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Articles

Synthesis and Characterization of Heterostructured $Mn_3GaN_{0.5}$/GaN Nanowires

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Heterostructured $Mn_3GaN_{0.5}$/GaN nanowires have been synthesized by a catalytic chemical vapor deposition method. $Mn_3GaN_{0.5}$/GaN nanowires exhibit ferromagnetic properties with a Curie temperature above room temperature. It was observed that the acceptor Mn^{2+}/Mn^{3+} (Mn 3d) levels superpose on the GaN energy levels, shifting the energy of 5.8 eV toward Fermi energy. Compared with GaN nanowires, the D^0X line of $Mn_3GaN_{0.5}$/GaN at 397.8 nm (3.117 eV) indicated the 33.4 nm red-shift due to hole-doping in the PL spectra. The synthesized $Mn_3GaN_{0.5}$/GaN nanowires had the relatively high yellow band emission of about 596.5 nm, due to the internal ${}^4T_1 \rightarrow {}^6A_1$ transition of Mn^{2+} ($3d^5$), which is permitted by selection rules. The sharp peak at 675.6 nm in $Mn_3GaN_{0.5}$/GaN nanowire PL spectra might correspond to the transition ${}^4T_2 \rightarrow$ defect state.

1. Introduction

The synthesis of GaN nanowires is important for both fundamental research and technology and opens up a variety of opportunities for industrial applications. The heterostructured MnGaN/GaN nanowire in which the doping is varied on a nanoscale is essential for integration in fundamental nanosystems. Han et al. first synthesized GaN nanorods through a reaction confined in a multiwalled carbon nanotube.[1] Since then, many research groups reported the synthesis of GaN nanowire using catalytic chemical vapor deposition (CCVD) appropriate to diverse nanosystems.[2,3] Recently, Gudiken et al. and Lauhon et al. announced GaP/GaAs superlattice structures and i-Si/p-Si and Si/Ge heterostructures by CCVD methods, respectively.[4,5] More recently, Cheng et al. reported Mg-doped GaN nanowires and Qian et al. announced n-GaN/InGaN/p-GaN nanowire heterostructures by CCVD methods.[6,7] However, there was no report on MnGaN/GaN heterostructured nanowires, so we have studied the synthesis of Mn-doped GaN and the coaxial structure of MnGaN/GaN nanowires for application to spintronic devices.

Recently, great interest has been focused on spintronics for electronic and photonic devices. The introduction of diluted magnetic semiconductors (DMS) becomes a fundamental issue in many spintronic applications due to compatibility with present semiconductor devices.[8] Many researchers have investigated GaN-based materials as magnetic semiconductors for applications to spintronics. Magnetic spintronic devices implement magnetic random access memory, magnetic sensors, the hard drive of a personal computer, spin light-emitting diodes, spin laser diodes, solar cells, optical switches, modulators, quantum computers, optical isolators, and electrically controlled ferromagnets.[9−11]

