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Growth and characterization of an InSb infrared photoconductor on Si via an AlSb/GaSb buffer

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ABSTRACT

A 99.6% relaxed InSb layer is grown on a 6° offcut (100) Si substrate via an AlSb/GaSb buffer using molecular beam epitaxy (MBE). A 200 nm GaSb buffer is first grown on Si and the lattice mismatch between them is accommodated by an interfacial misfit (IMF) array consisting of uniformly distributed 90° misfit dislocations. Si delta doping is introduced during the growth of GaSb to reduce the density of threading dislocation. Subsequently, a 50 nm AlSb buffer is grown followed by a 0.8 μm InSb layer. The InSb layer exhibits a 300 K electron mobility of 22,300 cm²/Vs. An InSb photoconductor on Si is demonstrated with a photoconductive gain from 77 K to 200 K under a 700 °C maintained blackbody.

1. Introduction

Indium antimonide (InSb) has a 300 K bandgap of 0.17 eV, which enables its application in the mid-infrared region (3-8 μm). Commercial InSb photodetectors have been widely used in the environmental monitoring, defense, thermal imaging and biomedicine [1]. The heteroepitaxial growth of InSb on Si can overcome the restrictions of the InSb substrate, which are its expensiveness, small size (< 2 inches in diameter), and low ruggedness [2]. The monolithic integration of InSb devices on Si is also one approach to realize emerging mid-infrared Si photonics. Mid-infrared Si photonics can extend the current operating wavelength (near-infrared) of Si photonics to mid-infrared, which provides additional channels for on-chip communication and enables Si photonics to become a fundamental platform of lab-on-chip technology [3-5].

Direct epitaxial growth of single crystalline InSb on (100) Si is challenging due to a weak chemical bond between In, Sb, and Si, which results in the formation of metallic In clusters instead of a continuous InSb layer on (100) Si [6]. Previously reported InSb grown on Si utilized GaAs and Ge as an intermediate buffer layer. However, their lattice mismatch is still high (14.6% for InSb/GaAs and InSb/Ge) and the quality of InSb is compromised [2, 7]. AlSb and GaSb are alternatives for intermediate buffers because of their ~6.3% lattice mismatch with InSb. Furthermore, the resistivity of AlSb is sufficiently high for the characterization of InSb electrical properties [8]. Moreover, GaSb and lattice-matched InAs are important material for the high electron mobility transistor (HEMT) [9], therefore, the development of the AlSb/GaSb buffer on (100) Si is also an approach to integrate InSb-based photonic devices and InAs- or GaSb-based HEMTs together on a Si platform.

The lattice mismatch between GaSb and Si can be accommodated by the interfacial misfit (IMF) array technique, where uniformly distributed 90° misfit dislocations are formed at the interface under carefully controlled growth conditions [10, 11]. A perfect IMF array can completely avoid the formation of threading dislocation because all strain energy caused by lattice mismatch is relieved by 90° misfit dislocations which are sessile on the (110) plane [12], however, the threading dislocation density of GaSb grown on Si using IMF array was reported to be $1 \times 10^8 \text{ cm}^{-2}$ [13]. The formation of threading dislocation is attributed to 1) the 60° misfit dislocation forms at the edge of islands under the Volmer-Weber growth mode [14, 15]; 2) one 90° misfit dislocation split into two 60° ones during the growth [16]. Therefore, other techniques to reduce the dislocation density are required because a high quality GaSb buffer is very important for the subsequent growth of InSb. It was reported that dopants effectively reduced the density of dislocation in the GaSb/GaAs [17], GaN/sapphire [18], GaN/Si [19] and Ge/Si [20]. Introducing doping impurities during layer growth limited the dislocation movement to prevent it to thread the entire layer [21, 22] or changed the orientation of dislocation movement to promote annihilation [19]. In this study, Si delta doping was introduced during the growth of GaSb on Si to reduce the density of threading dislocation. Si is a desirable dopant due to its non-toxic and highly purified Si source.

