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Characterization of selective quantum well intermixing in 1.3 μm GaInNAs/GaAs structures

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Rapid thermal annealing combined with SiO_2 caps deposited on the surface of samples by different techniques is used to selectively disorder 1.3 μm GaInNAs/GaAs multiquantum wells which have been preannealed *in situ* to the stage of blueshift saturation. After thermal annealing under specific conditions, a shift in band gap of over 170 meV has been obtained in sputtered SiO_2 -capped samples, while uncapped and plasma enhanced chemical vapor deposited SiO_2 -capped samples demonstrated a negligible shift. Quantum well intermixing in sputtered SiO_2 -capped samples originates from enhanced compositional interdiffusion due to the generation of point defects by ion bombardment during the sputtering process. Secondary ion mass spectrometry has confirmed that the enhanced blueshift was caused by the interdiffusion of group III atoms (In and Ga) between the quantum wells and barriers. Detailed photoluminescence and excitation spectroscopy were performed to study the optical properties of both intermixed and nonintermixed samples. © 2003 American Institute of Physics. [DOI: 10.1063/1.1590413]

I. INTRODUCTION

In recent years dilute nitride III–V materials have attracted a great deal of attention. The incorporation of a small amount of N into certain III–V group semiconductors strongly reduces the band gaps of matrix materials. This remarkable property makes it possible to reduce the band gap of InGaAs(N) material to 1.3 or even 1.55 μm optical fiber communication wavelengths.^{1,2} Compared to the InGaAsP/InP material system which is commonly used in this wavelength range, GaInNAs/GaAs has a larger conduction band offset, and so potentially much better high temperature performance. Moreover, GaInNAs can be grown coherently on GaAs substrates, so it is possible to monolithically integrate GaInNAs/GaAs heterostructures with Bragg reflectors composed of AlAs/GaAs. With these advantages, various devices based on this material system have been developed, such as ridge waveguide lasers,³ multijunction solar cells,⁴ vertical cavity surface emitting lasers,⁵ monolithically grown vertical cavity semiconductor optical amplifiers,⁶ and saturable Bragg reflectors for mode-locking applications.⁷ Despite these successes, all the devices reported so far are discrete, but future optical communication networks demand integrated devices which combine several active and/or passive

components with different functions. For this it is essential to realize selective modification of band gaps on a single substrate.

The quantum well intermixing (QWI) technique has proven to be one of the most advantageous approaches to modifying the properties of semiconductor quantum well structures in a controllable way.⁸ The typical origin of band gap modification in this case is that composition interdiffusion occurs between the barriers and quantum wells, so the energy diagram changes and therefore the transition energy increases. The primary advantage of QWI is that it avoids complicated regrowth processes, and therefore is potentially cost effective and reliable. In fact, QWI has been employed in various materials' systems for photonic integrated circuits.^{8–10}

It is desirable to extend the QWI technique to the technologically important GaInNAs/GaAs material system. However, this is still challenging as far as as-grown GaInNAs materials are concerned. The challenge arises from the “natural” variation in band gap induced by thermal annealing. There have been many studies on the effect of rapid thermal annealing (RTA) on as-grown GaInNAs/GaAs samples which were originally aimed at improving the luminescence efficiency. It has been commonly observed that as-grown GaInNAs/GaAs materials show some wavelength blueshifting of photoluminescence (PL) after thermal annealing.^{11–21} The fact that the shift in wavelength of PL