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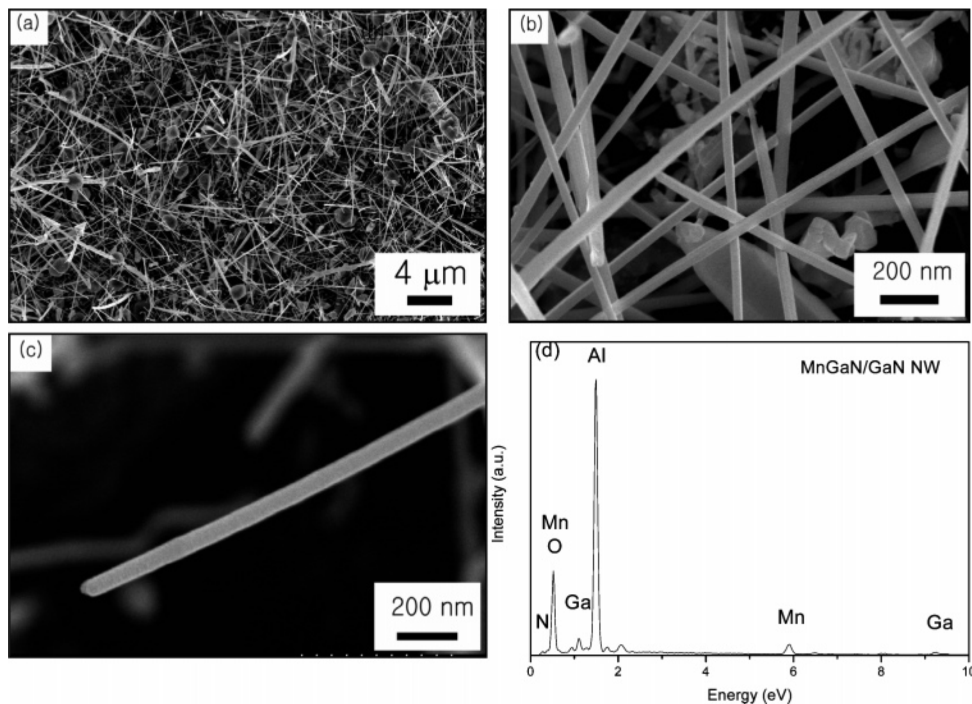

Figure 1. (a−c) SEM images of heterostructured MnGaN/GaN nanowires on alumina substrates. (d) EDX spectra of MnGaN/GaN nanowires on Al_2O_3 substrates. EDX indicates that the MnGaN/GaN nanowires are composed of Ga, N, and Mn, and Al and O are elements from Al_2O_3 substrate.

Spintronic devices must be controlled above room temperature without cryogenic temperature devices for practical applications. The key technology for creating practical spintronic devices is the increase of the Curie temperature above room temperature. The room temperature ferromagnetism of GaN-based materials has been investigated deeply after the theoretical prediction of GaMnN ferromagnetism above room temperature.[12−14]

Compared with MnGaN films, MnGaN nanowires have some novel advantages for spintronic devices. One advantage is that one-dimensional nanowire structures can promise nanoscale electronic and photonic devices. The second advantage is that the nanocharacteristics such as a quantum confinement effect can be applied effectively to the nanodevices. The third advantage is that electrically and magnetically controlled photonic structures can be constructed by assembling nanowires in periodic structures for the novel device applications. The one-dimensional properties of ferromagnetic MnGaN nanowires can control the spin injection to the spintronic devices precisely and effectively. Moreover, heterostructured nanowires are especially significant for nanosize spintronics because of the versatility and the flexibility of structural applications. One-dimensional heterostructured nanowires may open up numerous novel device applications in nanosize electronics and optoelectronics.

Previously, several reports have announced that the 3d transition metal Mn-doped GaN films show room-temperature ferromagnetism.[15−18] Reed et al. observed high-temperature ferromagnetism in a MnGaN film at 363 K Curie temperature.[16,17] Even if the first room-temperature ferromagnetism of MnGaN nanowires was realized by Deepak et al.[19] using carbon nanotubes as reaction template and Han et al.[20] reported the room-temperature ferromagnetism on 5% Mn-doped GaN nanowires, no reports have been announced about the synthesis of ferromagnetic heterostructured MnGaN/GaN nanowires by CCVD methods.[19] Therefore, it is still very desirable to synthesize ferromagnetic heterostructured MnGaN/GaN nanowires by CCVD.

In this work, we demonstrate the synthesis of heterostructured $Mn_3GaN_{0.5}$/GaN nanowires in situ by a CCVD method and also report the room-temperature ferromagnetism for the $Mn_3GaN_{0.5}$/GaN nanowires. We suggest that the room-temperature ferromagnetism of Mn-doped GaN nanowires originated from crystallized $Mn_3GaN_{0.5}$ structures.

