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with varying growth interruption time

Surface morphology and optical properties of InGaN quantum dots

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Abstract

The effect of different growth interruption time on the surface morphology and optical properties of InGaN quantum dots (QDs) grown on 2-inch silicon substrates is investigated. The surface becomes rougher and the photoluminescence intensity has been enhanced significantly when employing the growth interruption method. Temperature-dependent photoluminescence and excitation power-dependent photoluminescence both present unchanged peak energy and line-width of QDs. The sharp increase of PL intensity in medium temperature regime is attributed to the fingerprint of the existence of InGaN QDs. The shape of the QDs are further confirmed by the transmission electron microscopy with a size of 3 nm by 4 nm. Among the samples, a growth interruption time of 30 s gives the best optical performance.

Introduction

InGaN quantum dots (QDs) have attracted much research attention these years [1]. Due to defect-free and reduced quantum-confined Stark effect (QCSE), the nanometer-sized InGaN QDs are promising to be used in light emitting diodes (LEDs)[2, 3] and laser diodes (LDs) [4, 5]. A variety of methods have been developed to grow InGaN QDs. These methods include the surface treatment of the template before QDs growth [6–8], the growth parameters adjustment [9-13], the patterned template [14-16] and growth interruption method [17-19]. By using anti-surfactant or roughing the template surface one will get the InGaN QDs with relatively weak photoluminescence (PL) intensity (the PL intensity of the GaN bandgap peak is even comparable with the intensity of InGaN emission peak). With adjusting the growth parameters to realize 3-dimensional (3D) growth the InGaN QDs will suffer from the competing between 3D-growth and defects-emerging when the indium composition reaches a relatively high level [20]. To pattern the template is a rather complex technique and is hard to be used in the manufacture industrial production. Oliver et al reported the realization of micro-disk InGaN laser on sapphire substrates via growth interruption method [21]. Other research groups also employ the similar growth interruption methods to improve the optical properties of light emitting devices [22–25]. In this work, we investigated the effect of different growth interruption time on the surface morphology and optical properties of InGaN QDs grown on 2-inch silicon substrates. The surface morphology becomes rougher with increasing the growth interruption time which results in a more QD-like morphology. The PL intensity of the InGaN has also been enhanced significantly when undergone the growth interruption treatment. Temperaturedependent photoluminescence (TDPL) and excitation power-dependent photoluminescence show the unchanged peak energy and full-width at half-maximum (FWHM) of InGaN QDs both with increasing temperature and excitation power. The steep increase of the integral PL intensity in the medium temperature regime (\sim 160 K -200 K) exists in both QDs samples, which is attributed to the fingerprint of the existence of InGaN QDs. Fitted by the power law function, the change of the integral PL intensity of the InGaN QDs with



excitation power indicates fewer non-radiative recombination processes at 17 K. The size of the InGaN QDs were further confirmed by transmission electron microscopy (TEM).

Experiment

InGaN QDs with different growth interruption time were grown on the 2-inch silicon substrates by an AIXTRON 6 \times 2-inch close-coupled showerhead metal organic chemical vapor deposition (MOCVD) system. Trimethylgallium (TMGa), Trimethylgallium (TEGa), trimethylindium (TMIn), and ammonia (NH₃) were used as Ga, In, and N precursors, respectively. At first the reactor was heated to 1050 °C for 10 min in H₂ ambient followed with sillane treatment of the Si substrate for another 10 min. Subsequently, a 30-nm-thick low temperature aluminum nitride (LT-AlN) nucleation layer was deposited at 970 °C followed by a 120-nm high temperature AlN (HT-AlN) layer at 1100 °C. Then a 500-nm-thick GaN was grown on the AlN template at 1050 °C upon which a 3 nm InGaN layer was deposited at 680 °C. The ratio of mole flow rate of TEGa and TMIn was 2:1 with a V/III ratio of approximately 12300. Four samples with varying growth interruption time (0 s, 15 s, 30 s and 45 s) were fabricated and were labeled as A, B, C, and D, respectively. During the growth interruption phase, the injection valves of TEGa, TMIn and NH3 were switched off leaving the InGaN surface exposed to the N₂ ambient at the same growth temperature of InGaN QDs. The schematic diagram of the sample structure is illustrated in figure 1. The surface morphology of the samples was characterized by atomic force microscopy (AFM) using tapping mode and the TDPL properties were measured by a 325 nm He-Cd continuouswavelength laser with the temperature ranging from ~17 K to 300 K. Room-temperature PL spectra were also obtained by the He-Cd laser under the same condition at 300 K. Excitation power-dependent photoluminescence spectra were performed at 17 K by the same laser with some attenuator plates inserting in the optical path to get different excitation power. The size of the InGaN QDs was characterized by TEM.

