Selective area intermixing of III–V quantum-dot lasers grown on silicon with two wavelength lasing emissions

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Received 17 April 2019, revised 20 May 2019
Accepted for publication 24 June 2019
Published 5 July 2019

Abstract
The ability to tailor the bandgap of III–V compound semiconductors spatially, across the wafer is highly desirable for monolithically integrating photonic components with multi-functions. Using rapid thermal annealing with SiO₂ and TiO₂ capping layers as a selective area intermixing technique, we have demonstrated selective area bandgap tuning of III–V quantum dot (QD) material on a silicon (Si) substrate. Electrically pumped InAs/GaAs QD lasers directly grown on Si with dual-wavelength lasing emissions of 1275 and 1313 nm have been fabricated by this technique. This result indicates that the selective area intermixing technique can potentially be used in optical integrated circuits for Si photonics.

Keywords: quantum-dot laser, intermixing, silicon photonics

(Some figures may appear in colour only in the online journal)

1. Introduction
Silicon (Si) photonics-enabled optical interconnections is one of the strongest candidates to replace traditional copper interconnections in information systems. It allows information transfer via photons rather than electrons, thus with a much faster transmission speed, higher throughput and lower power dissipation [1–3]. Among the various kinds of well-studied Si optical components, Si-based light emitters, which had been considered as a road-block for Si photonics [4], have now been achieved by integrating III–V lasers on Si platforms, using wafer bonding methods [4–7] and epitaxial growth methods [8–12]. Although the former approach has already shown the nearly commercialised laser results, the latter one can provide ultimate integration density and the advantages of high volume manufacture [13]. The biggest hurdle for monolithic integration is the dissimilarities between III–V compounds and Si materials, which introduce a high density of defects, leading to reduced device performance and device lifetime limitations. By applying the combined strategies of mitigation of various defects and using III–V quantum dots (QDs) as the active region, practical and reliable InAs/GaAs QD lasers directly grown on off-cut Si/‘on-axis’ (001) Si with long lifetimes have been reported recently [10, 12]. The driving forces for the utilisation of QDs are not only their high efficient light generation rate (ultra-low threshold current) and temperature-insensitive properties, but also the enhanced tolerance to defects, due to the strong carrier localisation within the QDs [14]. Thus, it is clear that the QD is an ideal active structure for III–V lasers directly
grown on Si [15]. Recently, the dynamic properties of III–V/Si QD lasers have also been studied, in terms of relative intensity noise [16, 17], modulation bandwidth [18, 19], gain switching [20] etc. These results demonstrate the high potential of Si-based III–V QD lasers to be used as on-chip light sources for Si photonics.

To realise a monolithically integrated photonic circuit, the ability to engineer the bandgap across the wafer spatially is one of the critical points to reduce optical loss in waveguides [21]. In general, multiple bandgaps can be approached by two methods: selective area epitaxial regrowth or intermixing techniques [22]. The intermixing approach by rapid thermal annealing (RTA) is a relatively simple approach, compared with regrowth and widely used in modifying the wavelength of materials, either for quantum-wells [22–24] or QDs [25–29]. Among the various intermixing methods, impurity-free vacancy disordering assisted by dielectric layers is particularly attractive for optical integrated circuit fabrication, because it does not involve the introduction of extra impurities or damage [21]. The dielectric layers, which were deposited on the top of III–V epitaxial layers, play an important role in controlling the intermixing levels by their different thermal expansion coefficients. A thin layer of SiO\textsubscript{2} with thermal expansion coefficient of 0.52 × 10\textsuperscript{–6} °C\textsuperscript{−1} has been shown to produce a significant enhancement of the intermixing when it was capped on In\textsubscript{As}/Ga\textsubscript{As} QD structure [27]. Due to the thermal expansion coefficient mismatch between Ga\textsubscript{As} (∼6.5 × 10\textsuperscript{–6} °C\textsuperscript{−1}) and SiO\textsubscript{2}, a compressive strain is produced at the interface of Ga\textsubscript{As}/SiO\textsubscript{2} induces Ga atoms to migrate into the SiO\textsubscript{2} cap. Ga vacancies created by the enhanced Ga migration are then diffused through the III–V epi-structures and cause the intermixing process (blue shift) at the active region. In comparison with SiO\textsubscript{2}, the TiO\textsubscript{2} layer has a much larger thermal expansion coefficient of ∼8.2 × 10\textsuperscript{–6} °C\textsuperscript{−1} than Ga\textsubscript{As}, a tensile strain at the interface will restrict Ga vacancies produced during RTA, thus suppressing the inter-diffusion rate [30]. Therefore, the multiple bandgaps of III–V QD materials could be achieved by lateral patterning SiO\textsubscript{2} and TiO\textsubscript{2} on top before the RTA process, to provide a selective area intermixing technique. Using this technique, we can intentionally engineer the bandgap for individual photonic devices, such as QD lasers, electro-absorption modulators and low-loss waveguides, all monolithically integrated on the same platform. Although there are significant pioneering works on the selective area intermixing studies for QD materials on native substrates [27, 30], the intermixing effect of III–V QDs monolithically grown on a Si substrate, which may become more complicated due to the high-density defects involved, have not been reported in detail previously.

