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Growth and characterization of InAsSb alloys by metalorganic chemical vapour deposition*

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Low pressure metalorganic chemical vapour deposition (LP-MOCVD) growth and characteristics of InAsSb on (100) GaSb substrates are investigated. Mirror-like surfaces with a minimum lattice mismatch are obtained. The samples are studied by photoluminescence spectra, and the output is 3.17 μm in wavelength. The surface of InAsSb epilayer shows that its morphological feature is dependent on buffer layer. With an InAs buffer layer used, the best surface is obtained. The InAsSb film shows to be of n-type conduction with an electron concentration of 8.52×10^{16} cm^{-3}.

Keywords: metalorganic chemical vapour deposition (MOCVD), antimonides, semiconducting indium compounds

PACC: 7840K, 8115H, 0660E, 4270

1. Introduction

Among the various III–V materials, the ternary InAsSb solid solution is potentially an interesting system covering the two atmospheric windows, i.e. 3–5 μm and 8–12 μm. InAsSb compounds are attracting great interest for their applications, such as optical communications, environmental protection and remote sensing of atmosphere. InAsSb alloy covering a spectral region of 3–5 μm is a useful material system suitable for infrared source and detectors. The InAsSb alloys grown by using liquid phase epitaxy (LPE),[1] molecular beam epitaxy (MBE),[2,3] and metalorganic chemical vapour deposition (MOCVD) have been reported. Epitaxial growth of materials and the fabrication of devices were performed for InAsSb alloys mostly on InAs substrates.[4] But with InAs substrates used, the lattice mismatch increases rapidly with the increase of Sb concentration, and so it is difficult to epitaxially grow high-quality layers.

For InAsSb lattice-matched to GaSb substrates, GaSb has a wider bandgap of 0.72 eV than InAs, which could suppress Auger nonradiative loss. Successful growth of InAsSb lattice-matched to (100) GaSb, obtained by using MBE, has been reported.[2] And the $InAs_{1-x}Sb_x/InAs_{1-y}Sb_y$ strained-layer superlattice (SLS) has been proposed to be used for photodetection in a range of 8–12 μm.[5,6] But the GaSb substrates are thermally unstable with respect to the ternary metal. And the growth of InAsSb can be achieved only by fast supercooling, thereby leading to rapid growth but with a relatively low quality.[7,8]

Although many reports on InAsSb alloys have been published, to my knowledge, there can be found few detailed studies dealing with the growth property of InAsSb alloys grown on GaSb substrates by using LP-MOCVD. In this work, we report the results on growth of InAsSb alloys grown on GaSb substrate by using LP-MOCVD from the angle of electrical and optical properties.

2. Experiment

A self-made low pressure metalorganic chemical vapour deposition (LP-MOCVD) system with a horizontal reactor was used for the epitaxial growth. The trimethylgallium (TMG), trimethylindium (TMIn) and trimethylantimonide (TMSb) were used as metalorganic precursors, and 100% arsine was diluted

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by hydrogen as V group source. The temperatures for TMGa, TMIn and TMSb sources were –10, 17, and –10°C, respectively. Pd-diffused hydrogen was used as a carrier gas, and the total H_2 flowrate was 4 SLM. Te-doped GaSb (100) substrates were used. The growth was carried out at a low pressure between $9.3 \times 10^3 - 13.3 \times 10^3$ Pa in a rotating disc reactor at a rotation speed of 100 rpm. V/III ratio varied between 4 to 16. The growth temperature ranged from 550 to 620°C. GaSb substrates were protected from decomposing by using TMSb sources before reaching the growth temperature. The TMSb flux was first switched into the reactor at 350°C. The growth rate varied from 0.5 to 1.2 μm/h.

The epilayers were analysed by x-ray diffraction (XRD) measurements and PL spectra, and the surface morphologies of growth layers were studied by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The growth parameters, such as growth temperature and mole ratios of the input sources, were optimized to reduce the lattice mismatch between the InAsSb epilayer and the GaSb substrate. Table 1 presents the optimized growth parameters of $InAs_xSb_{1-x}$ grown on GaSb substrates.

