# Anodic–Oxide-Induced Intermixing in GaAs–AlGaAs Quantum-Well and Quantum-Wire Structures

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*(Invited Paper)*

***Abstract*— Anodic oxides of GaAs were shown to enhance the intermixing in GaAs–AlGaAs quantum wells (QW) during rapid thermal processing. Proximity of the anodic oxide to the QW has been shown to influence the photoluminescence (PL) energy shift due to intermixing. Anodic oxide induced intermixing has been used to enhance quantum-wire PL in the structures grown on V-groove patterned GaAs substrates. This has been attributed to enhanced lateral confinement in these structures. Injection of defects such as group-III vacancies or interstitials was considered to be driving force for the intermixing.**

***Index Terms*— Diffusion processes, oxidation, quantum wells, quantum wires, rapid thermal processes.**

## I. Introduction

QUANTUM-WELL intermixing (QWI) (interdiffusion) is very promising for postgrowth modification of materials properties and for fabrication of optoelectronic integrated circuits [1]–[4]. There are currently two kinds of interdiffusion techniques, one is the impurity-induced interdiffusion (IID) [5]–[7], another is the impurity-free interdiffusion (IFID) [3], [8]. IID usually introduces substantial undesired changes in the material resistivity and trap concentrations. IFID appears to be able to generate large bandgap energy shifts without such problems associated with IID. Usually, a dielectric layer is deposited on the surface of a quantum-well (QW) structure and the sample is annealed at elevated temperature. Depending on the dielectric material used, the encapsulant dielectric layer can enhance or suppress intermixing of the QW structure [9]. The dielectric layer can be deposited using chemical vapor deposition, electron–beam evaporation, or by other means [8]. In GaAs–AlGaAs system, $SiO_2$ and $Si_3N_4$ are commonly used to promote and reduce interdiffusion, respectively. $SiO_2$, however, reacts with Al when in direct contact with AlGaAs, and thus generates Si which behaves as an impurity source. $Si_3N_4$, on the other hand, causes considerable strain [3]. Alternatively, anodic oxide can be used as the encapsulant layer. Initial study showed that anodic oxide significantly increased the intermixing in GaAs–AlGaAs QW structures [10]. Anodic oxidization is an early way of making native oxide [11]. The pulsed anodization technique has recently attracted attention as a new way of forming current blocking layers for ridge-waveguide QW laser fabrication, since it was simple, reliable, and cost-effective [12]–[13]. We have recently applied this technique to enhance the photoluminescence (PL) and cathodoluminescence signals from V-grooved quantum wires and obtained both spectrally and spatially well-resolved light emission from quantum wires [14]. Like other intermixing techniques, this interdiffusion technique can be conveniently applied to the fabrication of multiple wavelength lasers and nonabsorbing mirrors for high-power laser applications.

In this paper, we review the effect of pulsed anodic oxide on the intermixing in GaAs–AlGaAs quantum structures. The experimental details will be given in Section II, and the results and discussions will be presented in Section III. The paper is summarized in Section IV.

## II. Experimental Details

The sample structures used for this work were grown by low pressure metal–organic chemical vapor deposition (MOCVD) on $p^+$GaAs substrates. The substrates were Zn-doped to a concentration of $1 \times 10^{19}$ $cm^{-3}$ and were of 2° off $\langle 100 \rangle$ toward $\langle 110 \rangle$. A GaAs buffer layer was grown on the substrate,

