An investigation of the growth of \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As} \) layers on \( \text{InP} \) by liquid phase epitaxy

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Abstract. Liquid phase epitaxial growth of lattice-matched \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As} \) layers on \( \text{InP} \) substrates is investigated with particular emphasis on the role of interface defects on layer quality. By differential Hall measurements it is established that a bad interface, resulting from the thermal decomposition of \( \text{InP} \) substrate prior to growth, degrades the electron mobility in all parts of the layer and the effect is most pronounced at regions close to the interface. However layers with much better physical and electrical characteristics are grown following steps to ensure substrate surfaces free from any thermal damage.

Keywords. Epitaxy; compound semiconductors; interface studies.

1. Introduction

\( \text{In}_{0.53}\text{Ga}_{0.47}\text{As} \) is an important material for fabricating microwave and optoelectronic devices. Low background impurity concentration as well as high electron mobility values are essential requirements for such devices. The liquid phase epitaxy technique (Nakajima 1985) is widely used to grow \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As} \) layers on \( \text{InP} \) substrates with properties suitable for the above applications. Different variations of the growth procedure have been used (Oliver and Eastman 1980; Bhattacharya 1981; Cook et al 1982; Kuphal and Pocker 1982) to achieve room temperature carrier concentrations in undoped layers around \( 5 \times 10^{14} \text{ cm}^{-3} \). It is generally believed (Penna et al 1984) that use of high purity starting materials followed by extensive baking of the growth melt and the etch melt are quite effective in reducing the unwanted impurity level in the grown layers.

Thermal decomposition of the substrate and of the layer is another factor that is known (Keramidas et al 1980; Mahajan et al 1982) to prevent growth of defect-free epi-layers and causes serious wetting problems (Logan et al 1983) during LPE regrowth. This effect is however reduced by maintaining an overpressure of phosphorus over the substrate during the whole growth cycle (Keramidas et al 1980, 1982; Beneking et al 1981; Logan 1987). Even then, some thermal damage to the \( \text{InP} \) substrate occurs which is removed by a shallow In etch just prior to growth. Reduction of impurities in the layer and improvement of the interface are both taken to be prime requirements for growing LPE layers with good electrical and structural qualities. But for improving the electrical qualities alone, it is necessary to assess the individual contributions of the above two process steps in controlling the resultant mobility and the carrier concentration in a grown layer.

We have grown \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As} \) layers on \( \text{InP} \) substrates by the conventional LPE technique, using methods to minimize both the unwanted impurities in the layer and the thermally generated defects at the interface. Using the differential Hall technique, we looked at the actual distribution of the mobility and the carrier concentration values along the growth direction of the layer starting from the interface. Our study revealed that the properties of the layer–substrate interface
play substantial roles in controlling the electrical qualities of In$_{0.53}$Ga$_{0.47}$As layers, other than their structural properties.

2. Experimental

All the starting materials namely In, InAs and GaAs were of 6N grade. They were cleaned in the usual manner and weighed in a balance having a resolution of 10 µg to prepare the composition for growing lattice-matched layers on InP. Indium for the etch-back melt and that for the growth melt were initially baked in pure H$_2$ at 740°C for 20 h. This was followed by adding InAs and GaAs to form the growth melt and baking the whole thing again at 680°C for another 24 h. During this second bake, the growth melt was covered by a graphite cap to prevent loss of arsenic. The liquidus temperature of the growth composition was typically 650°C and 3–4°C of supercooling was used before initiating the growth. To maintain supersaturation of the growth melt, a 0.15°C/min cooling ramp was used during growth. Epitaxial growth was usually carried out for 10 min resulting in a layer of 8–10 µm thickness. The phosphorus overpressure required to suppress the thermal decomposition of InP substrates has been reported to be provided by InP proximity caps (Keramidas et al 1980), by passing PH$_3$ gas through the reactor (Beneking et al 1980) or by using a substrate cap containing Sn–InP solution as a source of excess phosphorus (Keramidas et al 1982; Logan 1987). We have used the third technique but instead of an Sn–InP solution, which introduces Sn as another source of contamination, we used a number of small InP flakes as the source of phosphorus. The substrate cap so designed is shown in the sketch of figure 1. Since the effective surface area of the InP flakes is quite large and the space between the substrate surface and the bottom of the cap is kept quite narrow, sufficient phosphorus pressure could be developed and the layers grown were almost featureless as seen under a Nomarski Interface Contrast (NIC) Microscope.

