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# Growth and Characterisation of InAsSb Ternary Layers on (100) GaSb Substrates by LP-MOCVD*

Li Xiaoting[1,2], Wang Tao[1], Wang Jingwei[1], Wang Yiding[3], Yin Jingzhi[3],
Sai Xiaofeng[1], Gao Hongkai[1], and Zhang Zhiyong[4]

*(1 Xi'an Institute of Optics and Precision Mechanism, Chinese Academy of Sciences, Xi'an 710068, China)*
*(2 Chang'an University, Xi'an 710061, China)*
*(3 Department of Electronics and Engineering, Jilin University, Changchun 130023, China)*
*(4 Northwest University, Xi'an 710069, China)*

**Abstract:** InAsSb alloys are grown on n-(100) GaSb (Te-doped) and GaAs substrates by the MOCVD using TMIn, TMSb, and $AsH_3$ sources. The influence of growth parameters such as temperature, V/Ⅲ ratio, and buffer layer on the surface morphology and solid composition is studied. The surface morphology is observed by AFM and SEM. The As and Sb concentrations in the solid are characterized by electron microprobe analysis. The crystalline quality of the InAsSb epilayer is characterized by double-crystal X-ray rocking curve diffraction. The electrical properties are observed by the (Van der Pauw) Hall technique at room temperature. An InAsSb epitaxy layer with mirror-like surface and lattice mismatch of 0.4% is obtained.

**Key words:** LP-MOCVD; GaSb; InAsSb; growth
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## 1 Introduction

Semiconductor devices based on Sb compounds and their solid solutions such as GaInSb, GaInAsSb, and InAsSb have been found increasing practical use in recent years. The Ⅲ-Ⅴ ternary alloys $InAs_xSb_{1-x}$ covering the spectral region 3～5μm present important advantages over $Hg_{1-x}Cd_xTe$: better stability, higher electron and hole mobilities, and more availability of high-quality and low-cost substrates[1,2]. Among the various Ⅲ-Ⅴ materials, $InAs_xSb_{1-x}$ are potentially very important material systems suitable for the fabrication of light emitting diodes and detectors for middle-wave-length infrared applications. Applications in the spectral range 3～5μm are found in various domains such as pollutant detection, infrared thermal imaging, and lidars or optical countermeasures[3,4]. Despite the high potential and attractive properties of these and many other applications, industrial use is still in its infancy due to a number of challenges associated with the particular growth of Sb-compounds. First of all, most of the ternary and quaternary materials of interest are thermodynamically unstable and tend to show different forms of phase separation and ordering. This requires the crystal growth to be performed far from equilibrium, typically at low temperatures from 350 to 600 ℃. Further necessitating low-temperature growth are the

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Li Xiaoting female, was born in 1972, PhD candidate. She is engaged in research on material growth and photoelectric devices.

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low melting temperatures for some of the compounds. Unfortunately ,most of the commonly used metalorganic and hydride sources do not completely decompose at these low temperatures ,e. g. $AsH_3$ (650 ℃) . So the growth of Sb-compounds (e. g. InAsSb) is very difficult[5] . But the study of the growth of InAsSb ternary alloys is necessary.

In recent years ,there have been some reports on the $InAs_xSb_{1-x}$ ternary alloys grown by different methods, such as liquid phase epitaxy (LPE)[6,7] ,MBE[3,8～11] ,and MOCVD[12,13] on various substrates ,e. g. InAs , InSb , GaAs , GaSb , InP , and Si. However , very few growth characteristics and results are reported on InAsSb grown on GaSb by MOCVD. This paper aims at developing MOCVD growth of InAsSb layers on GaSb substrates. Special attention is devoted to elaboration on the conditions for the growth of good quality InAsSb layers.

