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Semicond. Sci. Technol. **20** (2005) 615–620 doi:10.1088/0268-1242/20/6/023

Luminescent properties of annealed and directly wafer-bonded InAsP/InGaAsP multiple quantum wells

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Received 5 February 2005, in final form 23 March 2005
Published 27 April 2005
Online at stacks.iop.org/SST/20/615

Abstract

InAsP/InGaAsP strain-compensated multiple quantum wells (SC-MQWs) were grown using gas source molecular beam epitaxy. The luminescent properties of the SC-MQWs after thermal annealing and direct wafer-bonding onto a GaAs substrate were studied. It is shown that photoluminescence (PL) intensities of the samples are improved by a factor of 4.1 upon annealing at 620 °C. The luminescence intensities of the samples bonded at 580 and 650 °C under ∼5 MPa pressure and 35 min annealing process are comparable with that of the as-grown ones. A proposed mechanism of defects annihilation relating to the evolution of nanoscale As-rich and P-rich clusters is then used to account for the dependence of PL intensities and the full width at half-maximum of InAsP/InGaAsP SC-MQWs on annealing temperatures.

1. Introduction

Serving as the optical gain amplification media, multiple-quantum-well (MQW) active regions are the key unit of vertical cavity surface emitting lasers (VCSELs). Due to the large Auger recombination and intervalence absorption [1] of quantum wells (QWs) in long wavelength (LW) and the low reflectivity of InP-based distributed Bragg reflectors (DBRs) [2], LW-VCSELs are trapped in the predicament of poor temperature characteristics, which has suspended their applications in middle and long-haul fibre-optic communication. To solve these problems, different methods have been proposed, such as monolithic InP-based VCSELs with AlGaAsSb/AlAsSb DBRs [3], GaAs-based VCSELs with diluted nitride like GaInNAs(Sb) MQWs [4, 5] and InGaAs quantum dot active regions [6], and direct wafer-bonded VCSELs [7]. Nitride MQWs can effectively confine electrons in quantum wells and thus realize high-temperature working VCSELs, but the degradation of crystal quality due to the incorporation of nitrogen challenges epitaxial material growth. InP-based and wafer-bonded VCSELs require QW active regions to be processed at high temperature. InGaAsP/InP MQWs have been widely used in edge emitting laser diodes, but their high-temperature performances are limited due to poor confinement of electrons as a result of low conduction-band-offset (CBO) ($0.35\Delta E_g$) [8, 9]. AlGaInAs-based MQWs with high CBO ($0.72\Delta E_g$) [10] are applied to VCSELs showing excellent device performances [3], in which wells and barriers can be strain-compensated, thus reducing Auger recombination and intervalence absorption [11]. However, the challenge to this material system is the stability and lifetime of Al-containing devices that should work in a severe environment, such as operating up to 85 °C in fibre-optic communications. An Al-free and possessing high-CBO material system is promising in promotion of VCSELs into the commercialization field.

InAsP/InGaAsP strain-compensated multiple-quantum-well (SC-MQW) structures having high CBO (0.5–$0.7\Delta E_g$) [12–14] are a prospective candidate for LW-VCSELs. They have shown high device performances in edge emitting lasers (EELs) [14–16]. As the composition of the group V element can be fixed for wells and barriers in the InAsP/InGaAsP material system, good control and qualities of QW interfaces can be achieved. In GaAs-based 850 nm VCSELs, great improvement of the device performance has been attained by the introduction of the Al-oxide current-confining aperture

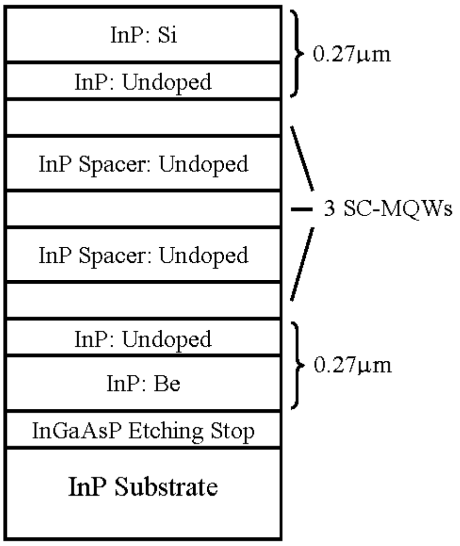


Figure 1. The schematic diagram of InAsP/InGaAsP SC-MQW structures for thermal annealing and wafer bonding.

technique [17]. But it encounters difficulties when applied to InP-based LW-VCSELs. The lattice-matched Al-containing materials grown on InP like AlInAs need high temperature and a long time to be oxidized [18], which may cause the degradation of QW active regions. Thus, it is necessary to have knowledge of the luminescent properties for InAsP/InGaAsP SC-MQWs upon the high-temperature annealing process. Besides, these studies are also very useful for direct wafer bonding techniques that require a high-temperature annealing process as well [7]. A complete report on this material system has been absent till now.

