Epitaxial Lift-Off of Thin InAs Layers

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We describe the use of the epitaxial lift-off technique to remove thin layers of InAs from the GaAs substrates on which they were grown and subsequently bonded to glass and silicon substrates. Lift-off was accomplished by taking advantage of the high etching selectivity between AlSb and InAs in an aqueous hydrofluoric acid etching solution, allowing a thin layer of AlSb to serve a sacrificial layer to facilitate the lift-off of the InAs. The InAs layers were transferred with little measurable effect on the electrical and structural properties of the films, as evidenced by Hall effect and x-ray measurements. The technique can easily be extended to transfer more-complex GaSb/AlSb/InAs structures.

Key words: AlSb, epitaxial lift-off, GaSb, InAs

INTRODUCTION

In 1987, Yablonovitch et al. demonstrated the ability to remove thin, epitaxially grown GaAs layers from their original substrates and transfer them to other substrates, providing greater flexibility for utilization of III-V semiconductor devices.[1] Since the initial demonstration of epitaxial lift-off, the technique has been used in the fabrication of a number of hybrid devices. Of particular importance is the transferral of high-quality GaAs optoelectronic devices to silicon substrates for integration with electronic circuitry.[2,3] A form of this lift-off technology has been extended to include InP-based materials.[4]

While epitaxial lift-off provides a potentially useful extension to current GaAs and InP technology, the method might possibly play a more fundamental role in the development of the device technology of the 6.1Å semiconductors (InAs, GaSb, AlSb, and related alloys). In the epitaxial growth of the 6.1Å semiconductors, one faces the choice of either using lattice-matched, but highly conducting substrates (GaSb or InAs), or of growing the epitaxial layers on semi-insulating but highly lattice-mismatched substrates like GaAs or InP. Epitaxial lift-off would provide the means by which lattice-matched layers could be grown and then subsequently transferred to more highly insulating substrates.

In this paper, we describe the successful removal of thin InAs layers from GaAs substrates by epitaxial lift-off. Lattice-mismatched GaAs substrates were used for practical considerations of cost and availability, and their use does not affect the results of the work. It should be a relatively straightforward matter to switch to lattice-matched InAs substrates. Our technique uses thin AlSb sacrificial etch layers to free

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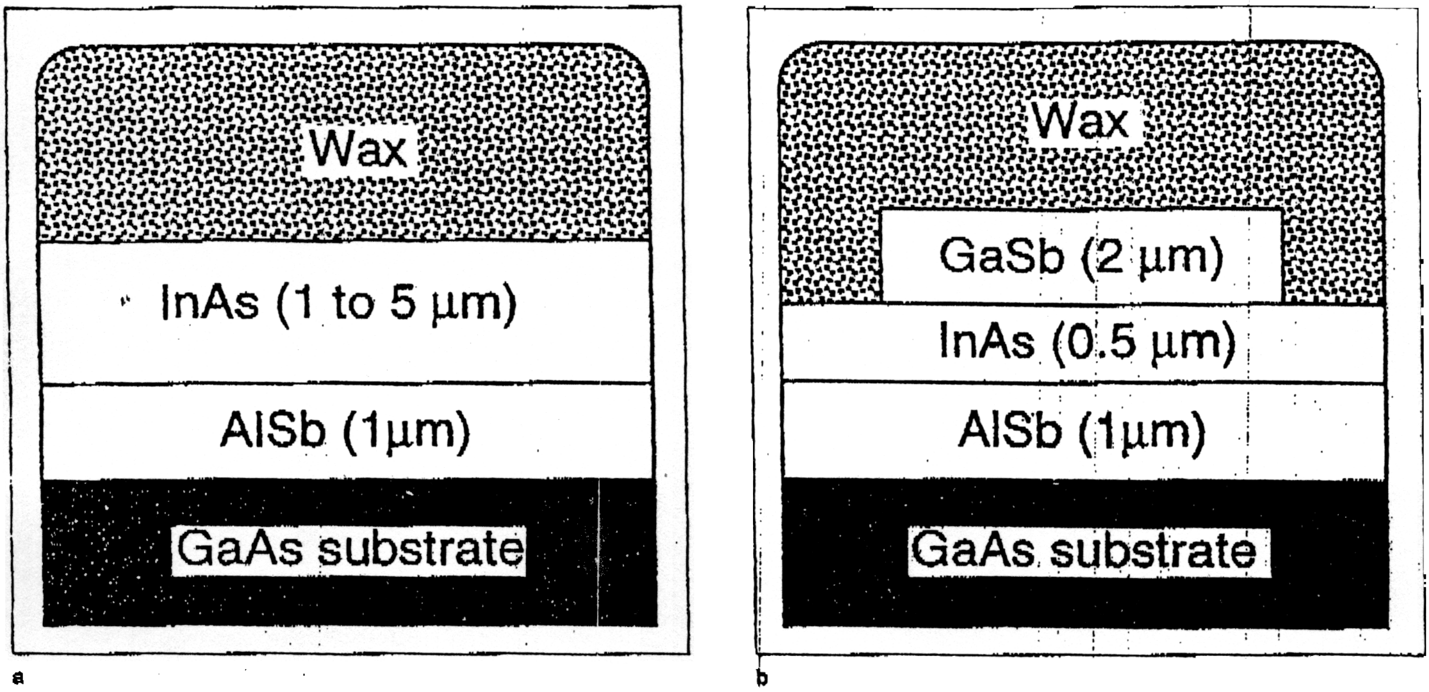

