KAPL-P-000232
(K96122)

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October 1996

NOTICE

KAPL ATOMIC POWER LABORATORY SCHENECTADY, NEW YORK 12321

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OMVPE Growth of Metastable GaAsSb and GaInAsSb Alloys Using TBAs and TBDMSb

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Abstract

Epitaxial layers of the metastable alloys GaAsSb and GaInAsSb have been grown by organometallic vapor phase epitaxy (OMVPE) using tertiarybutylarsine (TBAs) and tertiarybutyldimethylantimony (TBDMSb) with conventional group III sources. Layers with compositions well inside the miscibility gap were successfully obtained. The key parameters for obtaining these metastable alloys are the use of low V/III ratios ($\leq$ 1) and low growth temperatures. The Sb distribution coefficients for GaAsSb are close to unity on both GaSb and InAs substrates when V/III ratios of approximately unity are used. The distribution coefficients of Sb for GaInAsSb on GaSb and InAs substrates are slightly higher than unity for lower V/III ratios. The quality of the surface morphology is degraded as the composition moves further into the region of solid immiscibility. The hole concentrations of undoped GaInAsSb layers on GaAs substrates grown using conditions identical to those used for the InAs substrates are approximately 1 x 10^{17} cm^{-3} at room temperature. The concentrations of In and Sb in the GaInAsSb layers were 0.05-0.23 and 0.75-0.95, respectively, on GaSb substrates and 0.23-0.39 and 0.82-0.89, respectively, on InAs substrates. These compositions correspond to bandgap energies of 0.4-0.7 eV. Low temperature photoluminescence (PL) spectra were observed for GaInAsSb layers grown on GaSb and InAs substrates at wavelengths of approximately 1.9 and 2.0 μm with half-widths of 20 and 25.3 meV, respectively.

Introduction

Antimony based alloys have been developed mainly for optoelectronic applications in the mid to far infrared regions of the spectrum. For the far infrared applications at 8-12 μm, InAsSb [1,2], InSbBi [3], and InAsSbBi [4,5] alloys and GaInSb/InAs [6] and GaSb/InAs [7] superlattice structures have been explored. In addition, the GaAsSb and GaInAsSb alloys lattice matched to InP, GaSb, and InAs cover the spectral range from 1.3 to 4.3 μm. This mid infrared wavelength range has recently attracted increasing attention for potential device applications such as tandem solar cells [8], thermal photovoltaics [9], lasers and detectors for fiber optic systems [10], and atmospheric monitoring systems [11].

GaAsSb and GaInAsSb are known to have very large miscibility gaps with critical temperatures estimated from delta lattice parameter (DLP) model calculations to be 751 and 1467 °C, respectively [12-14]. Alloys with compositions inside the miscibility gap are virtually impossible to grow by the liquid phase epitaxy (LPE) technique [15-17]. Since the first organometallic vapor phase epitaxial (OMVPE) growth of GaAsSb was reported by Manasevit [18], there have been continuous efforts to obtain alloy compositions inside the miscibility gap. Cooper et al. [19] reported the OMVPE growth of $GaAs_{1-x}Sb_x$ with compositions of $0 \leq x \leq 0.26$ and $0.64 \leq x \leq 1$. Subsequently, Stringfellow and Cherng demonstrated the successful growth of GaAsSb and GaInAsSb with compositions throughout the miscibility gaps [13,14,20].

There has been rapid progress in the development of new organometallic precursors to replace the conventional sources, especially the group V hydrides that are highly toxic. Tertiarybutylarsine (TBAs) has been reported [21] to be much less hazardous than AsH_3. It has a decomposition temperature, T_{50}, of approximately 425 °C [22], much less than that for arsine (T_{50}~600 °C). This source also has a convenient vapor pressure for OMVPE growth.

Although trimethylantimony (TMSb) has been successfully used to grow several Sb-containing compounds and alloys by OMVPE, it is not convenient for low temperature growth due to the low decomposition rate at temperatures of 500 °C and below [23]. In addition, high carbon contamination levels are reported due to the CH_3 radicals produced [24]. In this regard, significant efforts have been devoted to developing new Sb precursors during the last several years. Two precursors, tertiarybutyldimethylantimony (TBDMSb) [25,26] and trisdimethylaminoantimony (TDMASb) [27,28], have been used for the growth of Sb-containing compounds by OMVPE in the temperature ranges of 325-650 and 275-600 °C, respectively. The layers grown using these two precursors showed good crystalline quality with no serious impurity contamination. For the growth of GaAsSb and GaInAsSb alloys, TDMASb was first used with TBAs, trimethylindium (TMIn), and trimethylgallium (TMGa). Unfortunately, parasitic reactions between these new group V precursors and the conventional group III precursors were observed. On the other hand, no parasitic reactions were observed when TBDMSb was used with TBAs, TMIn, and TMGa.

In this paper, the OMVPE growth of metastable GaAsSb and GaInAsSb alloys using TBDMSb and TBAs with the conventional group III sources, TMGa and TMIn, is reported. Good morphology layers with compositions inside the region of solid immiscibility were obtained. The distribution coefficients of In and Sb show trends similar to those reported previously.