In this work, a 0.8 μm InSb layer was grown on a 6° offcut (100) Si substrate via an AlSb/GaSb buffer using molecular beam epitaxy (MBE), and InSb photoconductors on Si were fabricated and measured. An offcut Si substrate was used to suppress the anti-phase boundary (APB) caused by the growth of polar III-V compound semiconductors on non-

polar Si [23]. The AlSb/GaSb buffer consists of a 5 nm AlSb prelayer on Si followed by 200 nm GaSb and 50 nm AlSb. The AlSb prelayer assists the formation of an IMF array at the GaSb/Si interface, and the top AlSb layer is used as a surface for the subsequent InSb growth. The growth process of GaSb on Si was optimized via introducing different Si delta doping concentrations. The lattice mismatch between InSb and AlSb is accommodated by the interfacial misfit dislocations.

2. Experimental details

Fig. 1. Schematic of (a) GaSb grown on (100) Si substrates with Si delta doping, (b) InSb grown on (100) Si substrates via an AlSb/GaSb buffer.

In this study, the experiment included two sets. The first set was optimizing the growth of GaSb on Si via Si delta doping. The second set was growing InSb on Si via an AlSb/GaSb buffer. In the first set, 200 nm GaSb layer was grown on (100) Si substrates with 6° offcut and Si delta doping was inserted at 30 nm, 40 nm, and 50 nm above the GaSb/Si interface as shown in Fig. 1(a). To obtain the optimized doping concentration, four samples were grown with different doping concentrations (0 , $1 \times 10^{12} \text{ cm}^{-2}$, $5 \times 10^{12} \text{ cm}^{-2}$, and $1 \times 10^{13} \text{ cm}^{-2}$). The Si substrate was etched with hydrofluoric acid (HF) to remove surface oxide. Then it was transferred to the a solid-state MBE system equipped with an antimony (Sb) valved

cracker to provide Sb_2 and heated to 950°C for 30 mins to form double layer steps on the Si surface, which is essential for the suppression of APB in compound semiconductors on Si [23]. All growth process was monitored using an in-situ high-energy electron diffraction (RHEED). After heating, a (2×2) (RHEED) pattern was observed, which indicated the formation of two double layer (biatomic) steps on a (100) Si surface [24, 25]. The temperature of the substrate was lowered to 560°C at $30^\circ\text{C}/\text{min}$ and the Si substrate was exposed to Sb_2 flux with a beam-equivalent (BEP) flux of about 1.2×10^{-6} Torr. After 4 min of Sb exposure, 5 nm AlSb was grown on Si with a growth rate of 0.1 monolayer per second and a V/III ratio of 10. During the growth of AlSb, the RHEED patterns were spotty, which indicated the formation of AlSb islands on Si.

Subsequently, the substrate temperature was lowered to 430°C at $20^\circ\text{C}/\text{min}$ under Sb overpressure for the growth of GaSb. The growth rate of GaSb was $0.6\ \mu\text{m}/\text{hrs}$ and V/III ratio was 5.5. At the initial growth stage, the RHEED pattern gradually changed from spotty to clear (1×3) in about 1 min indicating a switch from an island-like growth mode to a layer-by-layer one. After the growth of 30 nm GaSb, the shutter of Ga source was closed to interrupt the growth while the Sb valve was still opened to remain the Sb overpressure. The shutter of Si source was open for the delta doping. Different doping concentrations of 0, $1\times 10^{12}\ \text{cm}^{-2}$, $5\times 10^{12}\ \text{cm}^{-2}$, and $1\times 10^{13}\ \text{cm}^{-2}$ were introduced through controlling the time of Si exposure. During the delta doping, Ga shutter was closed. As shutter was opened to supply As flux. Si shutter was opened to supply Si atoms to the surface of sample. These Si atoms was bonded with As atoms and act as donors. The doping concentration depends on the opening time of Si shutter. After the doping, Si shutter was closed and Ga shutter was

opened to continuously grow GaSb. The other two delta dopings were carried out when the GaSb layer thickness was 40 nm and 50 nm, respectively, as shown in Fig. 1(a). For the same sample, the concentrations of three delta dopings keep consistent. The structural properties of samples were characterized using x-ray diffraction (XRD) and atomic force microscope (AFM).

