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Charge carrier generation, relaxation, and recombination in polytypic GaAs nanowires studied by photoluminescence excitation spectroscopy

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We report results of a study of polytypic GaAs nanowires using low-temperature photoluminescence excitation spectroscopy. The nanowire ensemble shows a strong absorption at 1.517 eV, as a result of resonant generation of heavy-hole excitons in the zinc-blende segments of the nanowires. Excitons then diffuse along the length of the nanowire and are trapped by the type-II quantum discs arising from the zinc-blende/wurtzite crystal structure alternation and recombine radiatively. Finally, experiments on single nanowires demonstrate that the energy of the Γ_7 conduction band to Γ_9 valence band exciton of wurtzite GaAs is 1.521 eV at 4 K.

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One-dimensional semiconductor nanostructures are currently attracting huge interest for optoelectronic applications, as they can improve efficiency of solar cells,^{1,2} and provide high-yield single photon emitters^{3–8} or low-threshold coherent light emitters.^{9,10} Non-nitride III-V semiconductor layers grow in a zinc-blende (ZB) crystal structure, but in the case of nanowires it is possible to grow III-V materials in a wurtzite (WZ) or a ZB form. The possibility of tuning the crystal phase along the length of nanowires offers additional degrees of freedom regarding the tuning of the electronic properties of nanowires. Not only do the WZ and ZB crystal phases of a III-V semiconductor have different electronic properties,¹² but it is also now possible to realize superlattices¹² or quantum dots¹³ based on the alternation of WZ and ZB phases along the length of nanowires. WZ GaAs nanowires have been grown in which the stacking faults are few, or entirely absent.^{14–16} Despite this technical progress, the band structure of WZ GaAs remains unclear. There is still controversy surrounding the value of the bandgap^{15,17–20} and of the electron effective mass,^{11,21–23} and the symmetry of the lowest energy conduction band has yet to be clarified.^{15,16,24} Non-resonant photoluminescence (PL) has been useful in elucidating the interplay between charge carrier relaxation and recombination mechanisms, but it cannot alone answer the questions regarding the band structure as the nanowire emission near the band-edge at low-temperature is dominated by the recombination of excitons bound to point^{25–27} and extended defects.^{22,28,29} Resonant techniques, such as photoluminescence excitation spectroscopy (PLE)²⁸ and resonant Raman scattering (RRS),^{15,16} are preferable for probing the band structure of nanowires with WZ crystal structure.

In this letter, we present a combined low-temperature PLE and PL study of polytypic GaAs nanowires grown by catalyst-free molecular beam epitaxy. Based on experiments

on ensembles and on single nanowires, we describe the generation, relaxation, and recombination processes of excitons for laser excitation energy in the 1.512–1.700 eV range. Finally, we deduce that the energy of the Γ_7 conduction band to Γ_9 valence band exciton of WZ GaAs is 1.521 eV at 4 K, and we discuss in terms of heavy-electron light-electron mixing the implications of this result for the electronic properties of GaAs WZ/ZB quantum discs.

GaAs nanowires have been grown by molecular beam epitaxy on SiO₂-coated (111)B GaAs. During the growth, the As partial pressure was increased from 8.8×10^{-7} to 3.3×10^{-6} millibars. The nanowires exhibit a length of a few microns and a diameter of typically 200 nm. The average volume fraction of material with ZB crystal phase is larger than 80%. The two ends of the nanowires exhibit faulted ZB and WZ structures, respectively, and the intermediate segments show a non-periodic alternation between WZ and ZB phases with spacing in the range 1–10 nm.^{17,22} The nanowires were capped by an Al_{0.33}Ga_{0.67}As shell in order to reduce the efficiency of non-radiative surface recombination.³⁰ For experiments on single nanowires, we dispersed the wires on a Si substrate. The ensembles and dispersed nanowire samples were held in a continuous flow He cryostat operating at 4 K. PL and PLE experiments were carried out using a continuous-wave Ti:Sapphire laser continuously tunable between 1.4 and 1.7 eV and with linewidth smaller than 160 μ eV. The laser power and wavelength were monitored with an (In,Ga)As photodiode and a Fizeau interferometer, respectively. The laser light was focused with a microscope objective of NA = 0.5 down to a 1.5 μ m diameter spot and the nanowire emission was collected by the same objective. For PL experiments, the signal was sent to a 64 cm focal length spectrograph equipped with 600 and 1200 grooves per mm gratings followed by a CCD. In the case of PLE experiments, the nanowire emission was sent to a 64 cm focal length triple spectrometer working in subtractive mode, allowing the analysis of emission energetically close to the laser line (closer than 1 meV). For PL and PLE experiments

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on ensembles and on single nanowires, the laser was polarized perpendicularly to the nanowires axis (i.e., polarization perpendicular to the [111] and [0001] crystallographic axes of ZB and WZ GaAs, respectively).