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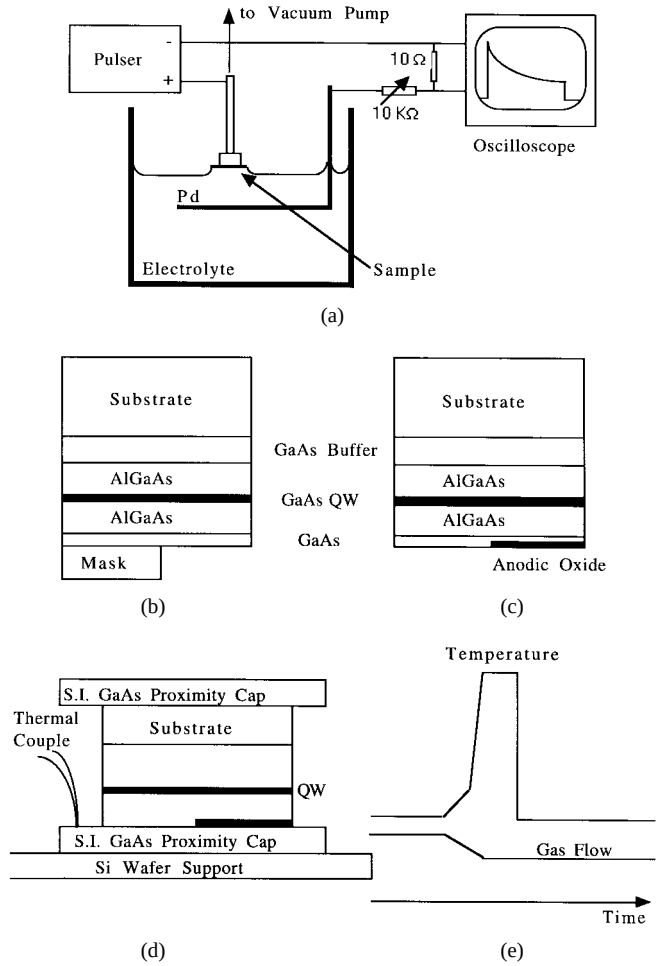


Fig. 1. (a) Experimental setup for pulsed anodic oxidization. The electrolyte was a solution of ethylene glycol:phosphoric acid:de-ionized water (40:20:1 by volume). The sample was held by the vacuum tweezer. The variable resistor was used to adjust the current density at the sample surface. The current was monitored with the oscilloscope. (b) Typical single QW sample with a mask on it to protect half of the surface before anodization. (c) Sample after anodization and removal of the mask. Half of the surface was converted into anodic oxide. (d) Sample load in the rapid thermal process chamber. Heating was provided by banks of lamp. (e) Typical programmed temperature and Ar gas flow.

and the thickness was between 100 and 1000 nm for different samples. For all the samples, the top layer was GaAs, the barrier layers were AlGaAs, and the QW layers were GaAs. All epilayers were nominally undoped and residual doping concentration was $\sim 10^{15}$ cm$^{-3}$.

The pulsed anodization was carried out at room temperature [15]. Fig. 1(a) schematically shows the anodization setup. For each sample, half of the surface was covered with thermal glue. A vacuum tube was used as the vacuum tweezer to hold the sample and conduct current. The electrolyte was made of ethylene glycol : phosphoric acid : de-ionized water (40 : 20 : 1 by volume). The voltage as measured across the two output terminals on the pulser was 50 V. The initial current was set according to the current density at the sample surface. The current density used in this paper was in the range from 40 to 160 mA/cm$^2$. The pulsewidth was 1 ms, while the pulse period was 12 ms. The total anodization time was fixed at 4 min. Under such conditions, the final anodic oxide thickness was

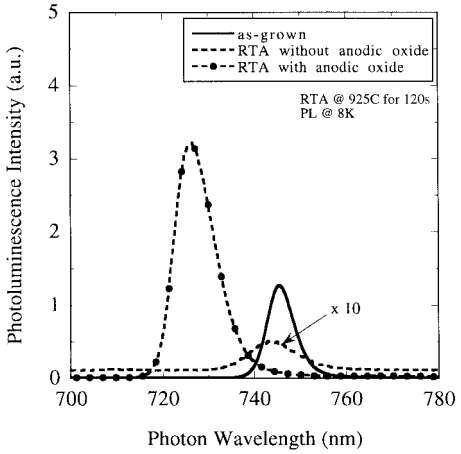


Fig. 2. PL spectra of a single GaAs–$Al_{0.3}Ga_{0.7}As$ QW. The QW was 3 nm thick, and the barriers were 50 nm thick. The solid line was from an as-grown sample. Another sample was annealed at 925 °C for 120 s with half area anodized (dashed–dotted line) and half area unanodized (dashed line). The anodization was carried out at 120 mA/cm$^2$.

in the range of 60–140 nm. Using higher voltage output from the pulser, higher current density, or higher pulse duty cycle, thicker anodic oxide can be obtained [16]. The area covered with thermal glue was unanodized, while anodization took place in the uncovered area to form an oxide layer, this is illustrated in Fig. 1(b). After anodization, samples were rinsed with de-ionized water, followed by acetone to remove the thermal glue, then by de-ionized water and $N_2$ blow dried [Fig. 1(c)].

The intermixing was carried out in a rapid thermal annealing (RTA) chamber with GaAs proximity caps in flowing argon ambient, as shown schematically in Fig. 1(d). The temperature was first slowly ramped up to 100 °C from room temperature (RT) in 1 min, and then was increased rapidly to the desired temperature (about 900 °C) at a ramp rate of 50 °C/s. After RTA, it cools down quickly [Fig. 1(e)]. PL measurements were performed at 8 K using an Ar–ion laser beam (514.5 nm), a spectrometer, and a Si CCD camera. Cares were taken to ensure the PL collection efficiency was about the same for each sample. For each sample, PL was measured both from the anodized area and unanodized area.

## III. Results and Discussions

We first study the effect of anodic oxide on the intermixing in a GaAs–AlGaAs single QW sample. The sample consisted of a 3-nm GaAs sandwiched between two 50-nm-thick $Al_{0.3}Ga_{0.7}As$ barrier layers. The top GaAs layer was 300 nm, while the GaAs buffer layer was 1000 nm. Fig. 2 compares the PL spectra from an as-grown sample, the anodized and unanodized areas of another sample. The sample was anodized at 120 mA/cm$^2$ and the thickness of anodic oxide was about 120 nm. Sample was annealed at 925 °C for 120 s. Relative to the as-grown sample, the unanodized area showed a marginal blueshift (5 meV) after annealing, while the anodized area showed much larger blueshift (44 meV).

Zn is well known to diffuse easily at elevated temperature and has been widely used as an impurity source in impurity-induced interdiffusion [17], [18]. But our data clearly

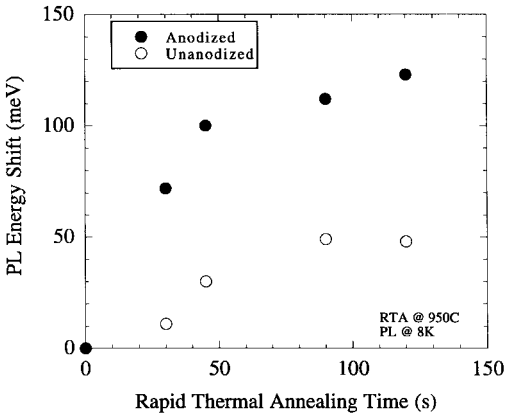


Fig. 3. PL energy shift relative to an as-grown sample against annealing time for a GaAs–$Al_{0.54}Ga_{0.46}As$ MQW structure. The sample was composed of four GaAs QW's, but for simplicity only the data for the 2.1-nm QW were shown here. The sample was annealed at 950 °C with half of the surface anodized (filled circles) and half unanodized (open circles). Similar trend held for other QW's of the structure.

demonstrated that the Zn dopants in the $p^+$-GaAs substrate did not play a dominant role under these annealing conditions.