In the second set of experiment, an 800 nm InSb layer was grown on a (100) Si substrate via a 250-nm-thick AlSb/GaSb buffer and the heteroepitaxial structure is shown in Fig. 1(b). In the first of experiment, the growth condition of GaSb on Si were optimized. 200 nm GaSb buffer was grown on Si under this optimized condition. Subsequently, the 50 nm AlSb was grown at 430 °C with a growth rate of 0.55 $\mu\text{m/hr}$ s and a V/III ratio of 10. The RHEED pattern remained as (1 $\times$ 3). Finally, the substrate temperature was lowered to 345 °C at 20 °C/min under Sb overpressure. A 0.8 μm InSb was grown on the AlSb surface with a growth rate of 0.73 $\mu\text{m/hr}$ s and a V/III ratio of 3. At the initial growth stage of InSb, a spotty RHEED pattern was observed and it changed to a clear (1 $\times$ 3) pattern after about 1 min of growth. The structural and electrical properties of InSb on Si were characterized using XRD, transmission electron microscopy (TEM) and Hall measurement. InSb photoconductors were fabricated using a standard metal lift-off technique with electron beam-evaporated Au/Ti (80 nm/30 nm) to form interdigitated electrodes on the InSb surface. The InSb photoconductors were mounted in a liquid nitrogen cooled optical cryo-stage for the measurement of photoresponsivity at different temperatures.

3. Results and discussion

Fig. 2. (a) Symmetric (004) ω - 2θ scans for GaSb on Si with different delta doping concentrations. (b) Doping concentration dependent full-width-half-maximum (FWHM) of (004) ω scans for GaSb on Si. (c) Doping concentration dependent r.m.s. surface roughness for GaSb on Si and the insets are AFM images (20 $\mu\text{m} \times 20 \mu\text{m}$).

Fig. 2(a) shows the (004) ω - 2θ scan of three doped samples and one non-doped sample. The relaxations of four samples are all nearly 100 % relaxed, therefore, the doping has no influence on the relaxation of GaSb layer on Si. Fig. 2(b) compares the full-width-half-maximum (FWHM) of (004) ω scans of four samples and the sample with the delta doping concentration of $5 \times 10^{12} \text{ cm}^{-2}$ has the lowest FWHM. **The delta doping process has been described in the section of experimental details.** The presence of threading dislocations broadens the FWHM of the symmetric (004) ω scan [26, 27]. Therefore, the threading

dislocation density in the sample with the delta doping concentration of $5 \times 10^{12} \text{ cm}^{-2}$ should be lowered than other samples. Compared with the non-doped samples, its FWHM is decreased by 12.2%. Fig. 2(c) shows the doping concentration dependent root mean square (r.m.s.) surface roughness of GaSb layers on Si. The insets are the AFM images of the samples. All samples exhibit a surface with a high density of small fluctuations which is attributed to the island-growth mode at the initial growth stage of GaSb on Si. Although the r.m.s. roughnesses of doped samples (3~4 nm) are slightly higher than that of non-doped sample (2.4 nm), this difference is negligible. Therefore, the doped samples are suitable for the subsequent growth of AlSb and InSb layers. In short, the optimal delta doping concentration for the growth of GaSb on Si is $5 \times 10^{12} \text{ cm}^{-2}$.

Fig. 3. (a) (004) and (b) (115) ω - 2θ scan of InSb grown on Si.

Fig. 3(a) and (b) show x-ray ω - 2θ scans of the InSb layer on Si at the (004) and (115) lattice plane. The ω value of the Si peaks are normalized to 0°. The out-of-plane ($a_{\perp}$) and in-plane ($a_{\parallel}$) lattice constants of InSb are calculated as 6.482 Å and 6.475 Å, respectively. The degree of relaxation (R) of the InSb layer is calculated using Eq. 1,

$$R = \frac{a_{\parallel} - a_s}{a_0 - a_s} \times 100\% \quad (1)$$

where a_s is the lattice constant of Si (5.431 Å) and a_0 is the lattice constant of bulk InSb (6.479 Å). The calculated relaxation of InSb is 99.6 %.