Results and discussion

The AFM images of samples A, B, C, and D are shown in figure 2. The surface of sample A is rather smooth with a root-mean-square (RMS) surface roughness of 0.268 nm as depicted in figure 2(a). The high clusters with a black hole in the center (pointed by the black arrows in figure 2(a)) are attributed to the high-indium-content InGaN clusters surrounding the threading dislocations. By employing a 15 s post-growth interruption time, we found the surface morphology changed obviously and the RMS roughness was increased to 0.664 nm (figure 2(b)). The consecutive InGaN surface in sample A was changed to GaN pits [black regions in figure 2(b)] [5] and fragmented InGaN pieces with InGaN QDs on tips [bright regions in figure 2(b)] [18]. The change of the surface morphology is mainly due to the thermal evaporation of the high-In-segregation regions. Such surface morphology is akin to that reported by Oliver *et al* Comparing figures 2(a) and (b), by introducing a 15 s post-growth interruption, it is found that the threading dislocations (black holes surrounded by high-indium-content clusters indicated by the black arrows) could not be seen from the AFM image, indicating the evaporation of







high-indium regions. Such high-indium-content-cluster regions are considered to act as the non-radiative recombination centers [26]. By further increasing the growth interruption time, we found the surface became even rougher. The RMS roughness of samples C and D increased to 0.707 nm and 0.787 nm, respectively.

Room temperature photoluminescence spectra of the four samples were measured under the same optical path condition and presented in figure 3. It is worth noting that the employment of a 15 s growth interruption will significantly enhance the PL intensity. By further increasing the growth interruption time to 30 s, we found the PL intensity increased again. However, the PL intensity started to decrease when the growth interruption time was increased to 45 s. All of the four samples contain a shoulder-peak in the long-wavelength side. The

relative intensity of the shoulder-peak to the main peak will be suppressed with the growth interruption treatment. The increase of PL intensity with prolonged growth interruption time coincides with the AFM morphology evolution shown in figure 2. During the InGaN growth period, the indium clusters are prone to accumulate around the dislocations which are depicted in figure 2(a). Such a behavior will deteriorate the optical property of the materials [26]. The optical property will become even worse when high-indium-composition materials are grown. The high-indium clusters will be etched away by the thermal annealing with the post-growth interruption technique. As the post-growth interruption time is increased, more indium complexes will be evaporated and a higher proportion of self-assembled quantum-dot-like indium regions located at the matrix with relatively higher crystalline quality will be reserved. Thus, the non-radiative recombination efficiency will be reduced to a large scale. It could be a little over-anneal with a 45 s post growth interruption treatment, which may rise the risk of deteriorating the InGaN quality.