Experimental Procedure

The epilayers were grown in an atmospheric pressure, horizontal, infrared heated OMVPE reactor described in detail elsewhere [29]. The carrier gas for the precursors was palladium-diffused hydrogen. Separate stainless steel tubing was used for the group III and V reactants in order to minimize possible parasitic reactions. TMGa, TMIn, TBAs,

and TBDMSb held in temperature-controlled bubblers at -10 to -14, 20, 0 to -11, and 20 °C, respectively, were the precursors. Exactly (100) oriented Te-doped GaSb and exactly (100) oriented InAs substrates were used. Semi-insulating GaAs substrates were used to produce samples for Hall-Effect measurements. The growth temperature was fixed at 550 °C for the growth of both GaAsSb and GaInAsSb on GaSb substrates. Growth temperatures of 550 and 500 °C were used on InAs substrates for GaAsSb and GaInAsSb, respectively. The total flow rate of the Pd-purified H_2 carrier gas was approximately 2200-2300 sccm.

All the substrates were prepared by first degreasing using trichloroethylene (TCE), acetone, and methanol, in that order. The GaAs substrates were then immersed in H_2SO_4 for 3 min followed by A-etch for 4 min. The GaSb substrates were etched using HF : H_2O = 1 : 5 for 10 min with immersion in HCl for 5 min before and after the etching solution to dissolve oxide layers. For the InAs substrates, a HF : H_2O = 1 : 1 etching solution was used prior to etching in a dilute Br-methanol solution. They were then immediately loaded into the reactor. In order to eliminate the residual air in the chamber, the quartz tube was purged with hydrogen for 30 min. After the temperature was raised to the final growth temperature, all the sources were introduced into the growth chamber. The InAs substrate was annealed under an As ambient for about 3 min at 400 °C, which is lower than the final growth temperature, before the organometallic sources were admitted into the reactor.

The surface morphologies of the epilayers were examined using a Nikon-AFX Nomarski interference contrast optical microscope. The composition of GaAsSb layers was determined using X-ray diffraction assuming Vegard's law. The solid composition of GaInAsSb was measured using a Cameca SX-50 electron microprobe. Room temperature Hall-Effect measurements were performed using the Van der Pauw geometry.

The photoluminescence (PL) was excited with an Ar^+ laser at a wavelength of 488 nm. A pair of off-axis paraboloidal reflectors focused the light onto the entrance slit of a SPEX M500 spectrometer. A longpass IR filter with cutoff wavelength at 950 nm was put

in front of the entrance slit to block reflected light. At the exit slit, an InSb photoconductor cooled to 77 K was used to detect the signal. The standard lock-in technique with a chopping frequency of 1.5 KHz was used to reduce the noise. The samples were held on a 15 K coldfinger. The excitation intensity was on the order of 40 W/cm^2.

Results and Discussion

Typical growth conditions and solid compositions for GaAsSb layers on GaSb and InAs substrates at a growth temperature of 550 °C are summarized in Table I. Similar to the results of Cherng [13,20] using AsH_3 (or TMAs) and TMSb, the input V/III ratio and growth temperature are critical factors for obtaining compositions inside the miscibility gap without phase separation when using TBAs and TBDMSb. Fig 1. (a) and (b) show the distribution coefficient of Sb on GaSb and InAs substrates, respectively. As described by Cherng et al. [13] for a III/V ratio of unity, the Sb distribution coefficient is nearly unity. Sb incorporation into the solid is reduced with increasing V/III ratio for values of > 1 as expected from previous results [14,20] using standard precursors. This result is also similar to a recent report of Watanabe et al. using TBAs and TESb [30].

Growth of GaAsSb on InAs substrates is more difficult at the same temperature of 550 °C. Huge liquid metallic droplets appear on the surface for InAs substrates with V/III ratios of greater than 1.6. This may be due to the local loss of As from the substrate surface before the beginning of growth. This would produce In-rich droplets on the surface. These droplets are expected to enhance the pyrolysis rate of the precursors. In other systems [31] this has been shown to result in vapor-liquid-solid (VLS) growth. Addition of the group V elements to the droplets may not result in the formation of a solid since 550 °C is above the melting temperature of InSb. In fact, the solidus line of the pseudobinary GaInSb phase diagram is nearly flat for a large composition range near InSb

[32]. This suggests that the growth temperature should be reduced to a value less than the melting point of InSb, approximately 525 °C. This problem was not solved, even by exposing the surface to flowing TBAs prior to growth.

Fig 2. shows the surface morphologies of several GaAsSb layers grown on GaSb and InAs substrates at 550 °C. A cross-hatched pattern is observed on the epilayers grown on GaSb due to a small lattice mismatch between substrate and epilayer. V/III ratios were adjusted to values between 1.3 and 1.36 to obtain shiny morphologies. The surface morphologies on InAs substrates are much worse than on GaSb. As discussed above, huge liquid droplets appear on the surface for a V/III ratio of 1.93 as seen in Fig 2. (d). The surface morphology is improved by decreasing the V/III ratio, but is still rough at a V/III ratio of 0.99, as seen in Fig 2. (e). It is necessary to lower the growth temperature to obtain good surface morphologies, as discussed below.