Fig. 4. (a) Cross-sectional STEM image of InSb on Si. (b) EDX profile of InSb on Si. The scanning line is indicated in (a). (c) STEM image of the GaSb/Si interface where four AlSb islands are indicated by black arrows.

Fig. 4(a) contains the scanning transmission electron microscopy (STEM) image of the InSb layer on Si. A Pt layer was deposit on the surface of the sample. Fig. 4(b) shows the energy dispersive x-ray (EDX) profile obtained along the red line in the STEM image. The GaSb/Si interface is flat, and the AlSb/GaSb and InSb/AlSb interfaces are rough. The existence of Pt signal is due to the top Pt layer. EDX shows the atomic percentage of elements in the InSb, AlSb, GaSb, and Si layers. About 20 atomic percentage of Al was observed at the GaSb/Si interface, which is attributed to the Al prelayer. The Si delta doping is unable to be detected using EDX due to its very low percentage (about 1%). As shown in Fig. 4(c), four AlSb islands indicating by black arrows can be seen at the GaSb/Si

interface, which agrees well with the spotty RHEED patterns observed during the growth of the AlSb prelayer [28]. AlSb islands on Si provided energetically favorable sites for the nucleation of GaSb islands [11]. The coalescence of these islands resulted in a continuous GaSb layer, which was consistent with the RHEED result.

Fig. 5. (a) Cross-sectional TEM image of InSb on Si, where the defects at the InSb/AlSb interface are indicated by a pair of black arrows and one threading dislocation in the InSb layer was indicated by a white

arrow. (b) SAED pattern at the InSb/AlSb interface and (c) SAED pattern at the GaSb/Si interface. (d) Cross-sectional TEM image of the GaSb/Si interface, where one threading dislocation is indicated by a white arrow. (e) HR-TEM image of the GaSb/Si interface where the interfacial misfit array is marked with black arrows. (f) Phase contrast image of the selected area marked labeled “(f)” in (e).

Fig. 5(a) shows the cross-sectional TEM image of InSb grown on Si via the AlSb/GaSb buffer. At the GaSb/Si interface, darker contrast areas are observed. The AlSb buffer has a low defect density and it provides a good platform for the subsequent growth of InSb. Few defects (shown by black dots and indicated by a pair of black arrows), which are distributed along the InSb/AlSb, and a threading dislocation indicated by a white arrow are observed on the left side of this image. Fig. 5(b) and (c) show the selected area electron diffractions (SAEDs) obtained at the InSb/AlSb and GaSb/Si interfaces, respectively. White arrows indicate the diffraction spots for InSb, AlSb, GaSb, and InSb, respectively. No satellite spots are observed: indicating the single crystalline nature of each layer and the absence of APB [29].

Fig. 5(d) shows the TEM image of the GaSb/Si interface with a higher magnification. White dots distribute along the interface which is a feature of IMF array [10] and their microstructures will be shown in Fig. 5(e) and (f). Some dislocations propagating from the interface are observed. In Fig. 5(d), the positions of 30 nm, 40 nm, and 50 nm above the interface are marked by red triangles, which indicate the position of delta doping. Most of dislocations disappear and only one threading dislocation indicated by a white arrow is observed when the GaSb layer thickness above 50 nm. This phenomenon is attributed to the pinning or bend of threading dislocation. In the previous research, delta doping was reported to terminate or change the orientation of threading dislocations [19], which can

prevent the propagation of threading dislocation or promote their annihilation through the combination of two threading dislocations with the opposite Burgers vectors [30]. The mechanism of bending or pinning threading dislocations may be due to the doping-related hardening where the impurities associated with doping suppress the motion of threading dislocations [19, 22]. Fig. 5(e) shows the high resolution TEM (HR-TEM) image of the GaSb/Si interface. An abrupt interface and black dots, which are distributed along the interface, are observed. The previous study suggested these dots are caused by the strain field of misfit dislocations [31]. The separation between these dots are uniform and about 3.5 nm. The relaxation of the GaSb buffer is calculated to be 99.2% using Eq. 1. If the entire lattice mismatch is accommodated by misfit dislocations, the separation (S) of the dislocations can be calculated using Eq. 2 [32],