Fig. 3 shows the blueshift relative to the as-grown sample versus annealing time for a QW of a four QW sample. For simplicity, only the 2.1-nm-thick QW data were shown. The $Al_{0.54}Ga_{0.46}As$ barriers were 50 nm thick. The GaAs top layer was 100 nm, and the GaAs buffer layer was 500 nm. The anodic oxide was about 60 nm thick, and the RTA was carried out at 950 °C. For the unanodized area, the blueshift was in line with the thermal stability study of GaAs–AlGaAs QW structures [19], and went up to 50 meV after annealing at 950 °C for 120 s. The anodized area, however, showed much larger blueshift (123 meV after annealing at 950 °C for 120 s). This suggests that anodic oxide is significantly enhancing the interdiffusion. Increased energy shift due to thermal intermixing in this case when compared to Fig. 2 (5–50 meV) may be due to increased temperature (from 925 to 950 °C) as well as increased Al content in the barriers from 0.3 to 0.54. In order to study the influence of the distance between the anodic oxide and the QW beneath it, we etched trenches into the top GaAs layer of the sample used in Fig. 2. This is shown in the insert of Fig. 4(a). The sample was first covered by thermal glue totally except an narrow stripe (window) region, and then was etched using phosphoric acid:hydrogen peroxide:de-ionized water (1:1:5 by volume) at 0.5 °C. After etching, the mask was removed and the process repeated to get more trenches. After etching, the whole sample was anodized except a small area for reference (denoted as "unanodized area"). The whole sample was then annealed at 950 °C for 120 s. The results were shown in Fig. 4(a). It can be seen that the unanodized area showed less blueshift compared to anodized area. And the QW beneath the trenches showed blueshift which increased with increasing trench depth, i.e., the closer the anodic oxide to the QW, the larger blueshift. If the enhanced blueshift were due to Zn diffusion from the heavily Zn-doped substrate, the blueshift should not have shown noticeable dependence on the trench depth. However, further work is under way to look at the effect of substrate

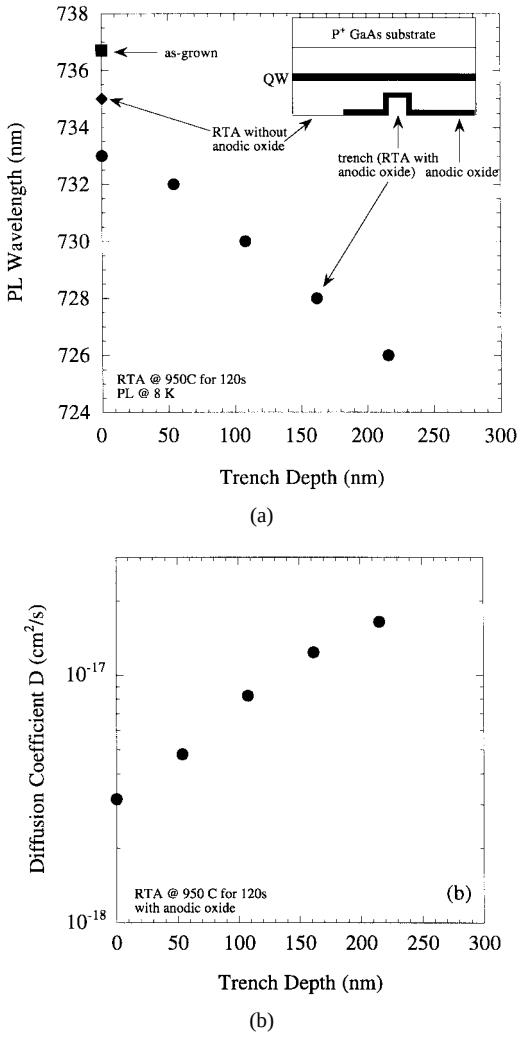


Fig. 4. (a) PL peak wavelength versus trench depth. The trench depth was measured before anodization. After the trenches with different depth were made by etching, the sample was anodized at 40 $mA/cm^2$ and then annealed at 950 °C for 120 s. Part of the sample surface was neither etched or anodized for comparison. PL peak wavelength for this area was shown as a diamond together with the PL peak wavelength for an as-grown sample (square). (b) Diffusion coefficient versus trench depth.

doping on intermixing. The observed blueshift dependence on the trench depth could find possible applications for the fabrication of multiple wavelength laser arrays.