Fig 3. (a) and (b) show the Sb and In distribution coefficients, respectively, for GaInAsSb layers grown on GaSb substrates at 550 °C. All the layers included in this figure have surface morphologies that are shiny to the naked eye. Sb distribution coefficients of greater than one were obtained using V/III ratios of approximately one. These results are generally consistent with that data for GaAsSb discussed above and similar to data presented by Watanabe and Iwamura [30] for the growth of GaAsSb using TESb and TBAs with TEGa at 470 °C. They reported that the Sb incorporation in the solid is increased by lowering the V/III ratio from 3 to 1 and reducing the growth temperature from 500 to 470 °C. Fig 3. (b) shows that the distribution coefficient of In is unity regardless of the growth parameters used.

The Sb and In distribution coefficients for GaInAsSb grown on InAs at 500 °C are presented in Fig 4. A growth temperature of 500 °C is used for InAs substrates to avoid the liquid droplets, as discussed above. Input V/III ratios of much lower than unity are necessary to obtain good surface morphologies. Sb and In distribution coefficients slightly higher than unity were obtained when using V/III ratios near 0.5.

The surface morphologies of GaInAsSb layers having several compositions grown on GaSb and InAs substrates are shown in Fig 5. Several typical growth conditions and solid compositions are summarized in Table I. The morphologies of the layers with compositions outside the region of immiscibility grown on GaSb are nearly smooth. Although the surface is shiny to the naked eye, tiny rectangular defects can be observed by interference contrast microscope as the composition moves to values inside the miscibility gap. Small liquid metallic droplets are observed for GaInAsSb on InAs substrates when the V/III ratio is higher than unity even at 500 °C, as shown in Fig 5. (d). Surface morphologies become shiny as the V/III ratio is reduced to about 0.5. Fig 5. (f) shows the cross hatch pattern due to a large lattice mismatch. Clearly, the key factors for obtaining good morphology layers are to use low V/III ratios, low growth temperatures, and to grow layers lattice matched to the substrate. The optimum V/III ratios for the growth of GaInAsSb layers are somewhat lower than for GaAsSb epilayers. For the growth of GaInAsSb on InAs substrates, a high TMGa/group III ratio in the input gas stream was used due to the incomplete decomposition of TMGa at the growth temperature of 500 °C, as shown in Table I. The incomplete decomposition of TMGa at 500 °C requires the use of relatively low V/III ratios for GaInAsSb layers on InAs substrates as compared to GaAsSb layers on InAs substrates with growth at 550 °C.

Fig 6 presents a diagram of the solid phase field for the GaInAsSb alloy system including the present data obtained using TBAs and TBDMSb with conventional group III sources. The 600 °C binodal isotherm calculated by Stringfellow and Cherng [34] is shown as the curved line and the iso-lattice constant lines representing alloys lattice matched to InAs and GaSb are included in Fig 6. Most of the layers grown on both GaSb and InAs substrates are positioned inside the region of solid immiscibility. For the GaInAsSb layers on GaSb substrates the concentrations of In and Sb are in the ranges 0.05-0.23 and 0.75-0.95, respectively. On InAs substrates the In and Sb concentrations are in the ranges 0.23-0.39 and 0.82-0.89, respectively. It can be seen that the bandgaps of

the GaInAsSb layers will range from 0.4 to 0.7 eV. None of the layers grown on InAs substrates are closely lattice matched, which results in some surface roughness, as discussed above.

The crystalline quality of the GaAsSb and GaInAsSb epitaxial layers was evaluated using X-ray diffraction measurements. X-ray diffraction peaks with half-widths similar to those reported previously [13,35] were obtained in these GaAsSb and GaInAsSb epilayers. As reported previously, the peaks become broader and their intensities decrease as the composition moves further inside the region of solid immiscibility.

Undoped GaInAsSb layers grown on semi-insulating GaAs substrates are *p*-type with hole concentrations of 8 x 10^{16} to 6 x 10^{17} cm^{-3} and hole mobilities of 100 to 150 $cm^2/Vsec$ at room temperature. Even with the large lattice parameter mismatch, the values of carrier concentration should be accurate, although the mobilities are probably much lower than would be obtained for homoepitaxial samples. Typical PL spectra at 15 K for a $Ga_{0.92}In_{0.08}As_{0.16}Sb_{0.84}$ layer grown on a GaSb substrate and a $Ga_{0.90}In_{0.10}As_{0.22}Sb_{0.78}$ layer grown on an InAs substrates are shown in Fig 7. The peak positions for the GaSb and InAs substrates were 660 and 610 meV, respectively. The intense and well resolved PL peaks are indicative of good material quality. The half-widths of 20 and 25.3 meV are comparable to those observed for GaAsSb [13] and rather narrower than previous results for GaInAsSb grown using conventional precursors [14].