In this paper, we present the luminescent properties of thermally annealed as well as wafer-bonded InAsP/InGaAsP SC-MQWs grown by gas source molecular beam epitaxy (GSMBE). The measurement of room-temperature photoluminescence (RT-PL) and high-resolution x-ray diffraction (HRXRD) rocking curves is carried out on samples with different annealing and bonding temperatures. The stronger PL performances are observed with annealing temperature up to 620 °C. A feasible annealing mechanism accounting for the interesting results is then discussed.

2. Experiments

InAsP/InGaAsP SC-MQWs were grown by the GSMBE method using a VG V90 system. Group III elements' (indium and gallium) molecular beams were evaporated from the effusion cells. Group V elements' (arsenic and phosphorus) beams were supplied by arsine (AsH_3) and phosphine (PH_3) cracked at high temperature (1040 °C). The flux of V elements was fixed during the growth of wells and barriers. The net strain of SC-MQWs was in the magnitude of 10^{-4} with 1.27% of compressive strain of InAsP wells and 1.19% tensile strain of InGaAsP barriers. The InAsP wells were tuned to 1.3 μm emissions and the band gap wavelength of InGaAsP barriers was 0.995 μm. The structure of InAsP/InGaAsP SC-MQWs is schematically shown in figure 1 which is designed n-type–intrinsic–p-type (NIP) (Si for n-type and Be for p-type) lineup and 2.5λ (λ = 1.3 μm) optical cavity. This structure can be used as active regions of InP-based and wafer-bonded VCSELs. Two undoped InP layers grown between doped InP and MQWs were served to block dopants diffusing into wells. The thickness of the InP cap layer (the distance from MQWs to the surface) was 0.27 μm.

Thermal annealing processes of InAsP/InGaAsP SC-MQWs were carried out at different temperatures between 580 and 680 °C, during which their surfaces were bared to the flowing N_2. For the wafer bonding process [19], 1 cm × 1 cm cleaved squares of InP-based SC-MQW structures and GaAs substrates along the [1 1 0] direction were used. After chemical cleaning and surface oxide removal, two samples were joined face-to-face and pressed by a stainless steel fixture with a pressure of ∼5 MPa. Then they were annealed in flowing N_2 ambient at 580 and 680 °C, respectively. The duration for thermal annealing and the wafer bonding process is fixed to 35 min. After bonding, the InP substrate and InGaAsP etch stop layer of the bonded samples are removed.

Photoluminescence (PL) of SC-MQWs excited by the 514.5 nm line of an Ar^+ laser with 15 mW pumping power was measured by NICOLET 860 Fourier transform infrared (FTIR) spectroscopy. And the measurement of HRXRD rocking curves was performed by Philips X'Pert materials research diffractometer (MRD).

3. Results and discussions

3.1. Optical properties of thermally annealed InAsP/InGaAsP SC-MQWs

The room-temperature photoluminescence (RT-PL) spectra of the InAsP/InGaAsP SC-MQW structures with bare surfaces annealed at 580 °C, 620 °C, 650 °C and 680 °C are shown in figure 2(*a*). Only one PL peak is observed which corresponds to the quantum transition from the first electronic sub-band to the first heavy hole sub-band (E_1–HH_1) [20]. Figure 2(*b*) depicts the integrated PL intensity and PL peak wavelength as functions of annealing temperature. After 580 °C thermal annealing, an intensified integrated PL intensity by a factor of 3.4 is measured and the PL peak wavelength blueshifts by 13.9 nm compared with those of as-grown samples. The annealing process with increased temperature continuously enhances the PL intensity and PL peak position blueshift till 620 °C, in which an intensified factor of 4.1 and a blueshift of 22.4 nm are observed. After that, their variations are the opposite. The PL intensities of 680 °C annealed samples are lower than that of the as-grown ones.

