

Research Paper

Surface Morphology of AlSb on GaAs Grown by Molecular Beam Epitaxy and Real-time Growth Monitoring by *in situ* Ellipsometry

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Abstract AlSb is a promising material for optical devices, particularly for high-frequency and nonlinear-optical applications. We report the effect of growth temperature on structural properties of AlSb grown on GaAs substrate. In particular we studied the surface of AlSb with the growth temperature by atomic force microscopy, and concluded that optimized growth temperature of AlSb is 530°C. We also show the result of real-time monitoring of AlSb growth by *in situ* ellipsometry. The results of the structural study are good agreement with the previous reported ellipsometric data.

Keywords: AlSb, molecular beam epitaxy, surface morphology, real-time monitoring, ellipsometry

I. Introduction

Sb-based high-electron-mobility transistors (HEMTs) have emerged as the next-generation HEMTs with advantages over conventional GaAs- or InP-based HEMTs [1]. Whereas the latter are utilized to GaAs or In(Ga)As channels and AlGaAs or InAlAs barriers, Sb-based HEMTs is applied to InAs channels and Al(Ga)Sb barriers. The advantages of this material system include the high electron mobility ($\sim 30,000$ cm²/Vs) and velocity ($\sim 4 \times 10^7$ cm/s) of bulk InAs at 300 K [2]. Researchers have implemented high-speed HEMT ($< 30,000$ cm²/Vs) with this material system and reported reduced operation voltage and power at high speed [1]. Furthermore the large conduction band offset (1.35 eV) between InAs and AlSb [3,4] causes a strong confinement of electrons and a large *g*-factor [5]. As a result this InAs/Al(Ga)Sb material system has been utilized to implement 2 dimensional electron gases (2DEGs) for spintronics applications [5-7]. Other good point of Sb-based material system is that the mobility is larger than conventional GaAs, InP and Si. Recently, researchers of Naval Research Laboratory in USA succeeded in the fabrication of Sb-based n- and p-channel heterostructure field effect transistor for high-speed and low-power application [8,9].

Although theoretical calculation predicted that the mobility of InAs/Al(Ga)Sb HEMT can reach up to $> 70,000$ cm²/Vs, the mobility of these HEMTs is limited up to less than the half of the calculation. These results from

defects such as imperfection of interface [10]. The defects are attributed mainly to the formation of AlAs-like bonds between InAs and Al(Ga)Sb interface and the propagation from wafers. Also lattice mismatch between InAs and AlSb may be contributed to the defects. In the former case, a migration-enhanced epitaxy method was used between InAs and AlSb to achieve InSb-like bonds [11,12]. However there is a big huddle to solve the latter because there is no high-quality insulating substrate for InAs, GaSb, and AlSb so-called as 0.61 nm lattice constant system. Although InAs and GaSb wafers are commercially available, those are not insulating substrates. Up to now, the only way to acquire the insulating substrate is metamorphic growth of AlSb on GaAs. The role of AlSb layer is of prime importance in overcoming the lattice mismatch -8% between GaAs substrate and InAs/AlSb 2DEG channel-and thereby improving the transport characteristics of the HEMT structures. Although techniques such as incorporation of short period superlattices in AlSb layer [13] helped to increase the quality of the wafer, the study of the growth of AlSb on GaAs is critical to enhance the quality of wafer and HEMTs.

However ex-situ analysis of AlSb is very difficult because of its high reactivity to oxygen, and only few studies of AlSb have been reported. In this work we report the effects of growth temperature on structural properties of AlSb layer on GaAs. In particular, surface morphology of AlSb layer grown on GaAs with various growth temperatures is studied using x-ray diffraction (XRD) and atomic force microscopy (AFM). Also, we performed the real-time monitoring of AlSb growth using *in situ* spectroscopic ellipsometry (SE).

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II. Experimental

The samples were grown about 1.5 μm thick on semi-insulating (0 0 1) GaAs substrate using Riber Compact 21E solid-source molecular beam epitaxy (MBE) system. We used the rotating compensator SE with the charge-coupled device (CCD) to measure the ellipsometric parameter for real time monitoring of growth process, and strain free windows to prevent distortions of the polarization states of the incoming and outgoing beams. The angle of incidence was 69.8° . The surface oxide of GaAs substrate was removed by heating the sample to 620°C under As_2 flux. Then about 200 nm thick buffer layer of GaAs was subsequently grown at 580°C . Changing the temperature from 580°C to the growth temperature (T_g), As_2 flux was maintained with the shutter for Ga being closed and the reflection high-energy electron diffraction (RHEED) pattern remaining at (2×4) . Reaching the preset T_g , As shutter was promptly closed with Sb shutter opened. It resulted in the change of RHEED pattern from (2×4) to (1×3) . We also performed the SE measurement and compared with the change of RHEED pattern. In the subsequent growth process of AISb layer with the Al shutter opened, the RHEED pattern of 3D appeared for about 20 s before recovering to (1×3) . We performed the experiment at the various T_g from 455 to 555°C .