Conclusions

The new group V precursors TBAs and TBDMSb have been used to grow GaAsSb and GaInAsSb epitaxial layers with compositions inside the miscibility gap. The efficient decomposition of the new group V precursors at low temperatures facilitates low temperature growth. The input V/III ratio and the growth temperature are found to be the crucial factors for obtaining good quality layers. The distribution coefficient of Sb is about

unity for GaAsSb layers on GaSb and InAs substrates when the V/III ratio is approximately unity. The Sb distribution coefficient for GaInAsSb layers is slightly higher than unity. The In distribution coefficient for GaInAsSb layers is nearly unity on GaSb substrates and slightly higher than unity on InAs substrates. Surface morphologies of GaAsSb and GaInAsSb layers grown on GaSb substrates deteriorate gradually as the composition moves further inside the region of solid immiscibility. It is much more difficult to obtain good morphology layers on InAs substrates. At the growth temperature of 500 ℃ only an extremely narrow range of V/III ratios yields good quality layers on InAs substrates. Undoped GaInAsSb layers are *p*-type with hole concentrations of ~1 x 10^{17} cm^{-3}. Intense and narrow PL spectra were measured at low temperature for GaInAsSb layers on GaSb and InAs substrates, indicating the high quality of the epitaxial layers.

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Figure Captions

Fig. 1 Sb distribution coefficient for GaAsSb layers grown on (a) GaSb and (b) InAs substrates at a temperature of 550 °C.

Fig. 2 Surface morphologies of $GaAs_{1-y}Sb_y$ epitaxial layers grown on GaSb and InAs substrates at Tg = 550 °C : (a) V/III = 1.3, y = 0.62, on a GaSb substrate ; (b) V/III = 1.3, y = 0.83, on a GaSb substrate ; (c) V/III = 1.36, y = 0.89, on a GaSb substrate ; (d) V/III = 1.93, y = 0.16, on an InAs substrate ; (e) V/III = 0.99, y = 0.66, on an InAs substrate.

Fig. 3 Distribution coefficients of (a) Sb and (b) In for GaInAsSb layers grown on GaSb substrates at the growth temperature of 550 °C.

Fig. 4 Distribution coefficients of Sb and In for GaInAsSb layers on InAs substrates at a growth temperature of 500 °C.

Fig. 5 Surface morphologies of $Ga_{1-x}In_xAs_{1-y}Sb_y$ epitaxial layers on GaSb and InAs substrates at Tg = 550 and 500 °C, respectively : (a) V/III = 1.0, x = 0.794, y = 0.938, on GaSb ; (b) V/III = 1.0, x = 0.099, y = 0.759, on GaSb ; (c) V/III = 1.05, x = 0.233, y = 0.796, on GaSb ; (d) V/III = 1.96, on InAs ; (e) V/III = 0.49, x = 0.275, y = 0.893, on InAs ; (f) V/III = 0.38, x = 0.394, y = 0.817, on InAs.

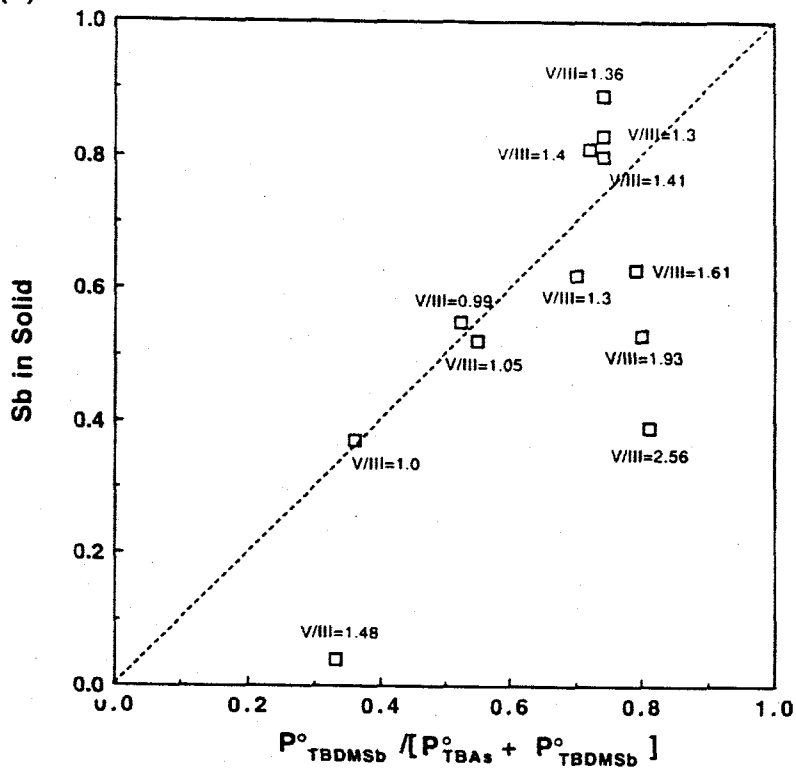
Fig.6 Solid phase field for GaInAsSb showing the alloy compositions grown on GaSb and InAs substrates. Calculated room temperature energy bandgaps [33] and the 600 °C binodal isotherm [34] are plotted. The iso-lattice constant lines for GaSb and InAs are also included.