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differs between samples, annealing conditions, and growth methods makes demonstration of intermixing problematic, because the uncontrolled natural variation in band gap induced by thermal annealing is not desirable for practical applications and can easily mask details of QWI. So far the detailed mechanisms behind the blueshift phenomenon remain under debate, and several possible mechanisms have been proposed, including improved uniformity or reduced Stokes shift,^{14,18} the interdiffusion of In and Ga between wells and barriers,^{11,12} the change in N-bonding configuration from a Ga-rich to an In-rich environment,^{16,17,19} and interdiffusion of N out of quantum wells due to interior defects.^{13,15,21} Whatever the mechanisms are, the shift in wavelength of GaInNAs/GaAs materials is known to saturate after a period of annealing, which provides the possibility of controlling the band gap by subsequent QWI. Here, we investigate QWI by means of RTA combined with depositing SiO₂-cap layers by different techniques, using well-characterized GaInNAs quantum well material which has been annealed *in situ* "to saturation." After RTA at 700 °C for ~180 s, controlled shifts in band gap at room temperature of up to ~180 meV have been observed in sputtered SiO₂-capped samples, while uncapped and plasma enhanced chemical vapor deposited (PECVD) SiO₂-capped samples demonstrated a negligible shift. This selective QWI of GaInNAs/GaAs QWs has been characterized by detailed photoluminescence (PL) and photoluminescence excitation (PLE) spectroscopy, and by secondary ion mass spectrometry (SIMS).

II. EXPERIMENT

The GaInNAs/GaAs multi-quantum well (MQW) structures investigated were grown by solid-source molecular-beam epitaxy on GaAs (001) substrates. Five period multiple QWs were sandwiched between two five-period AlAs (2 nm)/GaAs (2 nm) superlattice cladding layers plus 100 nm waveguide layers. The samples were capped with 100 nm of GaAs. Similar structures have demonstrated excellent lasing properties, and a series of reference samples grown under the same conditions has been characterized by spectroscopic techniques.^{22,23} The QW structures under investigation consisted of 30 nm GaAs barriers, and QWs of nominally fixed Ga_{0.62}In_{0.38}As_{0.985}N_{0.015} composition and well width of 7 or 9 nm, respectively. After growth, the samples were *in situ* annealed at 750 °C to activate efficient luminescence, which is known to produce associated blueshifts of several tens of nm. This annealing process assured blueshift saturation as demonstrated by the negligible additional blueshift upon further annealing. The PL peak wavelengths for the as-grown 7 and 9 nm QWs at room temperature (i.e., after *in situ* annealing) are at 1321 and 1290 nm, respectively. Because the 9 nm structure exhibits consistent trends in electronic transitions with 2.5 and 4 nm QWs of nominally equivalent composition,²² we infer from these PL values a small compositional difference from nominal of the 7 nm structure. The material was cleaved into samples ~3 mm × ~3 mm which were then left either uncapped (for reference) or coated with ~50-nm-thick SiO₂-capping layers deposited by either

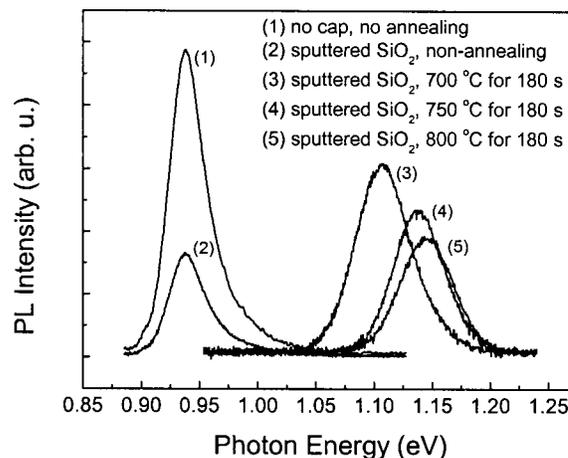


FIG. 1. Evolution of room temperature PL spectra of sputtered SiO₂-capped 7 nm QW samples with different thermal annealing conditions.

PECVD or rf sputtering. Then RTA was performed in a rapid thermal processing system. During thermal processing, the samples were kept in flowing nitrogen ambient, and a silicon or GaAs wafer covered the surface to protect the samples. The PL measurements were performed under excitation of a high power diode laser (670 nm). The PL signal was dispersed by a 0.46 m grating monochromator and detected by a thermoelectrically cooled Si/InGaAs detector using standard lock-in techniques. A variable temperature capability is provided by a liquid He cooled flow cryostat. For the measurement of PLE, the tunable excitation source was provided by a 250 W tungsten-halogen lamp dispersed by a 0.27 m grating monochromator, and scanned under computer control. A SIMS technique was used to measure the composition depth profile of selected samples. This measurement was performed using a Cs⁺ primary ion source as the sputtering gun and a positive secondary ion as the analysis source. The sputtering rate was determined by measuring the depth of the sputtered crater using a profilometer.