$$S = \frac{b}{f} \quad (2)$$

where b is the Burger vector of the misfit dislocation and f is the lattice mismatch, which is 12.24% for GaSb on Si. The misfit dislocation marked by a dotted square in Fig. 5(e) is analyzed in Fig. 5(f), where a Fourier filter is applied to extract its atomic arrangement. The red dashed loop indicates that the dislocation is a 90° type with a b of $\frac{a}{2}(110)$. a is the lattice constant of GaSb. Thus, the absolute value of b is 4.31 Å. The calculated separation, S , is 3.52 nm, which agrees well with the measured value. A uniformly distributed 90° misfit dislocation formed an IMF array at the GaSb/Si interface. The effect of AlSb islands can be neglected in this calculation due to their similar lattice constant with GaSb.

Fig.6. (a) HR-TEM image of the InSb/AlSb interface (the dislocations are marked with white arrows). (b) Phase contrast image of the selected area labeled “(b)” in (a).

Fig. 6(a) shows the HR-TEM image of the InSb/AlSb interface and four black dots indicated by white arrows. As mentioned above, these dots are attributed to the strain field of misfit dislocations [31]. Using the red dashed loop, the Burger vector of the misfit dislocation is measured to be $\frac{a}{2}(110)$ (90° type) and a is the lattice constant of InSb, as shown in Fig. 6(b). The STEM image shown in Fig. 4(a) indicates the InSb/AlSb interface is rougher than the GaSb/Si interface which is consistent with the observed rough interface in Fig. 6(a). Therefore, the separation of misfit dislocations at the InSb/AlSb interface was not as uniform compared to the separation of misfit dislocations at the GaSb/Si interface. The irregular distribution of misfit dislocations resulted in threading dislocations [33]. Moreover, 60° misfit dislocations always locate in the rough interface [34] which can glide to form threading dislocations [12]. A surface planarization step on the AlSb surface could improve the crystal quality of the InSb layer. The InSb layer exhibited a 300 K electron mobility and an unintentionally n-type doping concentration of $22,300 \text{ cm}^2/\text{Vs}$ and $3.17 \times 10^{16} \text{ cm}^{-3}$, respectively. This electron mobility is higher than that of the $0.8\text{-}1.0 \text{ }\mu\text{m}$ InSb layer grown on (100) Si via an AlSb buffer [8] and that of the $1.1 \text{ }\mu\text{m}$ InSb grown on (111) Si [35].

Fig. 7. (a) The photocurrent vs. DC applied voltage for the InSb photoconductor at different temperatures. The inset shows the output spectrum of a 700 °C black body. (b) The photocurrent vs. temperature for the InSb photoconductor at DC applied voltage of 1.0 V. The inset is an optical image of the photoconductor. (c) The 300 K transmission of the InSb layer on Si at different wavelengths.

Fig. 7(a) and (b) show the photocurrent of the InSb photoconductor on Si at different DC applied voltages and temperatures. The separation between two adjacent electrodes was about 18 μm, as shown in the inset of Fig. 7(b). A black body maintained at 700 °C acted as the light source and its output spectrum is shown in the inset of Fig 7(a). Unlike in a p-i-n photodetector, there was no p-n junction in the photoconductor to reduce the dark current [36]; leading to a high dark current. Therefore, a lock-in amplifier was used to filter out the dark current during the measurement of the light current [37]. The photoconductor demonstrated a linear increase in the photocurrent with the increasing applied voltage at 77 K, 120 K, 160 K, and 200 K, which is a feature of photoconductive gain [38]. The theoretical responsivity (R) of a photoconductor can be expressed as shown in Eq. 3 [37]

$$R = \frac{q\lambda}{hc} \frac{\eta\tau\mu_e V}{L^2} \left(1 + \frac{1}{b}\right) \quad (3)$$

where q is the electron charge, λ is the wavelength of incident light, μ_e is the electron mobility, η is the quantum efficiency, b is the electron-to-hole mobility ratio which depends on the effective mass of carriers in InSb, τ is minority carrier lifetime, L is the distance between two electrodes, and V is the applied voltage. