

Fig.2.5 SEM images of butt joint. (a) InGaAlAs-InP. (b). InGaAsP-InGaAsP. (c). InGaAlAs-InGaAsP

Fig.2.5 gives three examples of the butt-joint regrowth. Fig.2.5 (a) shows the image from [8, fabrication of]. The InGaAlAs core layer is buried in the regrown InP. Fig.2.5 (b) shows the image from [12]. The cores in both active region (left) and passive waveguide (right) consist of InGaAsP. Very little misalignment in the vertical direction was observed. Fig.2.5 (c) shows the image from [10]. The core of the left consists of InGaAlAs and that of the right consists of InGaAsP. The smooth connection in the core layers was obtained by in-situ cleaning.

Unfortunately, no papers working on InGaAlAs-InGaAlAs butt joint by ex-situ cleaning is found as far as I know. However this is what I will do in this section: butt joint connection for InGaAlAs-InGaAlAs core layers by using ex-situ cleaning before the regrowth.

2.4.1. Main issues

In order to reduce the coupling loss and improve the coupling efficiency, the connection of the interface between active region and passive region should be as perfect as possible. "Perfect" implies the following aspects:

Vertical alignment: the core layers of the active region and passive waveguide should be aligned at the same level so as to reduce the reflection and scattering.

Direct connection: in many cases, the core layers at the interface are separated by a void, as shown in Fig.2.9 (c).

Surface morphology: After the regrowth, the top surface of the regrown region should be kept at the same level. In other word, the overgrowth at the interface should be avoided or removed.

For example, Fig.2.6 shows the Scanning Electron Microscope (SEM) picture of the regrown sample(#3955) in our first trial. Before the regrowth, the InP layer and the core layer (100 nm USCH/130 MQW/100nm LSCH) was etched by Saturated Bromine Water (SBW) solution (SBW:HBr:H₂O=1:5:10) at room temperature. It offers several important lessons which must be taken into account for further improvement: 1) the misalignment of the bottoms of the active region and regrown passive region. This is caused by the imprecise control of the etching time. Another reason is that, in this case, the SBW is a non-selective solution and it continues to etch the InP substrate below the InGaAlAs core layer. Obviously, some selective solution is preferred to obtain better alignment. 2). Disconnection at the interface. The grown InP piles into the interface between the core layers of active and passive region and separate them. It will cause significant coupling loss due to the large refractive index difference between the InP and the other to core material. This disconnection may come from the small slope at the sidewall of active core layer. We think, the quaternary InGaAsP is very difficult to crystallize directly on the InGaAlAs surface due to the mismatch of the lattice constant. In order to avoid this, the sidewall of the InGaAlAs core should be as vertical as possible, so that the atom of InGaAsP can fill in the gap at the interface before the growth of InP buffer layer for the passive region. Other chemical solutions or ICP etching can be applied to meet this demand. 3). The undercut of the buffer layer/core layer beneath the SiO₂ mask. During the wet etching , the SBW solution etched the InP buffer layer vertically and horizontally simultaneously. Moreover, while etching the InGaAlAs core layer, the SBW kept on etching the sidewall of the InP buffer layer horizontally, resulting in more severe undercut, though the etch rate of the vertical etching and horizontal etching are different. Equivalently, the edge of the SiO₂ mask extended and

shadowed the sidewall of the InGaAlAs core. Thus fewer InGaAsP can reach the interface and form the crystallization. The undercut may also contribute to the disconnection mentioned above. In order to avoid this, several means can be applied. One is to reduce the thickness of the InP buffer layer because undercutting time is determined by the thickness of the buffer layer and core layer; this is one of the main reasons why two-step regrowth (for detail, refer to subsection 2.4.4) is applied in our experiments and many other groups^{??(ref)?}. Another method is to remove the extended part in an additional step before the regrowth.