III. RESULTS AND DISCUSSION

Figure 1 shows typical room temperature PL spectra of the 7 nm samples with or without sputtered SiO₂ caps, which are either unannealed or annealed for 180 s at different temperatures. Without thermal annealing, the samples have the same PL peak position before and after the sputtering process, which implies that the sputtering process only induces changes to the surface and does not change the structure of the quantum wells. It should be noted that the PL intensity of the sputtered SiO₂-capped sample is about one third that of the original sample. The decrease of PL intensity is ascribed to the reduction of absorbed laser power due to a change of surface conditions and/or absorption by defects at the interface generated by the bombardment of the sputtering process. As the annealing temperature increases, the PL peak shifts significantly towards shorter wavelength. As will be discussed later, this PL blueshift originates from the quantum well intermixing effect due to enhanced composition interdiffusion. It is noticeable that the PL intensity of the 700 °C annealed sample is higher than that of the unannealed sput-

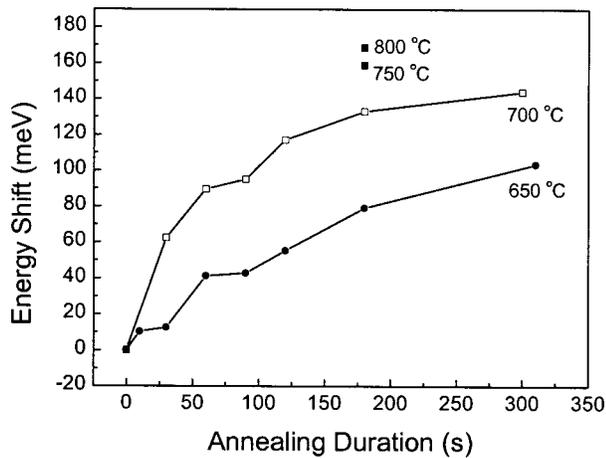


FIG. 2. Dependence of the PL blueshift of sputtered SiO₂-capped 9 nm QW samples on the thermal annealing conditions, measured at room temperature.

tered SiO₂-capped sample. The increase of the PL intensity upon annealing is attributed to further reduction of nonradiative recombination channels which are due to N-related defects and damage induced by N ions in GaInNAs QWs.^{12,13,15} Although the *in situ* annealing has saturated the PL wavelength, the reduction of the nonradiative centers may need further annealing (increased annealing temperature or duration) to fully saturate. Meanwhile, as the annealing temperature increases, the PL intensity decreases gradually (Fig. 1, graphs 3–5). This behavior is correlated to the variation in structure of the QWs induced by QWI. The postannealing process in sputtered SiO₂-capped samples results in enhanced atomic interdiffusion at the QW interfaces, which then moves the quantum-confined states upward in energy and reduces the capability of the QW with graded interfaces to collect and retain carriers and therefore decrease the PL efficiency.²⁴ Another reason for the decrease of PL intensity may originate from the thermal diffusion of point defects at the SiO₂ interface generated by the sputtering process into the QWs. Nevertheless, the decrease in PL efficiency is small and acceptable for device applications of intermixed samples.

Full systematics of the changes of the PL blueshift with the annealing conditions (temperature and time) were obtained in a 9 nm QW sample with similar composition from the same growth series, where more material was available. The dependence of the PL peak blueshift on the annealing conditions is shown in Fig. 2. Note the discrete points for 750 and 800 °C. The blueshift initially increases rapidly and then slows down as the temperature and/or annealing time increase, consistent with features expected for an interdiffusion process.