In order to further investigate the optical property of the samples undergone post-growth interruption treatment, we measured the TDPL for these four samples, the results of which are illustrated in figure 4. The TDPL spectra with normalized PL intensity of A, B, C, and D are present in figures 4(a)-(c) and (d), respectively. The peak wavelength of A vibrates with temperature while that of B, C, and D remain almost unchanged when the temperature ramps from ~17 K to 300 K. The spectra curves are fitted by the Gaussian function to attain the spectra parameters. The evolution trends with temperature of the peak energy, the normalized integral PL intensity and the FWHM are shown in figures 4(e), (f) and (g), respectively. The peak energy of A exhibits an S-shaped (red-shift then blue-shift then red-shift again) curve with the increasing temperature. It is an evidence of the existence of localized states in the InGaN quantum wells or QDs materials [27, 28]. The monotonic decay of the integral PL intensity with temperature as well as the expansion of FWHM in sample A is also a common phenomenon as the behavior of most InGaN materials. As for the three samples of B, C, and D, the peak energy remains almost unchanged and the FWHM even shrinks when the temperature being raised to a certain level. The integral PL intensity at first remains almost stable at relative low temperature (\sim 17 K - 130 K), then it increases drastically to a level nearly twice the intensity of that in ~17 K when the temperature reaches above ~150 K. The plunge of the PL intensity from ~200 K to 300 K is due to the activation of non-radiative recombination centers and the enhanced exciton-phonon coupling at relatively higher temperatures. The sudden increase of the PL intensity is so abnormal to the usual behavior of InGaN. But such similar novel behavior was also found by other researchers [29]. According to the research of Wu et al [30], there exists an energy level noted as E_a, below which all the energy states are occupied by carriers at rather low temperature. When the temperature increases, the carriers will escape to some higher energy states. If the density of states above E_a increases, the radiative recombination efficiency will be increased. However, the materials investigated by Wu et al were InGaN/GaN multiple quantum wells (MQWs), whose peak energy changing with temperature undoubtedly represented an S-shape curve. While in InGaN QDs, the curve of peak energy versus temperature will be some different. Considering the Dirac function of the density-of-states in the zero dimensional materials (e.g. quantum dots) [31], if the E_a locates near below the peak energy of the density-of-states, the thermalactivated carriers will come to the energy state of which the density-of-states increases to nearly infinity, resulting in a narrowing FWHM and increasing radiative intensity. As the energy gap between the two states is so close, thus the shift of the peak energy is negligible. The narrowing FWHM and abnormal increasing of PL intensity of samples B, C, and D are coincident well with the QDs behavior explained above, which provides a firm proof of the existence of InGaN QDs in our samples treated with post-growth interruption. It also should be noted that Oliver et al considered the InGaN QDs were formed mainly during the growth of the GaN capping layer [17]. Whereas according to our results, we would like to confirm that the InGaN QDs have already been formed after the post-growth annealing. As revealed by moon *et al* there remain two types of indium-rich clusters in the InGaN. One is the self-assembled quantum-dot-like indium-rich region located in the center of InGaN materials which contributes to the radiative recombination. While the other is the indium agglomerate aggregating around the defects near the surface which acts as non-radiative recombination center thus exacerbating the optical properties [32]. In sample A, as illustrated in figure 2(a), the indium clusters accumulating around the black holes which refer to the dislocation are attributed to act as non-radiative recombination centers. While treated by using post-growth anneal technique, such indium clusters are disappeared, remaining most self-assembled quantum-dot-like indium-rich regions to enhance the photoluminescence. Combining the TDPL results, we exclaim such self-assembled quantum-dot-like indiumrich regions are the exact InGaN QDs. The almost unchanged peak energy and FWHM with ramping temperature further confirms the existence of InGaN QDs in samples B, C, and D [13, 33]. Here, the almost unchanged peak energy with ramping temperature doesn't mean that the peak energy keeps to a constant value with temperature. Examining carefully from figure 4(e), we could find that the peak energy at 300 K is certainly lower than that at 20 K of both samples A, B, C, and D, which coincides well the bandgap shrinkage with increasing temperature which acts as a principle law according to the semiconductor physics. However, the variation of peak energy with temperature changing from 20 K to 300 K in B, C, and D is pretty small compared





to that in sample A. And according the model proposed by Wu *et al* the increase of PL integral intensity should be accompanied by the blue-shift of peak energy and narrowing of FWHM. Such phenomena could also be found in B, C, and D from figures 4(e) and (g), though the values of blue-shift of peak energy in B, C, and D are rather small.

Although the behaviors of samples B, C, and D are similar, there remain some elaborate differences. The largest variations of peak energy with temperature of A, B, C, and D are 25.0 meV (8.20 nm), 4.5 meV (0.90 nm), 4.4 meV (0.89 nm) and 7.8 meV (1.58 nm), respectively. As the narrow vibration of peak energy with temperature indicates the good uniformity of the quantum dots [13], we conclude B and C have the superior uniformity to the other samples. And we attribute the increasing at a certain high temperature to be the fingerprint of InGaN QDs. Even A has a little increase point of PL intensity at 170 K, which is regarded as the evidence of the existence of InGaN QDs in A. The weak optical property of A is mainly due to the existence of large density of non-radiative recombination centers. Such centers will be significantly diminished by the postgrowth interruption treatment. The temperature (noted as T_c) possessing the highest integral PL intensity is 180 K, 160 K and 200 K in samples B, C, and D, respectively. The higher T_c may refer to the higher energy that enables the carriers to hop into the state with highest density-of-states. As to B and D, the integral PL intensity presents a slow increase below 50 K then slowly drops down before the sudden increase near T_c. The decrease phase in samples B and D is due to the capture of thermal-activated carriers by the non-radiative centers surrounding the QDs [29]. While in sample C, the PL intensity increases monotonically straight up to the highest point along the temperature from 17 K to T_c, which reveals a significant reduction of non-radiative centers besieging the QDs regions. The sudden decrease of the FWHM at T_c also coincides with the significantly increased density-of-states at T_c (a large amount of carriers populate together at the same energy state will significantly reduce the radiative FWHM). Sample C reserves the narrowest variation of FWHM (as small as 3.8 meV, namely less than 1 nm) with temperature among the four samples. We also calculate the ratio of the integral PL intensity at 300 K (I_{300k}) divided by the strongest integral PL intensity at T_c (I_{Tc}). Such a ratio will indicate the quantum efficiency of the InGaN QDs [29]. The ratio of I_{300k}/I_{Tc} is 6.13%, 15.92%, 12.11% for samples B, C, and D, respectively. According to the TDPL results, sample C gains the best optical performance.

