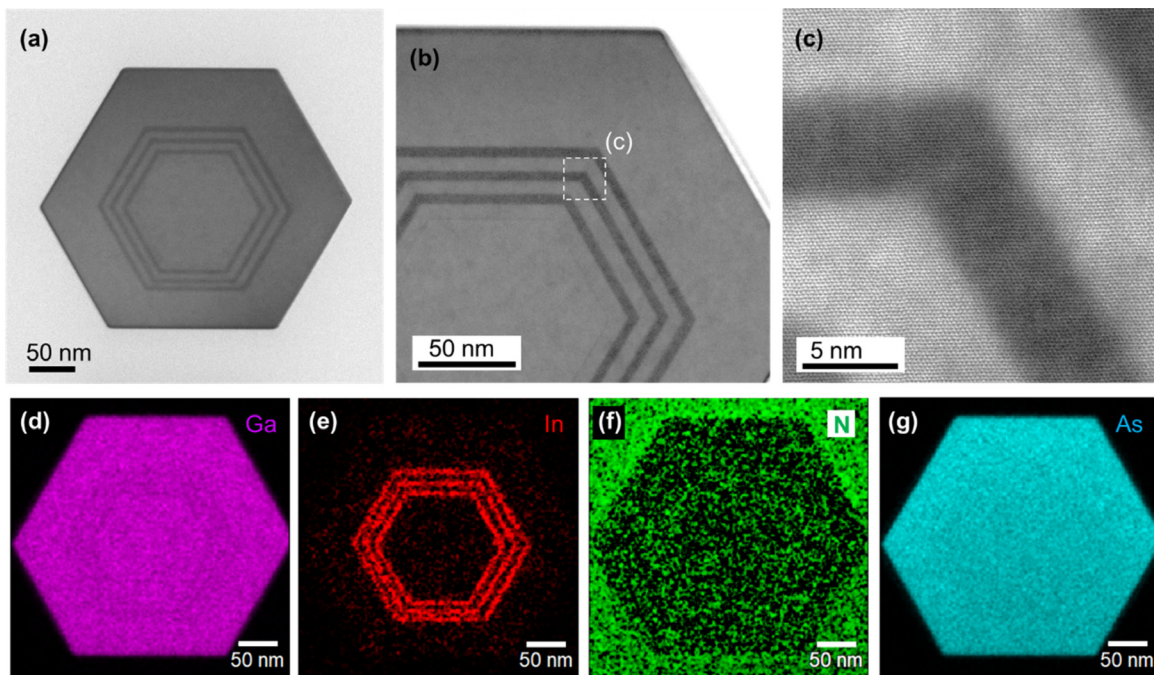


FIG. 4. (a) Cross-sectional BF-STEM images of the GaInNAs nanowire nominally containing 20% In and 2% nitrogen. (b) Higher magnification of the BF-STEM image around the upper right shell area in (a) and (c) its further enlarged image at the GaInNAs quantum wells delimited in (b). (d)–(g) EDS elemental mappings of Ga, In, N, and As, respectively.

of GaAs/GaInNAs/GaAs quantum-well layers forming a triple quantum-well structure. The dimensions of the corresponding layers are approximately equal to 140 nm (the GaAs core), 9 nm (the width of the GaInNAs quantum wells), 11 nm (the width of the GaAs barriers), and 70 nm (the width of the outermost GaAs shell). Figure 4(b) shows a higher magnification image of the upper right part of (a), and Fig. 4(c) presents an enlarged view at the area delimited in (b). The obtained high-resolution images confirm that the nanowire contains a well-formed GaAs/GaInNAs triple quantum-well structure in the shell.

