Thermal stability of strained $\ln_x Ga_{1-x} As / \ln_v Al_{1-y} As / \ln P$ heterostructures

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We investigated the thermal stability of strained layers of InGaAs and InAlAs on InP. Epilayer and interface quality was assessed by high-resolution x-ray diffraction and electron mobility measurements as a function of annealing cycle. Both techniques show that pseudomorphic heterostructures retain their high crystalline quality at annealing temperatures of up to 700– 800 °C, despite exceeding the Matthews–Blakeslee [J. Cryst. Growth 27, 118 (1974)] critical layer thickness by as much as a factor of 4–8. On the other hand, layers which are partially relaxed (incoherent) as-grown relax further during annealing. These findings demonstrate that layers which are beyond the predicted critical thickness, but coherently strained after growth, are stable to normal device processing and operating temperatures and hence may be suitable for use in device heterostructures.

Semiconductor alloys of $In_xGa_{1-x}As$ and $In_yAl_{1-y}As$ are of interest for both optical and electronic devices. The In_{0.53}Ga_{0.47}As and In_{0.52}Al_{0.48}As alloys have been applied extensively because they can be grown lattice matched to InP substrates. The use of mismatched epitaxial layers, however, allows much greater freedom to design heterostructure devices with desired optical and electronic properties.¹ If an epitaxial layer is sufficiently thick and mismatched, the formation of misfit dislocations at the substrate/epilayer interface is energetically favored. These dislocations relieve strain, allowing the distorted epilayer lattice to relax back toward its natural cubic symmetry. However, lattice relaxation via misfit dislocations generally degrades the structural, electrical, and optical quality of epitaxial layers, often making them unsuitable for device applications.

The point at which misfit dislocations begin to form is known as the *critical layer thickness*. Although several theories have been proposed to predict the critical layer thickness, most device designers rely upon the model of Matthews and Blakeslee (MB).² Recent experimental work has shown, however, that high-quality InGaAs/InP and InAlAs/InP heterostructures³⁻⁵ and devices^{6,7} can be grown beyond the MB limit, $t_{c,MB}$. Such results are often attributed to metastability.

A potential problem with the use of metastable layers in devices is that layers may relax during high-temperature processing steps or device operation. Experimental work on thermal stability in the InAlAs/InGaAs/InP materials system is limited to studies of lattice-matched heterostructures.^{8–10} Stability studies of strained layers have been reported for the InGaAs/GaAs system, but the results are contradictory.^{11–13} For example, Peercy *et al.*¹¹ annealed AlGaAs/InGaAs/GaAs heterostructures in which the InGaAs layer exceeded $t_{c,MB}$. They observed a dramatic decrease in photoluminescence intensity after the anneal, suggesting lattice relaxation. A similar photoluminescence experiment by Bertolet *et al.*¹² yielded the opposite results: strained InGaAs layers were thermally stable, despite exceeding $t_{c,MB}$. To our knowledge, there are no reports on the thermal stability of mismatched $In_xGa_{1-x}As/In_yAl_{1-y}As/InP$ heterostructures. This letter addresses that gap.

For this study, epitaxial layers of $In_xGa_{1-x}As$ and $In_yAl_{1-y}As$ were grown on semi-insulating (001) InP substrates by molecular beam epitaxy (MBE). The substrate temperature, measured by a thermocouple and optical pyrometer, was 460–510 °C. The V:III beam-equivalentpressure ratio was between 15:1 and 25:1. Growth rates ranged from 0.6 to 0.9 μ m/h. Samples were annealed in an AG Associates Heatpulse 210 system with a nitrogen ambient. Samples were placed facedown on a GaAs wafer during the anneal to minimize thermal decomposition.¹⁴ The temperature was measured by a thermocouple attached to a Si wafer and is believed to be accurate to within 10 °C.

The samples were characterized by high-resolution x-ray diffraction (HRXRD) to determine layer composition, strain, and crystalline quality. The rocking curves were measured with Cu $K\alpha$ radiation and an InP first crystal oriented for the (004) reflection. Electron mobility and density were measured as a function of annealing cycle by the van der Pauw technique at 77 and 300 K.

As a reference, we first studied a nearly latticematched $In_{0.537}Ga_{0.463}As$ layer, sample 1331, in a sequential annealing experiment. The layer thickness is 4000 Å, about one-half of $t_{c,MB}$. Hence, we do not expect misfit dislocations to form during growth or annealing. HRXRD scans were taken after each anneal. The as-grown (004) scan, shown in Fig. 1, reveals an InGaAs peak width approximately equal (within 10%) to the theoretical value predicted from simulations.⁴ Pendellosung (interference) fringes are also present. Both of these facts indicate high crystalline quality in the epilayer. After 60-s anneals up to 850 °C, the rocking curve is unchanged except for a decrease in fringe intensity on the low-angle side which we attribute to interdiffusion at the InGaAs/InP interface.

We also measured mismatched layers which relaxed substantially during growth. Annealing such layers causes additional relaxation, with the parallel lattice mismatch increasing and the perpendicular mismatch decreasing.

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FIG. 1. HRXRD scans of a nearly lattice-matched reference InGaAs sample as a function of annealing cycle. The peaks at 0 and -80 arcsec correspond to the InP substrate and InGaAs epilayer, respectively.

The result is a decrease in the epilayer-substrate separation in (004) HRXRD scans. For example, a 1.0 μ m layer of In_{0.61}Ga_{0.39}As on InP ($t/t_{c,MB} = 33$) was 52% relaxed asgrown and exhibited poor crystalline quality, with the HRXRD peak width exceeding the theoretical value by a factor 20. After a 60 s anneal at 850 °C, the layer relaxation increased to 67%.