Fig. 7 Low temperature (15 K) PL spectra for $Ga_{0.92}In_{0.08}As_{0.16}Sb_{0.84}$ on a GaSb substrate and $Ga_{0.90}In_{0.10}As_{0.22}Sb_{0.78}$ on an InAs substrate at Tg = 550 and 500 °C, respectively.

Table I Growth Conditions and Solid Compositions for $GaAs_{1-y}Sb_y$ and $Ga_{1-x}In_xAs_{1-y}Sb_y$ Layers

Growth Temperature (°C)	Substrate	Molar Flow Rates (μmole / min)				V/III Ratio	In Solid Composition (x)	Sb Solid Composition (y)
		TMGa	TMIn	TBAs	TBDMSb			
550	GaSb	21.29		7.33	20.34	1.3		0.83
550	GaSb	21.29		7.6	21.39	1.36		0.89
550	InAs	21.29		8.4	32.62	1.93		0.16
550	InAs	21.29		9.99	10.99	0.99		0.66
550	GaSb	16.9	1.64	6.86	11.6	1.0	0.099	0.759
550	GaSb	8.1	2.84	3.9	7.62	1.05	0.233	0.796
500	InAs	18.54	2.84	3.42	7.01	0.49	0.275	0.893
500	InAs	16.59	2.5	3.6	8.56	0.64	0.319	0.878

(a)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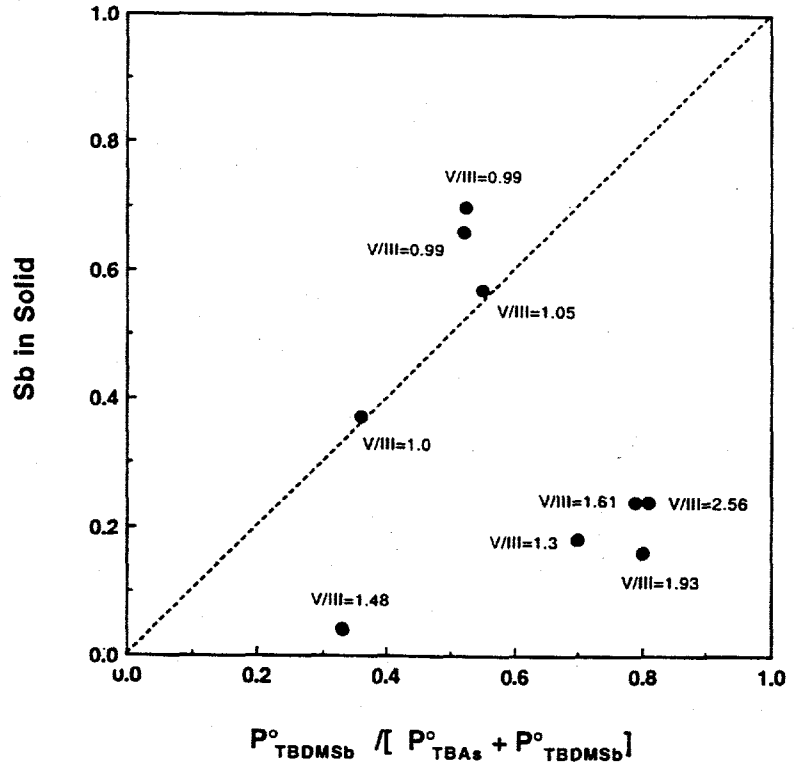
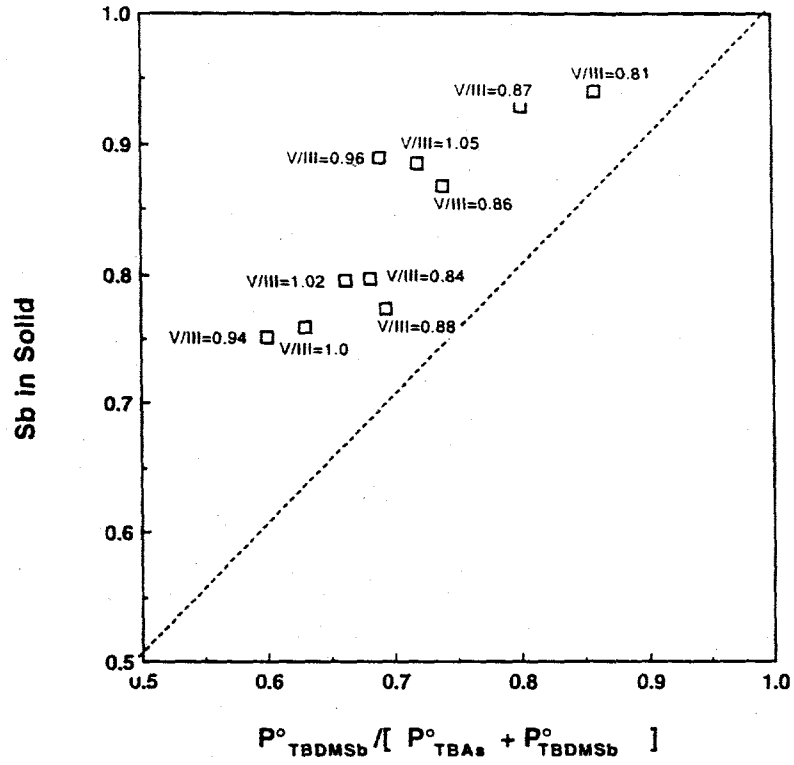
0.8
0.6
0.4
0.2
0.0
Sb in Solid
V/III=1.36
V/III=1.3
V/III=1.4
V/III=1.41
V/III=1.61
V/III=1.3
V/III=0.99
V/III=1.05
V/III=1.93
V/III=1.0
V/III=2.56
V/III=1.48
0.0
0.2
0.4
0.6
0.8
1.0
P°TBDMSb /[P°TBAs + P°TBDMSb]