The “selective” nature of the QWI is demonstrated by distinctive blueshift characteristics observed in samples with SiO₂ caps produced by different fabrication processes. In Fig. 3 we compare the room temperature spectra of an unannealed sample and samples with the same annealing conditions (700 °C for 180 s) but under different capping conditions. Compared with the unannealed reference sample, the annealed samples without caps have a negligible blueshift

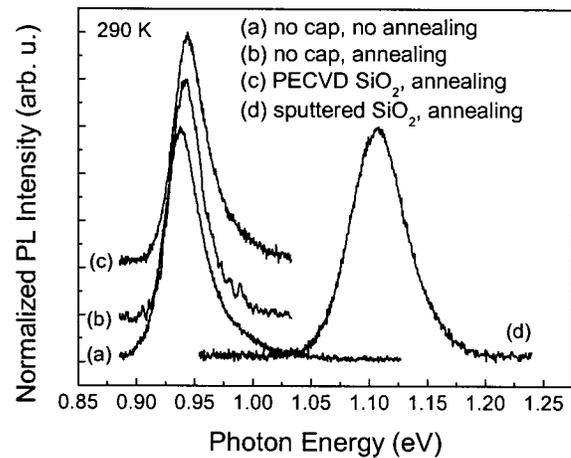


FIG. 3. Normalized PL spectra of 7 nm QW samples with different capping annealed under the same conditions (700 °C for 180 s), measured at room temperature.

(~4 meV), demonstrating that they have indeed been annealed to saturation of the blueshift as long as the annealing temperature is below 800 °C. It is noticeable that the sample capped with PECVD SiO₂ shows a blueshift of only ~7 meV. However, in marked contrast, the sample capped with sputtered SiO₂ shows a blueshift of ~170 meV.

In order to elucidate the origin of the substantial increase of transition energy in the sputtered SiO₂-capped sample, we performed an analysis of the composition profile using SIMS. Figure 4 shows the composition profiles of In, Ga, and N of annealed samples covered with PECVD (thin curves) and sputtered SiO₂ (thick curves). The five QWs and barriers can be seen clearly by the sharp change of both In and Ga composition with the depth. It is believed that little interdiffusion occurred in the PECVD SiO₂ encapsulated sample. This is confirmed by the small blueshift of PL and the near-square shape of the Ga composition in the barriers. The nonsquare shape in the QWs is due to limited depth-resolution and the ion beam mixing effect during SIMS analysis. The relative compositions of In and N in the quantum wells and the layer thicknesses are consistent with the nominal values for the structure designed and what was determined by other techniques (high resolution x-ray diffraction). However, in the sputtered SiO₂-encapsulated sample, apparent compositional interdiffusion of In and Ga has taken place between the QWs and the barriers, while the structure period remains unchanged. This can be clearly seen by the change of profile shape, i.e., the composition of In decreases whereas the composition of Ga increases in the QWs. The change in composition in the QWs is compensated for by corresponding changes in adjacent barriers. However, as shown in Fig. 4(b), there is no observed change in N composition in the above two samples (note that curve 2 was offset vertically for clarity). This suggests that the diffusivity of N and As is very small and cannot account for the QWI. Therefore our results provide unambiguous evidence that the PL blueshift originates from the QWI effect mainly due to the interdiffusion of In and Ga between the QWs and barriers.