Most advice applications require pseudomorphic (coherently strained) layers of high crystalline quality. We now focus on the thermal stability of pseudomorphic layers beyond the Matthews–Blakeslee limit. In Fig. 2, we show the effects of annealing on sample 1879 which contained a 3000 Å layer of $In_{0.477}Ga_{0.523}As$. The InGaAs layer is in tension, with $t/t_{c,MB} = 7.3$. The only effect of 60 s anneals up to 850 °C is a slight loss in fringe intensity, similar to 1331, the reference. HRXRD should be able to detect relaxation of less than 5% for this sample. We conclude that the strained InGaAs layer is not relaxing appreciably during the anneals.

We also used HRXRD to examine the structural stability of InGaAs in compression. In this case, the pseudo-



FIG. 2. HRXRD scans of sample 1879 before and after annealing. The sample contains a pseudomorphic InGaAs layer with $t/t_{cMB} = 7.3$.



FIG. 3. HRXRD scans of a MODFET heterostructure (inset) before and after annealing. The pseudomorphic InGaAs channel exceeds $t_{c,MB}$ by a factor of 2.6.

morphic InGaAs layer was the channel of a modulationdoped field-effect transistor (MODFET). The cross section of MODFET 4089 is shown as an inset in Fig. 3. All the layers are nominally lattice matched to the InP substrate except the 500 Å $In_{0.64}Ga_{0.36}As$ channel, with $t/t_{c,MB} = 2.6$. HRXRD scans for as-grown and after a 60 s anneal at 800 °C are shown in Fig. 3. The crystalline quality is essentially unchanged by annealing, demonstrating the thermal stability of this device structure. We also examined an 1800 Å pseudomorphic layer of $In_{0.44}Al_{0.56}As$ $(t/t_{c,MB} = 8.6)$ with HRXRD and found it to be thermally stable to 850 °C.⁵

To obtain a measure of thermal stability which is more sensitive than HRXRD, we measured the electron mobility of pseudomorphic MODFET heterostructures before and after annealing. The mobility of modulation-doped structures is a strong function of layer and interface quality, particularly at low temperatures. Hence, lattice relaxation via misfit dislocations is expected to severely degrade electron mobility.^{3,6}

In addition to sample 4089, we examined MODFETs 4168 with an InAs mole fraction in the channel, x, of 0.53 (lattice matched) and 4084 with x=0.68 (t/t_{cMB} = 3.8). Except for the channel composition, these samples were identical to 4089 (Fig. 3 inset). In Fig. 4, we show the electron mobility at 300 and 77 K as a function of annealing cycle. We also plot the 77 K sheet carrier concentration; the 300 K values were similar. The 300 K mobility remains between 8000 and 11 000 cm²/Vs for all three samples for anneals ranging from 5 s at 700 °C to 60 s at 800 °C. The small changes in mobility apparently result from as-grown water nonuniformities. For all three samples, the 77 K electron mobility remained between 40 000 and 50 000 cm²/Vs after 700 °C anneals, but decreased after anneals at 800 °C. This decrease in mobility is accompanied by a decrease in carrier density.

The fact that even the lattice-matched sample experiences a drop in low-temperature mobility after an 800 °C anneal suggests that strained layer relaxation is not respon-



FIG. 4. Electron mobility at 300 K (a) and 77 K (b), and sheet carrier concentration at 77 K (c) for MODFETs as a function of annealing cycle. x is the InAs mole fraction in the 500 Å $In_xGa_{1-x}As$ channel.

sible. Instead, interdiffusion of In, Ga, and Al across the InGaAs/InAlAs interface could reduce the interface abruptness and degrade the mobility. In addition, Si dopant atoms might diffuse toward the channel and contribute to ionized impurity scattering. The loss in carrier density could be due to the amphoteric nature of Si in III-Vs, with additional Si atoms occupying group V sites after annealing.¹⁵ An alternative explanation, supported by recent work on lattice matched InGaAs/InAlAs/InP structures,¹⁰ is that Si donor atoms diffuse toward the surface, reducing the charge transfer into the channel.

 $In_xGa_{1-x}As/In_yAl_{1-y}As/InP$ heterostructures exceeding $t_{c,MB}$ by as much as a factor of 4–8 are thermally stable. We attribute the stability to two factors. First, at the temperatures of interest, there is insufficient energy for the homogeneous nucleation of dislocation half-loops.^{5,16} Second, due to the high quality of the substrate and MBE lavers, there is an insufficient number of sites, such as threading dislocations in the substrate, or impurity clusters or oval defects in the epilayers, for heterogeneous nucleation of dislocations to take place. Hence, the thermal stability of pseudomorphic layers may be a function of not only lattice mismatch and layer thickness but also substrate preparation and the growth system. Variations in the density of heterogeneous nucleation sites can explain the apparent discrepancies in the literature.^{11,12} In the case of samples which are partially relaxed as-grown, the severe lattice mismatch results in roughness and possibly three-dimensional growth. Associated defects (e.g., stacking faults) could serve as a source of nucleation sites for misfit dislocations during growth and annealing.

In summary, we have found that pseudomorphic $In_xGa_{1-x}As/In_yAl_{1-y}As/InP$ heterostructures are thermally stable at annealing temperatures of up to 700–800 °C, well above typical device processing or operation temperatures. This is important for advanced device design because the parameter space available for layer composition is considerably wider than generally recognized.

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The key finding of our work is that pseudomorphic

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