(b)


1.0
0.8
0.6
0.4
0.2
0.0
Sb in Solid
V/III=0.99
V/III=0.99
V/III=1.05
V/III=1.0
V/III=1.61
V/III=2.56
V/III=1.3
V/III=1.93
V/III=1.48
0.0
0.2
0.4
0.6
0.8
1.0
P°TBDMSb /[P°TBAs + P°TBDMSb]

(a)
(b)
(c)
(d)
(e)
40 µm

(a)


Sb in Solid
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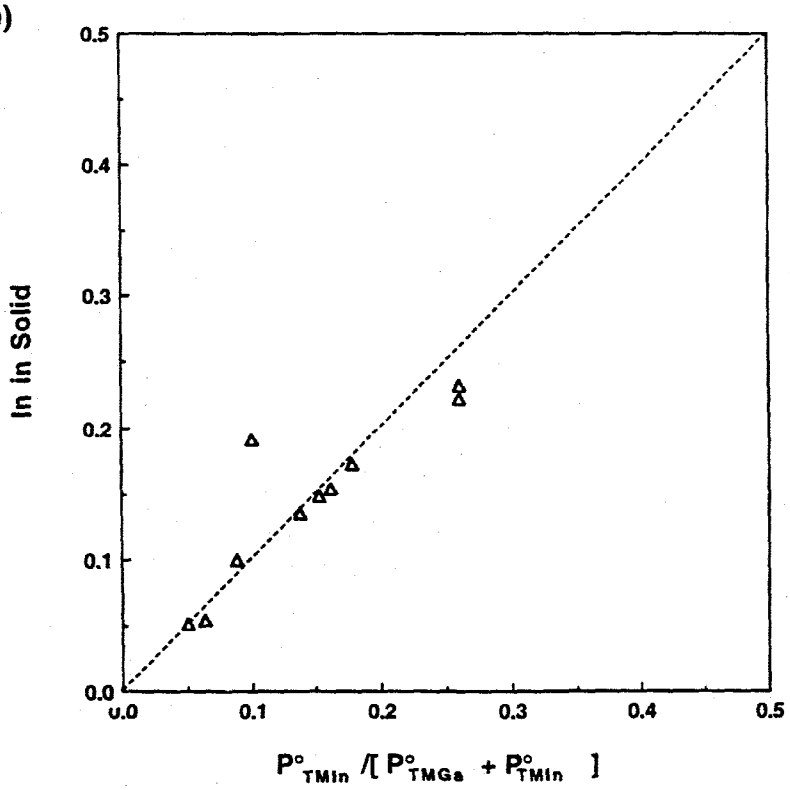
