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Release of Epitaxial Layers Grown on InAs Substrates

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We present a process for selectively releasing large area epitaxial layers from InAs substrates. The technique employs the extreme selectivity between InAs and GaSb when etched by hydrochloric acid. The etch-stop layer is a strain-compensated superlattice containing GaSb. It is sandwiched between the desired epitaxial layer and the InAs substrate to effectively stop the etch process, leaving the desired epitaxial layer with high surface and crystal quality after the process. The etch-stop layer can be further etched away. The epitaxial thin film had been successfully grafted to a GaAs substrate by van der Waals bonding.
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Grafting an epitaxial semiconductor film onto a new substrate and fabricating devices on this film has been proposed for many high-performance devices. A technique that is often used to realize this is the epitaxial lift-off (ELO) process.[1,2] In this paper, we extended the ELO process to manufacture devices on epitaxial layers that were grown on InAs wafers, of interest to midinfrared light emitting devices, photodetectors, and electronic devices.[3-5]

In 1995, Fastenau *et al.* reported a technique to lift-off thin InAs layers grown on a GaAs substrate by using an AlSb sacrificial layer.[6] An alternative way is to etch off the substrate to release the epitaxial film, similar to that described by Yi-Yan *et al.* for the InGaAs/InP material system.[2] The latter approach is generally faster, more reliable, and more suitable for large samples. Our implementation for InAs substrates is based on the latter concept, and makes use of the extreme etch selectivity that can be achieved between InAs and GaSb in hydrochloric acid.

Experimental

Before we describe the details of the processing, we list all the agents and etch mixtures used in the experiment. Where the symbol HCl is used, it should be understood as hydrochloric acid with 37% concentration of hydrogen chloride. Analogously, HF is hydrofluoric acid with 49% hydrogen fluoride. H_2SO_4 is sulfuric acid with 96% hydrogen sulfate. H_2O_2 is 30% hydrogen peroxide. All agents are taken from freshly opened bottles and at room temperature. Two etch mixtures were prepared using some of the agents listed above and deionized water (H_2O). Mixture A, $H_2SO_4:H_2O_2:H_2O = 1:8:15$. Mixture B, $H_2SO_4:HF:H_2O_2:H_2O = 1:1:4:50$. All the ratios quoted here are in volume and at ambient temperature. Since the reactions of mixing are exothermic, both mixtures are made at least 4 h before the experiments to ensure that they have returned to ambient temperature. All the experiments were carried out in a class 1000 clean room with ambient temperature at 21-22°C. Unless otherwise stated, all the agents and mixtures are stirred at a speed of 200 rpm during the experiments.

Two types of semiconductor substrates, InAs and GaSb, are used in the experiments. The crystallographic orientations for both of them are (100).

In order to establish the process, the etch selectivity of HCl on InAs and GaSb was determined first. The first step was to find out the etch speed of InAs by HCl. A piece of Si-doped InAs wafer was first patterned by standard photolithography and then immersed in HCl for 60 s. The sample was then carefully rinsed in H_2O and the photoresist was stripped. This etching results in a very rough surface; the etch depth measured by a Sloan IIA Dektak is about 800 nm. In the second step, a similar experiment was carried out to determine the etch speed of HCl on a Te-doped GaSb wafer. After an etching of 60 min, no measurable etch profile was found when measured by the Dektak, which has a minimum resolution in the tens of nanometers range. Even by using a Nomarski phase-contrast microscope, which can reveal a surface roughness as small as several nanometers, no etch profile could be detected. We can therefore safely assume that the etch depth in GaSb is less than 10 nm, and hence that the selectivity of HCl when etching InAs *vs*. GaSb is at least greater than 4800.