To investigate structural stabilities of samples upon thermal annealing, their HRXRD rocking curves are measured and shown in figure 3. Satellite peaks are the result of x-ray diffraction from periodic QWs. The modulation superposed on the satellite peaks is attributed to the x-ray diffraction from the periodic structure consisting of InP spacer and quantum wells. The well-marked XRD peaks of the as-grown samples indicate their sharp interfaces and good control of each layer thickness. As can be seen from figure 3, no obvious quality degradation of MQW structures occurs on the annealed samples with temperatures varied from 580 to 650 °C.

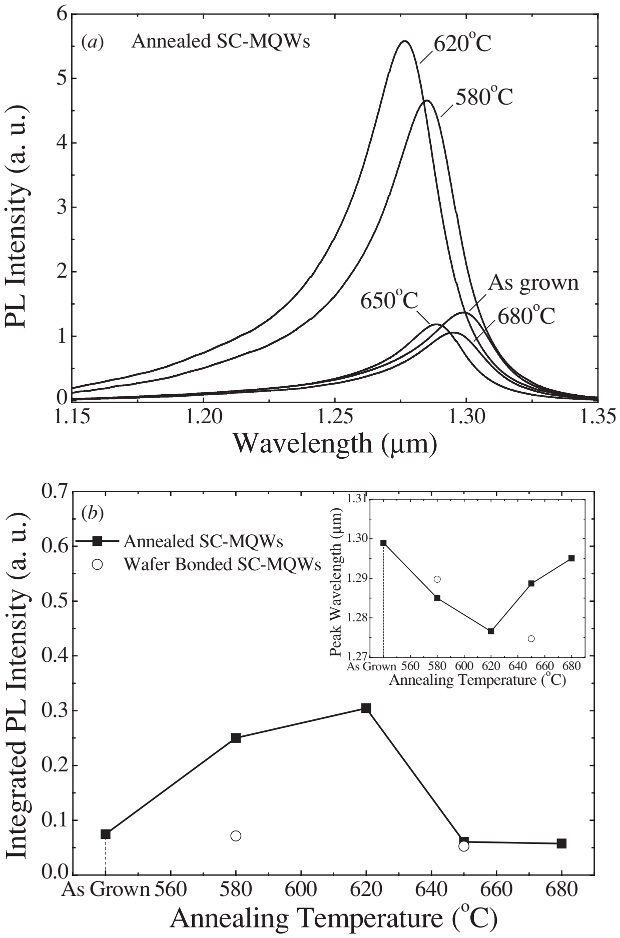


Figure 2. (*a*) RT-PL spectra of the as-grown and thermally annealed samples and (*b*) integrated RT-PL intensity of the as-grown, thermally annealed and wafer-bonded samples. The inset is the PL emission wavelength as a function of annealing temperature.

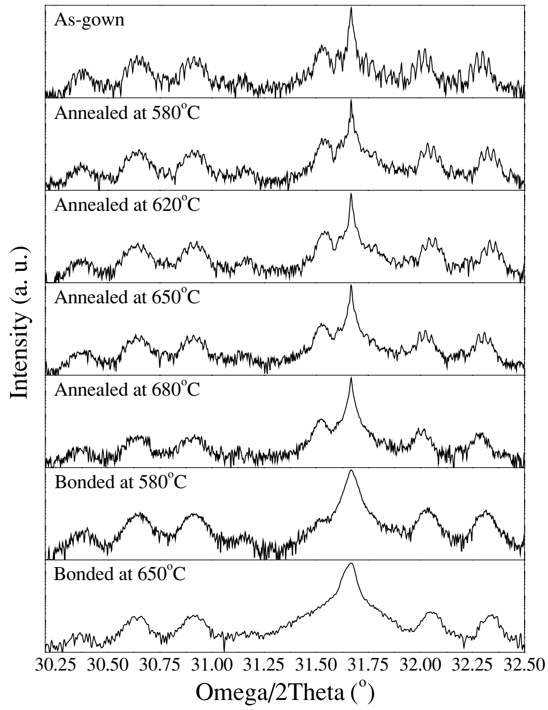


Figure 3. HRXRD rocking curves of as-grown, thermally annealed and wafer-bonded samples.

Upon 680 °C thermal annealing processes, the XRD rocking curve reflects the deterioration of the MQW structures.