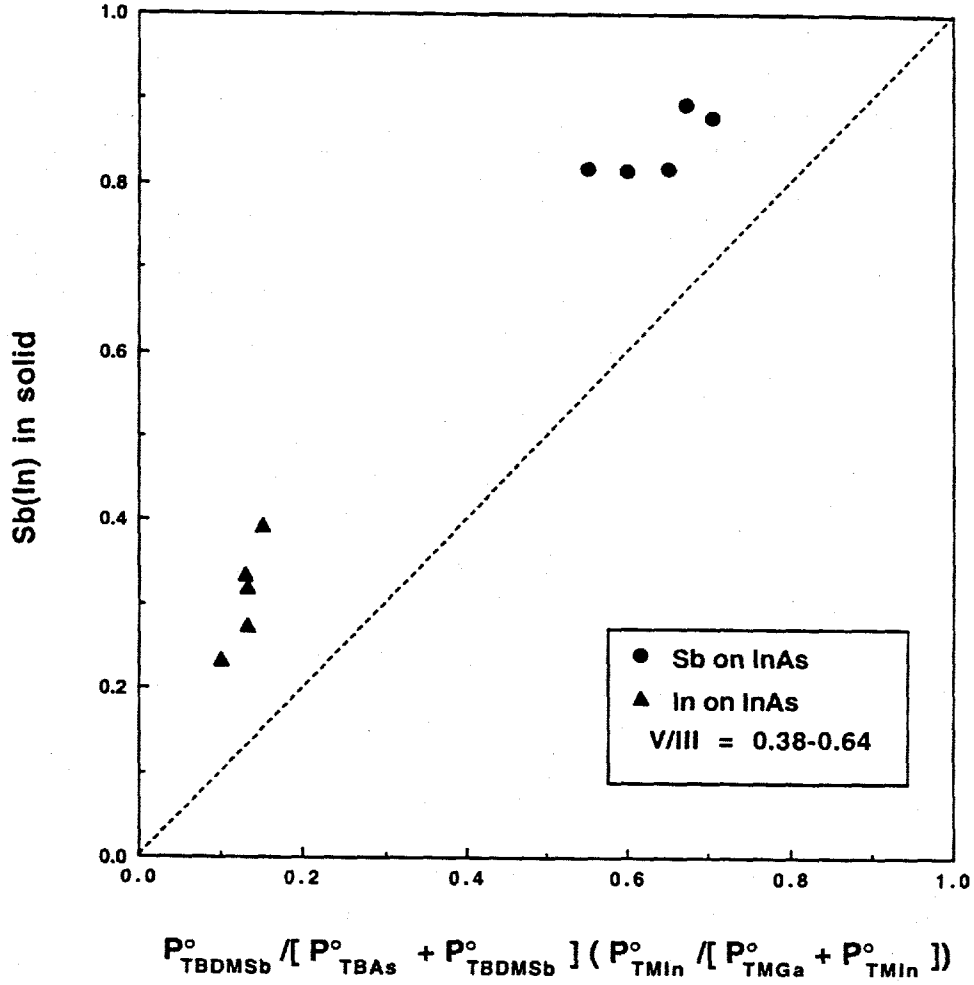
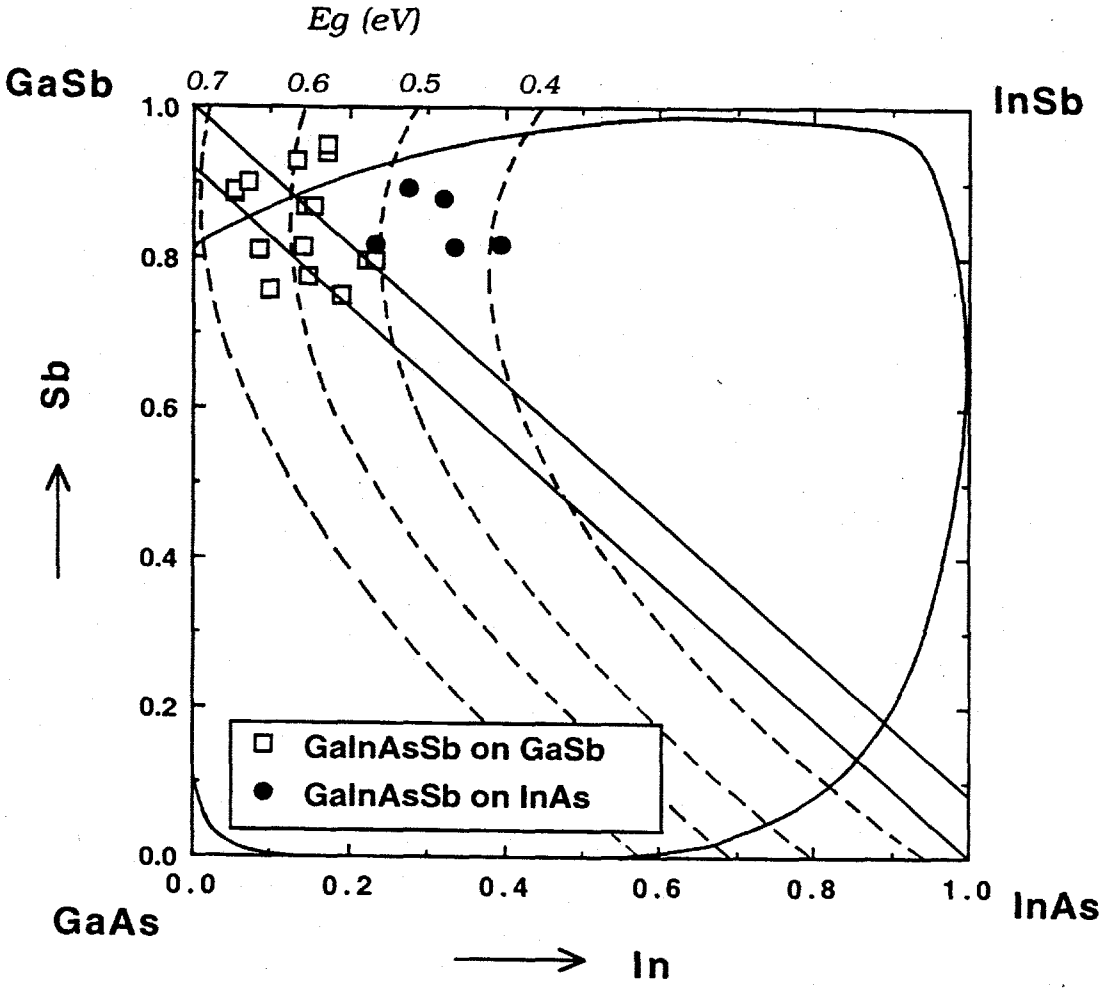
0.9
0.8
0.7
0.6
0.5
V/III=0.81
V/III=0.87
V/III=1.05
V/III=0.96
V/III=0.86
V/III=1.02
V/III=0.84
V/III=0.88
V/III=0.94
V/III=1.0
0.5
0.6
0.7
0.8
0.9
1.0
P°TBDMSb /[P°TBAs + P°TBDMSb]

