

Effect of growth interruption time on the quality of InAs/GaSb type-II superlattice grown by molecular beam epitaxy*

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We systematically investigate the influence of growth interruption time on the properties of InAs/GaSb type-II superlattices (T2SLs) epitaxial materials grown by molecular beam epitaxy (MBE). X-ray diffraction (XRD) and atomic force microscope (AFM) are used to characterize the material quality and morphology. The full width at half maximum (*FWHM*) of the XRD 0th satellite peaks ranges from 32" to 41", and the root mean square (*RMS*) roughness on a 5 $\mu\text{m} \times 5 \mu\text{m}$ scan area is 0.2 nm. Photoluminescence (PL) test is used to reveal the influence of the growth interruption time on the optical property. Grazing incidence X-ray reflectivity (GIXRR) measurements are performed to analyze the roughness of the interface. The interface roughness (0.24 nm) is optimal when the interruption time is 0.5 s. The crystal quality of T2SLs can be optimized with appropriate interruption time by MBE, which is a guide for the material epitaxy of high performance T2SL infrared detector.

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InAs/GaSb type-II superlattices (T2SLs) have emerged as one of the top candidates for the third-generation infrared (IR) detection technology^[1-3]. The InAs/GaSb heterostructure presents an unusual type-II "misaligned" band alignment in that the top of the valence band of GaSb is higher than the minimum conduction band of InAs located at 150 meV^[4,5]. Thus, there is a tunable energy band, leading to a broad wavelength spectrum ranging from the mid wavelength infrared (MWIR) to the very long wavelength infrared (VLWIR) regime controlled by the thickness ratio of InAs and GaSb. The reduction of the Auger recombination rate due to the spatial separation of electrons and holes in the T2SL decreases the dark current, which makes it possible for the device to operate at high temperature^[6,7].

The accurate thickness of binary materials can be obtained by switching the shutter of the source furnace of the molecular beam epitaxy (MBE) system. There is no common atom between InAs and GaSb layers, and "Ga-As" or "In-Sb" bonds may be formed at the alternate interfaces (IFs)^[8-10]. InSb and GaAs interfaces have different effects on the quality of the T2SLs material^[11,12].

Besides, SRH defects which are related to minority carrier lifetime are generally considered to be generated during material growth^[13-15]. Therefore, the careful control of the shutter sequence is very important to the overall performance of InAs/GaSb T2SLs. Some studies on interface control method have been reported to improve the quality of the epitaxy layer, such as interrupted growth methods, V-group (Sb or As) soak methods, and they are mainly concentrated on the device or superlattices performance under the corresponding shutter sequence^[16,17]. However, there are few discussions related to the optimization of shutter sequence time.

In this paper, we systematically analyzed the quality of the InAs/GaSb T2SL for the MWIR range by varying the growth interruption time which is an important factor in the shutter sequence control. Firstly, we introduce the growth process of the InAs/GaSb T2SLs in detail. Then we study the effect of growth interruption time on the structural and optical characteristics of T2SLs.

The InAs/GaSb T2SL materials are grown on n-type doped GaSb(100) substrates by a multi-chamber ultra-high vacuum MBE system, equipped with thermal

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evaporation cells of In and Ga and valved cracker cells of As and Sb. Firstly, the GaSb substrate is deoxidized at 520 °C for 10 min, and a 500-nm-thick GaSb buffer layer is grown at 460 °C. Then 120 periods of superlattice with 9 monolayers of InAs and 8 monolayers of GaSb are deposited at 380 °C, which is approximately 10 °C higher than the 1×3 to 2×5 surface reconstruction temperature, recognized from the reflection high-energy electron diffraction (RHEED), as shown in Fig.1. The beam equivalent pressure (BEP) of Sb is 4.07×10^{-6} mbar, and the V/III BEP flux ratio is 4 during the GaSb growth. The BEP of As is 5.74×10^{-6} mbar, and the V-III BEP flux ratio is 8 during the InAs growth.

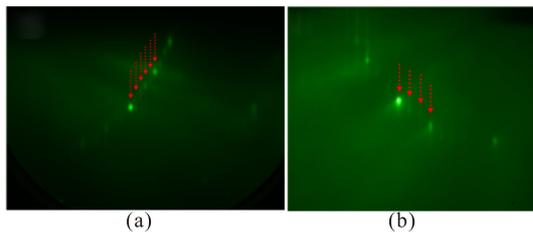
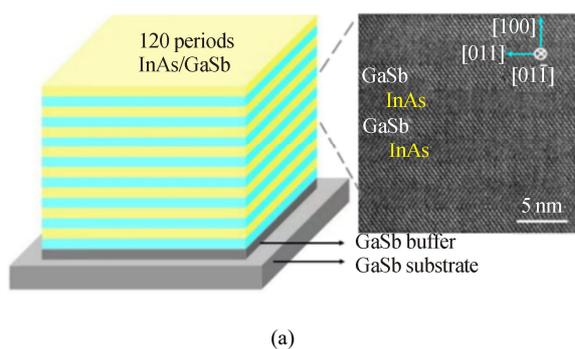