GaSb however cannot be used straightforwardly as etch stop on InAs, because the lattice mismatch between InAs and GaSb would not allow growing high quality epitaxial layers of GaSb of sufficient thickness on InAs. We therefore designed a lattice matched etch-stop layer as $In_{0.91}Ga_{0.09}As/GaSb$ strained layer (but strain compensated) superlattice with identical thickness of each constituent layer. Several samples with different etch-stop thickness have been grown to explore the process technology. The actual layer grown on top of the etch-stop layer is a midinfrared (MIR) light emitting diode (LED) structure designed to emit at around 4.3 μm. A schematic drawing of the full epitaxial structure is shown in Fig. 1 and details of the etch-stop layer structures of the three samples are listed in Table I. The wafers employed have thicknesses of about 375 μm typically and were only polished on the epi side. The surface roughness of the other side is estimated to be in the order of several micrometers.

The sample was first glued on a glass carrier wafer with Apiezon W wax. The wax had to be applied very carefully without covering the edge of the sample. This is critical for successfully grafting the thin film to a foreign substrate. The wax protects the top surface and provides support for the thin film after the epitaxial release process. The glass carrier wafer facilitates the handling of the sample.

The sample was then immersed into mixture A for about 130 min. This step etches about 337 μm of InAs substrate at an average etch speed of 2.59 μm per min, leaving a film of about 40 μm. After careful rinsing, the sample was then put into HCl for about 30 min without stirring. Once the whole surface appears shiny, the etch stop is reached and the sample can be taken out and rinsed. This process worked very well for samples B and C but appeared to be unsuccessful for sample A. Samples B and C were left in the etchant for an extra 20 min to ensure that the substrate was totally etched away, eliminating the need for visual inspection of the sample surface after the substrate etchback process.

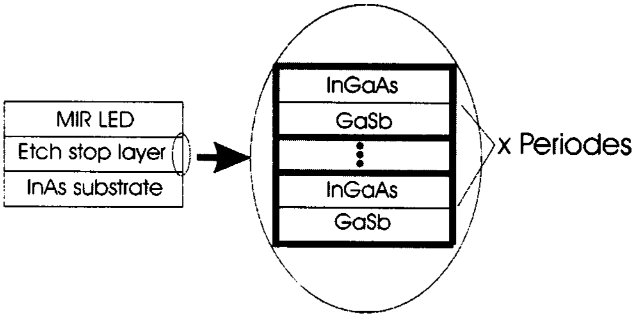

Figure 1. Schematic structure of the test samples.

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Table I. Detailed list of the etch-stop layer structures of three samples.

Sample	Thickness of each constituent layer (nm)	Periods	Total thickness of etch-stop layer (nm)
A	20	2	80
B	20	10	400
C	10	10	200

In order to find out the reason of the lack of success of sample A, we repeated the process and carefully observed the surface during the process. We found that some tiny holes first randomly appeared on the surface when the substrate was etched away in HCl at most parts of the sample. We presume that these holes are etch pits related to defects on the substrate. When such etch pits are deep enough to penetrate the etch-stop layer and reach the LED structure, which contains mostly InAs, the etchant starts to attack the LED structure and expands underneath the etch-stop layer. It is thus appears mandatory to use an etch-stop layer of at least 200 nm.

If desired, the etch-stop layer can also be removed. Mixture B was used for this purpose. Addition of hydrofluoric acid is needed to etch GaSb while the sulfuric acid helps to etch InGaAs. During the etch process, we observed the typical color changes corresponding to the subsequent removal of the GaSb and InGaAs layers. On samples B and C, we counted 10 periods, proving that all 10 periods of the etch-stop layers were left after the HCl etch. The estimated etch speed of the etch-stop layer in mixture B was around 0.5 μm/min. The attack of the InAs layer of the LED structure can be controlled within a range of tens of nanometers and could be further improved by using a higher dilution of mixture B.

The obtained thin film was then successfully bonded to a GaAs wafer using van der Waals bonding.[1] In the next step, the Apiezon W wax was dissolved in chloroform followed by rinsing and drying with a nitrogen gas pistol.

This process has been applied successfully to samples as large as a quarter of a 2 in. wafer and should be applicable to even larger samples. Applications of this technology include resonant-cavity emitters[7] and detectors,[8] surface-textured emitters[9] and detectors,[10] etc.