LPE growth was carried out under three conditions. In one case, the substrate cap was not introduced and a deep In etch (6–7 s) was used to remove the thermally damaged layers. In the second case, growth was carried out under the substrate cap but without any In etch-back step. In the third case, both substrate cap and a shallow (about 3 s) In etch was used. The same growth melt was used in these three

![Figure 1. Cross-sectional diagram of the modified substrate cap.](image-url)
Growth of \textit{In}_{0.53}\textit{Ga}_{0.47}\textit{As} layers on \textit{InP} by liquid phase epitaxy

runs and other growth parameters were kept the same. The layers were first checked for physical perfections using an NIC microscope and their electrical properties were evaluated using conventional Van Der Pauw, Hall and differential Hall techniques.

3. Results and discussion

The average values of Hall mobility obtained in the above three cases are presented in table 1. It is clear that the absence of the substrate cap in the first case had a very deleterious effect on the mobility of the layers and even with the substrate cap, a shallow etching of the substrate was necessary to get a respectable value of the mobility. This result corroborates with the physical observations of the surfaces which show rapid decrease in the number of thermal pits from case 1 to case 2, the pits being almost absent in case 3. The importance of having a damage-free substrate surface using both phosphorus overpressure and in-situ etch is more evident from the results of the differential Hall measurements in figure 2, carried out on the layers grown under the second and third cases. Successive layer removal in this measurement was done with an etchant composed of 1 part of HF, 1 part of H$_2$O$_2$ (30%) and 10 parts of H$_2$O which has an etching rate of 0.63 \( \mu \text{m/min} \). For the layer grown with both substrate cap and In etch, the mobility is above 8000 cm$^2$/V.s in most parts along the thickness. The mobility however dropped both at the surface and at the interface. For the layer grown without any In etch to remove the top damaged layer of the substrate, the degradation of mobility at the

<table>
<thead>
<tr>
<th>Sample</th>
<th>Grown under</th>
<th>( \mu_h ) (cm$^2$/V.s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No substrate cap, in-situ In etch</td>
<td>6000</td>
</tr>
<tr>
<td>2</td>
<td>Substrate cap, no In etch</td>
<td>6500</td>
</tr>
<tr>
<td>3</td>
<td>Substrate cap and in-situ In etch</td>
<td>7400</td>
</tr>
</tbody>
</table>

![Table 1](image)

**Figure 2.** Profiles of Hall mobility (\( \mu_h \)) and carrier concentration (\( n \)) along the growth directions of \textit{In}_{0.53}\textit{Ga}_{0.47}\textit{As} layers grown under two different conditions.
interface is much more prominent. The overall value of mobility is also lower in this case and this is as expected since substrate defects are very easily transported through the grown layers. In both cases, very poor mobility is observed in a narrow region near the surface. We believe that this is due to the loss of As and subsequent disordering of the layer surface during cool-down of the furnace. The carrier concentration is plotted for the better layer and it is more or less uniform.

We conclude that both the presence of impurities in the layer and the interface damages play significant roles in the electrical properties of LPE grown In$_{0.53}$Ga$_{0.47}$As layers. The degraded surface region is quite shallow and a short etch is sufficient to remove it. However this region may become important if another epi-layer is subsequently regrown over the already grown layer.

4. Conclusion

Good quality In$_{0.53}$Ga$_{0.47}$As layers were grown on InP by LPE using prolonged baking schemes to reduce impurities and using phosphorus overpressure over the substrate to improve interface qualities. By profiling mobility values along the growth direction, it is shown that the interface has a very prominent role in controlling the overall electrical quality of a layer. A poorly processed substrate surface results in severe degradation of mobility at the interface and also at other parts of the layer. Thus care must be taken in LPE growth, not only to prevent impurities from getting into the layer but also to protect the substrate surface from thermal and other damages in order to ensure a defect-free interface.

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