Table 1. Growth conditions of $InAs_xSb_{1-x}$ films on GaSb substrates.

growth temperature/°C	550–620°C
growth pressure	$9.3 \times 10^3 - 13.3 \times 10^3$ Pa
TMIn flux (mol/min)	$(1.5–3)\times10^{-5}$
TMSb flux (mol/min)	$(3.5–5)\times10^{-5}$
AsH_3 flux (mol/min)	$(1.5–2.5)\times10^{-4}$
total H_2 flux (l/min)	4
V / III mole ratio	4–16
Sb/V mole ratio	0.08–0.15

Under these conditions the solid compositions of the epilayers ended up with $x = 0.93$. The thicknesses of the epilayers were in a range of about 0.6–1.2 μm. The surface morphologies were examined by using optical microscopy, SEM and AFM. The solid composition and lattice mismatch were measured by electron microprobe analysis and XRD, respectively. XRD was also used to evaluate the crystalline quality of the epilayer. The electrical properties of the InAsSb epilayers were tested by using room temperature Hall measurements and the van der Pauw technique. The majority-carrier type and concentration were determined by the InAsSb layers grown on semi-insulating GaAs substrates. AFM images of selected sample surfaces were obtained by tapping mode.

3. Results and discussion

3.1. X-ray diffraction

InAsSb epilayers were grown on GaSb substrates. The epilayers were mirror-like. Typical XRD results for several samples of InAsSb/GaSb are shown in Fig.1. A minimum lattice mismatch between InAsSb and GaSb was achieved to be 1%, which implies that the homogeneous InAsSb can be grown successfully inside the empirical miscibility gaps. The component was measured as $InAs_{0.93}Sb_{0.07}$ that is nearly lattice matched to (100) GaSb substrate.

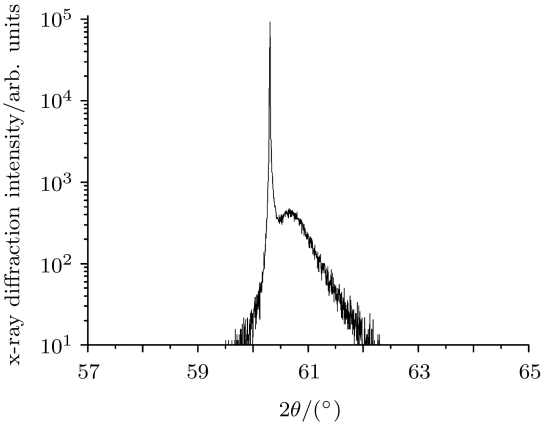


Fig.1. The double XRD pattern of the $InAs_xSb_{1-x}$/GaSb sample, where III/V ratio is 0.22, Sb/V ratio is 0.17, growth temperature is 580°C, and solid composition thickness d is 1 μm.

3.2. Surface morphology

The detailed surface morphology was examined by optical microscopy, SEM and AFM. Figures 2 and 3 show respectively a series of SEM and AFM ($2\,\mu m \times 2\,\mu m$) images of $InAs_{0.93}Sb_{0.07}$ film surface morphologies, prepared at different growth parameters. The results show that growth parameters can affect the surface morphologies of $InAs_{0.93}Sb_{0.07}$ films. The $InAs_{0.93}Sb_{0.07}$ films were all grown at temperatures between 550 and 620°C and the thickness was 0.8 μm. Figures 2(a)–2(c) correspond to Figs.3(a)–3(c) respectively, i.e. the images in Fig.2(a) and Fig.3(a) are obtained from the same sample. Despite the SEM image having low distinguishability, Fig.2 presents a set of almost mirror-like finishing surface images. It is evident that the surface morphology in Fig.2(c) is better than those in Figs.2(a) and 2(b).