## 2　Experiment

The epitaxial growth was performed in a horizontal ,low pressure MOCVD reactor. The group-Ⅴ precursors were trimethylantimony ( TMSb) and 10 % arsine diluted by hydrogen. The group-Ⅲ precursors were trimethylgallium ( TMGa) and trimethylindium ( TMIn) . The source temperatures for TMGa , TMIn , and TMSb were - 17 , 5 , and - 5 ℃,respectively. The carrier gas was Pd-purified $H_2$ . The substrates were (100)-oriented , Te-doped GaSb and semi-insulating GaAs. GaSb and GaAs substrates were kept from decomposing by TMSb and arsine sources before reaching the growth temperature.

The growth parameters , such as growth temperature and the ratios of Ⅴ/ Ⅲ,were optimized to decrease the lattice mismatch between the InAsSb epilayer and the GaSb substrate. The growth temperature was in the range of 520～630 ℃,and Ⅴ/ Ⅲ ratios were varied from 5 to 25. The total gas flowing into the reactor was 4. 3L/ min.

After InAsSb epitaxy was obtained on (100)-oriented GaSb and GaAs substrates by a low pressure MOCVD system ,the characteristics of InAsSb epitaxy were investigated by means of X-ray diffraction ,optical microscopy ,atomic force microscopy (AFM) , scanning electron microscopy ( SEM) , and electron microprobe analysis. Standard Hall measurements were performed to determine the electrical properties of the InAsSb epilayers.

## 3　Results and discussion

In the MOCVD growth of Sb-based compound ,temperature is a critical parameter determining the surface morphology and the composition of the epilayer.

In order to obtain an epilayer with the lattice matching the GaSb substrates ,we must control the composition , i. e. the $x$ value of $InAs_xSb_{1-x}$. According to Vergard ’s laws , $InAs_{0.91}Sb_{0.09}$ with a composition corresponding to $x = 0.91$ has a lattice that matches GaSb substrates. In order to study the influence of growth temperature on the solid composition ,the growth temperature was varied from 520 to 630 ℃ while keeping the other growth parameters constant ,such as Ⅴ/ Ⅲ ratios of 10 and a growth pressure of 133kPa. The growth electron microprobe analysis result shows that the solid composition of $InAs_xSb_{1-x}$ is dependent on growth temperature ,as shown in Fig. 1. In the range of 520 ～600 ℃, $x$ increases with increasing growth temperature. This may be due to the different pyrolysis efficiencies of TMSb and $AsH_3$ at different temperatures. It is known that TMSb is nearly completely pyrolysed above 500 ℃, but the pyrolysis of $AsH_3$ still increases with increasing growth temperature over 500 ℃. The real ratio of As and Sb in the vapor phase consequently increases , resulting in the increase of As composition $x$. Above 600 ℃, $x$ decreases with increasing growth temperature. This might be due to the stronger parasitic reaction between TMIn and $AsH_3$ in the vapor phase[14] .

The Ⅴ/ Ⅲ ratio is another critical parameter for MOCVD growth of Sb-based compounds ,as we

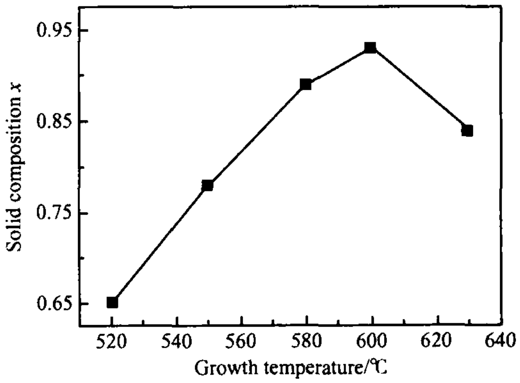

Fig. 1 Solid composition $x$ of InAs$_x$Sb$_{1-x}$ versus growth temperature

see in Fig. 2 the surface morphologies of InAsSb in different Ⅴ/Ⅲ ratios by means of SEM.