2. Experimental Section

Heterostructured MnGaN/GaN nanowires were prepared in situ by a catalytic chemical vapor deposition (CCVD) method using gallium metal (Ga, 99.999%, Sigma-Aldrich) and manganese chloride ($MnCl_2$, 99.99%, Sigma-Aldrich) sources. The nickel nitrate/ethanol solution with the concentration of 0.01 M was soaked

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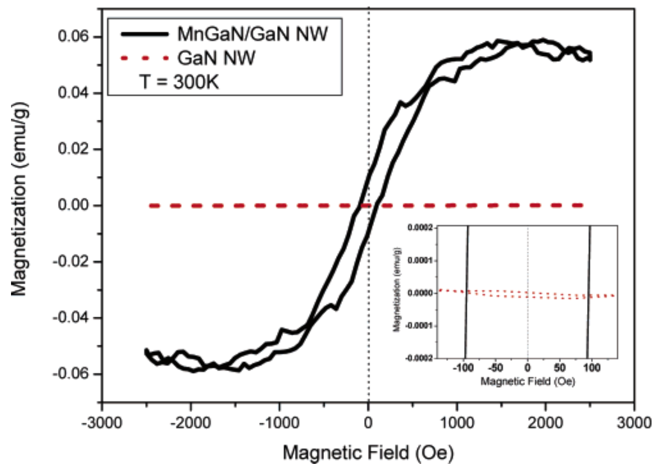

Figure 2. VSM spectra for GaN and heterostructured MnGaN/GaN nanowires measured at room temperature. The spectra clearly show the ferromagnetic properties of MnGaN/GaN nanowires. The inset shows an enlargement near the origin.

on the surface of alumina (Al_2O_3) substrates (10 × 10 mm in size). After drying the substrates in air, the catalyzed alumina substrates were placed vertically on the side of a quartz boat loaded with Ga powder. The upright separation distance between the Ga source and Ni-catalyzed alumina substrate was about 10 mm. The quartz boat that contained the Ga source and the catalyzed alumina substrates were located downstream of the Mn source inside the quartz tube of a CVD system. The separation distance between Mn source and the substrate was about 5 in. The quartz tube was evacuated to less than 0.01 Torr. A carrier gas of Ar was kept flowing through the quartz tube at a flow rate of 1000 sccm, while the temperature was increased from room temperature to 900−1000 °C at a rate of 35 °C/min. In the first step, the GaN nanowires were synthesized in the temperature range of 900−1000 °C for 40 min under the constant flow of 100 sccm ammonia (NH_3) and 100 sccm hydrogen (H_2). In the second step, Mn-doped GaN nanowires were synthesized by reacting with $MnCl_2$ for 20 min. After the reaction, light red materials were deposited on the surface of alumina substrate. The morphologies and microstructures of the deposited GaN nanowires were studied by scanning electron microscopy (SEM) (Hitachi S-4700), transmission electron microscopy (TEM) (Hitachi H-9000 NAR), X-ray diffraction (XRD) (Rigaku DMAX PSPC MDG 2000), vibrating sample magnetometry (VSM), X-ray photoelectron spectroscopy (XPS) (VG Scientific ESCA-LAB 220i), energy-dispersive X-ray spectroscopy (EDX) (Hitachi S-4700), and photoluminescence (PL) measurements using the 325-nm line of a He−Cd laser as excitation source.

3. Results and Discussion

SEM observation shows that the synthesized nanowires have an average diameter of 50 nm and length of several tens of micrometers, as shown in Figure 1a−c. The as-synthesized nanowires have uniform morphology and clean surface, as shown in Figure 1a−c. Energy-dispersive X-ray spectroscopy (EDX) analysis indicates that the synthesized nanowires are MnGaN nanowires on an alumina (Al_2O_3) substrate, consisting of Ga, N, and Mn elements, as shown in Figure 1d.