The growth rate of AISb and InAs were 0.32 ML/s and 0.23 ML/s, respectively. We used the Riber's valved cracker arsenic source with the tip temperature of 900°C to produce As_2 , and the Veeco's (formerly Applied EPI) valved cracker antimony source with the tip temperature of 900°C to produce Sb_2 . Beam equivalent pressure of As_2 is $530\sim 540^\circ\text{C}$. It is noted that this value of the RMS roughness is comparable to the typical value of 0.4 nm for the surface of GaAs substrate. At the T_g lower than 530°C the RMS roughness is seen to increase substantially with decreasing T_g -exhibiting a three-fold increase below 480°C . Moreover, in Fig. 3, closer examination of the AFM images for the T_g lower than 530°C appears "ripple" patterns lying parallel to the direction of $(0\ 0\ 4)$. The formation of these stripe-shaped ripples may have been caused by inhomogeneous bonding between Al and Sb atoms due to shorter mean-free-path of Sb atoms at below 530°C . The similar phenomenon was reported in Ref. 16. 1.5 nm thick GaSb on InAs with In-Sb like interface grown at 350°C -where the optimal temperature of GaSb on InAs is 400°C -shows ripples on the AFM image. Although the thickness of 1.5 nm is too thin compared with the one of 1.5 μm of AISb, the observation of the ripple with the large RMS value (~ 1.2 nm) only at lower T_g of 350°C and small Ga/Sb ratio (0.81) compared to unity support that this phenomenon is attributed to the short mean-free-path of Sb, because we can assume that the mean-free-path of Ga is nearly identical at both T_g of 350 and 400°C and the ripple is related to localized element with shorter mean-

III. Results and Discussion

Figure 1 show the real time measured SE spectra of AISb on GaAs in the growth process. In Fig. 1(a) the change of ellipsometric parameter Δ is observed. The Δ contains the thickness information of the sample, and it changes in a periodic from 0 to 180° with growth. The significant change of Δ at ~ 0.4 min means that the growth of AISb has been started. Figure 1(b) shows the changes of dielectric function with time from 0.74 to 6.45 eV. These changes mean phase transition from dielectric function of GaAs to AISb at ~ 0.44 min. Figure 2 show the observed RHEED patterns in the growth process. The observed RHEED pattern of GaAs is (2×4) , and the pattern changed into (1×3) with starting the growth of AISb. This means that the RHEED pattern is in agreement with the results of SE measurement.

The surfaces of AISb layers grown at the various T_g were measured by AFM as shown in Fig. 3. The root mean

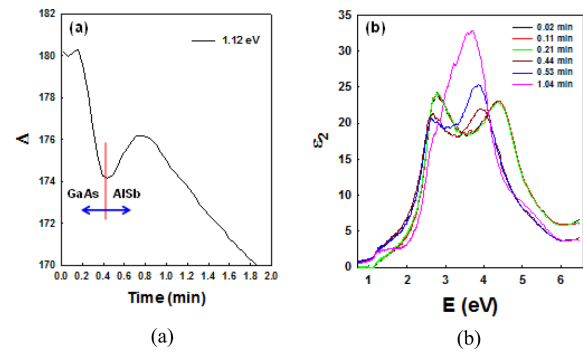


Figure 1. (a) Ellipsometric parameter Δ at 1.12 eV during AISb growth, (b) Pseudodielectric functions for different thicknesses of AISb on GaAs at growth temperature.

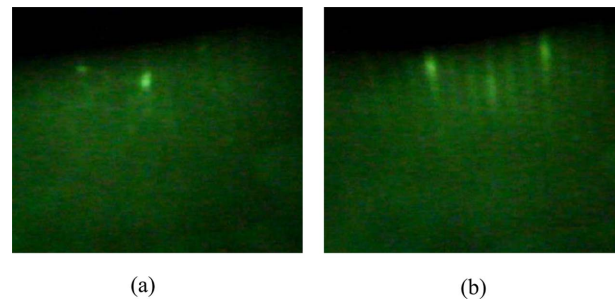


Figure 2. (a) RHEED pattern from GaAs substrate before AISb growth, (b) RHEED pattern from AISb layer after AISb growth.