We show in Figure 1(a) a PL spectrum of an ensemble of as-grown nanowires taken with excitation at 1.650 eV and a power of 3 nW. The wire PL shows several emission peaks in the 1.440–1.515 eV range. The number and the positions in energy of these emission lines vary from place to place on the sample and their linewidths range typically between 500 μ eV and a few meV. When increasing the excitation density up to 8 μ W, the shape of the multi-peak spectrum evolves towards a Gaussian centred at 1.495 eV with a full width at half maximum of 35 meV (Figure 1(b)). On its high-energy side, we detect an additional emission band centred at 1.516 eV with a 7 meV broadening. In agreement with previous reports,^{17,22} we attribute the PL emission between 1.440 and 1.515 eV to the recombination of excitons in

polytypic segments of the nanowires. The narrowest lines observed here arise from the recombination of excitons bound to single WZ/ZB quantum discs. The emission energy of such excitons depends strongly on the local coupling scheme between WZ and ZB GaAs, as discussed in Refs. 22 and 31. When the excitation density is increased, filling of available quantum disc states and population of the nearly fault-free WZ and ZB segments result in a broadening of the discs PL and in the appearance of the 1.516 eV band, respectively (Figure 1(b)). Finally, we note that for an excitation power larger than 2 μ W, heating of the sample leads to a red-shift of the nanowire PL, and analysis of the high-energy side of the 1.516 eV PL band also indicates an increase in effective carrier temperature (from 25 to 100 K, when the excitation power is increased from 2 μ W to 3 mW).

We show in Figure 2(a) a typical micro-PL scan taken along the length of a single nanowire and obtained with a laser excitation at an energy of 1.650 eV. The energy of