With the strongest PL intensity by a factor of 4.1 at 620 °C and structural stabilities up to 650 °C of InAsP/InGaAsP SC-MQWs, they are promising for application in wafer-bonded and Al-oxide current-confining aperture-based LW-VCSELs. As a comparison, Syrbu *et al* [21] studied the thermal stabilities of InAsP/InGaAsP SC-MQWs grown by chemical beam epitaxy (CBE). In their experiments, samples annealed at different temperatures between 500 °C and 700 °C exhibit a significant degradation of PL intensity (by a factor of up to 50) and large blueshift of emission wavelength (up to 150 nm). In contrast to the maximum blueshift of 22.4 nm in our annealed GSMBE grown structures, the thermal annealing process remarkably modifies the QW structure of CBE grown samples and thus PL performances. So CBE grown InAsP/InGaAsP SC-MQWs have a limit to their application in the VCSEL technologies concerning a high-temperature annealing process. The structural difference stemming from the epitaxy difference between GSMBE and CBE may be responsible for the PL properties of annealed SC-MQWs. In section 3.3, we will propose a thermal evolution process of nanoscale clusters in our samples to explain their PL performances and structural stabilities.

Generally, the PL behaviour of MQWs upon thermal annealing can be explained by two mechanisms: nonradiative centres removal [22] and quantum-well intermixing (QWI) [23, 24], which have been extensively studied on a variety of material systems [21–26]. Nonradiative centres are related to point defects, strain-induced dislocations, etc. The annealing process annihilates (regenerates) defects thus improving (degrading) material qualities. Approximately, the variation of PL intensity is inversely proportional to the full width at half-maximum (FWHM) of PL spectra [26]. In the annealing of InAsP/InGaAsP SC-MQWs, however, a complex dependence of FWHM on annealing temperatures suggests a different defect curing mechanism which will be discussed in detail later. QWI results from the interdiffusion of group III [23] or V [24] atoms upon a thermal process, which is triggered by high temperature and the surface stoichiometry of MQWs. Due to the same group V compositions of wells and barriers, high temperature causes interdiffusion of group III atoms (indium from wells to barriers and gallium from barriers to wells) and makes the blueshift of emission wavelength. A phosphorus-poor surface generates group V vacancies as a result of material decomposition. These vacancies pass through the InP cap layer and diffuse into QW regions promoting the structural evolution of wells. To a large extent, the PL behaviour of the annealed sample with bare surface is related to the surface stoichiometry [27]. Upon the modification of surface stoichiometry, different PL behaviour can be achieved. In the case of wafer bonding, a pressure is imposed on samples, which makes atomic-scale contact of surfaces. Thus, the decomposition of surface materials can be suppressed. Much smaller QWI effects and better preservation of QW structures are expected to be realized by a pressure applied on the surface of annealing samples.

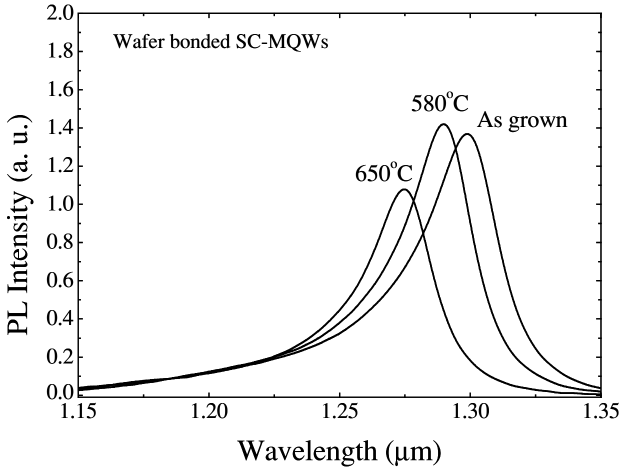


Figure 4. RT-PL spectra of the as-grown and wafer-bonded samples.