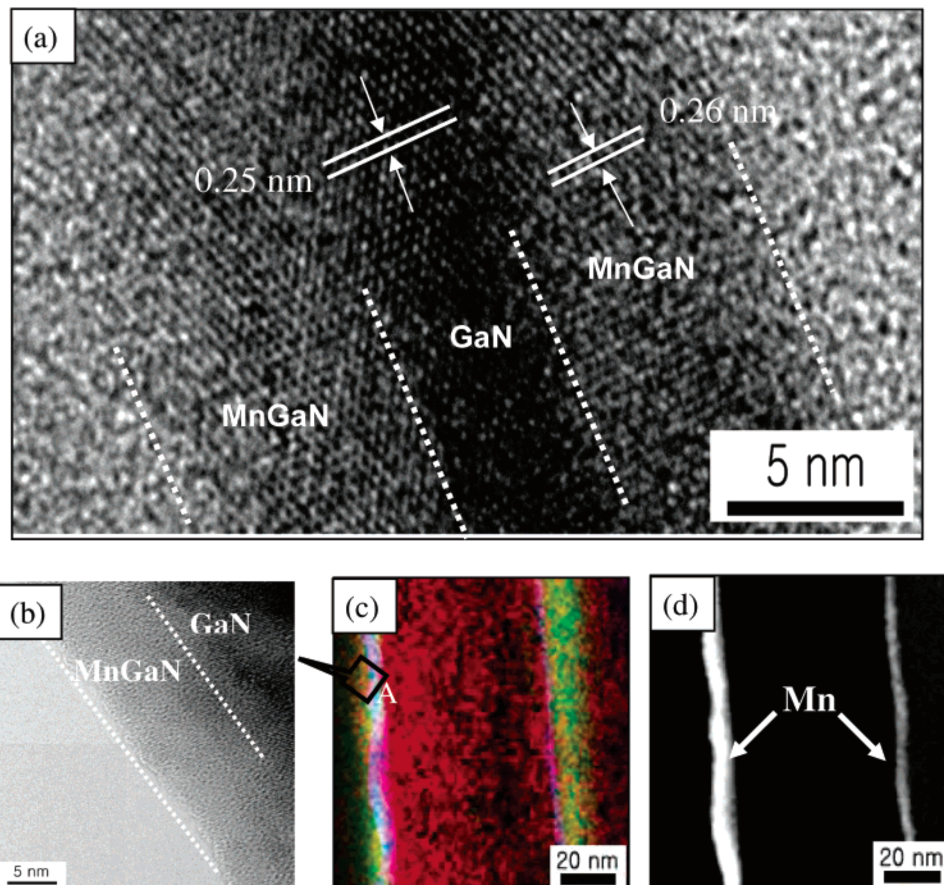

Figure 3. TEM images and mapping of heterostructured MnGaN/GaN nanowires: (a) a high-resolution TEM image of a heterostructured MnGaN/GaN nanowire, (b) a high-resolution TEM image of another heterostructured MnGaN/GaN nanowire (an enlargement of region A in Figure 3c), (c) final mapping of a heterostructured MnGaN/GaN nanowire, and (d) Mn elemental mapping of a heterostructured MnGaN/GaN nanowire.

Figure 2 shows vibrating sample magnetometer (VSM) magnetization curves at room temperature for the GaN and heterostructured MnGaN/GaN samples. The hysteresis curves of heterostructured MnGaN/GaN nanowires show clear ferromagnetic behaviors of the Mn-doped nanowires, while the curves of GaN nanowires show nearly a flat line with little magnetization. The remnant magnetization and the coercive field intensity for the heterostructured DMS nanowires are 0.01 emu/g and 108 Oe, respectively. This hysteresis curve shows that these heterostructured DMS nanowires are composed of soft ferromagnetic material. The inset indicates that the remnant magnetization of the GaN nanowires is 0.000014 emu/g, which originated from Ni catalysts.

TEM shows that the inner layer (GaN) indicates crystalline structures, but the outer layer (MnGaN) presents some dislocations or stacking faults, as shown in Figure 3a,b. TEM observation confirms that the as-synthesized nanowires have coaxial heterostructures that consist of the Mn-doped MnGaN layer outside and GaN layer inside. The lattice spacing of the GaN inner layer and MnGaN outer layer is estimated to be 0.25 and 0.26 nm from the lattice fringes of the TEM image in Figure 3a, respectively. The TEM image shows the lattice mismatch between GaN and MnGaN in Figure 3a. Figure 3b shows the TEM image of another MnGaN/GaN nanowire's outer layer. Final elemental mapping for the synthesized MnGaN/GaN nanowire supports that the produced nanowires consist of heterostructures, as shown in Figure 3c, and Mn mapping shows that Mn elements only appear outside of that nanowire, as shown in Figure 3d.

The heterostructured MnGaN/GaN and GaN nanowires were characterized with Cu Kα radiation by X-ray diffractometer (XRD). In Figure 4a, the XRD spectrum shows the peaks of (100), (002), (101), (102), (110), (103), indicating that the GaN and MnGaN/GaN nanowires have hexagonal wurtzite structures. The other peaks are identified as the rhombohedral Al_2O_3 substrate. There is no Mn metal peak in the XRD of heterostructured MnGaN/GaN nanowires, indicating that no Mn metal is covered on the outside layer of MnGaN/GaN nanowires. The XRD reveals that the synthesized GaN and MnGaN/GaN nanowires have well-crystalline structures. The relatively dominant (101) and (110) peaks of GaN nanowires are observed, implying that most of GaN nanowires are aligned toward (101) and (110) directions, whereas the relatively dominant (002) and (110) peaks of MnGaN/GaN nanowires are observed, implying that most of MnGaN/GaN nanowires are aligned toward (002) and (110) directions and most MnGaN outside layers are grown in (002) directions. In Figure 4b, compared with GaN nanowires, MnGaN/GaN nanowires exhibit a slight decrease in peak positions from 36.88° to 36.80° in the (101) direction. $Mn_3GaN_{0.5}$ compound is identified at the extra peak at 40.46°.

Figure 5 shows the X-ray photoelectron spectroscopy (XPS) spectra of Mn $2p_{1/2}$, Mn $2p_{3/2}$, and Ga 3d core levels and the valence-band levels of heterostructured $Mn_3GaN_{0.5}$/GaN nanowires. In this work, the $Mn_3GaN_{0.5}$/GaN nanowires consist of 10% Mn mass compositions, considering the integration and the atomic sensitivity factors of Mn $2p_{3/2}$, as shown in Figure 5a. The Ga−Mn bonds are revealed by the XPS spectra of Ga 3d, as shown in Figure 5b. The Ga 3d spectrum of the heterostructured $Mn_3GaN_{0.5}$/GaN nanowires is deconvoluted with Ga−N, G−O, and Ga−Mn bonds.[21] The Ga atoms in the $Mn_3GaN_{0.5}$/GaN nanowires are composed of bonding with N, O, and Mn atoms, such as Ga−N, Ga−O, and Ga−Mn bonds, respectively.[21] The relative atomic ratio of Ga/N is 1.7, whose value is determined from the integration of intensities in the Ga 3d and N 1s spectra. The valence level spectra of $Mn_3GaN_{0.5}$/GaN nanowires and GaN nanowires were shown in Figure 5c. The electronic density of states of $Mn_3GaN_{0.5}$/GaN nanowires is greater than that of GaN nanowires near the Fermi energy. This result indicates that the acceptor Mn^{2+}/Mn^{3+} (Mn 3d) levels superpose on the GaN energy levels, shifting the energy of 5.8 eV toward the Fermi energy, as shown in Figure 5c.[13−15]