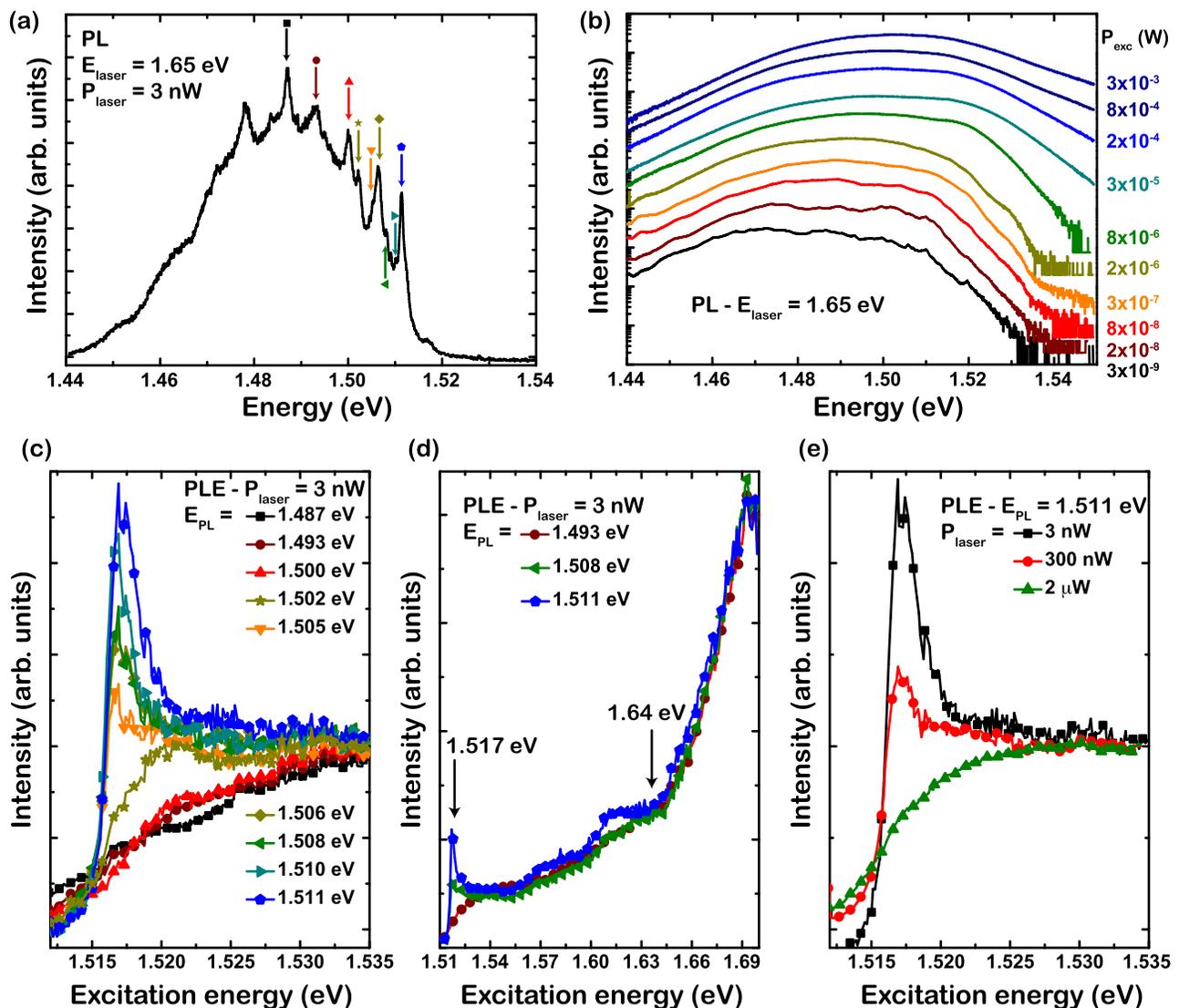


FIG. 1. (a) PL spectrum taken on an ensemble of as-grown nanowires with a laser excitation energy and power of 1.65 eV and 3 nW, respectively. Symbols show the detection energies for the PLE scans in (c) and (d). (b) PL spectra for excitation power between 3 nW and 3 mW (from bottom to top). (c) and (d) PLE spectra taken with a laser excitation power of 3 nW and a laser excitation energy between 1.512 and 1.535 eV (c) and between 1.512 and 1.700 eV (d). For clarity, PLE scans in (c) and (d) have been normalized to the emission intensity detected under laser excitation at 1.535 and 1.700 eV, respectively. (e) PLE scan of the 1.511 eV emission taken with a laser excitation power of 3 nW, 300 nW and 2 μ W (black, red, and green lines, respectively). The PLE scans have been normalized to the emission intensity detected under laser excitation at 1.535 eV.

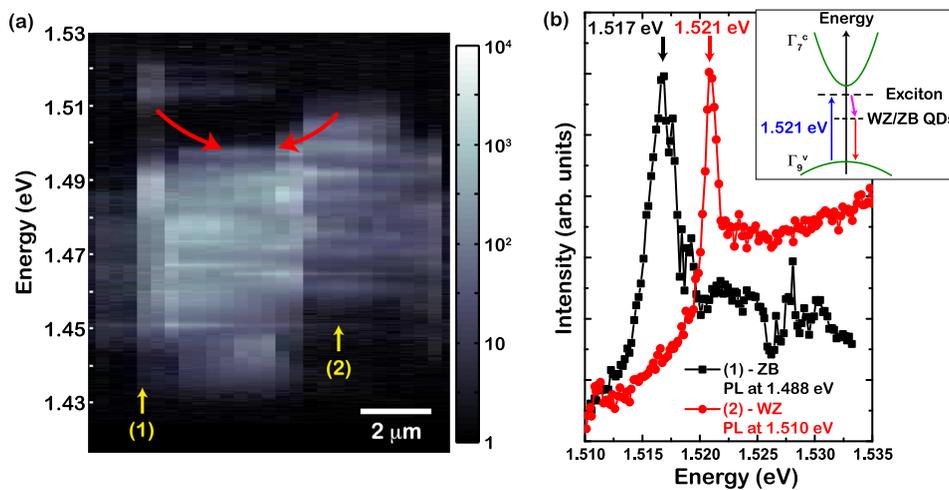


FIG. 2. (a) Micro-PL mapping taken along the length of a single GaAs nanowire. Red arrows highlight the redshift of the PL when going from the extremities to the central segment of the nanowire. (1) and (2) indicate where the PLE scans displayed in (b) have been acquired. (b) Normalized PLE scans taken at positions (1) and (2) (black and red spectra, respectively). Inset: Schematic band structure of WZ GaAs. The energy of the Γ_7^v valence band to Γ_7^c conduction band exciton of wurtzite GaAs is 1.521 eV at 4 K. After excitation at 1.521 eV, excitons are generated resonantly in WZ GaAs segments (blue arrow), they diffuse and get trapped by ZB quantum discs (QDs) (purple arrow), where they recombine radiatively (red arrow).