Fig. 1. Epitaxial lift-off structures with the wax as a support for the epilayers: (a) InAs, and (b) GaSb.

above-lying InAs layers, which ranged in thickness from 1 to 5 µm. The layers can be transferred with little measurable effect on the electrical and mechanical properties of the film, as evidenced by Hall effect and x-ray measurements.

LIFT-OFF PROCESSING

The critical element to the success of epitaxial lift-off is the extreme selectivity in etching rates of the InAs and AlSb epilayers in an aqueous hydrofluoric (HF) acid solution. The lift-off structure, shown in Fig. 1 and grown by molecular beam epitaxy (MBE), consists of an AlSb sacrificial layer between the substrate and the InAs lift-off layer. In HF solutions, the AlSb is completely etched without any significant etching of the InAs layer, thus freeing it from the GaAs substrate. The wax shown on top of the structure is applied separately after MBE growth and provides a support for the lift-off layers.

The process of applying the support wax (Apiezon W) followed the approach of Yablonovitch et al.[5] First, a large sample of the as-grown lift-off structure (typically one-quarter of a two-inch wafer) was rinsed in acetone, methanol, and deionized (DI) water. Then the central area of the sample was covered with 1 mm diameter chunks of the hard wax. The sample, with the wax on top, was then heated to approximately 135°C, at which point the wax melted across the sample. Surface tension prevented the wax from flowing over the sides of the wafer. Additional wax could be added to ensure total coverage to a depth of 0.5 to 1.0 mm. The resulting wax layer was smooth and uniform across the sample except at the edges where it was slightly rounded. Once the wax had cooled and hardened, the large piece was diced into smaller samples, typically on the order of 5 × 5 mm. Dicing the samples after applying the wax ensured that the AlSb sacrificial layer was exposed to the etch on the sample edges. However, if some of the edge pieces appeared to have wax on the sides that may block the etch, the wax was removed by wiping the sample with a cotton swab soaked with 1,1,1 trichloroethane (TCA).

The wax-coated samples were immersed in an H_2O:HF solution for etching. In our various trials, we used etching solution concentrations of either 20:1 or 40:1 (H_2O to HF) and the etches were always carried out at room temperature. In addition to providing a support for the InAs epilayers, the wax also aided the etching process. The wax induced a compressive stress in the lift-off layer[1] so that as the AlSb layer etched and the InAs was undercut, the edge of the InAs film curled up slightly. This curling permitted better diffusion of the reactants away from the etching zone and allowed the etch to proceed at a faster rate. The films typically lifted off after less than one full day of etching.

Once the AlSb sacrificial layer was completely etched away, the wax served as a support for the fragile InAs layer during subsequent processing. The removed layer was rinsed in DI water and then bonded to another substrate or a glass slide. Two methods of bonding were attempted: Van der Waals (VDW) bonding and gluing the layers with an adhesive. We found that the VDW bonding method, described by Yablonovitch for GaAs-based devices,[5] was generally not reliable for our samples. The InAs layers would not consistently bond to the new substrate. Typically, the center would bond but not the edges, resulting in breakage at the edges. Also, the pressure applied to induce bonding frequently cracked the InAs epilayer.