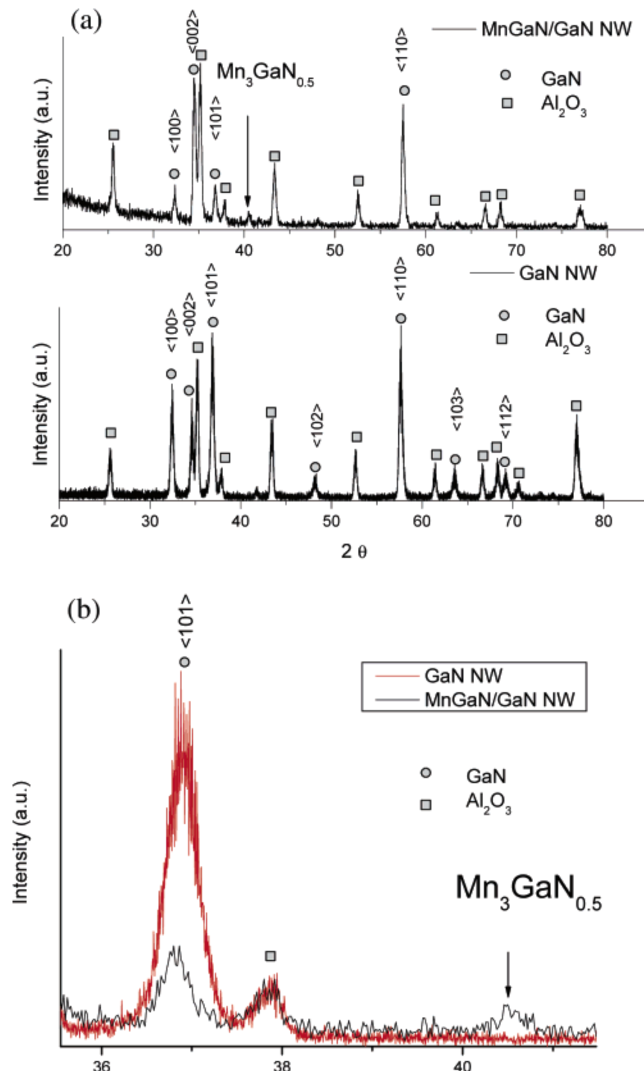

Figure 4. XRD diffraction patterns of heterostructured MnGaN/GaN and GaN nanowires on Al_2O_3 substrates. (a) X-ray diffraction (XRD) spectrum shows the peaks of (100), (002), (101), (102), (110), (103), (112), identifying that the GaN and MnGaN/GaN nanowires have hexagonal wurtzite structures. The other peaks identify a rhombohedral Al_2O_3 substrate. (b) Comparison of the XRD spectrum of heterostructured MnGaN/GaN nanowires and of GaN nanowires exhibits a slight decrease in peak positions from 36.88° to 36.80° in the (101) direction. $Mn_3GaN_{0.5}$ compound is identified as the extra peak at 40.46° (JCPDS card No. 36-145).