emission from the quantum discs at the extremities of the wire is 1.490 and 1.495 eV (spectra (1) and (2) in Figure 2(a), respectively), but the central segment of the nanowire shows a PL peak between 1.460 and 1.480 eV. The emission energy is correlated with the local twinning and stacking fault density.¹⁷ In particular, where the local fault density is high, coupling between successive crystal-phase quantum discs leads to a decrease of the exciton energy of the quantum disc.³¹ In agreement with the transmission electron microscopy measurements performed on similar nanowires,¹⁷ the micro-PL scan in Figure 2(a) confirms the model that the two tips of our wires show ZB and WZ crystal structures with low twinning and stacking fault densities, respectively, and the central segments of the wires exhibit repeated alternation between WZ and ZB crystal structures.

We display in Figures 1(c) and 1(d) PLE scans taken with an excitation power of 3 nW on WZ/ZB quantum discs emitting between 1.487 and 1.511 eV. As the laser energy is scanned from 1.512 to 1.535 eV, we detect a strong enhancement of the 1.511 eV emission when exciting at 1.517 eV (Figure 1(c)). Since the nanowires studied show an equivalent volume fraction of ZB crystal phase larger than 80%, we attribute the increase in emission intensity for excitation at 1.517 eV to the resonant generation of heavy-hole excitons in ZB GaAs, followed by their trapping in the quantum discs. The 3 meV inhomogeneous broadening of the heavy-hole exciton is the result of local variations of the strain state of the ZB phase along the length of the wire.³² We note that even when exciting the nanowire ensemble with a laser excitation energy below 1.516 eV, we detect emission from the quantum discs. This is evidence of light absorption by the WZ/ZB discs. However, this process is inefficient, as only a weak PL signal is detected in this case (Figure 1(c)). When going from an excitation energy of 1.530 to 1.640 eV, the PL intensity at 1.511 eV increases by a factor of ~ 2.5 (Figure 1(d)). Then, for higher laser energy, we observe an abrupt increase in emission intensity, which we attribute to the onset of absorption in the $\text{Al}_{0.33}\text{Ga}_{0.67}\text{As}$ shells.³³ All discs, irrespective of their emission energy, exhibit a similar PLE profile for laser energy larger than 1.530 eV (Figure 1(d)), but this is not the case for excitation energy in the 1.512–1.530 eV range. As shown in Figure 1(c), the lower the PL energy of the disc, the less pronounced the heavy-hole

exciton resonance at 1.517 eV. We will demonstrate in the following that the disappearance of the exciton resonance in the PLE scans taken on low-energy emitting quantum discs is a direct consequence of the microstructure of our nanowires.

As discussed above, after excitation at 1.517 eV, excitons are generated resonantly in the ZB GaAs segments with low stacking fault density. Conversely, the longer the ZB segment and the lower the local stacking fault density, the larger the nanowire absorption cross-section at 1.517 eV. At low excitation density, we observe almost no PL signal at 1.516 eV (Figure 1(a)). Excitons generated in the ZB segments therefore diffuse (the exciton diffusion length in undoped GaAs can be of the order of a μm ³⁴) and get trapped by the nearby quantum discs, where they recombine radiatively. In agreement with the micro-PL scan in Figure 2(a), WZ quantum discs in nearly fault-free ZB GaAs segments give rise to the emission in the higher-energy part of the spectrum in Figure 1(a). Consequently, only discs emitting at higher energy may exhibit an increase in PL intensity due to resonant excitation at 1.517 eV, which is in agreement with the data in Figure 1(c). The evolution of the PLE spectrum of the quantum disc emitting at 1.511 eV as a function of the excitation power corroborates this picture. As depicted in Figure 1(e), increasing the excitation density leads (i) to an increase of the PL signal when exciting below 1.516 eV and (ii) to a disappearance of the excitonic resonance at 1.517 eV. We underline that even for the higher excitation power used here (i.e., $2 \mu\text{W}$), our excitation conditions remain below the Mott transition for excitons.³⁵ As a matter of fact, strong excitation leads to filling of the available WZ/ZB discs states (Figure 1(b)) even for excitation below 1.516 eV. As a result of this filling, the transfer of excitons from the ZB GaAs segments to the discs is hindered and no clear resonance is observed when exciting at 1.517 eV (Figure 1(e)). Instead, carriers recombine radiatively in the fault-free ZB segments, giving rise to the 1.516 eV emission band (Figure 1(b)).