In this paper, a comparative study of the intermixing effect in In\textsubscript{As}/Ga\textsubscript{As} QDs directly grown on Si capped by separated SiO\textsubscript{2} and TiO\textsubscript{2} layers, to determine the optimal thermal annealing conditions, is reported first. Detailed analysis of annealed QDs with different temperatures of the two capping layers, in terms of physical shape, sizes and size distributions, have been evaluated by using high-resolution aberration corrected scanning transmission electron microscopy (STEM). Based on the optimised annealing conditions from each individual study, the III–V/Si QD structure material with double bandgaps has been achieved by using the selective area intermixing technique. Thus, electrically pumped In\textsubscript{As}/Ga\textsubscript{As} QD lasers directly grown on Si with two different emission wavelengths of 1275 and 1313 nm have been fabricated from this thermally processed wafer, showing that this selective area intermixing technology can be used in optical integrated circuits for Si photonics without severe degeneration of the QD material quality.

2. Experiment methods

2.1. Optimisation of RTA conditions for SiO\textsubscript{2} and TiO\textsubscript{2} capped samples

In this work, as shown in figure 1(a), the III–V QD structure was grown by a molecular beam epitaxy system on the n-doped Si (001) substrate with an oriented offset of 4° towards the [110] direction. A 6 nm Al\textsubscript{Ga}As nucleation layer was deposited first to provide a good interface between Si and III–V materials [31]. After a 1000 nm Ga\textsubscript{As} buffer layer and three layers of five periods of In\textsubscript{Ga}As/Ga\textsubscript{As} strained-layer superlattices as dislocation filter layers, the threading dislocation (TD) density was reduced from the order of 10\textsuperscript{10} cm\textsuperscript{–2} to the order of 10\textsuperscript{7} cm\textsuperscript{–2}; detailed descriptions can be found in [9, 32]. To gain a better understanding of the emission properties of the QDs from photoluminescence (PL) measurement, very thin Al\textsubscript{Ga}As upper and lower cladding layers of 100 nm are used for this test sample. In the active region,
five layers of dot-in-a-well (DWELL) structure was grown, separated by 50 nm GaAs spacer layers. Each layer of DWELL consists of 2 nm InGaAs wetting layer, 3ML InAs QDs and 6 nm InGaAs capping layer. On top of the sample, uncapped 3ML InAs QDs were grown for QD density measurement using an atomic force microscope (AFM). Figure 1(b) shows the AFM image of QD density, which is around $3 \times 10^8$ cm$^{-2}$.

Before the thermal annealing process, the sample was cleaved into two and capped with a 200 nm SiO$_2$ film by plasma-enhanced chemical vapour deposition and 200 nm TiO$_2$ film by physical vapour deposition (PVD75), respectively. The two samples were then cleaved into many pieces for further comparison studies. For the SiO$_2$-capped samples, an RTA duration study was implemented for 0, 5, 30, 60 s at 700 °C. Based on the optimal annealing duration, different annealing temperatures of 700 °C, 725 °C, 750 °C, 775 °C and 800 °C were trialled. The annealing temperature study for TiO$_2$-capped samples was then executed at 700 °C, 725 °C, 750 °C and 775 °C. PL measurements of each as-grown and annealed sample were conducted under excitation using a solid-state laser of wavelength 532 nm at room temperature (RT).