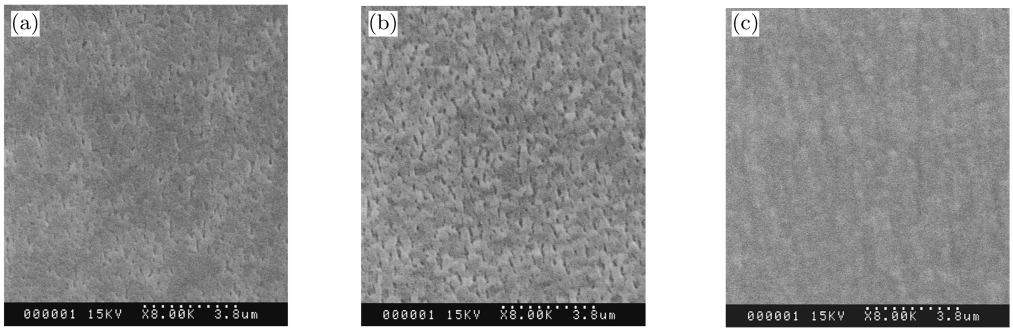


Fig.2. SEM images of InAsSb layers grown on GaSb(100) at 580°C without any buffer layer (a), at 500°C with a 30nm thick InGaAsSb buffer layer (b) and at 580°C with a 30nm thick InAs buffer layer (c).

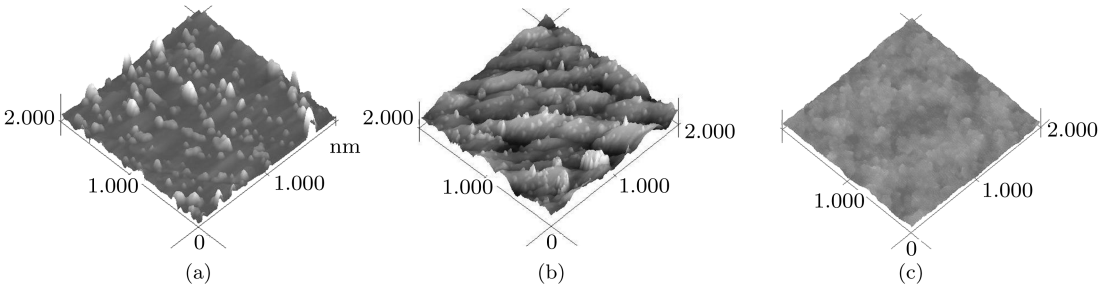


Fig.3. AFM images of InAsSb layers grown on GaSb(10 0) at 580°C without any buffer layer (a), at 500°C with a 30 nm thick InGaAsSb buffer layer (b), and at 580°C with a 30 nm thick InAs buffer layer (c).

Without any buffer layer, the $InAs_{0.93}Sb_{0.07}$ surface exhibits many hillocks as indicated in Fig.3(a). The surface was mirror-like with very small hillocks and had an arithmetic mean roughness (R_a) of 2.707 nm, i.e. $R_a = 2.707$ nm. With an $In_{0.97}Ga_{0.03}As_{0.90}Sb_{0.10}$ buffer layer grown at 500°C, there appeared many ridges and small hillocks on the InAsSb surface as shown in Fig.3(b), which had an arithmetic mean roughness (R_a) of 5.541 nm, i.e. $R_a = 5.541$ nm. Previously reported results have shown that small hillock defects on surface often occur in the growth of antimonide compounds such as GaInSb[9] and GaInAsSb.[10−12] With a low temperature InAs buffer layer, InAsSb layer had a mirror-like surface as shown in Fig.3(c) and an arithmetic mean roughness (R_a) of 1.411 nm, i.e. $R_a = 1.411$ nm. The surface morphology with an InAs buffer layer was better than those with and without InGaAsSb buffer layers.