Characterization

The process was modified slightly by replacing the Apiezon W wax with standard white wax to provide a hard surface to be measured by scanning probe microscope (SPM). Figure 2 shows an SPM micrograph of sample B before the van der Waals bonding. The measured root mean square roughness is about 1.9 nm over 2 μm scanning range, showing an extremely high surface quality. Photoluminescence measurements showed no deterioration in luminescence efficiency of the MIR LED structure, proved that the high crystal quality was maintained after the process.

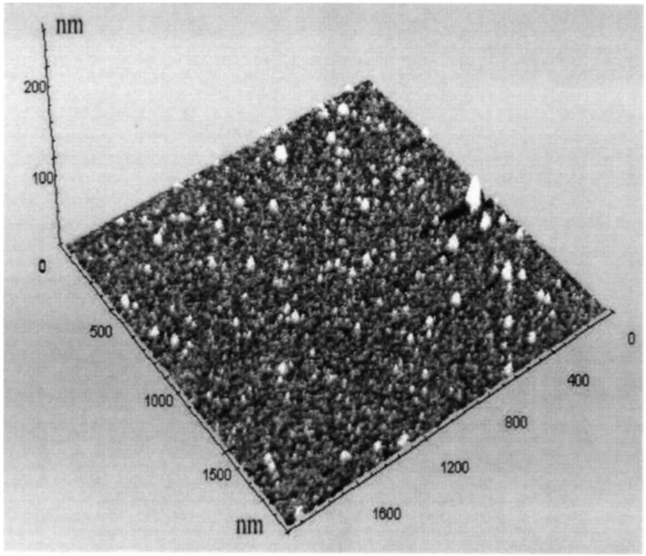

Figure 2. SPM micrograph of the surface of sample B after the ELO process.

Conclusion

We developed a novel technique to release epitaxial layers from InAs substrates. The process is applicable to any wafer size. The integrity of the epitaxial layers is conserved by the process.

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References

1. E. Yablonovitch, T. Gmitter, J. P. Harbison, and R. Bhat, *Appl. Phys. Lett.*, **51**, 2222 (1987).
2. A. Yi-Yan, W. K. Chan, C. K. Nguyen, T. J. Gmitter, R. Bhat, and J. L. Jackel, *Electron. Lett.*, **27**, 87 (1991).
3. A. Wilk, M. El Gazouli, M. El Skouri, P. Christol, P. Grech, A. N. Baranov, and A. Joullié, *Appl. Phys. Lett.*, **77**, 2298 (2000).
4. X. Y. Gong, H. Kan, T. Makino, T. Yamaguchi, T. Nakatskasa, M. Kumagawa, N. L. Rowell, A. Wang, and R. Rinfret, *Cryst. Res. Technol.*, **30**, 603 (1995).
5. Tl. Voronina, T. S. Lagunova, M. P. Mikhailova, K. D. Moiseev, and Yu. P. Yakovlev, *Semiconductors*, **30**, 523 (1996).
6. J. Fastenau, E. Özbay, G. Tuttle, and F. Laabs, *J. Electron. Mater.*, **24**, 757 (1995).
7. R. Zhu, M. C. Hargis, J. M. Woodall, and M. R. Melloch, *IEEE Photonics Technol. Lett.*, **13**, 103 (2001).
8. A. Shen, H. C. Liu, M. Gao, E. Dupont, M. Buchanan, J. Ehret, G. J. Brown, and F. A. Szmulowioz, *Appl. Phys. Lett.*, **77**, 2400 (2000).
9. R. Windisch, C. Rooman, B. Dutta, A. Knobloch, G. Borghs, G. H. Döhler, and P. Heremans, *IEEE J. Sel. Top. Quantum Electron.*, **8**, 248 (2001).
10. K. J. Weber, A. W. Blakers, and K. R. Catchpole, *Appl. Phys. A: Mater. Sci. Process.*, **69**, 195 (1999).