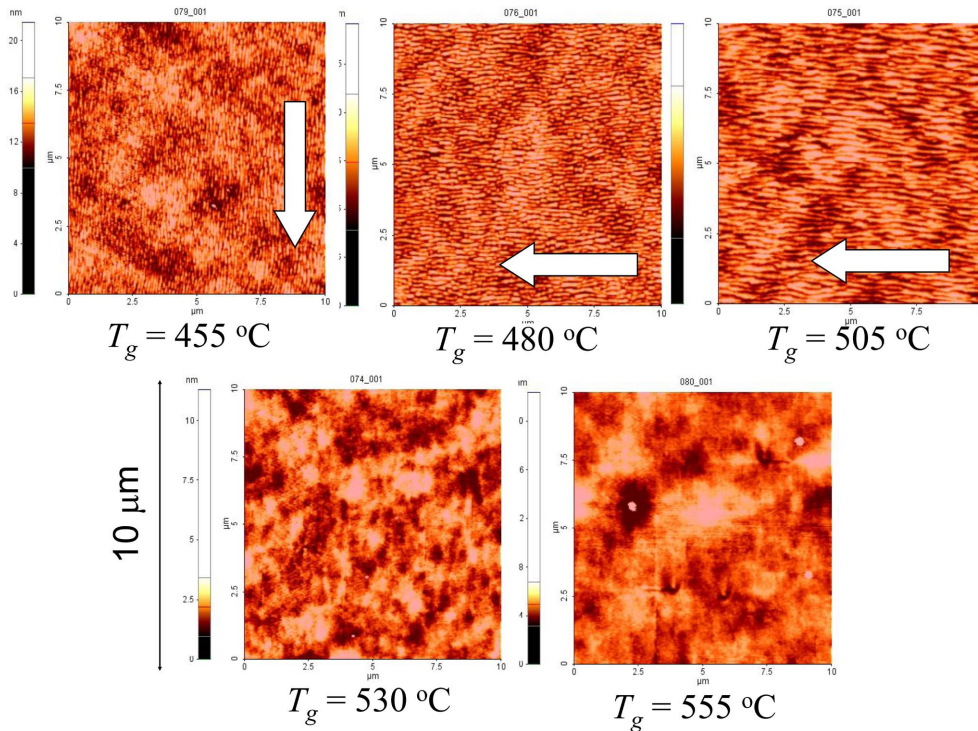


Figure 3. 10 μm × 10 μm AFM images of 1.5 μm thick AlSb layers grown on GaAs at the various growth temperatures (T_g). No ripples were found at $T_g \geq 530^\circ\text{C}$. The arrows in the figures show the direction of ripples laying parallel to (1 1 0).

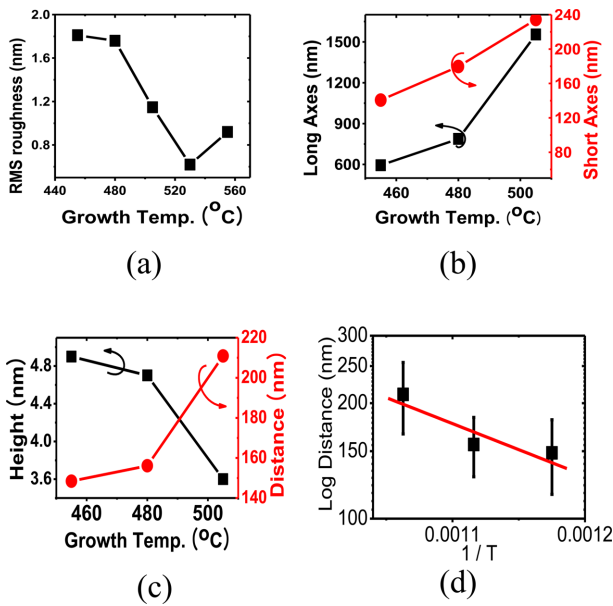


Figure 4. (a) Plot of the RMS roughness versus T_g , (b) plot of length (long axis) and width (short axis) of the observed ripples versus T_g below 530°C , and (c) plot of height and distance of ripples versus T_g below 530°C . No ripples were found at $T_g \geq 530^\circ\text{C}$. (d) Arrhenius plots of distance between ripples vs. growth temperature in K for 460-500°C grown samples.

free-path, which, in turns, shows lower Ga/Sb ratio. It is also mentioned that Ga/Sb ratio is nearly unity (~0.98) at $T_g = 400^\circ\text{C}$ with flat GaSb/InAs surface at this T_g [16].