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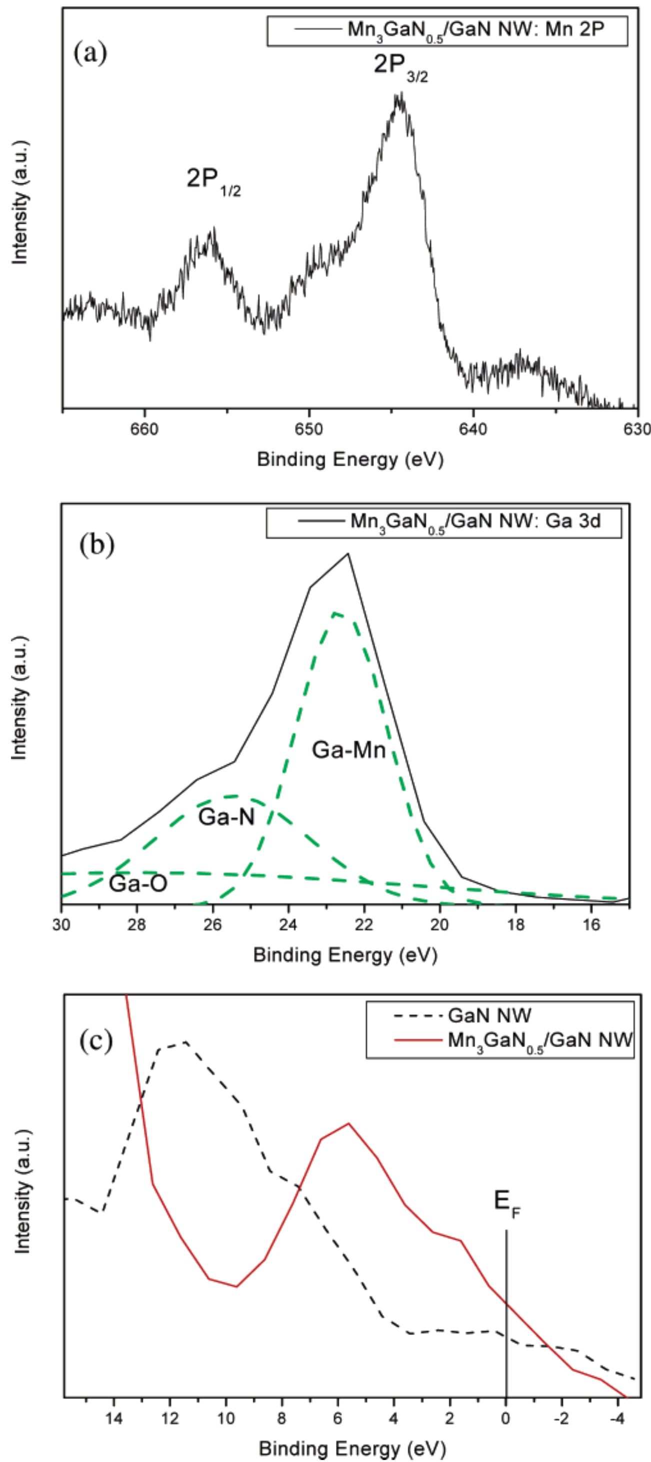

Figure 5. XPS spectra of heterostructured $Mn_3GaN_{0.5}$/GaN nanowires: (a) The Mn 2p spectra of $Mn_3GaN_{0.5}$/GaN nanowires. (b) The Ga 3d spectra of $Mn_3GaN_{0.5}$/GaN nanowires. The spectra are deconvoluted with Ga−Mn, Ga−N, and Ga−O bond curves. (c) The valence band spectra of $Mn_3GaN_{0.5}$/GaN nanowires and GaN nanowires. The Fermi levels of $Mn_3GaN_{0.5}$/GaN nanowires and GaN nanowires were determined by the Fermi level of a Cu foil, defined as the level of zero binding energy.

Some reports announced that the formations of Ga−Mn and Mn−N phases lead to the ferromagnetic properties of MnGaN nanowires, because GaMn is ferromagnetic and Mn_xN_{1-x} is ferrimagnetic.[22−25] However, our results for XRD measurements identify none of these phases but indicate only $Mn_3GaN_{0.5}$ structures. The XRD results confirm the intrinsic ferromagnetic structures of $Mn_3GaN_{0.5}$ through the observation of the (111) reflection for $Mn_3GaN_{0.5}$. Reed et al. also observed that room-temperature ferromagnetic properties were the result of Mn_3GaN structures.[16] In this work, XPS shows the Ga−Mn bonds in the deconvoluted spectra of Ga 3d. We believe that these Ga−Mn bonds originated from $Mn_3GaN_{0.5}$ phases rather than GaMn phases because no GaMn phases have been shown in our XRD measurements.

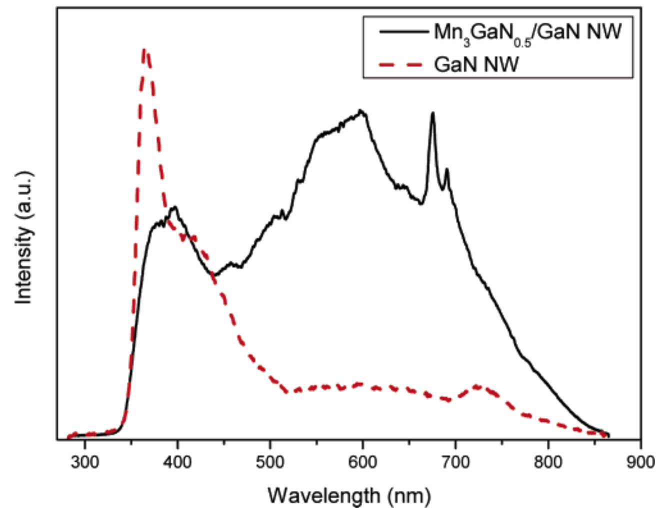

Figure 6. PL spectra of heterostructured $Mn_3GaN_{0.5}$/GaN and GaN nanowires measured at room temperature. D^0X lines of GaN and $Mn_3GaN_{0.5}$/GaN nanowires are 365 and 409 nm, respectively. $Mn_3GaN_{0.5}$/GaN nanowires have relatively higher yellow bands than GaN nanowires at about 600 nm. The sharp peak at 675.6 nm in the $Mn_3GaN_{0.5}$/GaN nanowires' PL spectra might correspond to the transition $^4T_2 \rightarrow$ defect state.

The photoluminescence (PL) spectrum for the $Mn_3GaN_{0.5}$/GaN heterostructured nanowire was obtained at 300 K using a He−Cd laser with excitation at 325 nm, as shown in Figure 6. PL indicates that the neutral-donor-bound-exciton (D^0X) line at 365 nm (3.397 eV) of GaN nanowires leads to a near-band-edge transition, that is, band to band recombination of free excitons bound to neutral donors. Compared with the D^0X line of GaN nanowires, the D^0X line of $Mn_3GaN_{0.5}$/GaN nanowires at 397.8 nm (3.117 eV) indicates that the 33.4-nm red-shift is due to hole-doping.[19,26] D^0X bands have asymmetrical broad line shapes due to the one-dimensional nanosize structures of GaN and $Mn_3GaN_{0.5}$/GaN nanowires. Compared with GaN nanowires, the synthesized $Mn_3GaN_{0.5}$/GaN nanowires have the relatively high yellow band emission of around 600 nm, because of the internal $^4T_1 \rightarrow {}^6A_1$ transition of Mn^{2+} ($3d^5$), which is permitted by selection rules.[19,26−28] The sharp peak at 675.5 nm in $Mn_3GaN_{0.5}$/GaN

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nanowires' PL spectra might correspond to the transition $^4T_2 \rightarrow$ defect state.[28]

4. Conclusions

We have synthesized heterostructured MnGaN/GaN nanowires using a catalytic chemical vapor deposition method. The MnGaN/GaN nanowires consist of 10% Mn mass compositions on the outer layer. The hysteresis curve generated by VSM analysis confirms the room-temperature ferromagnetic properties of Mn-doped GaN nanowries. Ferromagnetic MnGaN nanowires with Curie temperatures above 300 K allow room-temperature spintronic device operation without external magnetic field.

The synthesized MnGaN/GaN nanowires consist of $Mn_3GaN_{0.5}$/GaN and have Ga−Mn bonds, which originated from a crystallized $Mn_3GaN_{0.5}$ structure on the outside layer. We suggest that the ferromagnetic properties of MnGaN/GaN nanowires originated from $Mn_3GaN_{0.5}$ structures.

Compared with GaN nanowires, the D^0X line of $Mn_3GaN_{0.5}$/GaN nanowires at 397.8 nm (3.117 eV) indicates the 33.4-nm red-shift due to hole-doping in the PL spectra. The synthesized $Mn_3GaN_{0.5}$/GaN nanowires have the relatively higher yellow band emission of about 600 nm than the GaN nanowires, due to the internal $^4T_1 \rightarrow {}^6A_1$ transition of Mn^{2+} ($3d^5$).

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