Molecular-beam epitaxial growth of high-quality InSb on InP and GaAs substrates

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Epitaxial layers of InSb have been grown in InP and GaAs substrates by molecular-beam epitaxy. The dependence of the epilayer quality on flux ratio, J_{Sb_4}/J_{In}, was studied. Deviation from an optimum value of J_{Sb_4}/J_{In} (~ 2) during growth led to deterioration in the surface morphology and the electrical and crystalline qualities of the films. Room-temperature electron mobilities as high as 70 000 and 53 000 cm^2/V s were measured in InSb layers grown on InP and GaAs substrates, respectively. Unlike the previous results, the conductivity in these films is n type even at $T = 13$ K, and no degradation of the electron mobility due to the high density of dislocations was observed. The measured electron mobilities (and carrier concentrations) at 77 K in InSb layers grown on InP and GaAs substrates are 110 000 cm^2/V s (3×10^{15} cm^{-3}) and 55 000 cm^2/V s (4.95×10^{15} cm^{-3}), respectively, suggesting their application to electronic devices at cryogenic temperatures.

I. INTRODUCTION

The smallest band-gap III-V materials, InSb and the $InAs_xSb_{1-x}$ alloys, have received a great deal of attention due to their potential application in far-infrared sources and detectors.[1] Kurtz *et al.*[2] demonstrated a feasibility of Osbourn's proposed idea by making a photodiode consisting of a *p-n* junction embedded in a $InAs_{0.09}Sb_{0.91}$/InSb strained-layer superlattice, exhibited photoresponse out to a wavelength of 8.7 μm at 77 K. The expected superior transport properties might eventually prove useful for low-temperature electronic devices also.

In order to study the electrical properties of these materials, as well as to explore their suitability for integration with devices based on various different semiconductors, the heteroepitaxial growth of these materials on wide-band-gap semi-insulating substrates is necessary. Limited studies have been reported on heteroepitaxial growth of InSb using MBE. Chyi *et al.*[3,4] have reported room-temperature electron mobilities as high as 57 000 cm^2/V s, measured in an unintentionally doped InSb layer about 5 μm thick grown on GaAs and Si substrates, where the mobility decreases as the temperature decreases. Williams *et al.*[5] reported an electron mobility of 66 000 cm^2/V s at 290 K in 10-μm-thick InSb film on GaAs substrates where the carrier type changes to *p* type, as the temperature is lowered, as a result of freeze-out behavior. These observed results have generally been attributed to the large number of dislocations in the films due to a large lattice mismatch.[3-5] However, it is important to grow high-quality InSb epilayers without any degradations at low temperatures since most applications of low band-gap semiconductors such as InSb are envisaged to be at cryogenic temperature.

In this paper we report on our studies on the heteroepitaxial growth of undoped InSb layers on InP ($\sim 11\%$ mismatch) and GaAs ($\sim 14\%$ mismatch) substrates. We have not used any special techniques to control the propagation of dislocations generated by the lattice mismatch, such as the incorporation of a strained-layer superlattice buffer, use of a misoriented substrate, or *in situ* and *ex situ* annealing.

II. MOLECULAR-BEAM EPITAXIAL GROWTH

Heteroepitaxial InSb layers were grown on undoped GaAs and Fe-doped InP (100) substrates in a RIBER 2300P MBE system. Arsenic and Sb fluxes, generated from elemental sources, were in the form of tetramers (As_4 and Sb_4). After the oxide desorption under an As_4 overpressure, the growth of InSb was initiated at substrate temperatures ranging from 350 to 400 °C, as measured by an infrared pyrometer. The growth rate was 1 μm/h, and the flux ratio J_{Sb_4}/J_{In} was varied in the range of 0.3–2.5. Hall measurements were performed on van der Pauw samples formed on the unintentionally doped InSb layers to assess their electrical properties. The mesa patterns were formed by photolithography and mesa etching in a 5:1 solution of lactic and nitric acids.

The observed spotty nature of the reflection high-energy electron diffraction (RHEED) pattern along the [110] azimuth at the initiation of the growth indicated three-dimensional growth due to a large lattice mismatch between the film and the substrate. After growth of ~ 200 Å, the spots changed into streaks with a (1×3) reconstruction, indicating continuous coverage and smoothing of the growth front.