Fig.1 RHEED patterns of (a) GaSb \times 5 reconstruction and (b) GaSb \times 3 reconstruction

Fig.2(a) is the schematic diagram of the InAs/GaSb T2SLs multilayer heterostructure. InAs and GaSb layers can be clearly seen from the transmission electron microscope (TEM) image on the right of Fig.2(a). The shutter sequence for the T2SLs growth is shown in Fig.2(b). The GaSb epitaxial layers are soaked by Sb for 6 s, followed by an artificially added InSb interface layer. The InAs epitaxial layers are followed by As soak for 6 s. To reveal the regulation of interruption time on the quality of T2SLs, different growth interruption time (0 s, 0.5 s, 1.0 s, 1.5 s) is implemented for superlattices growth before InAs and GaSb layer epitaxy, and samples are denoted as A, B, C, and D, respectively.

Fig.3(a) shows the high-resolution XRD spectra of the 2θ - ω scan around the GaSb (004) reflection for four samples. The fifth satellite peak exists, and the satellite peak is sharp, indicating excellent crystal quality. Fig.3(b) displays the changes of substrate peak's full width at half maximum (*FWHM*), 0th satellite peak's *FWHM*, strain and period thickness with the interruption time. The *FWHM* of the 0th satellite peak for Sample B



(a)

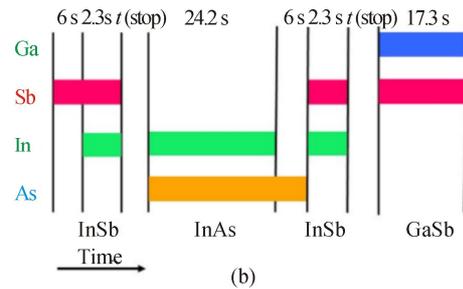


Fig.2 (a) Schematic of InAs/GaSb T2SLs epitaxial structure; (b) Diagram of the shutter sequence during epitaxy growth of InAs/GaSb T2SLs

measured in Ω mode is $32''$, confirming a better crystal quality. The T2SL structures suffer from compressive strain. It can be regulated by controlling the amount of InSb, and the strain adjustment was discussed in our previous reports^[18]. As the interruption time increases, the period thickness is roughly the same, slightly decreasing.

The surface morphology has been observed by atomic force microscope (AFM) on a $5 \mu\text{m} \times 5 \mu\text{m}$ scan area, as shown in Fig.4, and all the samples showed a minor root mean square (*RMS*) roughness of 0.2 nm. The atomic steps can be observed, and the width is about $1 \mu\text{m}$. The white embedded diagram shows the height change in the direction of atomic steps. For Sample B, the atomic steps are more uniform and the edges are more continuous.

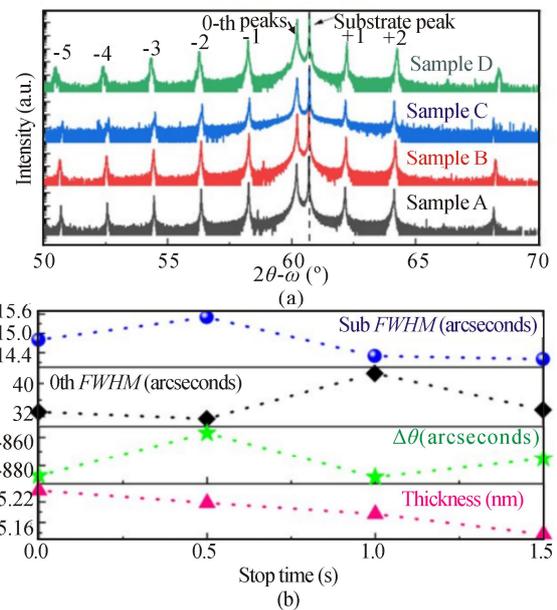


Fig.3 (a) XRD patterns of the 2θ - ω scan around the GaSb (004) reflection for four samples; (b) Changes of substrate *FWHM*, 0th *FWHM*, strain and period thickness with the interruption time

Fig.5 shows the grazing incidence X-ray reflectivity (GIXRR) curve with different interruption time. GIXRR measurements are performed on a high-resolution X-ray diffractometer. It can characterize the thickness and roughness of samples by using the refraction and reflection of X-ray in materials and the mutual interference between reflected rays. In Fig.5(a), the reflection intensity decreases as

the incident angle increases. The reflection intensity is very sensitive to the roughness of the interface, and the rough interface causes X-ray scattering. For Sample B and Sample D, the reflection strength decreases more slowly than that of Sample A and Sample C. The intensity of the Bragg peak of Sample B is stronger, and there still exist prominent Bragg peaks in large reflex angles. Moreover, as shown in Fig.5(b), the interface roughness is the lowest of 0.24 nm when the interruption time is 0.5 s, according to fitting results from the curve of Fig.5(a).