The low volatility of Sb is also the reason that the antimonides are grown at a low Ⅴ/Ⅲ ratio. The Ⅴ/Ⅲ ratio is also shown to have an important influence on the morphology. 3D island growth occurs under certain conditions. The 2D nucleation is only observed for a small range of Ⅴ/Ⅲ ratios. Too high a Ⅴ/Ⅲ ratio (Ⅴ/Ⅲ= 25) will cause excess Sb to stay on the sample surface, which results in the formation of Sb droplets, thus causing rough morphology (Fig. 2(a)). An appropriate Ⅴ/Ⅲ ratio (Ⅴ/Ⅲ= 10) will result in InAsSb alloys with smooth and mirror-like surface (Fig. 2(b)). Too low a Ⅴ/Ⅲ ratio (Ⅴ/Ⅲ= 5) will lead to unstable growth and poor crystal quality (Fig. 2(c)). In the extreme case, intolerable In droplets may be generated on the sample surface.

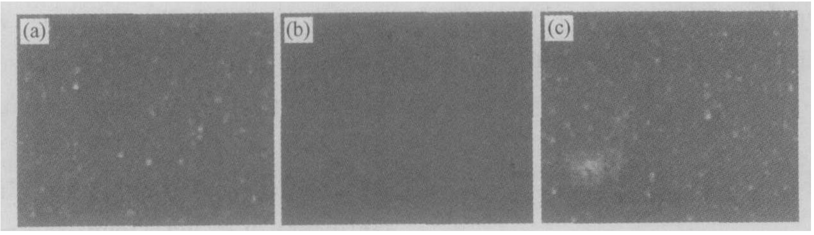

Fig. 2 Surface morphologies of InAsSb for different Ⅴ/Ⅲ ratios (a) Ⅴ/Ⅲ= 25; (b) Ⅴ/Ⅲ= 10; (c) Ⅴ/Ⅲ= 5

We observed the surface of InAsSb with and without a GaSb buffer by means of AFM. The result shows that the epitaxial layer without a buffer has a smoother surface than that with a buffer, as shown in Fig. 3. The phenomenon is in accord with the results of Nouaouraj[15]. The GaSb surface stoichiometry is less stable than that of GaAs, and is easy to change through annealing. When As exists in the reactor, the control of the structural and chemical properties of arsenide-antimonide interfaces is not easy. The use of two different elements As and Sb can result in a decrease of the interface quality. For $T < 400$ ℃, As-Sb exchanges in the surface layer occur; for $T > 450$ ℃, the desorption of Sb atoms changes the formation of GaAsSb microphases. This surface reconstruction strongly affects surface morphology and crystal quality.

Figure 4 shows the double-crystal X-ray rocking curve of the (400) diffraction of InAsSb. An

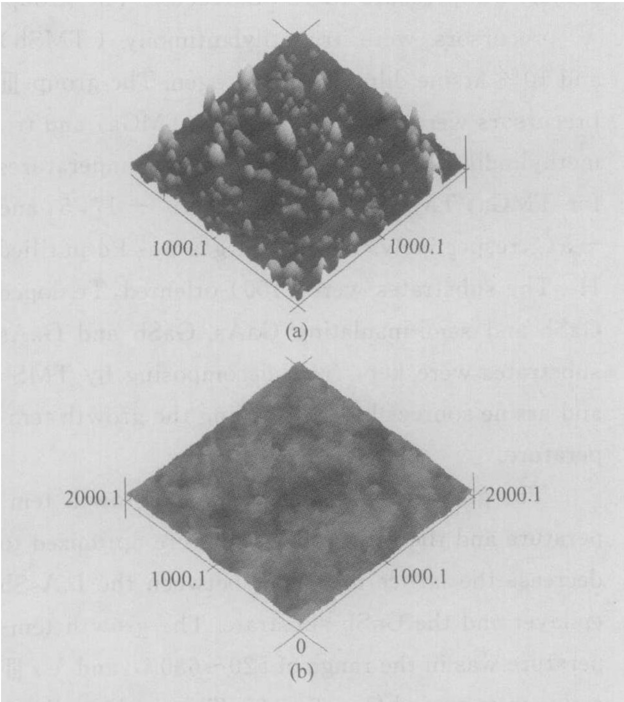

Fig. 3 Surface morphologies of InAsSb with buffer layer (a) and without buffer layer (b)

InAsSb epitaxy with a lattice mismatch of 0.4% is obtained, and the crystal quality is still poor. This may be because Sb has a much weaker chemical bonding and low volatility, Sb-based compounds are difficult to grow, and high-quality crystal of Sb-based compounds are difficult to obtain.