Figures 4(d)–4(g) show the energy dispersive x-ray spectroscopy (EDS) elemental mapping of Ga, In, N, and As, respectively. The enhancement of the In-related EDS signal can be clearly seen in Fig. 4(e), providing the direct experimental proof for the presence of In within the GaInNAs shell layer. However, the introduction of N in the GaInNAs shell layer cannot be clearly seen from Fig. 2(f), probably due to the small concentration of N close to the detection limit, which is about 3%.³⁰ The In mapping in Fig. 4(e) shows lateral inhomogeneities. Moreover, in the BF-STEM image in Fig. 4(c), which is sensitive to strain, contrast variations of the same scale are visible. The feature is considered to be phase separation, which is observed for GaInNAs alloys with high In and N contents.^{53,54}

Further EDS investigations were carried out to reveal the information on N in the GaInNAs quantum well. In the high-angle annular dark field (HAADF) image shown in Fig. 5(a), the GaInNAs shell can be recognized since the large atomic number of In provides a largely bright contrast.⁵⁵ Figure 5(b) shows the EDS line profiles for Ga, In, N, and As elements across the GaAs/GaInNAs triple quantum-well, as

indicated by the arrow in Fig. 5(a). The line profiles clearly indicate the formation of three quantum wells, as recognized by the corresponding peaks of In and the slight dip of Ga. In addition, the existence of N with its density close to the detection limit, which is estimated to be at 2%–3%,³⁰ is also confirmed. The outer-shell side nitrogen signal, indicated by the green arrow, could be related to the extinction of plasma source with the termination of nitrogen gas, which occasionally splashed active nitrogen species.⁵⁶

The optical quality of the grown NWs was evaluated using PL spectroscopy at RT. The results of the RT-PL measurements on the series of samples containing different amounts of nitrogen are summarized in Fig. 6. The PL spectrum of the N-free NW structure contains a single peak at 1080 nm. Based on the PL peak position in this case and by neglecting the quantum confinement effects, the indium content of the GaInAs layers can be estimated at approximately 18%.⁵⁷ This represents the lowest limit of the In concentration as quantum confinement effects will increase the energy of band-to-band transitions. Therefore, the PL results confirm the intended In composition of approximately 20%. The PL peak position shifts to 1170 and 1255 nm with an increasing nitrogen content [N]. This is accompanied by a decrease in the PL linewidth and a decrease in its intensity in the sample with the highest [N]. The observed narrowing of the PL spectra upon nitrogen incorporation could be attributed to an improved uniformity of the forming alloy and a local strain compensation by co-doping, since introduction of small nitrogen atoms can compensate strain near large In atoms. Moreover, a decrease in the PL intensity in the NWs with the highest nitrogen content is likely indicative of the formation of point defects, which is common in thin films.⁵⁸

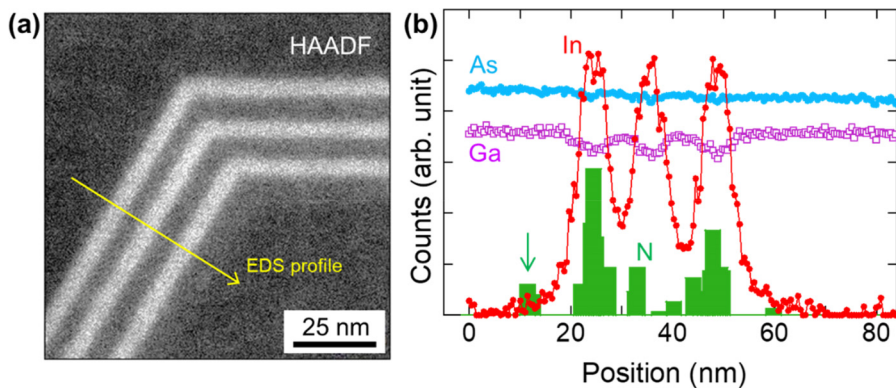


FIG. 5. (a) HAADF-STEM images of the GaAs/GaInNAs/GaAs core-multishell nanowires nominally containing 20% In and 2% N in the GaInNAs shell. (b) EDS scan profile for the elemental intensities of Ga, In, N, and As across the GaAs/GaInNAs triple quantum wells along the arrow indicated in (a). An unintentional nitrogen signal introduced by the plasma extinction is indicated by the green arrow in (b).

Nevertheless, the PL emission remained very bright even in this structure. Therefore, the results of the RT-PL measurements clearly prove the growth of high optical quality GaInNAs/GaAs heterostructure NWs with nitrogen composition approaching 2% and emitting close to the optical communication window of 1.3 μm .

In summary, we reported the growth of triple quantum-well GaAs/GaInNAs/GaAs core-multishell nanowires (NWs) having 2% N with 20% In. The NWs were grown by selective area plasma-assisted MBE on patterned Si(111) substrates with SiO₂ mask holes. The nucleation and the growth of the GaAs nanowire core were carried out by the constituent Ga-induced vapor–liquid–solid growth at the open holes. We then obtained finely controlled regular core-multishell NWs containing GaAs/GaInNAs triple quantum wells with uniform wire length and diameter with an overall 96% yield. The nitrogen concentration of the GaInNAs shell was noted to vary nominally at 0%, 2%, and 3%. The GaInNAs NWs showed a spectral red shift relative to the peak from GaAs NWs with their emission up to 1.26 μm of telecommunication wavelengths with increasing N content, making them a promising tool in telecommunication light sources.

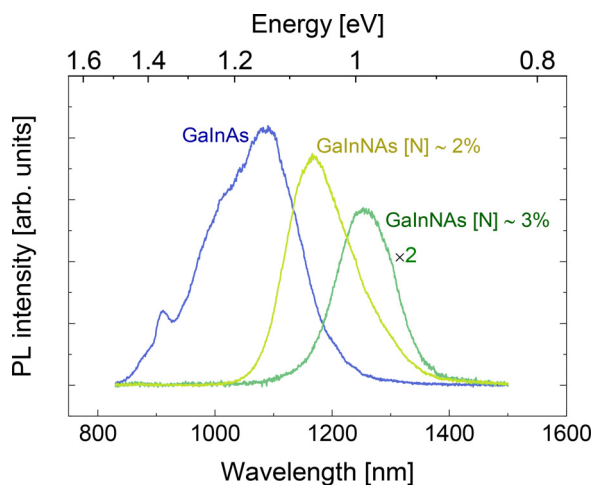


FIG. 6. PL spectra of GaAs/GaInNAs/GaAs core-multishell nanowires acquired at room temperature. The PL intensity in the structure with the highest nitrogen content is scaled by a factor of two for clarity.

See the supplementary material for details regarding SEM images of the initial nucleation of the GaAs core on the patterned substrate after a 5-min growth at different square mask openings and SEM images for nanowires comprising GaInAs quantum wells, GaInNAs quantum wells containing 2% nitrogen, and GaInNAs quantum wells containing 3% nitrogen.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Kaito Nakama: Conceptualization (lead); Data curation (lead); Formal analysis (lead); Investigation (lead); Writing – original draft (lead). **Fumitaro Ishikawa:** Conceptualization (equal); Data curation (equal); Formal analysis (equal); Funding acquisition (lead); Writing – original draft (equal); Writing – review & editing (lead). **Mitsuki Yukimune:** Data curation (supporting); Formal analysis (supporting); Investigation (supporting); Writing – original draft (supporting). **Naohiko Kawasaki:** Data curation (supporting); Investigation (supporting). **Akio Higo:** Data curation (supporting); Funding acquisition (supporting); Investigation (supporting). **Satoshi Hiura:** Data curation (supporting); Investigation (supporting). **Akihiro Murayama:** Data curation (supporting); Investigation (supporting). **Mattias Jansson:** Data curation (supporting); Formal analysis (supporting); Investigation (supporting). **Weimin M. Chen:** Data curation (supporting); Formal analysis (supporting); Funding acquisition (supporting);

Investigation (supporting). **Irina A. Buyanova**: Data curation (supporting); Formal analysis (supporting); Funding acquisition (supporting); Investigation (supporting).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding authors upon reasonable request.

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