The average lengths of long- and short-axes of the ripples were plotted versus the T_g below 530°C in Fig. 4(b). The average height and the distance between ripples are

also plotted in Fig. 4(c). In Fig. 4(b) and Fig. 4(c) the average size of ripples and the separation distance between them decrease with decreasing T_g below 530°C while the average height increase. These result in more uneven surface with the greater RMS roughness. This phenomenon of local piling up of AlSb reflects that the materials on the surface have a lower energy to travel across the surface at the lower T_g .

On the other hand, at the T_g higher than 530°C , the appearance of drops and dislocations was observed in the AFM images in Fig. 3. The formation of these defects is the consequence of re-evaporation of Sb atoms from the surface leading to anion deficiency, i.e. V/III ratio less than unity. In addition, the RMS roughness at the T_g higher than 530°C increases in Fig. 3(a).

According to the results of surface-morphological study, we conclude that the most appropriate T_g is $\sim 530^\circ\text{C}$ for the growth of AlSb on GaAs. Here it should be pointed out that our result is a good agreement with the result of the researchers in Naval Research Laboratory [14,15] because we used the same Sb valved cracker and the same flux of Sb_2 . Therefore if the flux of Sb_2 and type of Sb source are modified, the optimal T_g will be changed. However the trend of surface roughness as the function of T_g revealed in the article will be valid again. In addition it is a good agreement that the optimized T_g is 530°C by previous reported SE data of AlSb [17]. From the figure 4(d), the activation energy of surface diffusion can be extracted using the following equation; $D = D_0 \exp(-E_s/(K_b T))$ where D , D_0 , E_s , K_b and T stand for distance between ripples, constant, surface diffusion activation energy,

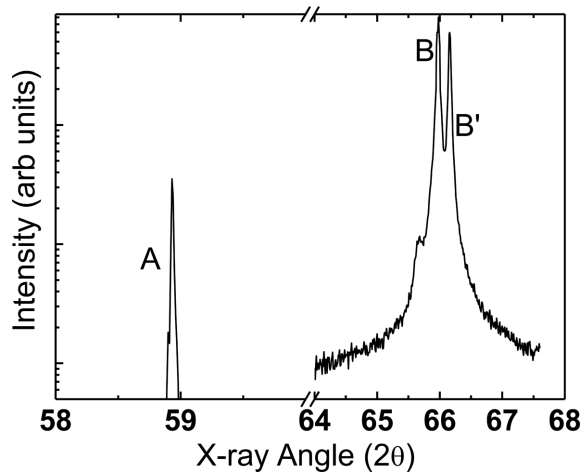


Figure 5. XRD curve of 1.5 μm thick AlSb layer grown on GaAs buffer layer.

substrate temperature in K, respectively. Calculated E_s is 1.356 eV from AlSb ripples.

XRD measurement of the (0 0 4) reflection for typical, 1.5 μm thick AlSb layer on GaAs is shown in Fig. 5. The largest double peaks (marked as B and B') near 66° are from GaAs. The splitting is caused by $K_{\alpha 1}$ and $K_{\alpha 2}$ lines of unfiltered x-ray source, and the dips within the peaks are due to saturation of the detector. The single, strong peak at 58.9° (marked as A) is from AlSb. Since the simulated position of (0 0 4) peak is 60.3° for single-crystalline AlSb with 100% relaxation while the theoretical estimation of that is 55.6° for fully-strained AlSb on GaAs substrate with 0% relaxation, the observed peak at 58.9° in Fig. 5 corresponds to a coherent relaxation of about 71%. This value is smaller than that of Ref. 14 (~89%), and comparable with that of Ref. 18 (~70%). Variations of the T_g in the range of $455\sim 555^\circ\text{C}$ did not have any noticeable effect on XRD spectra. In the fabrication of all 2DEGs, the T_g of 530°C was therefore used for the growth of AlSb on GaAs.

Also the full width at half maximum of XRD peak for 1.5 μm thick AlSb is $\sim 0.02^\circ$. This result is comparable with the results of Ref. 15 for 1.7 μm thick AlSb with 0.0638° of peak and Ref. 19 for 1.0 μm thick AlSb with 0.13° of peak.