For better understanding the effect of intermixing on the QDs capped by SiO$_2$ and TiO$_2$, high angle annular dark field (HAADF) STEM images were obtained using a C-EG F JEOL R005 double aberration corrected TEM/STEM operating at 300 kV. Lattice resolved STEM HAADF Z-contrast images, were then obtained with a convergence semi-angle of 21 mrad and a STEM inner annular collection angle of 62 mrad.

2.2. Selective area intermixing and laser device fabrication

For the selective area intermixing, the real laser structure of InAs/GaAs QDs on Si substrate was grown as shown in figure 2(a). The epitaxy layers and growth method were identical to the previous test sample, except for the two thick cladding layers of 1.5 μm and a highly doped 300 nm GaAs contact layer on the top. A quarter of the wafer was then covered by 200 nm SiO$_2$ and TiO$_2$ thin film half and half as shown in figure 2(b). A trade-off RTA condition of duration and temperature was applied from the previous optimisation studies for both SiO$_2$ and TiO$_2$ caps. After thermal annealing, PL mapping was executed to check the bandgap changes of the QD materials. The dielectric layers were then removed using hydrofluoric acid (HF) before further device fabrication. 25 μm width broad area lasers were fabricated by standard photolithography, wet chemical etching and metallisation. Ti/Pt/Au and Ni/GeAu/Ni/Au were deposited for p-type and n-type ohmic contacts, respectively. The laser devices were then cleaved into 3 mm length cavities, without any facet coatings, and mounted on the copper heatsinks.

3. Results and discussion

3.1. SiO$_2$ capping

3.1.1. PL spectra. Figure 3(a) shows RT PL spectra of the intermixing duration study of DWELL structure capped by SiO$_2$ for as-grown (A.G.), 5, 30 and 60 s RTA. The inset of figure 3(a) shows the PL peak intensity as a function of the annealing duration, showing an increase with longer annealing time and reaching the maximum point when the duration is 30 s. The increased PL intensity is contributed to by the reduced TD density after the high RTA. The high-temperature annealing enhances the mobility of dislocations.
and increases the probability of interaction and self-annihilation of the TDs with each other [33]. As shown in figure 3(b), the peak wavelength of PL was decreased with increased annealing time. This small blue shift is due to the inter-diffusion of In and Ga atoms between the interface of InAs QDs and GaAs barrier layers. The change of full width at half maximum (FWHM) of PL for the duration study is also shown in figure 3(b), the FWHM is raised at first when the annealing time is 10 s, and then decreases with longer annealing times. The decreased FWHM indicates an improved QDs size homogeneity when the annealing time is longer than 30 s. By considering the peak intensity, wavelength and FWHM, an annealing time of 30 s was chosen for the subsequent annealing temperature study, as it shows a relatively larger blue-shift and peak PL intensity.

![Figure 3](https://example.com/figure3.png)

**Figure 3.** (a) PL spectra of SiO2 capped InAs/GaAs DWELL structure grown on Si substrate with different duration times at 700 °C. Inset: the PL intensity against the annealing durations. (b) Plots of PL peak wavelength and FWHM against to the annealing durations at 700 °C. (c) PL spectra of SiO2 capped InAs/GaAs DWELL structure grown on Si substrate with 30 s duration at different temperatures. Inset: the PL intensity against to the annealing temperature. (d) Plots of PL peak wavelength and FWHM against to the annealing temperatures with 30 s duration.

[34]. The high temperature also causes a bigger blue shift of PL emission, and a comparison of peak wavelengths at different temperatures is shown in figure 3(d). It should be noticed that the PL spectra become broader when the annealing temperature is raised from 725 °C to 775 °C. However, the linewidth narrows at a very high temperature of 800 °C from figure 3(d). The broader linewidth accompanied by the large blue-shift is the contribution from an inhomogeneous diffusion of Ga atoms at relatively low annealing temperature [35]. Therefore, we presume that the RTA leads to a distinct change of emission peak and an increased linewidth at relatively low temperatures with SiO2 cap. At 800 °C, the influence of annealing on size distribution is more dominant. As a result, the FWHM reduces again, and the blue shift of the PL spectrum becomes larger than at other temperatures. This conclusion is further confirmed by our STEM observations.