III. RESULTS AND DISCUSSION

Figure 1 shows the surface morphologies of InSb films (1 μm thick) grown on (100) InP substrates with different partial pressure ratios P_{Sb_4}/P_{In}, as observed under a Nomarski phase contrast microscope. Specular surfaces are obtained in the sample grown with a partial pressure ratio of ~ 6 [Fig. 1(a)]. With excess Sb_4, a slightly hazy surface is obtained [Fig. 1(b)]. On the other hand, lack of Sb_4 leads to a rough surface [Fig. 1(c)] or the formation of In droplets on the surface [Fig. 1(d)]. Measured room-temperature Hall mobilities in these samples are shown in Fig. 2 as a function of the partial pressure ratio. All the samples are *n* type and the highest electron mobility can be observed in the sample grown with a P_{Sb_4}/P_{In} ratio of ~ 6 and a correspond-

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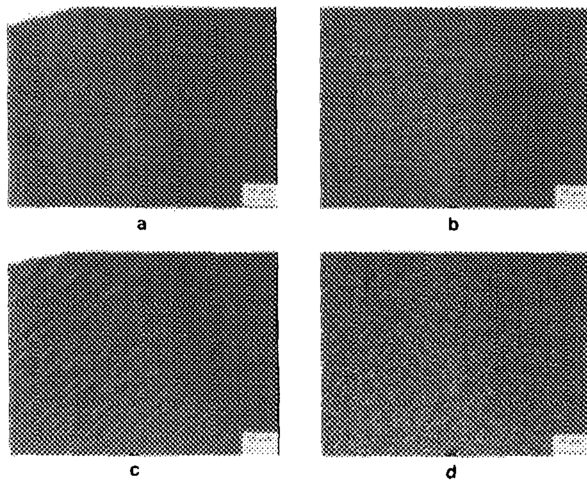

FIG. 1. Surface morphologies of 1-μm MBE InSb films grown on InP at 380 °C at a rate of 1 μm/h for various values of the partial pressure ratio P_{Sb_4}/P_{In}: (a) 6, (b) 10, (c) 4, and (d) 2.

ing beam flux ratio J_{Sb_4}/J_{In} of ~ 2. This suggests that the sticking coefficient of tetramer Sb_4 source at the growth temperature of 375 °C is ~ 0.5.[6] It may be noted that according to the growth model of GaAs using a tetramer source, the maximum sticking coefficient for As_4 on GaAs is also 0.5. The above observations suggest that the flux ratio, J_{Sb_4}/J_{In}, plays a crucial role in determining the surface morphology and the electron mobility in heteroepitaxial InSb films due to the high sticking coefficient of Sb_4 molecules at the growth temperature used. Similar results were obtained in the InSb films grown on (100) GaAs substrates.

In order to investigate the microcrystalline quality of these films, we have made x-ray diffraction measurements using a conventional x-ray diffractometer with Cu$K\alpha_1$ and $K\alpha_2$ lines. It is found that there exists an optimum value of J_{Sb_4}/J_{In} flux ratio (~ 2) where the x-ray linewidth becomes minimum, and the intensity becomes more than that of the substrate, as shown in Fig. 3. Deviation from this optimum point leads to an increase in x-ray linewidth, indicating a deterioration in the crystal quality.