3.2. Optical properties of directly wafer-bonded InAsP/InGaAsP SC-MQWs

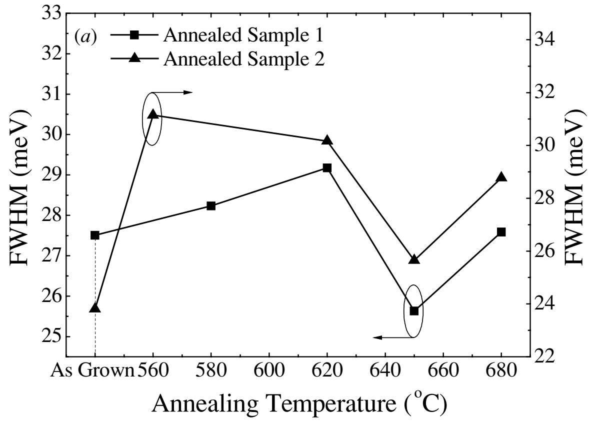
Wafer bonding techniques have achieved outstanding device performances [2] in the development of LW-VCSELs. In this study, we use the GSMBE grown InAsP/InGaAsP SC-MQWs as the active regions of wafer-bonded structures. Figure 4 shows their RT-PL spectra. The PL peak intensity of bonded SC-MQWs is first intensified at 580 °C and then decreased at 650 °C. The comparisons of PL performances of bonded samples with annealed ones are depicted in figure 2(*b*). We can see that, though the integrated PL intensity of 580 °C annealed SC-MQWs is improved by a factor of 3.4, the samples bonded at the same temperature display comparable PL intensity with the as-grown ones. This result tells us that wafer bonding processes have a great degradation effect on the luminescent efficiency of MQWs. The diffusion of interfacial defects between InP and GaAs surfaces such as voids, inclusions and point defects [28] partly contributes to this degradation. But a main factor affecting PL degradation may be related to the bonding pressure, which has not been very clearly understood till now. The HRXRD rocking curves of the samples bonded at 580 and 650 °C are shown in figure 3. We can see that the modulation superposed on the satellite peaks in the MQW structures annealed up to 650 °C is completely evened out in both the bonded samples. The deterioration of material qualities presumably results from the application of bonding pressure, which is to be further investigated. In addition, the PL emission wavelength of 580 °C bonded samples does not show blueshift compared with that of the annealed ones at the same annealing temperature (figure 2(*b*)). So bonding pressure may largely induce defects in QW regions but not remarkable QWI. Defects produced in bonded structures upon different pressure as well as the dependence of PL intensity on pressure and the InP cap layer have been investigated by Ohiso *et al* [29, 30]. According to their results, PL intensity degrades to ∼70% when bonding pressure and the thickness of InP cap layer are 5.6 kPa and 0.27 μm, respectively. But it degrades to ∼18% with the bonding pressure increased to 9.8 kPa (annealing temperature and time are 600 °C and 1 h, respectively). The increase of bonding pressure greatly degrades material qualities. Considering a 0.27 μm thick InP cap layer and ∼5 MPa bonding pressure in our experiment which is a relatively high value [28–31], the comparable PL intensities of the bonded InAsP/InGaAsP SC-MQWs at 580 °C and 650 °C with that of the as-grown ones indicate superior PL performances of this material system in contrast to other groups' work [30, 31]. Furthermore, upon Ohiso's studies, the PL performances of our samples can be still improved by moderately decreasing the pressure. InAsP/InGaAsP SC-MQWs should be a prospective active layer for achieving high performances of LW-VCSEL structures. Detailed device studies on wafer-bonded VCSELs are under investigation.

In addition, we can find another result from figure 2(*b*) that the variation of PL intensity and peak wavelength of InAsP/InGaAsP SC-MQWs with wafer bonding temperature is much slower than that of thermally annealed samples. This result tells us that SC-MQW structures are well preserved and thus thermally stable in the wafer bonding process, which results from the stable stoichiometry interface between InP and GaAs in the bonding case. In the presence of bonding pressure (∼5 MPa in this work), the gap between InP and GaAs surface contact reached an atomic scale and the surface decomposition of group V atoms, such as arsenic and phosphorus, is very small. Thus, QWI formed by the diffusion of interfacial atoms and vacancies is much less than that of the annealing case with bare surface. So it is observed that the emission wavelength of 580 °C bonded samples is longer than that of annealed samples at the same temperature (figure 2(*b*)). However, the PL spectra of 650 °C bonded samples show a blueshift of emission peak compared with that of annealed ones. The reason is that massive vacancies produced in the bare InP surface of annealed samples diffuse into the QW regions, which leads to serious atomic intermixing between wells and barriers. But stable stoichiometry interfaces of the bonded samples reduce this vacancy-diffusion effect and make the QW structure unchanged. Thus, the interdiffusion of group III elements (In, Ga) in the MQWs is the main reason of the observed blueshift of the PL peak. In fact, QW regions sandwiched between top and bottom DBRs in monolithic VCSELs are expected to achieve higher annealing temperature with stable structure than 650 °C, since the diffusion sources of interfacial atoms and vacancies should no longer appear in such structures.