The purpose of these studies is to find the most suitable RTA conditions, in terms of annealing time and temperature, of the SiO2-capped DWELL structure for a relatively large wavelength shift and an appropriate QDs optical emission without serious material degradation. By considering results from both annealing time and temperature studies, the RTA with 30 s duration at 725 °C causing ∼34 nm blue-shift in PL
peak position and $\sim$15.9% increases in PL intensity is the most useful choice, which is the reference for the further RTA study for the TiO$_2$-capped DWELL and selective area intermixing.

### 3.1.2. QD morphologic and size distribution analysis

HAADF STEM images of as-grown and annealed specimens were obtained at relatively low magnification (at $\sim$600kX, for size distribution analysis) while detailed STEM images of individual QD’s have been taken at high magnification (at $\sim$4MX) for comparison. As the image contrast in the HAADF image is approximately proportional to the square of the mean atomic number, assuming pure Rutherford scattering, such images offer an insight into the relative local chemistry of the features.

Figure 4(a) shows the HAADF STEM images for SiO$_2$ capped DWELL of the as-grown, 700 °C and 800 °C annealed samples and a corresponding schematic diagram of the morphological evolution. As the images are shown, QDs in the as-grown sample exhibit a sharp interface and inhomogeneous dot size. After 700 °C annealing, the interface of QDs and the surrounding matrix became less distinct, possibly due to the thermally intermixing process. However, after 800 °C annealing, the QD uniformity had significantly improved, with an increase in the average dot-size.

Approximately 100 QDs have been measured for each of the as-grown, 700 °C annealed and 800 °C annealed samples using HAADF imaging at $\sim$600 k magnification to determine the width and height of the QDs. The widths and heights of the QDs were measured by drawing intensity line profiles across the HAADF images. Table 1 displays the widths, heights and height/width ratio of QDs in the as-grown, 700 °C and 800 °C annealed samples.

**Figure 4.** (a) C$_s$ corrected HAADF STEM images of SiO$_2$ capped DWELL as a function of annealed temperature and related schematic diagram. (b) Histogram of SiO$_2$ capped QDs widths in as-grown, 700 °C annealed and 800 °C annealed samples.

| Sample     | Width (nm)  | Height (nm) | Height/width ratio |
|------------|-------------|-------------|--------------------|
| As grown   | 28.2 ± 4.4  | 8.8 ± 1.0   | 0.31               |
| 700 °C     | 28.8 ± 3.7  | 9.0 ± 0.8   | 0.31               |
| 800 °C     | 36.0 ± 3.3  | 9.5 ± 0.9   | 0.26               |

3.1.2. QD morphologic and size distribution analysis

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A more detailed width distribution of QDs capped by SiO$_2$ is given by the histogram presented in figure 4(b). The standard derivation of the 100 QDs size from the as-grown sample is around 4.4 nm. After the high-temperature thermal annealing, the standard derivation is decreased to 3.7 nm, and 3.3 nm from 700 °C and 800 °C annealed samples, respectively. These results are evidence to support our previous assumption from the PL measurement. The improved uniformity of QD size distribution after the RTA is the contribution of the decrease of FWHM at 700 °C and 800 °C.

Figure 5 shows a series of lattice resolved HAADF STEM Z-contrast images (presented in false colour) of typical single QDs as a function of annealing temperature. The increased contrast brightness in the region of the QD reflects the higher mean atomic number due to the presence of Indium. While the Z-contrast images are not specifically indium concentration maps, they highlight the changes in the QD morphology, which is likely to be associated with localised indium diffusion. The typical as-grown dot is $\sim$25 nm in diameter. After 700 °C annealing, the image contrast becomes more uniform across the dot region, and the QD size increased lateral. After 800 °C annealing, the lateral dot size further increased. It is important to note that although the overall aspect ratio appears to change as a function of annealing temperature, the basic shape morphology of QDs remains essentially the same.
3.2. TiO₂ capping