IV. Conclusions

Antimonide-based InAs/AlSb HEMT structures were fabricated using MBE and morphological study of AlSb buffer layers and transport properties of various structures were carried out. We performed the real-time monitoring of AlSb growth by using SE measurements, and it is a good agreement with the results of RHEED pattern. This fact proves the importance and the sensitivity of SE technique

for the real-time monitoring of film growth using the ellipsometry. From morphological study by AFM, it is concluded that AlSb layer on GaAs has the smoothest surface with the RMS roughness of 0.619 nm when the growth temperature is 530°C . Drops and misfit dislocations are developed when the growth temperature is higher than 530°C , and stripe-shaped pattern of ripples are observed below 530°C . Analysis of XRD pattern measurements yield about 71% relaxation of AlSb layer grown on GaAs buffer layer. We compared this structural study of AlSb with the previous reported SE data, and concluded a good agreement that the optimized temperature of growth is 530°C . These results will be useful database for Sb-based optoelectronic applications and high speed devices.

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References

- [1] B. R. Bennett, R. Magno, J. B. Boos, W. Kruppa, and M. G. Ancona, *Solid-state Electronics*, 49, 1875 (2005); this paper reviews Sb-based semiconductors for electronic devices.
- [2] Z. Dobrovolskis, K. Grigoras, and A. Krotkus, *Appl. Phys. A: Solids Surf.* 48, 245 (1989).
- [3] I. Vurgaftman, J. R. Meyer, and L. R. Ram-Mohan, *J. Appl. Phys.* 89, 5815 (2001).
- [4] A. G. Milnes and A. Y. Polyakov, *Mater. Sci. Eng. B* 18, 237 (1993).
- [5] Yu. G. Sadofyev, A. Ramamoorthy, B. Naser, J. P. Bird, S. R. Johnson, and Y-H. Zhang, *Appl. Phys. Lett.* 81, 1833 (2002).
- [6] P. R. Hammar, and M. Johnson, *Phys. Rev. Lett.* 88, 066806-1 (2002).
- [7] A. Zakharova, I. Lapushkin, K. Nilsson, S. T. Yen, and K. A. Chao, *Phys. Rev. B* 73, 125337 (2006).
- [8] J. B. Boos, B. R. Bennett, N. A. Papanicolaou, M. G. Ancona, J. G. Champlain, R. Bass, and B. V. Shanabrook, *Electronics Letters* 43 (2007) 834.
- [9] B. R. Bennett, M. G. Ancona, and J. B. Boos, *Mrs Bulletin* 34 (2009) 530.
- [10] H. Rodilla, T. González, D. Pardo, and J. Mateos, *J. Appl. Phys.* 105, 113705 (2009).
- [11] G. Tuttle, H. Kroemer and J. H. English, *J. Appl. Phys.* 67, 3032 (1990).
- [12] B. R. Bennett, B. V. Shanabrook, and E. R. Glaser, *Appl. Phys. Lett.* 65, 598 (1994).
- [13] S. H. Shin, J. Y. Lim, J. D. Song, H. J. Kim, S. H. Han, and T. G. Kim, *J. Korean Phys. Soc.* 53, 2719 (2008).
- [14] B. P. Tinkham, B. R. Bennett, R. Magno, B. V. Shanabrook, and J. B. Boos, *J. Vac. Sci. Technol. B* 23, 1441 (2005).
- [15] B. R. Bennett, B. P. Tinkham, J. B. Boos, M. D. Lange, and R. Tsai, *J. Vac. Sci. Technol. B* 22, 688 (2004).
- [16] A. Tahraroui, P. Tomasini, L. Lassabatère, and J. Bonnet, *Appl. Surf. Sci.* 162-163, 425 (2000).
- [17] Y. W. Jung, T. H. Ghong, J. S. Byun, Y. D. Kim, H. J. Kim, Y. C. Chang, S. H. Shin, and J. D. Song, *Appl. Phys. Lett.* 94, 231913 (2009).
- [18] Y. C. Lin, H. Yamaguchi, E. Y. Chang, Y. C. Hsieh, M. Ueki, Y. Hirayama, and C. Y. Chang, *Appl. Phys. Lett.* 90, 023509 (2007).
- [19] C. Nguyen, B. Brar, C. R. Bolognesi, J. J. Pekarik, H. Kroemer, and J. H. English *J. Electronic Materials* 22, 255 (1993).