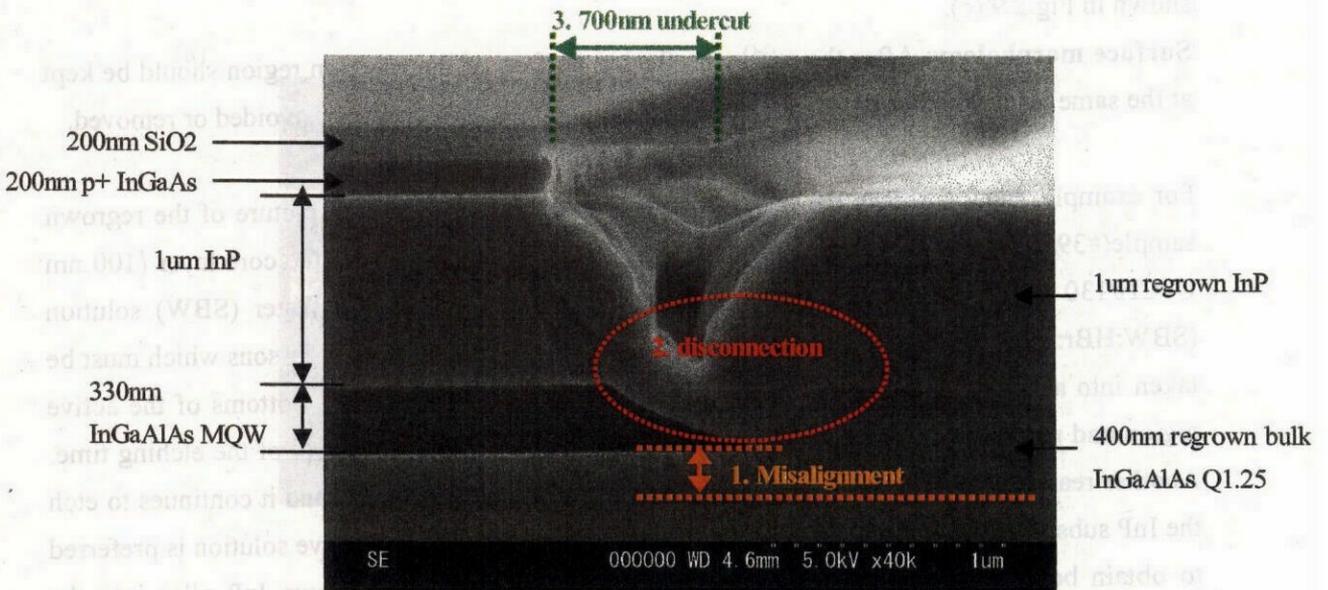


Fig.2.6 Scanning Electron Microscope (SEM) image of the cross section view of the sample #3955 after regrowth . The InP buffer layer and the InGaAlAs core layer were etched all by SBW (Saturated Bromine Water:HBr:H2O=1:5:10) at room temperature.

Detailed discussion is given in subsection 2.4.3 and 2.4.4. In all the following experiments, we use the same stack for the core layers, as shown in Fig. 2.7. In the active region, the layer stack consists of an n-InP substrate, a 100nm non-intentionally doped lower SCH of InGaAlAs with the PL wavelength of 1100nm, a 130nm MQW core layer with the PL wavelength of 1490 nm, a 100nm upper SCH of InGaAlAs, a InP cladding layer, and sometimes a p⁺-doped InGaAs contact layer. The thickness and doping of the InP cladding layer change in different cases. In the passive region, the core layer is composed of 400nm bulk InGaAsP with the PL wavelength of 1250nm and non-intentionally doped InP cladding layer.

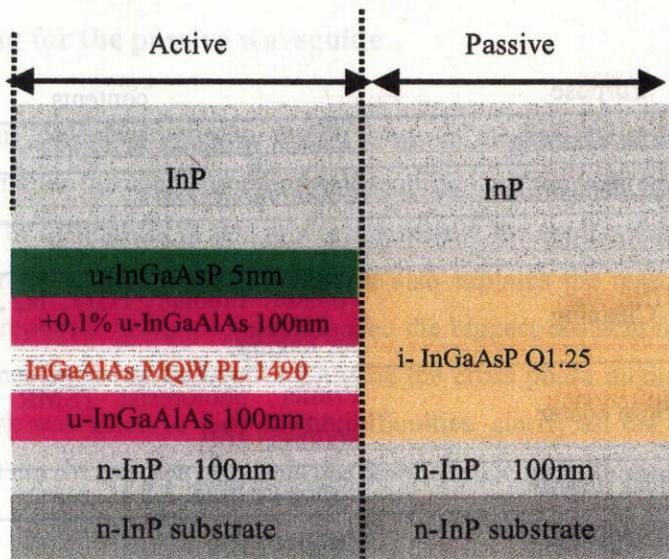


Fig. 2.7 Layer stacks of the active region and passive region. The thickness and doping of the InP buffer layer change in cases.

2.4.2. Treatment before regrowth

Table 2.4 shows the processing steps before regrowth. The first step after the growth of the active device is the deposition of a 200nm SiO₂ mask layer with a magnetron sputtering machine (ULVAC). The deposition is done at 250 °C, and for 15 minutes. Then the active region will be patterned by the photoresist by means of photolithography. The types of photoresist depend on the following etching method. S1805 is used on for wet etching and TSMR 8900 can be used for both wet etching and dry etching. In order to obtain vertical sidewall of SiO₂, we use CHF₃ Inductively Coupled Plasma (ICP) to etch the SiO₂ mask with CHF₃ /Ar/O₂ plasma (gas flow ratio 1:9:0.3, source power 200W and bias power 25W). Finally, the remaining photoresist is removed using the ICP O₂ plasma ashing before InP material is etched. In order to remove the active layer stack where the passive waveguide will be grown, both wet etching and dry etching can be applied (detailed in 2.4.2.3).

SiO₂ deposition

Step	Purpose	contents
1	Wafer cleaning	Aceton, Ethonal, H ₂ O, N ₂ .
2	SiO ₂ deposition	600nm/hour, 250°C

Photolithography

5	Cleaning	Aceton, Ethonal, H ₂ O, N ₂ . 90°C oven for baking.
6	Spin coater	Primer, photoresist TSMR (500rps:5s, 6000rps: 40s)
7	Mask aligner	Exposure time: 3.8 s
8	Development	NMD-3: 20 s

ICP dry etching for SiO₂

9	SiO ₂ etching	CHF ₃ /Ar/O ₂ : 1/9/0.3, room temperature
10	Photoresist removal	O ₂ ashing

Passive region definition

11	Wet etching/dry etching	2.4.3.1/2.4.3.2
----	-------------------------	-----------------

Cleaning right before the growth

12	Wet etching/dry etching	
----	-------------------------	--

Table 2.4 Processing steps before regrowth.

Before regrowth, the samples are etched in C₆H₈O₇ –H₂O₂ –H₂O (citric acid) for about 5 seconds. This step removes about 7 nm of InAlGaAs and 2 nm of InP. Afterwards, a 3-min H₂SO₄ cleaning step is employed. Then the sample is immersed into the solution of 3.2 ml (NH₄)₂S_x/ 45 ml H₂O at 50 °C under illumination of a halogen lamp for about 10 minutes. After this the sample is rinsed in water for about 20 minutes to remove the excess sulfur from the surface. Due to the sulfur passivation, the oxidation of the sidewall can be prevented.

2.4.3 Etching for the passive waveguide

The etching in the defined passive region is one of the key issues of high quality regrowth. It directly determines the coupling efficiency/coupling loss between the active device and passive waveguide. The difficulties in this processing restrict the application of the butt-joint regrowth, especially for InGaAlAs material system. It also explains the reason why in-situ cleaning by MOVPE is proposed. In my project, it is also the biggest obstacle for the fabrication of the all optical switches by using InGaAlAs EAM as the cross phase modulation medium. Long time and hard work was taken to face all the difficulties, clarify all the issues, and try all possible means to find out the best way towards the dawn of success.

2.4.3.1 Wet etching

Etchant dependence:

From subsection 2.4.2, we know that different solutions bring different sidewall profile of InGaAlAs core layer and consequent connection perfection. We have tried several solutions including SBW, SH and citric acid. From Fig.2.6, we know that SBW etches both InP and InGaAlAs effectively, and the core layer of the passive waveguide cannot match well with that of active layer, leading to mode mismatch and large scattering loss. Fig. 2.8 shows the SEM images of the cross section view by using different solutions. Both SH ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}=1:1:10$) and citric acid ($\text{C}_6\text{H}_8\text{O}_7:\text{H}_2\text{O}_2=5:1$) are selective solutions, so they stop etching the InP substrate beneath InGaAlAs core layer. Flat and clear horizontal interface appears in both cases. Compared to SBW, they offer the match between the core layers of the active and passive parts, and consequently increasing the coupling efficiency and reducing the insertion loss due to mode mismatch. Furthermore, the side wall etched by the citric acid is sharper than that etched by SH. This is mainly caused by the faster side etching of the InGaAlAs SCH layer than that of MQW layer. The difference of the side etch rate may come from the difference of the composition of Al in the SCH with rich Al and MQW with effectively poor Al. This is not in contradiction with the results in ref.[13], because the crystal orientations are different.