To determine the diffusion coefficient dependence on the trench depth, we first calculated the subband energies and wavefunctions in an intermixing modified single nonsquare QW [20], and then compared the calculated interband transition energy to the PL data. The confinement profile of this interdiffused QW was nonlinear and was modeled here by an error function. The spatially dependent electron effective mass was taken into consideration using a nonparabolic band model derived from a fourth order expansion in $k$ with the coefficients determined using a 14-band calculation, and excitonic effect was also considered. The details of the modeling was given in our previous work elsewhere [15] where the calculated Al profile after intermixing was in good agreement with measured data. Fig. 4(b) shows the resulting diffusion coefficient as a function of the trench depth.

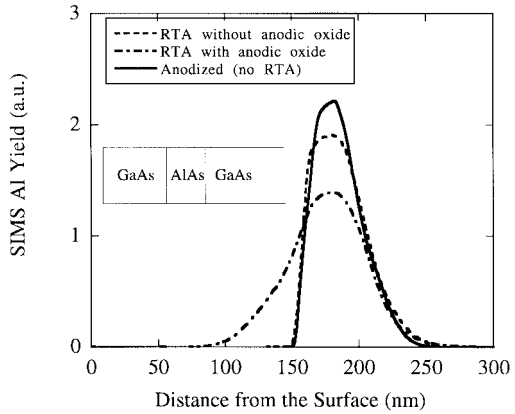


Fig. 5. Al profile from a GaAs–AlAs–GaAs substrate structure measured by SIMS. The solid line represents the Al profile from an anodized but not annealed sample. For the sample annealed at 950 °C for 60 s, the Al profiles were taken from the anodized area (dashed-dotted line) and unanodized area (dashed line).

To further confirm the effect of anodic oxide on the intermixing, we profiled Al from a GaAs–AlAs–GaAs substrate structure by secondary ion–mass spectroscopy (SIMS). The GaAs buffer layer was 1000 nm thick. The results were plotted in Fig. 5, showing in-depth profiles of Al, taken under identical SIMS conditions, from an anodized, but not annealed, sample and from an annealed sample with half surface anodized and half surface unanodized. The Al profile for the anodized but not annealed sample revealed the original Al profile. The asymmetric behavior in this case is characteristic for SIMS profiles of thin buried layers and reflects the mixing induced by the ion beam used for the SIMS analysis [21], which was 10-keV $O_2^+$ beam in this work. For the unanodized area of the annealed sample, the Al profile was slightly broadened on both sides of the AlAs layer, presumably due to the thermal interdiffusion in the sample. For the anodized area of the annealed sample, however, the Al profile was clearly seen broadened significantly toward the surface, i.e., toward the anodic oxide, confirming the strong effect of anodic oxidation on the intermixing in GaAs–AlAs layers.

We have also applied the anodic–oxide-induced intermixing to the PL and cathodoluminescence study of GaAs–AlGaAs quantum-wire structures grown by MOCVD on V-groove patterned $p^+$GaAs substrates. The quantum-wire structure used in this work was schematically shown in Fig. 6. The epitaxial layers consist of a GaAs buffer layer (100 nm), a lower $Al_{0.5}Ga_{0.5}As$ barrier $(1_m)$, a GaAs layer, an $Al_{0.5}Ga_{0.5}As$ upper barrier (100 nm), finally a GaAs layer (150 nm). On the bottom of the V-groove, a GaAs quantum wire was formed. Cross-sectional transmission electron microscopy (TEM) was used to check the structural properties and to measure the dimension of the structure. The vertical thickness of the quantum wire at the bottom of the crescent was measured to be 9 nm, and the sidewall QW thickness was 2.3 nm. The first electron to heavy hole transition was evaluated [22] to be 1.558 eV (796 nm), using the vertical quantum-wire crescent thickness of 9 nm and a lateral potential well width of 67 nm, and a lateral potential of 101 meV in the conduction band and 68 meV in the valence band. Fig. 7 showed the PL from the quantum-wire

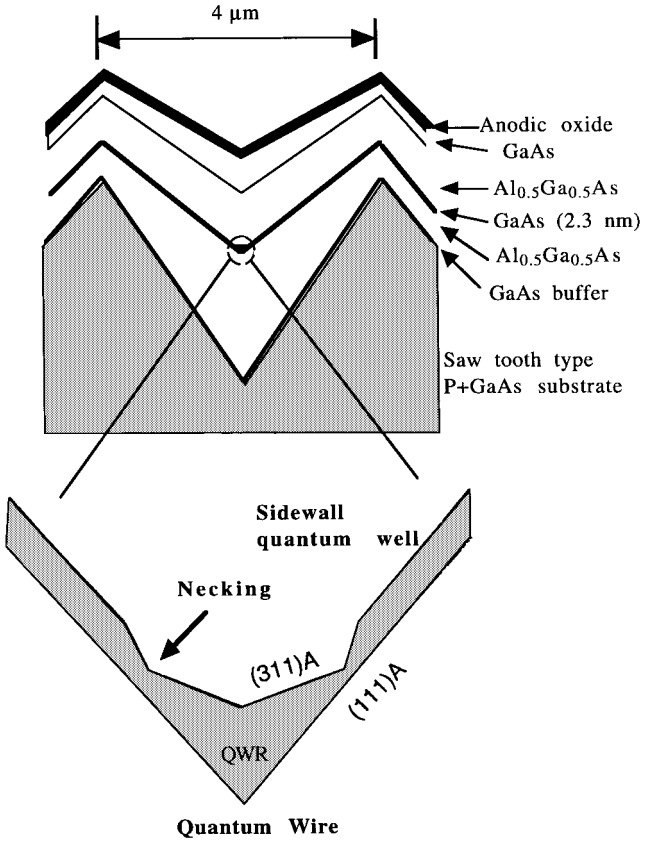


Fig. 6. Schematic diagram of the GaAs–AlGaAs quantum-wire sample used in this paper. The quantum-wire region was enlarged and shown in the lower part of the graph.

structure. No characteristic PL signal from quantum wire was seen for the as-grown sample. A hint of the quantum-wire PL was barely seen from the annealed area without anodic oxide. This is due to the fact that the volume of the quantum wire is much smaller than that of the side-wall QW's (SWQW's), so most of the photogenerated carriers were collected by the SWQW's and recombine there. The quantum-wire PL from the anodized area of the annealed sample is very clearly seen in Fig. 7. Although the annealing time was only 1s, it should be pointed out that the ramp time from 100 °C to 900 °C was 16 s, and it took a few seconds for the sample to cool down from 900 °C to below 700 °C. The arrow in the graph indicates the calculated transition wavelength in the as-grown quantum wire. The assignment was also confirmed by cathodoluminescence done on similar samples annealed with anodic oxide [14]. In our earlier study, quantum-wire PL was not observed until RTA temperature was above 850 °C from anodized sample [14], this coincides with the fact that anodic–oxide-induced intermixing in QW's emerges around 850 °C. The likely reason for the anodic–oxide enhanced quantum-wire PL signal could be that the anodic–oxide-induced intermixing in the SWQW's [23] increased the SWQW's subband energy and thus reduces the carrier confinement in the SWQW's, so more photogenerated carriers could move to the quantum wire before they recombine in the SWQW's, as the quantum wire is less affected by the anodic–oxide-induced intermixing in the vertically due to its larger thickness.

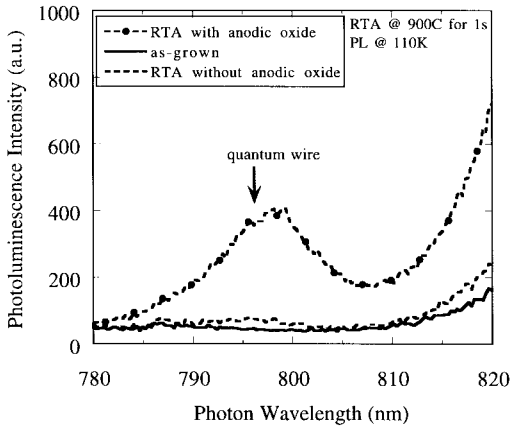


Fig. 7. PL spectra of the quantum-wire structure. The solid line was from an as-grown sample. A sample was annealed with half area anodized (dashed-dotted line) and half area unanodized (dashed line). The arrow indicates the calculated quantum-wire PL position.