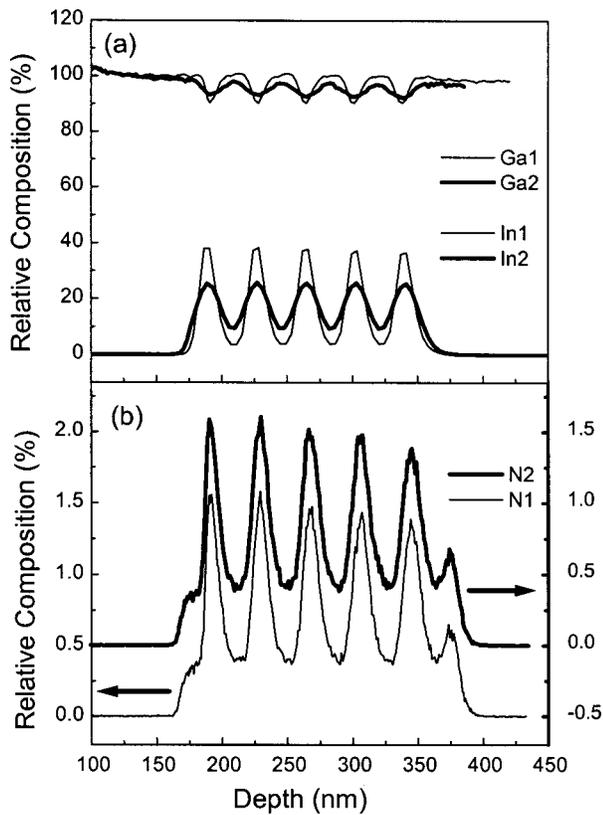


FIG. 4. Composition profiles measured by SIMS for 1, a PECVD SiO₂-capped 7 nm QW sample annealed at 700 °C for 180 s; 2, sputtered SiO₂-capped 7 nm QW sample annealed under the same conditions. Note that in (b) curve 2 was shifted vertically for clarity.

It is interesting to note that the enhanced QWI is not correlated to specific dielectric layers used (which are all SiO₂) but is strongly dependent on the fabrication process. In view of the different influence induced by sputtering and PECVD processes on the sample surface, it is believed that the driving force of enhanced interdiffusion in sputtered SiO₂ capped samples originates from the generation of increased point defects by ion bombardment during the sputtering process.²⁵ In the PECVD process, few point defects are expected to be generated near the sample surface. However, the difference in thermal expansion coefficient between SiO₂ and the semiconductor may generate some defects during the annealing process which promote composition diffusion to some extent, and this can explain the small blueshift in PECVD SiO₂-capped samples. As was pointed out by Kowalski *et al.*,²⁵ the process employed in this work is different from impurity free vacancy disordering (IFVD) which uses a PECVD SiO₂ dielectric cap layer to enhance the intermixing effect. The IFVD process has been ascribed to the outdiffusion of Ga into the SiO₂ cap during the annealing process. This results in the generation of Ga vacancies close to the semiconductor surface which then thermally diffuse into the active region during the annealing process and promote the interdiffusion of atoms in wells and barriers.²⁶ Previously, it was commonly accepted that Ga vacancies mainly promote the interdiffusion of group III atoms, but recent experiments prove that interdiffusion of group V atoms can also be enhanced by Ga vacancies.^{27,28} By looking into de-