Shown in Fig. 4(a) are the temperature-dependent Hall mobilities measured in a 10-μm-thick unintentionally doped InSb layer grown on InP under the optimum growth condition discussed earlier. The measured mobilities are 70 000 and 110 000 cm^2/V s at 290 and 77 K, respectively. It may be noted that the electron mobility steadily increases as the temperature is lowered from 300 to 77 K. The electron mobility in this temperature range in bulk InSb is predominantly limited by electron-hole scattering due to the very heavy hole mass (~ 30 times the electron mass).[7] Below 100 K, it is evident that the mobility is very weakly limited by impurity scattering because of the small electron mass. The behavior of the carrier concentration as a function of temperature [Fig. 4(b)] is characteristic of n-type materials, and the increase in n_H above 200 K indicates the onset of intrinsic conduction. The carrier concentration measured below 200 K implies an ionized donor concentration of approximately 3×10^{15} cm^{-3}. Measured 300- and 77-K Hall mobilities and (carrier concentrations) of undoped 2.5-μm-thick InSb layer grown on GaAs substrate with a flux ratio of ~ 2 are 53 000 cm^2/V s ($n_{300\,K} = 1.75\times 10^{16}$ cm^{-3}) and 55 000 cm^2/V s ($n_{77\,K} = 4.95\times 10^{15}$ cm^{-3}), respectively. These values are somewhat lower than those obtained for the 10 μm-thick InSb grown on InP, probably due to the improved quality of epilayer away from the InSb/substrate interface. It must be noted that the electron mobilities of InSb layers grown on both GaAs and InP substrates at 77 K are much

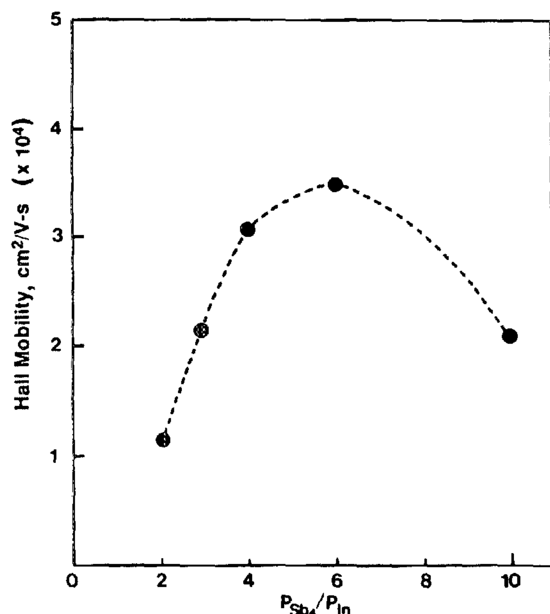

FIG. 2. Variation of measured Hall mobilities with P_{Sb_4}/P_{In} in 1-μm InSb films grown on InP at 380 °C and at 1 μm/h. The dashed line is a join of the data points.

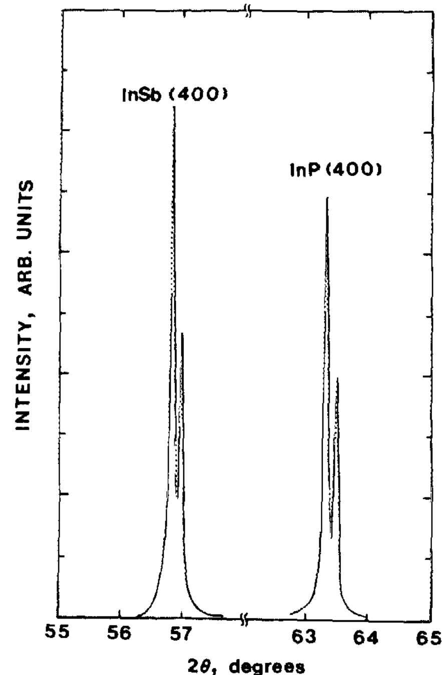

FIG. 3. X-ray diffraction data of 1.5-μm InSb grown on InP with $P_{Sb_4}/P_{In} = 6$. Also shown is the diffraction from the InP substrate.