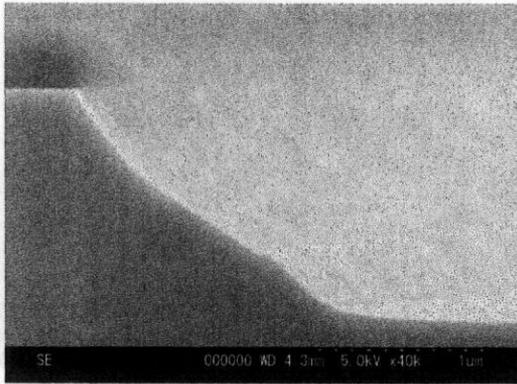
Orientation dependence:

The wet etching quality depends not only the solutions, but also the orientation. Fig. 2.9 shows

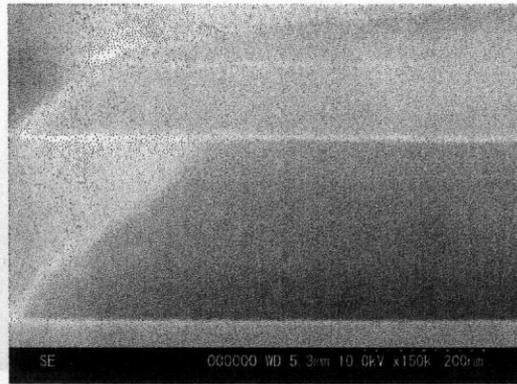
the orientation dependence of the wet etching and regrowth. Fig.2.9 (a) is for reverse ridge structure and Fig.2.9 (b) is for forward ridge one, and the core layer of both ridge structures are etched by the same SH solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}=1:1:5$ at 5°C) for 30 seconds. Focusing on the profile of the side wall of the core layer, we find that the undercut for reverse ridge (about 300 nm) is more severe than that for forward ridge (about 100nm). As a result, a void region appears after the regrowth for the reverse ridge structure while good connection is obtained for forward ridge type. The void is due to the shadowing effect caused by the upper InP cladding layer. This phenomena is similar with that caused by SiO_2 , as mentioned above. The void has very negative influence on the coupling loss, lasing performance and etc. How to eliminate the void and avoid the shadowing effect is the key issue for the regrowth of the reverse ridge structure. After all, it has very vertical sidewall of InP cladding layer, which is beneficial to avoid the overgrowth and achieve flat surface after the regrowth. On the other side, the undercut is slighter in forward ridge case. However, the quaternary InGaAsP Q(1.25) material grows in $[0 -1 1]$ and $[0 1 -1]$ direction, thereby providing a base from which InP can rapidly grow in the $[1 0 0]$ direction, causing the overgrowth at both side of the SiO_2 mask. The thickness and the height of the overgrown part are almost the same with the thickness of InP cladding layer, which is 1 μm . This overgrowth cause more difficulty in the following fabrication and degrade the device performance. One way to eliminate the overgrowth is to use ICP to etch the thick InP cladding layer. We will discuss this in the following subsection. Another way is to reduce the thickness of the InP cladding layer so that the overgrown can be suppressed. This is another reason to apply two-step regrowth, as well as that mentioned in section 2.4.1.

In conclusion, for one-step regrowth, reverse ridge type is preferred due to its vertical sidewall of InP cladding layer and avoidance of the overgrowth. In this case, the core layer can be firstly etched by the ICP to suppress the undercut, then followed by selective wet etching to define achieve perfect horizontal alignment. For two-step regrowth, forward ridge type is beneficial because of slighter under cut and suppression of the overgrowth due to the thin InP cladding layer.

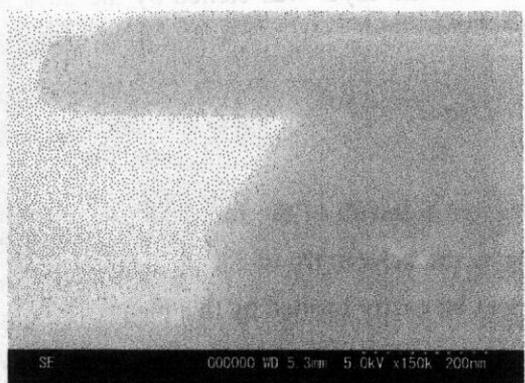
In this thesis, reverse ridge denotes $[0 1 1]$ orientation and forward ridge denotes $[0 -1 1]$ orientation.