3.2.1. PL spectra. Due to the larger thermal expansion coefficient of TiO₂, the inter-diffusion rate inside of the TiO₂ capped DWELL structure is restricted during the RTA. Figure 6(a) shows the comparison of the PL spectra between the as-grown and TiO₂-capped DWELL structures with 30 s anneal duration at different temperatures from 700 °C to 775 °C. Since the annealing temperature of 800 °C is too high to maintain the material quality for SiO₂ cap, 800 °C is not employed in the following studies. The inset of figure 6(a) is the plot of the peak intensity of PL as a function of annealing temperature. The peak intensity is first raised and reached its maximum point at 700 °C, then reducing as the temperature increased. The intensity trace of the TiO₂ cap is similar to the SiO₂ annealing temperature study, where an appropriate annealing temperature could enhance the self-annihilation of the dislocations, thus improving the PL intensity. As the temperature further increased, the material quality and QDs will be degenerated or destroyed, gradually causing the PL intensity to drop. Comparing with the SiO₂-capped DWELL, the TiO₂-capped DWELL shows a much smaller blue-shift under the same annealing conditions, as demonstrated in figure 6(b). The blue-shift of TiO₂-capped DWELL structure is only ∼6.4 nm by RTA for 30 s at 725 °C. Also, the FWHM has a slight decrease at temperature up to 725 °C owing to the narrower size distribution of QDs.

3.2.2. QD morphologic and size distribution analysis. Figure 7(a) shows the HAADF STEM images of the DWELL of the as-grown, 700 °C annealing 775 °C annealing samples capped by TiO₂, with a corresponding schematic diagram of morphological evolution shown at the left-hand side. Comparing with the SiO₂ capped DWELL, the QDs capped by TiO₂ still retained the sharp interface and inhomogeneous dot size even after 700 °C annealing. When the annealing temperature increased to 775 °C, the surrounding matrix of dots became less distinct, and the average dot size was slightly increased due to the intermixing process at a very high temperature. This intermixing phenomenon has happened at 700 °C for SiO₂ capped QDs, which evidences that the TiO₂ with a larger thermal expansion coefficient could effectively restrict the intermixing process.

Table 2 shows the average size of QDs in width and height for as grown, 700 °C and 775 °C annealed samples. The widths of TiO₂ capped QDs for 700 °C, and 775 °C annealed samples are increased by 1.9% and 11.5%, respectively, compared with the as-grown sample. The error bars were calculated from the standard deviation, where the insignificant changes for different annealing temperature samples mean the size distribution of TiO₂ capped QDs for each sample remained almost the same. A detailed histogram of TiO₂ capped QDs widths in as-grown, 700 °C annealed and 775 °C annealed samples is shown in figure 7(b).

The HAADF STEM Z-contrast images of typical QDs as a function of annealing temperature are shown in figure 8. The indium distribution of a single dot is highlighted with contrast brightness. For TiO₂-capped QDs, the image contrasts of the single QDs of the as-grown sample and the 700 °C annealed sample are almost unchanged. Only a slight indium diffusion happens at the edge of QD at 700 °C annealing due to the intermixing process. After 775 °C annealing, the indium diffusion became more pronounced. Although it still keeps a basic shape morphology, the dot lateral dimension increases further as with the SiO₂ capped QD annealed at temperatures above 700 °C. The TEM results are consistent with the observation from the PL measurements in figure 6.

3.3. Selective area intermixing and laser results

Based on the previous studies of the annealing conditions of SiO₂ and TiO₂ caps, the RTA process with 30 s duration at 725 °C was implanted for selective area intermixing. Figure 9(a) shows the PL mapping of the peak wavelength for a QD wafer sample after the RTA with the SiO₂ and TiO₂ capped regions labelled. A clear square pattern could be found on the right side of the sample, which was covered by TiO₂. As expected from the previous studies, the SiO₂ capped region shows a bigger wavelength shift in PL measurement comparing with the TiO₂ capped region. Also, an even larger wavelength shift and a reduced intensity were obtained on the edge of the SiO₂ capped region. It is likely that the RTA caused serious degradation of the material quality at the wafer edge. Figures 9(b) and (c) show the comparisons of the PL spectra at the centres of SiO₂ and TiO₂ capped regions before and after RTA, respectively. Table 3 summarises the wavelength-dependent results of the PL spectra. There is a ∼40 nm blue shift of wavelength at the SiO₂ capped region after RTA, and only ∼3.4 nm peak wavelength shift at the TiO₂ capped
This RTA introduces a broader linewidth of the PL spectra for both of SiO\textsubscript{2} and TiO\textsubscript{2} capped regions. The SiO\textsubscript{2} region has a bigger change in FWHM and a larger blue shift compared with TiO\textsubscript{2}. It should be noticed that, compared with the test samples in the previous studies, the real laser samples have much thicker cladding layers with high doping levels. Although we kept the other epitaxial layers and growth methods the same as the test sample, the high concentration of the dopants from the cladding layers causes an unpredicted effect on the intermixing process, with a slight material degeneration from both SiO\textsubscript{2} and TiO\textsubscript{2} capped wafer.