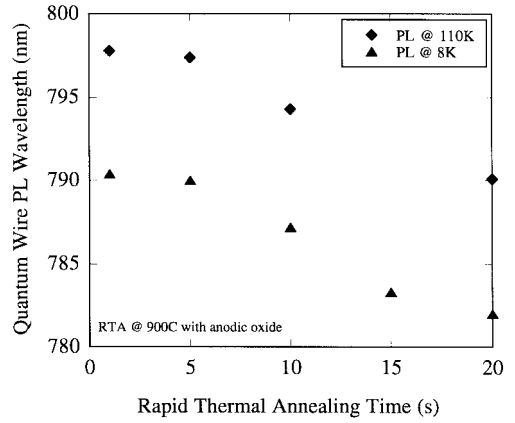


Fig. 8. Quantum-wire PL peak wavelength as a function of annealing time. The anodized samples were annealed at 900 °C. PL data were taken at 110 K (diamonds) and at 8 K (triangles).

Fig. 8 shows the quantum-wire PL peak wavelength as a function of the RTA time for samples annealed at 900 °C. Two sets of data were given for the same set of samples, one for the 8 K PL and another for the 110 K PL. It is clear that in the RTA time range, the PL from the quantum wire was blueshifted. Since no quantum-wire PL data were obtained without the anodic–oxide-induced intermixing in the SWQW's, it is difficult to say whether or not the quantum-wire PL blueshift was affected significantly by the presence of the anodic oxide.

Compared to other impurity free induced intermixing techniques, anodic–oxide-induced intermixing technique is very simple and cost-effective. Increasing the output voltage of the pulser, or increasing the current density or duty cycle, or a combination of all of these parameters, we have found that the anodic oxide thickness may be increased significantly. It is expected that increased anodic oxide thickness would lead to larger blueshift. Using different electrolyte also showed encouraging results. Work is in progress to optimize the anodic–oxide-induced intermixing.

So far, the mechanism of the anodic–oxide-induced intermixing is still not very clear. It is believed that the enhanced intermixing in the anodized samples was associated with the anodic oxide, not directly to the Zn in the Zn-doped $p^+$-GaAs substrates. Efforts are under way to look at the role of doping of the substrate and epilayers in anodic oxide induced intermixing. Our studies showed that the residual water in the anodic oxide plays some role, but is not the main cause [15]. The interdiffusion results suggests that the concentration of a native point defect, i.e., a group-III vacancy or interstitial, is increased at elevated temperatures when annealing under an anodic oxide. We consider it is likely that at high temperatures the oxide causes the injection of these native defects which enhance the interdiffusion. For the impurity-free enhanced interdiffusion induced in GaAs by a deposited $SiO_2$ layer [3], it is often assumed that an increased Ga vacancy concentration enhances the interdiffusion. However, the chemical interactions between GaAs and a hydrated mixture of Ga and As oxides are presumably quite different than those between GaAs and $SiO_2$ [24]. Work on the mechanism is in progress.

## IV. Conclusion

We have shown anodic–oxide-induced intermixing in GaAs–AlGaAs QW and quantum-wire structures. It has been demonstrated that the intermixing is significantly promoted by the presence of an anodic–oxide on the top of the structure. This effect is stronger if anodic oxide is placed closer to QW. Zn diffusion alone (without anodic oxide) induced impurity interdiffusion due to large amount of Zn in the heavily $p^+$-GaAs substrates was not the cause of the anodic–oxide-induced intermixing. However, this does not rule out the possibility that interaction between Zn and the point defects produced by anodic oxidation may play an important role in the anodic–oxide-induced intermixing. We have applied the anodic–oxide-induced intermixing to enhance the quantum-wire PL signal in GaAs–AlGaAs quantum-wire structure grown on V-grooved substrate.

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