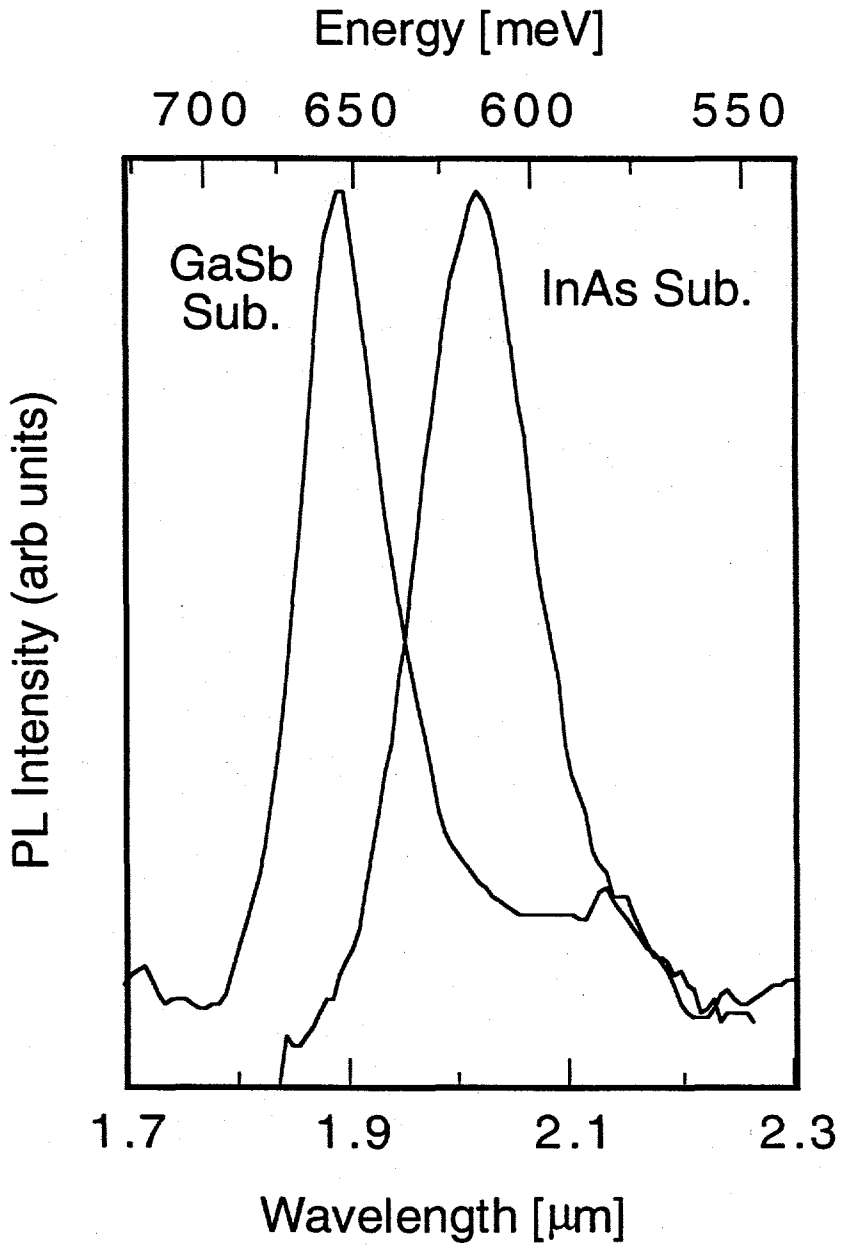
(b)


In in Solid
0.5
0.4
0.3
0.2
0.1
0.0
0.0
0.1
0.2
0.3
0.4
0.5
P°TMIn /[P°TMGa + P°TMIn]


1.0
0.8
0.6
0.4
0.2
0.0
0.0
0.2
0.4
0.6
0.8
1.0
Sb(In) in solid
Sb on InAs
In on InAs
V/III = 0.38-0.64
$P^{\circ}_{TBDMSb} / [P^{\circ}_{TBAs} + P^{\circ}_{TBDMSb}] (P^{\circ}_{TMIn} / [P^{\circ}_{TMGa} + P^{\circ}_{TMIn}])$

(a)
(b)
(c)
(d)
(e)
(f)
40 μm


Eg (eV)
0.7
0.6
0.5
0.4
GaSb
InSb
GaAs
InAs
Sb
In
1.0
0.8
0.6
0.4
0.2
0.0
0.0
0.2
0.4
0.6
0.8
1.0
GaInAsSb on GaSb
GaInAsSb on InAs


Energy [meV]
700
650
600
550
GaSb
Sub.
InAs Sub.
PL Intensity (arb units)
1.7
1.9
2.1
2.3
Wavelength [μm]