Figure 6. PL spectra of TiO\textsubscript{2} capped InAs/GaAs DWELL structure grown on Si substrate with 30 s duration RTA at different temperatures. Inset: the PL intensity against to the annealing temperatures. (b) Plots of PL peak wavelength and FWHM against to the annealing temperatures with 30 s duration.

Figure 7. (a) High angle annular dark field (HAADF) STEM images of TiO\textsubscript{2} capped DWELL as a function of annealed temperature and related schematic diagram. (b) Histogram of TiO\textsubscript{2} capped QDs widths in as-grown, 700 °C annealed and 775 °C annealed samples.

Figure 8. Cs corrected high resolution HAADF STEM images (shown in false colour) of a single QD of TiO\textsubscript{2} capped DWELL as a function of annealed temperature with 30 s duration.

| Sample      | Width (nm) | Height (nm) | Height/width ratio |
|-------------|------------|-------------|--------------------|
| As grown    | 26.9 ± 3.9 | 8.7 ± 0.8   | 0.32               |
| 700 °C      | 27.4 ± 3.8 | 8.8 ± 0.8   | 0.32               |
| 775 °C      | 30.0 ± 3.4 | 9.0 ± 0.9   | 0.3                |

Table 2. Summary of the widths, heights and height/width ratio of TiO\textsubscript{2} capped QDs in as grown, 700 °C and 800 °C annealed samples.

region. This RTA introduces a broader linewidth of the PL spectra for both of SiO\textsubscript{2} and TiO\textsubscript{2} capped regions. The SiO\textsubscript{2} region has a bigger change in FWHM and a larger blue shift compared with TiO\textsubscript{2}. It should be noticed that, compared with the test samples in the previous studies, the real laser samples have much thicker cladding layers with high doping levels. Although we kept the other epitaxial layers and growth methods the same as the test sample, the high concentration of the dopants from the cladding layers causes an unpredicted effect on the intermixing process, with a slight material degeneration from both SiO\textsubscript{2} and TiO\textsubscript{2} capped wafer.

Figure 9(d) shows the comparison of the lasing spectra of the Si-based QD laser devices fabricated by this thermally processed wafer. Two different lasing wavelengths indicating
the SiO₂ and TiO₂ capped regions are shown, which gives ∼37 nm lasing wavelength difference. The inset of figure 9 is the light–current (L–I) curves of QD lasers on Si for as-grown and after the RTA process in SiO₂ and TiO₂ capped region under pulsed operation. Table 3. The comparison of PL parameters, in term of peak wavelength, intensity, FWHM and blue shift, for SiO₂ and TiO₂ capped DWELL structure before and after RTA with 30 s duration at 725 °C.

4. Conclusion

In this paper, we have demonstrates a comparative study of intermixing effects on InAs/GaAs QDs directly grown on Si capped by separated SiO₂ and TiO₂ layers. The optimisation of annealing conditions in terms of temperature and duration and STEM analysis were implemented for both cap materials. Electrically pumped InAs/GaAs QD lasers directly grown on Si with two different emission wavelengths of 1275 nm and 1313 nm were achieved from one single wafer by the selective area intermixing method. These results indicate that selective area intermixing is a promising technique for realising Si-based optical integrated circuits.

Acknowledgments

The authors acknowledge financial support from UK EPSRC under Grant No. EP/P006973/1, and EPSRC National Epitaxy Facility. European project H2020-ICT-PICTURE (780930), Beijing Natural Science Foundation Z180014; Royal Academy of Engineering (RF201617/16/28). ML thanks the Chinese Scholarship Council for funding her research studies. SC thanks the Royal Academy of Engineering for funding his Research Fellowship.
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