A possible explanation is supposed to be that the formation of GaAsSb micro-phase is caused by exchange between As and Sb during the growing of InAsSb at higher temperatures.[13] The GaAsSb may be mainly responsible for the defects such as small hillocks. The low temperature InAs buffer layer could preclude the exchange between As and Sb, so that mirror-like surface can be achieved.

We also observed that the surface morphology of InAsSb epilayers was to a great extent determined by the incorporation of arsenic and group-III elements, thereby avoiding the occurrence of hillocks. The V/III ratios used ranged from 2 to 5. A low V/III ratio is necessary for the growth of high-quality InAsSb, and excess As is prone to causing surface defects. The surface morphology turned rough quickly with the increase of As element. The optimum ratio was primarily dependent on growth temperature. The growth rate was linearly dependent on the quantity of group III elements flowing into the reactor. The optimum V/III ratio was found to be almost unchanged when the growth rate varied. The growth temperature of InAsSb layer could also strongly affect the surface morphology. When growth temperature was higher than 620°C, the surface morphology became rough. Similar results were obtained for the growth of InAsSb with rougher surfaces occurring at temperatures lower than 500°C. The surface morphology was improved with a low temperature InAs buffer layer. That may be beneficial to further device processing.

3.3. Electrical properties

The electrical properties of InAsSb layer were measured by using the Hall method and the Van der Pauw technique. We observed that the InAsSb films were of n-type conduction. A room temperature electron mobility (μ_{H}) and electron concentration (n) were observed to be $411\,\mathrm{cm}^2\cdot\mathrm{v}^{-1}\cdot\mathrm{s}^{-1}$ and $8.5\times10^{16}\,\mathrm{cm}^{-3}$ under the doping condition, respectively, i.e. $\mu_{\mathrm{H}}=411\,\mathrm{cm}^2\cdot\mathrm{v}^{-1}\cdot\mathrm{s}^{-1}$ and $n=8.5\times10^{16}\,\mathrm{cm}^{-3}$

The photoluminescence peak of InAsSb grown on GaSb substrate appears at $3.17\,\mu$m in wavelength at 77 K (see Fig.4). The photoluminescence wavelength $3.17\,\mu$m is shorter than that corresponding to the bandgap of $InAs_{0.93}Sb_{0.07}$ ($4.13\,\mu$m), which is thus supposed to be due to the type II transition. The full width at half maximum (FWHW) of PL spectrum is about 41.8 meV at 77 K. That can be explained as the band curved near the interface between InAsSb and GaSb, and the transition happening on interface has a higher probability than that inside. Thus it can be developed into a potential infrared emitting material.

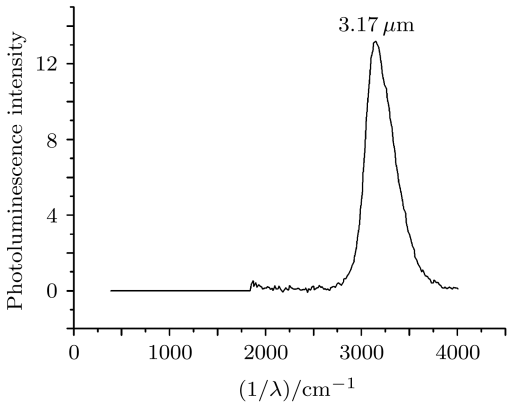


Fig.4. PL-spectra of InAsSb/GaSb at temperature 77 K.

4. Summary

The layers of $InAs_xSb_{1-x}$ with $x=0.93$ are grown on GaSb substrates by using low pressure MOCVD. The surfaces are smooth and mirror-like. Their detailed surface morphologies and electrical properties were investigated. The concentration of TMSb greatly affects the surface morphology. The surface morphology is improved with using a low temperature InAs buffer layer. The samples are studied by photoluminescence spectra. The photoluminescence wavelength $3.17\,\mu$m is shorter than the bandgap of $InAs_{0.93}Sb_{0.07}$ ($4.13\,\mu$m), which, we supposed, is due to the type II transition.

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