Gluing the lift-off layers to glass substrates gave better results. A general-purpose, low-viscosity, cyanoacrylate adhesive (Permabond 910) was used for this process. A glass microscope slide was rinsed in acetone, methanol, and DI water to remove any dust particles or other contaminants. The glue was spun onto the glass to give a thin, even layer, and then the InAs was positioned on top. Slight pressure was applied to ensure total contact of the InAs with the glue and glass, and after a brief drying period, the bond was set. The wax layer was then removed by soaking the sample in TCA. Once the wax dissolved, the sample was cleaned by rinsing in acetone, methanol, and DI water. The InAs layer on its new substrate was then ready for characterization and testing.

In order to take advantage of epitaxial lift-off in the 6.1Å semiconductor materials system, one must be able to remove structures more complex than simple InAs layers. These structures or devices might typically include GaSb and/or AlSb layers, which would also be etched by the HF solution used to remove the AlSb sacrificial layer. To demonstrate the wider applicability of the lift-off technique, we modified the process described above to show that more complex, mesa-etched structures could also be transferred intact. Our approach is similar to the combination of mesa etching with epitaxial transfer previously demonstrated in transferring (Al,Ga)As light-emitting devices to silicon[5] and glass[6] substrates. In order to test this modified process, a new structure was grown, consisting of a 0.5 μm InAs etch-stop layer between the 1. 0 μm AlSb sacrificial layer and a 2.0 μm GaSb lift-off layer. Square 5 × 5 mm mesas were formed in the GaSb using standard photolithography and a solution of 2:1:20 HF:H_2O_2:H_2O to etch the GaSb down to the InAs layer. The locations of the mesas were marked on the back of the wafer, and then a flat layer of wax was applied to the face of the wafer as described above. Using the marked mesa locations, the wafer was diced into 6 × 6 mm chips so that a 0.5 mm wide InAs border region surrounded the GaSb mesa. The dicing also ensured the exposure of the edges of the AlSb sacrificial layer. The whole structure was placed into the dilute HF solution to etch away the AlSb layer. The top-side wax and underlying InAs etch-stop layer protected the GaSb from the HF solution. Using this slightly modified technique, we were able to transfer composite GaSb/InAs structures to glass slides. The technique can easily be adapted for transferring more complex structures like resonant-tunneling diodes or heterostructure field effect transistors.

CHARACTERIZATION

The actual mechanics of epitaxial lift-off simply take advantage of the etching selectivity between the sacrificial layer and the desired lift-off layer. However, in order for this process to be useful, the characteristics of the layer or device removed must not degrade or change during the transfer. The first and simplest test of the lift-off layers on their new substrates was a visual examination. Using a microscope at 300x magnification to compare lift-off samples to ones on original GaAs substrates, it was impossible to tell the difference between the two. No cracks or other damage were visible in the lift-off samples, indicating that no gross mechanical damage resulted during the transfer process.

Visual examination was an easy way to determine initial success, since a damaged expitaxial layer is not worth further processing and testing, but more importantly the electrical properties of the layers had to be tested. We found that there was very little change in the low-field transport properties of InAs layers during the transfer. The mobility and carrier concentration of two InAs films were measured at room temperature using van der Pauw techniques. The 2