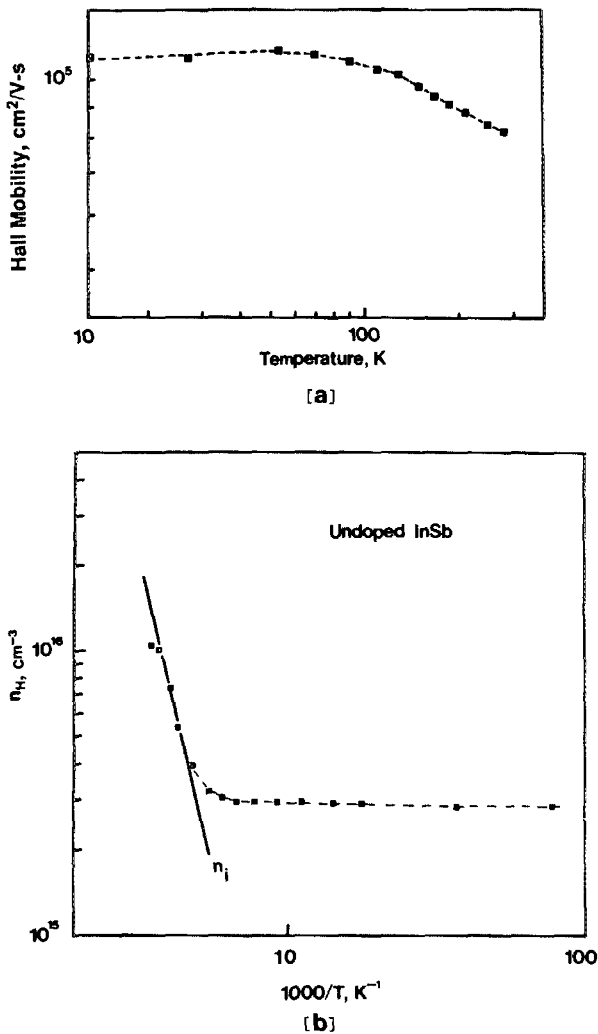

FIG. 4. Measured temperature-dependent Hall data showing (a) electron mobility, and (b) carrier concentration in a 10-μm InSb layer grown on InP. The intrinsic carrier concentration profile is also indicated in (b).

higher than those reported previously.[3–5] It must also be noted that in our samples any special techniques have not been applied to control dislocations generated by the lattice mismatch, implying that improved electrical properties of our epilayers are simply related to the bulk properties of the epilayer, such as lower autocompensation and better stoichiometry control, rather than to the heterointerface and its related effects to accommodate the lattice mismatch between the two materials. However, these values are still much lower than that from the best homoepitaxial film with the impurity concentration of 1×10^{14} cm^{-3} ($\mu_{77\,\mathrm{K}} = 1\,100\,000$ cm^2/V s).

The electrical properties of Si-doped InSb grown on GaAs (100), (311)A, and (311)B substrates were measured to determine the effects of impurity incorporation and compensation. Three substrates with (100), (311)A, and (311)B orientations were mounted alongside on the molybdenum block. The growth temperature ($T_g = 380\,^\circ$C), growth rate (1 μm/h), and flux ratio ($J_{\mathrm{Sb}}/J_{\mathrm{In}} = 2$) were held constant, while the silicon source temperature (T_{Si}) was adjusted in the range 800–950 °C to change the Si-doping level in each growth run. Carrier concentration determined from Hall measurements show that intentionally Si-doped InSb layers are n type at all doping levels. Figure 5 shows the electron concentration in (100), (311)A, and (311)B-oriented layers as a function of silicon source temperature. As can seen in this figure, the electron concentration in (100) and (311)B layers is approximately the same, but that in (311)A layers is 2–3 times lower, suggesting that there is a higher compensation of Si dopants in (311)A-oriented layers. This feature is similar to previous observation[8] on Si incorporation for different orientations of GaAs, except the fact that the conduction type of Si-doped (311)A GaAs layers was p type at all doping levels. Our results suggest that most of the Si atoms get incorporated in In sites rather than Sb sites even though the dangling bond configuration in the (311)A orientation favors Si incorporation in the Sb sites. A possible explanation is that in our MBE-grown InSb layers the growth parameters favor preferential Si incorporation on In sites.