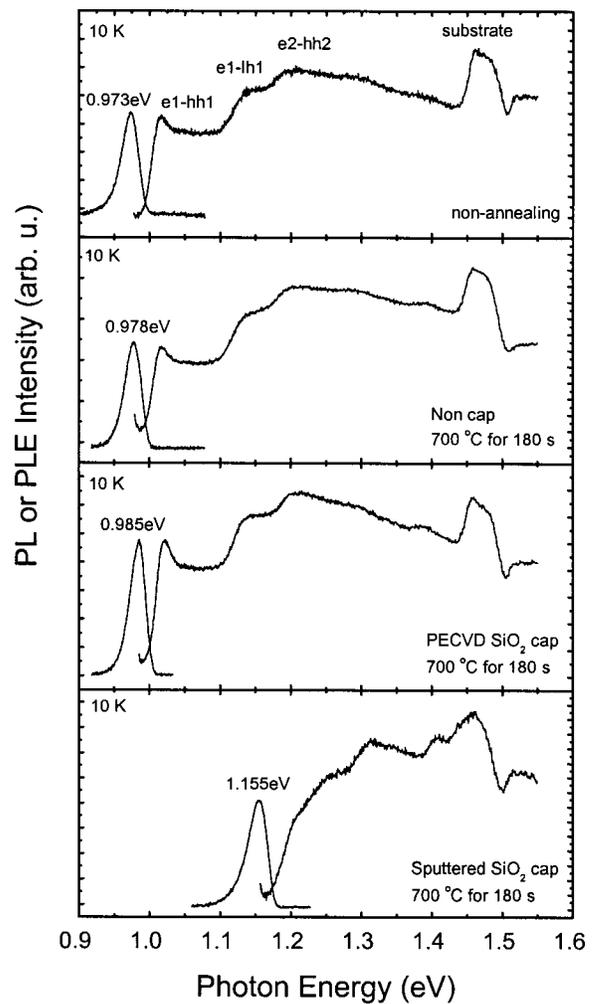


FIG. 5. PL and PLE spectra of 7 nm QW samples with different capping, annealed at 700 °C for 180 s. The PL spectra were taken under excitation of light dispersed from the lamp.

tails of previous experiments of ordinary IFVD,^{26–28} it is found that this process usually occurred at very high temperature (850–950 °C). This may imply that the diffusion of Ga atoms into PECVD SiO₂ layers requires high temperature. In fact, we also observed a blueshift of over 100 nm in PECVD SiO₂-capped samples after 850 °C annealing. Although the diffusion mechanism needs further investigation, the current method is believed to be advantageous over ordinary IFVD. First, the temperature required for sputtered SiO₂-induced QWI is much lower than ordinary IFVD, which is favorable for device processing; second, the different QWI features due to sputtering and PECVD processes can be employed to precisely tune the band gap, because PECVD SiO₂ layers with different thicknesses can be considered as inhibiting control of the point defect densities generated by the subsequent sputtering process.²⁵

To gain deeper insight into the effect of QWI on the optical properties, we focus on a comparison of the PL and PLE properties of the reference sample and annealed samples capped with PECVD and sputtered SiO₂ layers, respectively. The PLE spectra of four differently processed samples were measured at 10 K, as shown in Fig. 5. The PL spectra shown in Fig. 5 were excited by 670 nm wavelength dispersed from

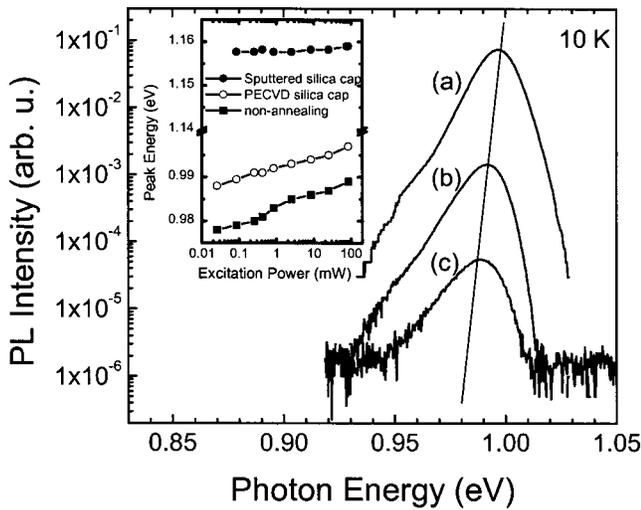


FIG. 6. PL spectra of a PECVD SiO₂ capped sample annealed at 700 °C for 180 s recorded at 10 K under various excitation powers: (a) 84, (b) 0.84, and (c) 0.025 mW; the straight line is a guide for the eye. The inset shows the PL peak energies as a function of the excitation power for different samples.

the lamp/monochromator source, and thus represent very low intensity excitation. As can be seen from the spectra, the photoluminescence in all four samples is from the band edge of the lowest transitions of the QWs. The Stokes shift of ~40 meV between the PL peak energy and lowest absorption peak is an indication that the low temperature PL is from localized states. Consistent with the room temperature PL results, the lowest transition energies (e1-hh1) in the PLE spectra of the uncapped sample and the PECVD SiO₂-capped sample increased only 0 and 5 meV, respectively, relative to the unannealed sample. However, the corresponding transition energy of the sputtered SiO₂-capped sample increased substantially. Similarly, we also observed the shift in energy of other transitions (e1-lh1, e2-hh2) but by a reduced amount, which is qualitatively consistent with the features of modification of transition energies by quantum well intermixing.²⁹ Precise calculation of the transition energies of samples after QWI is complicated by uncertainties in material properties and the exact composition and potential profile, and work to elucidate this is still underway. Currently, our PLE spectra provide direct evidence that the PL blueshift is correlated with the increase of the lowest transition energy in the quantum wells, and only takes place with sputtered-SiO₂ capping. The broadening of the lowest exciton absorption peak in the sputtered-SiO₂ capped sample results from inhomogeneous broadening due to asymmetric diffusion. Nevertheless, compared to the reference sample the absorption edge has a significant blueshift, which is crucial for practical applications.