In brief, PL intensity enhancing effects by the thermal annealing process have made the luminescent properties of InAsP/InGaAsP SC-MQWs upon wafer bonding be comparable with those of the as-grown ones. Despite some degrading of PL performances by imposed pressure in the bonding processes, much better stabilities of bonded SC-MQWs with constant stoichiometry interfaces are achieved. Superior luminescent properties and structural stabilities should be achieved in VCSELs with these SC-MQWs. We will then discuss the internal annealing mechanism in support of these characteristics of this material system.

3.3. Evolution of nanoscale As-rich and P-rich clusters in the annealing process

The integrated PL intensity and FWHM are two principal parameters characterizing optical qualities of luminescent



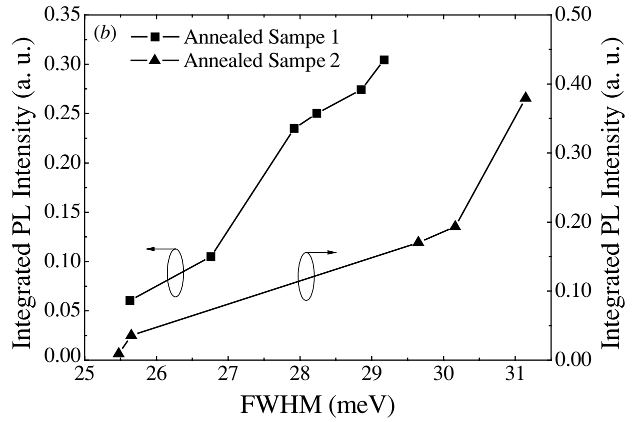


Figure 5. (*a*) The RT-PL FWHM as a function of annealing temperature and (*b*) the integrated RT-PL intensity as a function of FWHM.

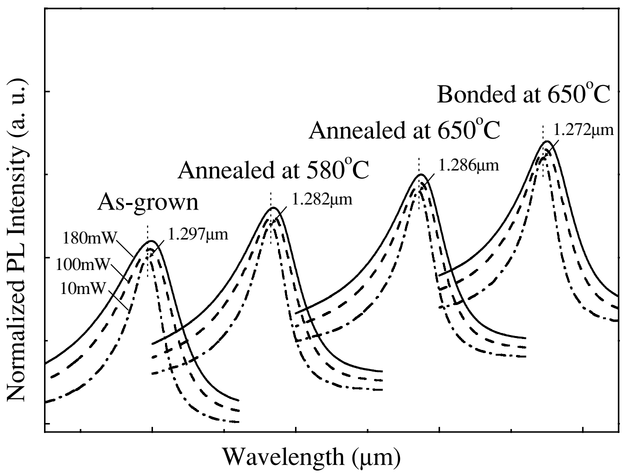


Figure 6. Excitation laser power dependence of RT-PL spectra of as-grown, thermally annealed and wafer-bonded InAsP/InGaAsP SC-MQW structures.