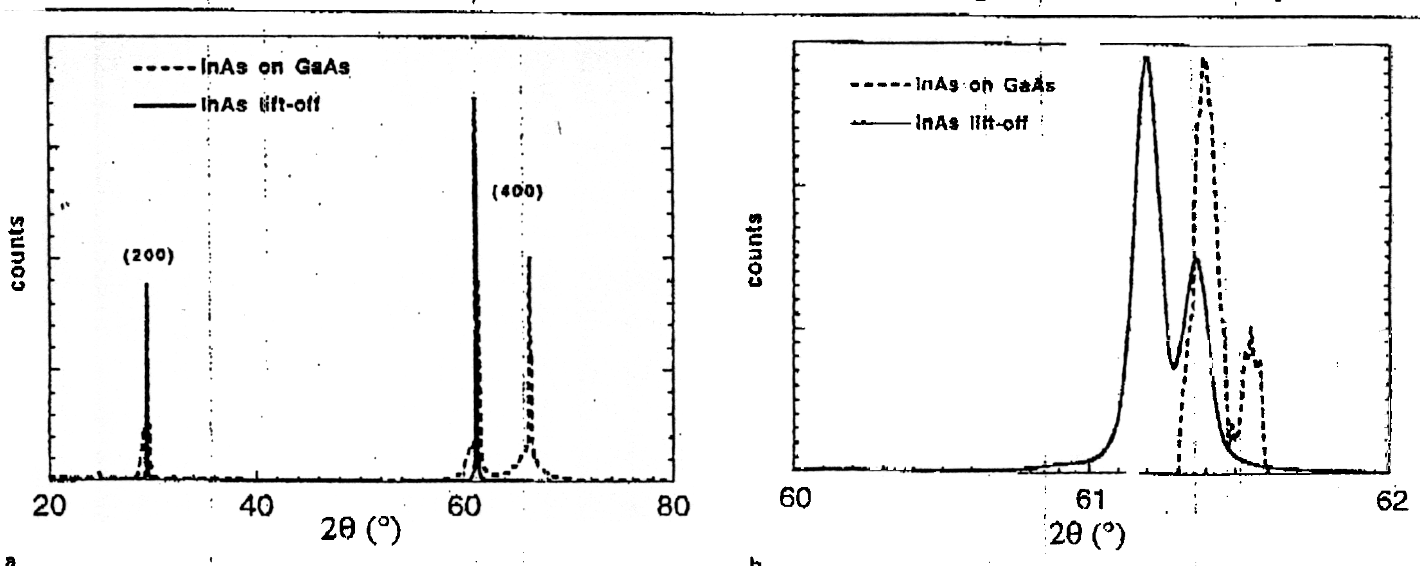

Fig. 2. (a) Comparison of x-ray θ-2θ scans for 2 μm InAs layers on the GaAs substrate and after lift-off on a glass substrate. The peak at 63 degrees comes from the GaAs substrate, and (b) a detailed measurement of the x-ray diffraction intensities around the location of the (400) plane for 2 μm InAs samples on the GaAs substrate and after lift-off on a glass substrate. The double peaks for each sample come from the $K\alpha_1$ and $K\alpha_2$ peaks from the copper x-ray source.

μm thick films were initially from the same substrate, but one had subsequently been transferred to a glass slide. Measured mobilities were 11000 $cm^2/V\cdot s$ for the transferred sample and 10500 $cm^2/V\cdot s$ for the InAs layer still on the GaAs substrate. Corresponding electron concentrations were 1.76×10^{16} cm^{-3} and 1.74×10^{16} cm^{-3}, respectively.

Measurements of the x-ray characteristics of thin InAs films were performed using a Phillips diffractometer with a copper x-ray source. Figure 2a shows wide-angle θ-2θ scans for an InAs layer on the original GaAs substrate and a layer that had been transferred to a glass slide. There is very little difference in the two except for the absence of a GaAs peak for the transferred sample. An expanded plot of the (400) reflections, shown in Fig. 2b, shows some subtle differences. The peak from the transferred sample was shifted to lower angles by 0.2 degrees in comparison to the peak from the as-grown sample. We attribute this shift to relaxation of residual strain in the InAs epitaxial layer after lift-off. The shift represents a 0.018Å increase in the InAs lattice constant that is normal to the interfaces. Through the Poisson effect, this implies that the in-plane lattice constant decreased slightly. The direction of the changes suggests that the underlying AlSb layer has a stronger influence on residual strain in the InAs epilayers than the severely lattice-mismatched GaAs substrate. If the underlying substrate had been the primary cause of the strain, the shift in the lattice constants would have been in the opposite direction. In any case, the strain relaxation is not a large effect.

CONCLUSIONS

The extension of epitaxial lift-off to the 6.1Å semiconductor materials has been demonstrated in this work. Thin layers, 1 to 5 μm, of InAs have been successfully removed from their original GaAs substrates and bonded to glass and silicon substrates. The technique has also been slightly modified to remove layers of GaSb and AlSb, which will allow lift-off to be applied to various devices grown in this material system. Lift-off was accomplished by taking advantage of the high etching selectivity of AlSb over InAs in an HF etching solution. AlSb sacrificial layers allowed lift-off to be applied to lattice-matched materials such as InAs or GaSb. Electrical and x-ray measurements demonstrate that the physical characteristics of the epilayers are unchanged by the lift-off process.

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