We performed PL measurements at different excitation and various temperatures for these samples. In Fig. 6, we show PL spectra at 10 K under different excitation powers for a PECVD SiO₂-capped sample. It is clearly seen that with an increase of excitation power the PL peak energy shifts towards higher energy. The dependence of the peak energies on the excitation power for three different samples is shown in the inset of Fig. 6. The PECVD SiO₂-capped

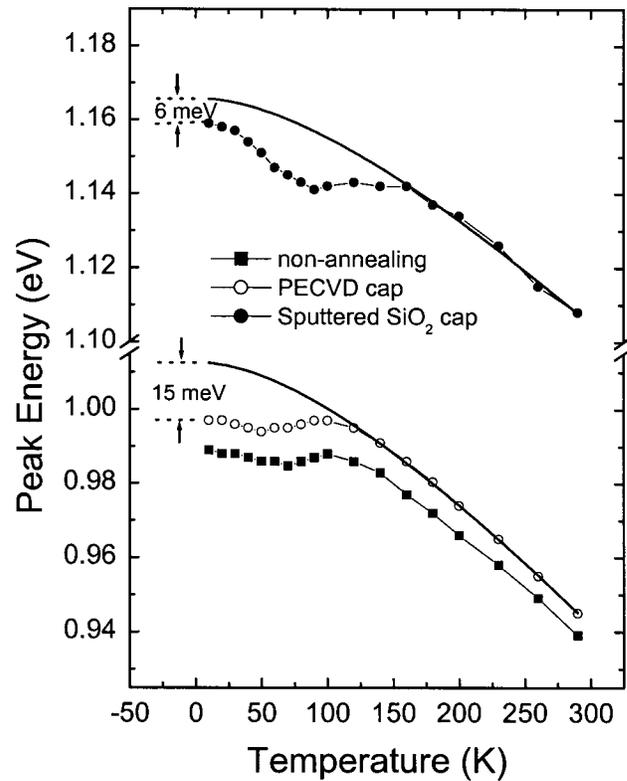


FIG. 7. Dependence of PL peak energies on the temperature for different samples under excitation of a 84 mW laser diode; the solid lines are curves fitted to the Varshni equation.

sample demonstrated the same behavior as the unannealed one. However, to the contrary, the sputtered SiO₂-capped sample showed negligible peak shifting under excitation power that spanned three orders (inset of Fig. 6). The phenomenon that the PL peak energies at low temperature depend on excitation power is ascribed to the gradual filling of strongly localized states within the band tail. These strongly localized states may originate from fluctuations in the composition and/or well width in the QW plane. Although composition fluctuation may be relieved in part by the 750 °C *in situ* annealing, complete elimination (if such is possible) may need higher temperature.¹³ The elimination of PL blueshift with the excitation power in the sputtered SiO₂-capped samples implies that the sputtering process promotes in-plane compositional diffusion in addition to interdiffusion between QWs and barriers. This is further confirmed by the temperature dependence of the PL peak energy.