(a)

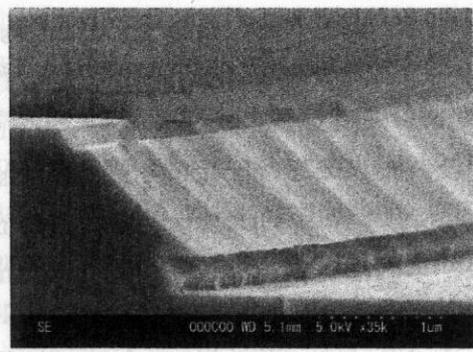
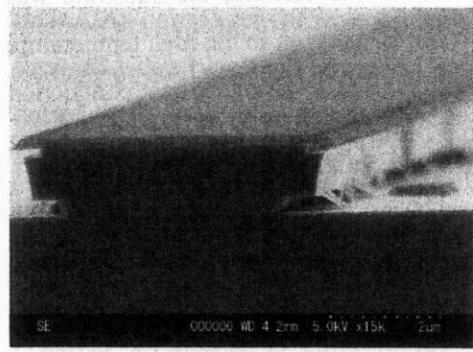


(b)

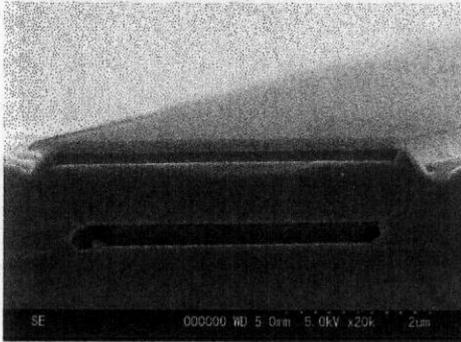


(c)

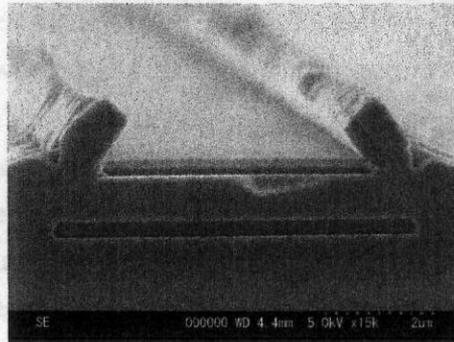
Fig. 2.8 SEM images of the cross section with sidewall etched by (a) SBW (SBW:HBr:H₂O=1:1:10) (b) SH (H₂SO₄:H₂O₂:H₂O=1:1:10) and (c) Citric Acid (C₆H₈O₇:H₂O₂=5:1)



a. Reverse ridge before regrowth



b. Forward ridge before regrowth



c. Reverse ridge after regrowth

d. Forward ridge after regrowth

Fig. 2.9 Orientation dependence of the regrowth. The InP layer was etched by the 20% HCl for 90 seconds and the core layer is etched by $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}=1:1:5$ (SH) for 30 seconds.

2.4.3.2 Dry etching

The main purpose of the dry etching is to obtain vertical sidewall of the InP cladding layer and suppress the undercut of the core layer. Nevertheless, the selectivity of the ICP is worse than wet etching, thus flat and clear bottom interface cannot be easily formed by this means.

The etching of the InP/InGaAs/InGaAlAs material can also be done in the same ICP etching machine with Cl_2/Ar plasma. The etching condition for low damage and vertical, smooth sidewall has to be experimentally found. At an elevated temperature of 220°C , which was the ideal temperature to efficiently remove any indium chlorides formed in the etching, the gas ratio of $\text{Cl}_2/\text{Ar} = 2:8$ sccm was found to be ideal. At source power 200W and bias power 25W and under 1Pa ambient gas pressure, the etching rate and selectivity of InP over SiO_2 were 350nm/min and 1:5 respectively. The etching rate of InGaAlAs was 400nm/min. In this etching, the substrate was bonded to the Si carrier wafer using the silver paste. This practice is common in the high temperature etching process with our ICP etching machine. The silver paste is used to facilitate heat conduction to the substrate surface and to avoid sample of small size from being blown away during the etching process. The total area of the etching surface has to be made same for ever etching run to ensure a same etching condition since our ICP etching machine shows considerable loading effect to the etching profiles. This was done by placing dummy InP substrate of appropriate sizes to conserve a same etching area.