One may have some doubt about the excitation laser source as a photon with a wavelength of 325 nm will also excite the carriers in the GaN matrix. And if there remains a p-n junction or some piezoelectric field in the GaN region, they will cause the carrier-transition phenomenon from GaN to InGaN [27]. However, here we only added an InGaN QDs layer on a GaN matrix. In principle, none of the external fields exists in our samples. For further confirm, we also measured the TDPL of our samples with a pulse laser of 400 nm and found the similar abnormally-enhanced PL intensity at some medium temperature in the InGaN QDs samples (the results are not presented in this paper.).

To further confirm the existence of InGaN QDs as well as to investigate the characteristics of the QDs, excitation power-dependent PL were performed at 17 K. The normalized PL spectra of samples A, B, C, and D are shown in figures 5(a)-(c), and (d), respectively. All the spectra present a small peak wavelength change as the excitation power increased from 0.024 mW up to 1.245 mW. With the Gaussian fitting, the spectra parameters of the peak energy, the integral PL intensity and the FWHM are extracted in figures 5(e), (f), and (g), respectively. The peak energy of A performs a blue-shift of 20.0 meV with increasing the excitation power. While the blueshift values of B, C, and D are 5.0 meV, 4.4 meV and 7.0 meV, respectively. The insensitive of peak energy to excitation power of B, C, and D reveals the characteristic of high density-of-states in InGaN QDs [13, 34]. Sample C demonstrates the lowest blue-shift of peak energy indicating its superior carrier restriction ability among the other samples. Generally, the integral PL intensity is proportional to the excitation power with a power index, which could be expressed as $I \propto P^{m}[35, 36]$, where I is the integral PL intensity and P is the excitation power with a power index m reflecting the various recombination processes. If m is equal to 1, the radiative recombination becomes dominant. While if m is larger than 1, it means there remain some nonradiative recombination processes such as Shockley-Read-Hall recombination. The summarized relationship between the excitation power and integral PL intensity is depicted in figure 5(f). The fitted values of m of A, B, C, and D are 1.27, 1.14, 1.01 and 1.00, respectively. Such results coincide well with our above analysis that C and D possess the superior radiative recombination efficiency to the other samples. According to figure 5(g), the variations of FWHM of A, B, C, and D are 23.0 meV, 5.6 meV, 0.5 meV and 0.9 meV, respectively. The extremely small changes of FWHM of C and D further confirm the existence of high density-of-states in the InGaN QDs.

To further investigate the size of the InGaN QDs, we fabricated another sample noted as sample E. The structure and growth conditions were the same as sample D except an approximately 60-nm-thick GaN layer on top. Sample E was grinded and then thinned by ion beam milling. The TEM was performed to characterize the size and morphology of InGaN QDs and the TEM images are shown in figure 6. One could see the InGaN QDs distribute in the active region clearly from figure 6(a). The magnified images of the QDs are shown in







Figure 6. The TEM images of InGaN QDs. (a) The distribution of the InGaN QDs. (b) The magnified image of the InGaN QDs. (c) The zoom-in picture of one InGaN QD.

figures 6(b) and (c). The size of the dots is about 3 nm in height and 4 nm in width. Such a height is coincident with the value we designed in figure 1. The relatively small size reveals the good shape of the InGaN QDs.

Conclusion

At this paper, we investigate the surface morphology and the optical properties of the InGaN QDs by employing post-growth interruption method with varying interruption time. With the growth interruption treatment, the sample surface becomes rougher and the indium clusters surrounding dislocations are disappeared from the AFM images. The room temperature witnesses a significant improvement of PL intensity of the samples undergone growth interruption treatment. TDPL results present a sudden increase of integral PL intensity at a certain relatively higher temperature and extremely small variation of peak wavelength and FWHM with temperature. Such characteristics reveal the existence of InGaN QDs with excellent optical properties. Excitation power-dependent PL results show unchanged peak energy and FWHM with changing excitation power of the samples comprised of InGaN QDs. The fitting results of the integral PL intensity with changing excitation power confirms that the radiative recombination dominates in the InGaN QDs. The TEM images reveal the InGaN QD has a spherical size of 3 nm in height and 4 nm in width. Comparing the optical performance of different interruption time, we conclude adopting a 30 s growth interruption time will gain the best optical performance of the InGaN QDs.

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