Shown in Fig. 7 is the temperature dependence of the PL peak energies for three different samples. The PL peak of PECVD SiO₂-capped sample shows exactly the same behavior as the reference. The “S-shape” features are due to the relaxation dynamics of localized carriers/excitons at low temperatures.³⁰ Carrier localization is a common phenomenon for GaInNAs/GaAs QWs and shows different low-temperature features depending on the distribution of localized states.^{31–33} However, at higher temperatures (over 100 K) the PL properties should be governed by delocalized carrier states, usually described by the Varshni empirical equation. However, as the temperature decreases, the PL energies

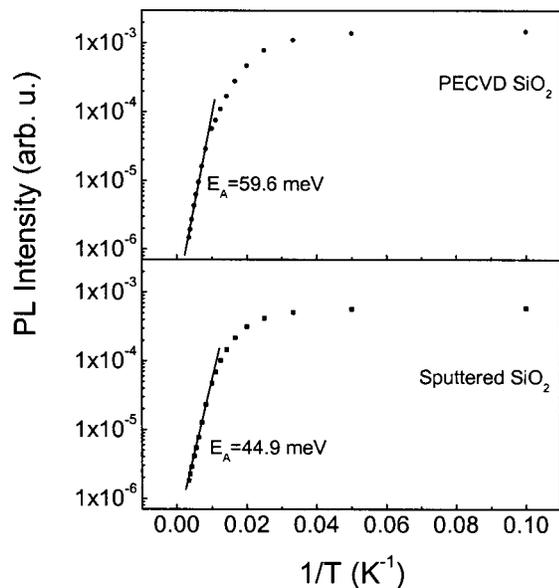


FIG. 8. Arrhenius plot of the integrated PL intensity for two different samples under excitation of a 84 mW laser diode: 1, PECVD SiO₂-capped sample annealed at 700 °C for 180 s; 2, sputtered SiO₂-capped sample annealed at 700 °C for 180 s.

gradually deviate from the Varshni equation. The difference in PL energy between experiment and theory at lowest temperature usually represents the depth of localization. The localization energies of PECVD and sputtered SiO₂-capped samples are 15 and 6 meV, respectively. Again the reduction of the localization energy can be attributed to improvement of composition uniformity due to the in-plane diffusion of In and Ga, consistent with the dependence of peak energies on the excitation power.

The temperature dependence of the integrated PL intensity is shown in Fig. 8. At high temperature, the thermal emission of the carriers out of the quantum well into the barriers plays an important role in the thermal quenching of PL.³⁴ The activation energy of the thermal emission process can be determined by the slope in an Arrhenius plot in the high temperature region, as shown in Fig. 8. The determined activation energies of the reference sample and annealed samples with PECVD and the sputtered SiO₂ cap are 59.6, 59.5, and 44.9 meV, respectively. It is not surprising that the PECVD SiO₂-capped sample has similar activation energy to the reference sample because the PECVD process and RTA do not change the QW structure. However, in the sputtered SiO₂-capped sample, due to the strong quantum well intermixing effect, the confinement energy of the carriers has been significantly reduced, which leads to a decrease in activation energy.³⁴ This result is consistent with that shown in Figs. 4 and 5. It should be pointed out that, apart from the competition between the QW capture and thermal emission into the barriers, the carriers may also undergo nonradiative loss via various defects, which would reduce the activation energy. Since the samples investigated only differ in the deposition type of the dielectric layers, the reduction of activation energy in the sputtered SiO₂-capped sample may result from point defects that diffused into the QWs from the interface between the sample surface and the dielectric layers

generated by ion bombardment. Whatever the reasons, this reflects the importance of the role played by point defects in sputtered SiO₂-induced QWI.

IV. SUMMARY

In conclusion, we reported the investigation of controlled QWI in 1.3 μm GaInNAs/GaAs multiquantum wells. By applying sputtered SiO₂ capping and suitable rapid thermal annealing, blueshifting of PL up to 170 meV was achieved, while negligible blueshifting was observed in PECVD SiO₂-capped or noncapped samples. This selective modification of the band gap of GaInNAs/GaAs by QWI is imperative for a wide range of photonic integrated circuit and advanced device applications. Secondary ion mass spectrometry gives direct evidence that the band gap tuning mechanism results from the interdiffusion of In and Ga between the quantum wells and the barriers. Detailed comparative studies using temperature dependent PL and PLE spectra were performed for different samples. All the results indicate that point defects generated in the sputtering process not only promote interdiffusion between the QWs and barriers but also in-plane compositional diffusion.

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