We show in Figure 2(b) PLE scans taken at the two extremities of the nanowire in Figure 2(a) for an excitation energy between 1.512 and 1.535 eV. When exciting the left-hand extremity of the wire (position (1) in Figure 2(a)), the disc emission is maximum for a laser energy of 1.517 eV (Figure 2(b)). In agreement with the data obtained on the

wire ensemble (Figure 1), we deduce that the crystal structure of this wire segment consists of faulted ZB. When carrying out a PLE scan at the opposite tip of the wire (position (2) in Figure 2(a)), which exhibits accordingly a faulted WZ structure, we detect a resonance centred at 1.521 eV with a 2 meV linewidth. This resonance arises from the resonant generation of excitons in WZ GaAs followed by their trapping into the nearby ZB quantum discs. The fundamental conduction band of usual semiconductor compounds with WZ crystal structure, such as a GaN or ZnO, is Γ_7^c ,³⁶ *ab initio* calculations have predicted either a Γ_8^c (Ref. 11) or a Γ_7^c symmetry³⁷ for the lowest-energy conduction band of WZ GaAs. Although both the transitions from Γ_7^c and Γ_8^c to the heavy-hole valence band Γ_9^v are allowed for light polarized perpendicular to the wire axis,³⁸ in practice the oscillator strength of the $\Gamma_8^c \rightarrow \Gamma_9^v$ transition is almost zero.³⁷ Consequently, we attribute the exciton transition at 1.521 eV to $\Gamma_7^c \rightarrow \Gamma_9^v$, in agreement with the RRS experiments on a single nanowire reported in Ref. 15. We note that the line broadening observed here for the $\Gamma_7^c \rightarrow \Gamma_9^v$ transition is 2 meV, to be compared with the 8 meV extracted by RRS in Ref. 15. It is at first surprising that the exciton linewidth obtained from a coherent technique is larger than what we observe by PLE. However, the excitation density used for the experiments in Figure 2(b) (0.17 W/cm²) is lower than what is used usually for RRS and we speculate that the difference in exciton linewidth originates from a power-induced broadening of the exciton transition. Finally, we would like to mention that excitons bound to GaAs WZ/ZB quantum discs exhibit scattered values of diamagnetic coefficient²³ and a large scattering rate with acoustic phonons.²² While Γ_7^c electrons are expected to show an effective mass similar to that of the Γ_6^c conduction band of ZB GaAs,^{11,37} the reports in Refs. 22 and 23 suggest rather that the electron mass is heavy. Since the Γ_7^c and Γ_8^c states show a light and heavy electron mass, respectively,^{11,36} we propose that electronic confinement in WZ/ZB GaAs crystal-phase quantum discs leads to a heavy-electron light-electron mixing, in a way similar to the heavy-hole light-hole mixing reported in Ref. 39. The resulting conduction state would exhibit a high effective mass as well as a non-zero oscillator strength for recombination to the valence band, in agreement with experimental reports. For such a mixing to be possible, the energy separation between the Γ_7^c and Γ_8^c conduction bands must be small. This agrees qualitatively with theoretical calculations of the band structure of WZ GaAs, which predict systematically that the energy difference between the Γ_7^c and Γ_8^c bands is smaller than 85 meV.^{11,17,37}

In conclusion, we have performed PL and PLE experiments on polytypic GaAs nanowires both individually and in ensembles. At 4 K, the nanowire PL is dominated by the recombination of excitons bound to WZ/ZB quantum discs. The PLE of ensembles of quantum discs shows a strong resonance at 1.517 eV, as a result of resonant generation of heavy-hole excitons in the ZB GaAs segments followed by their trapping by the discs. For higher excitation density, filling of the discs states results in the disappearance of the 1.517 eV resonance. Instead, we observe a higher-energy emission band centred at 1.516 eV that we attribute to the recombination of carriers in nearly fault-free ZB GaAs wire

segments. We have then deduced from micro-PLE scans taken along the length of single nanowires that the energy of the $\Gamma_7^c \rightarrow \Gamma_9^v$ exciton in WZ GaAs is 1.521 eV at 4 K. Coupled with previous reports, these results suggest that the lowest energy electron state in GaAs crystal-phase quantum discs results from the mixing between the Γ_7^c and Γ_8^c conduction bands of WZ GaAs.

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