Representative experimental mobility versus carrier concentration data of MBE-grown InSb layers on (100) GaAs substrates measured at 300 K are shown in Fig. 6, together with the calculated solid curve based on the empirical expression[9]:

$$\mu = \mu_0/[1 + (n/n_0)^m], \qquad (1)$$

assuming $\mu_0 = 10^5$ cm^2/V s, $n_0 = 1.48\times10^{16}$ cm^{-3}, and $m = 0.756$. The data in Figure 6 based on measured results obtained by using Si as the n-type dopant, but also include results from undoped layers grown under different conditions. The measured carrier concentration of undoped InSb layers varied between 10^{16} and 10^{18} cm^{-3}, strongly depending on the flux ratio and the substrate temperature. The low-

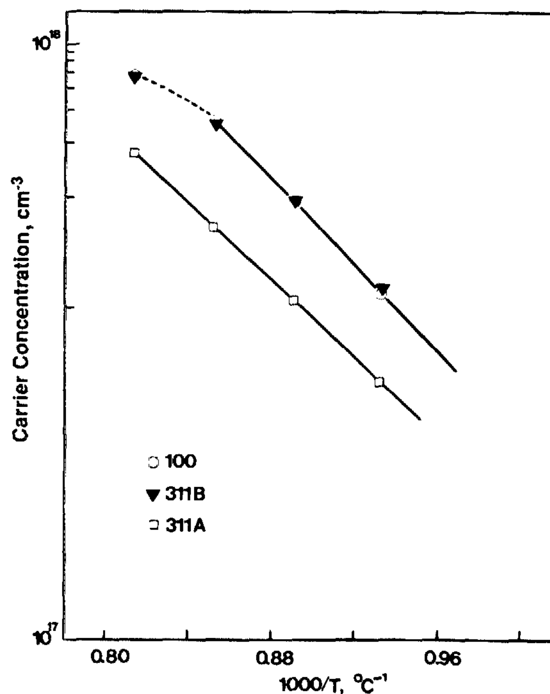

FIG. 5. Measured electron density vs inverse Si source temperature in Si-doped InSb layers grown on GaAs substrates of different orientations.

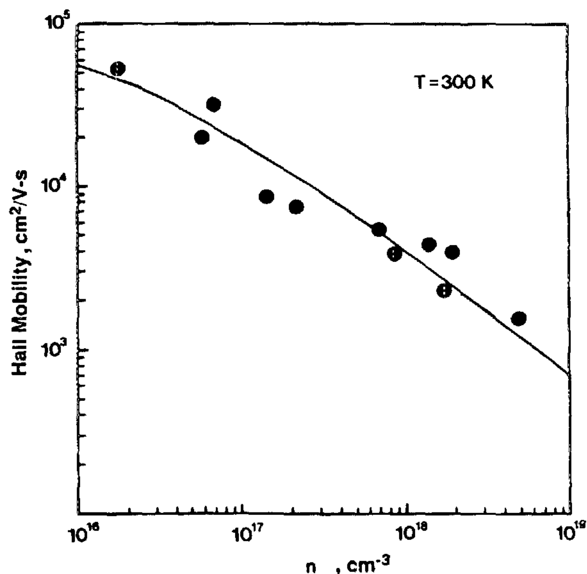

FIG. 6. Measured variation of mobility with net donor density in *n*-type undoped and Si-doped InSb layers at 300 K. The solid line is a best fit to the data using Eq. (1).

est 300-K carrier concentrations lie in the range from $(1.5–5)\times 10^{16}\ \mathrm{cm}^{-3}$ with a Hall mobility of 53 000 cm^2/V s at the lower concentration. The data in Fig. 6 also indicate that the mobility is a strong function of carrier concentration, suggesting that the control of background impurities in the epilayers is the key in growing high-quality material. It should be remembered also that at 300 K the intrinsic carrier concentration is $2\times 10^{16}\ \mathrm{cm}^{-3}$.

IV. CONCLUSIONS

We demonstrate the heteroepitaxial growth of high-quality InSb layers on GaAs and InP substrates by molecular-beam epitaxy without the electron mobility degradation and the carrier freeze-out at low temperatures. The flux ratio, J_{Sb_4}/J_{In}, plays a crucial role in determining the surface morphology, the microcrystalline property, and the electron mobility in heteroepitaxial films due to extremely high sticking coefficient ($\sim$0.5) of Sb_4 molecules. To improve the quality of InSb heteroepitaxial layers, the background impurity must be identified and reduced. However, the fact that extremely high mobilities can be measured at low temperatures suggest that FET-like devices operating at cryogenic temperatures can be conceived.

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