materials. FWHM is related to structural defects of QWs such as interface roughness, random alloy disorder and well width fluctuations, which leads to the smearing of band edges and the formation of band tails [32]. Generally, the narrowed PL linewidth (FWHM) of samples after annealing indicates the improved material qualities and the increased PL intensities [26]. The dependence of FWHM on annealing temperatures and the integrated PL intensity as a function of FWHM are plotted in figures 5(*a*) and (*b*), respectively. Sample 1 is the annealed SC-MQWs sample and its PL results are shown in figure 2(*a*). Sample 2 is for the inspection of the measurement repetition. We can see that a high PL intensity corresponds to large FWHM in our annealing experiment. This phenomenon cannot be explained simply by the annihilation or regeneration of point defects. Some other annealing mechanism may be responsible for it. A similar relation is observed by Feng *et al* [33] in the annealing investigation of low-temperature grown AlGaAs/GaAs MQWs. In their studies, excess arsenic atoms aggregate to clusters upon thermal annealing, and then become large and the space between neighbouring clusters is increased, which increases the carrier lifetime and the PL intensity [34, 35]. They attributed the broadening of FWHM to the roughening of interfaces. Nanoscale compositional variations including As-rich and P-rich clusters in GSMBE grown InAsP/InP QWs were observed by Zuo *et al* [36]. So we propose that these nanoscale clusters appear in the wells of our GSMBE grown SC-MQW structures, and they evolve upon a thermal annealing process accounting for the result of figure 5. First, As-rich and P-rich clusters amalgamate atoms, little clusters and point defects during a relatively low temperature stage, such as 580 °C. This process makes these clusters large and distant, thus increasing the carrier lifetime and PL intensity. With the well-pronounced boundary of nanoscale clusters, the degree of alloy disorder in wells is enhanced and PL linewidth is increased. When these nanoscale clusters grow up to the maximum, they then start to decompose. At this point, the PL intensity, peak wavelength and linewidth start to evolve in the opposite direction. There is an active energy decided by annealing temperature to decompose these clusters. When As-rich and P-rich clusters are dissolved into wells upon the 650 °C thermal annealing process, uniform composition distribution in wells is achieved, which narrows PL linewidth. In figure 3, well-marked XRD peaks of samples up to 650 °C indicate the maintenance of smooth interfaces of MQWs. Thus, interface roughening does not fairly contribute to the PL spectra broadening in this temperature stage. With the decomposition of these clusters, the carrier lifetime is reduced and PL intensity is decreased. An existent thermal energy for dissolving nanoscale clusters accounts for the structural stabilities of InAsP/InGaAsP SC-MQWs up to the 650 °C annealing process. Finally, a higher temperature annealing process such as 680 °C leads to serious atomic interdiffusion and roughening of interfaces as observed in the XRD rocking curve of figure 3, which degrades PL performance and broadens FWHM as shown in figure 5(*a*).

Although nanoscale clusters are present in InAsP wells, they do not greatly degrade the optical qualities of SC-MQWs. As can be seen from figure 5, the FWHM of all samples are smaller than 30 meV and their variation upon thermal annealing is in the range of 5 meV, indicating good qualities of GSMBE grown SC-MQWs structures even after thermal annealing and wafer bonding up to 650 °C. Dependence of PL behaviour of the SC-MQWs on the high excitation laser powers can also verify this result. Alloy disorder causes the smearing of band edges and the formation of band tails that lead to the blueshift of PL spectra pumped by high laser power [37]. Different laser power pumped PL spectra of InAsP/InGaAsP SC-MQWs are shown in figure 6, in which the PL intensity is normalized and spectra of different samples are shifted for clarity. No blueshift of emission wavelength is observed. The contrarily small redshift (2–3 nm) results from the heating by high power of laser. So luminescence degradation effects of alloy disorder due to the presence of nanoscale clusters on GSMBE grown InAsP/InGaAsP SC-MQWs are limited and do not observably degrade their PL performances.

4. Conclusion

In summary, we have studied the optical and structural properties of GSMBE grown InAsP/InGaAsP SC-MQWs emitting at 1.3 μm upon thermal annealing and direct wafer bonding. The thermally annealed structures with annealing temperature lower than 620 °C exhibit stronger PL intensity (by a factor of up to 4.1) and blueshift of peak wavelength (up to 22.4 nm). The surface stoichiometry of SC-MQW structures has an important effect on their annealing conditions achieving optimal material qualities. Wafer-bonded InAsP/InGaAsP SC-MQWs with annealing temperature between 580 and 650 °C have slower variation of PL intensity and emission wavelength than those of annealed samples with bare surfaces, which indicates that much better stabilities of SC-MQWs upon thermal processes can be achieved with constant stoichiometry of surface or interface such as in monolithic VCSELs. The comparable PL intensities of bonded samples with as-grown ones prove the superior optical performances of this material system. The proposed evolution mechanism of nanoscale As-rich and P-rich clusters is responsible for luminescent properties of InAsP/InGaAsP SC-MQWs upon thermal processes. The FWHM of all samples is smaller than 30 meV and its variation upon thermal annealing and wafer bonding is in the range of 5 meV, indicating good luminescent efficiency of our GSMBE grown samples. Thus, InAsP/InGaAsP SC-MQWs should be promising for wafer-bonded and monolithic grown VCSEL structures as the active regions.

Acknowledgments

This work is supported by the National Key Basic Research and Development Program of China under grant no 2003CB314903 and the special funding of Shanghai Nano-Promotion Center under grant no 0352NM092.

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