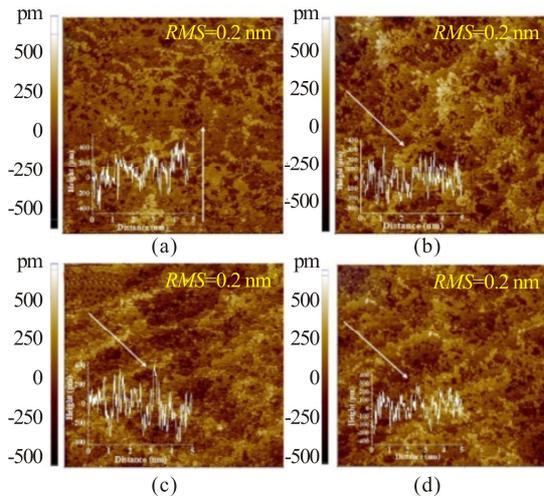


Fig.4 AFM morphology images for 5 μm×5 μm area: (a) Sample A; (b) Sample B; (c) Sample C; (d) Sample D

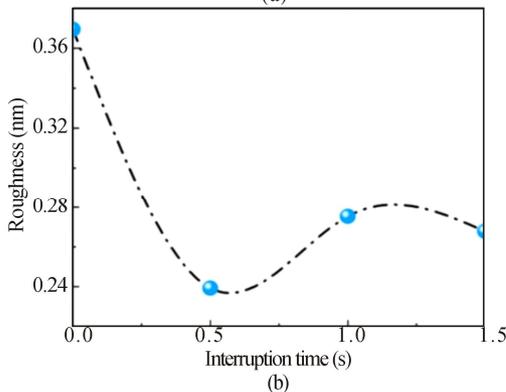
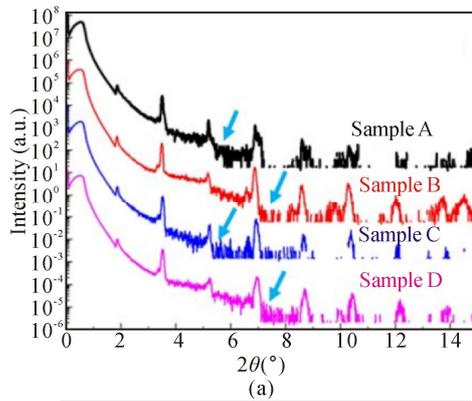


Fig.5 (a) GIXRR curves with different interruption time; (b) Change of interface roughness with interruption time

The photoluminescence (PL) spectra at 77 K for different interruption time are displayed in Fig.6. The wavelength of superlattice first decreases and then increases with the interruption time. Different growth interruption time brings subtle changes in the interface structure, and the interface structure may affect the superlattice's effective band gap^[4]. The PL spectrum can also reflect the quality of the interface. The *FWHMs* of PL are about 30—48 meV. When the interruption time is 0.5 s, the value of PL *FWHM* is lower and PL spectrum curve is smooth. The slight irregular PL spectrum of Sample D may be related with the improper settings of integration time, chopper frequencies, and band pass frequency^[19].

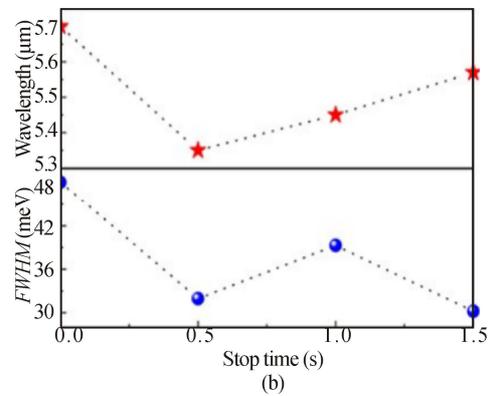
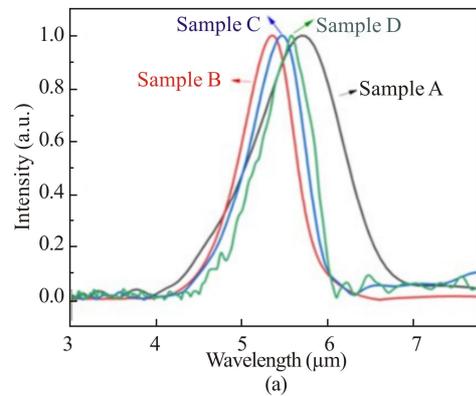


Fig.6 (a) Normalized PL spectra at 77 K; (b) PL *FWHM* and wavelength for different interruption time

In conclusion, we systematically investigate the influence of growth interruption time on the quality of InAs/GaSb T2SLs, which is an important factor in the precise control of epitaxy growth. The crystal quality, surface morphology, interface roughness, and optical characteristics of four samples with different interruption time are studied by GIXRR, HRXRD, AFM and PL spectroscopy. The optimized growth interruption time is 0.5 s, and the *FWHM* of XRD 0th satellite peak and PL spectrum are small. The GIXRR reflection intensity attenuation is the slowest, and it has an atomic flat surface morphology at this time. It is of great significance to optimize the shutter sequence for the precise control of material growth, and the discussion of the interruption time provides controllable growth parameters for the

epitaxy growth of MBE in detail.

Statements and Declarations

The authors declare that there are no conflicts of interest related to this article.

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