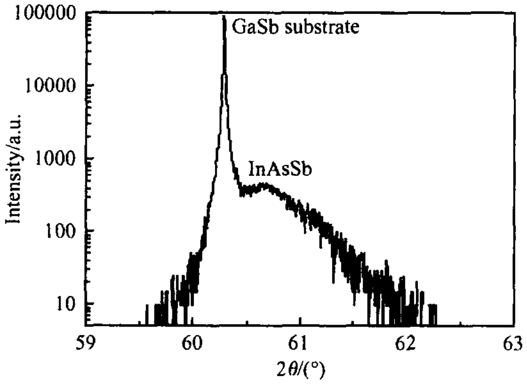

Fig. 4　X-ray diffraction of InAsSb/GaSb sample

The electrical properties, mobility, and carrier density were determined by Hall measurements using the Van der Pauw method with a mesa structure on an undoped InAsSb layer deposited on GaAs substrates. The sample is formed to be n-type with room temperature carrier concentrations of $8.52\times10^{16}cm^{-3}$ and mobilities of $411cm^2/(V\cdot s)$. A possible explanation for n-type conduction might be that carbon occupies group-Ⅲ element sites as a donor in the materials[14]. The mobilities of the sample are low. This may be explained by the crystalline quality, which is better when the epitaxy layers are grown to match the lattice closely. In fact, when the layers are grown on GaAs, the lattice mismatch also plays a role, and microphases and large crystal defects could be present, thus degrading the crystal quality. The result is a decrease in mobility.

## 4　Conclusion

InAsSb alloys with smooth and mirror-like surfaces have been successfully grown on GaSb substrates by LP-MOCVD using TMIn, TMSb, and $AsH_3$ as source materials. The detail surface morphology is observed by SEM and AFM. The result shows that buffer layer, growth temperature, and Ⅴ/Ⅲ ratio affect the surface morphology. The solid concentrations were characterized by electron microprobe analysis, and the result shows that the $x$ value of $InAs_xSb_{1-x}$ increases with increasing growth temperature; but above 600 ℃, $x$ decreases with increasing growth temperature. The results of double-crystal X-ray rocking curve diffraction indicate that the growth window of InAsSb alloys is very narrow. On the other hand, the electrical properties are investigated, and an electron mobility of $411cm^2/(V\cdot s)$ with a concentration of $8.52\times10^{16}cm^{-3}$ is achieved.

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# GaSb 衬底上 InAs$_x$Sb$_{1-x}$合金的低压 MOCVD 生长和表征*

李晓婷[1,2] 汪 韬[1] 王警卫[1] 王一丁[3] 殷景致[3] 赛小锋[1] 高鸿楷[1] 张志勇[4]

(1 中国科学院西安光学精密机械研究所, 西安 710068)
(2 长安大学理学院, 西安 710061)
(3 吉林大学电子工程系, 长春 130023)
(4 西北大学, 西安 710069)

**摘要**: 采用自制的低压金属有机化学气相淀积设备,用三甲基镓、三甲基铟作为Ⅲ族源,三甲基锑和砷烷作为Ⅴ族源在(100)面 GaSb 和 GaAs 单晶衬底上分别外延生长了 InAsSb 材料. 用 X 射线双晶衍射、原子力显微镜、扫描电镜和电子探针能谱仪等对材料特性进行了表征,研究了生长温度、Ⅴ/Ⅲ比、过渡层等生长参数对外延层质量的影响. 获得了与 GaSb 衬底晶格失配度为 0.4%的表面光亮且晶体质量较好的 InAs$_{0.85}$Sb$_{0.15}$外延层.

**关键词**: LP-MOCVD; GaSb; InAsSb; 生长

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李晓婷 女,1972 年出生,讲师,博士研究生,主要从事Ⅲ-Ⅴ族材料生长和光电器件的研究.

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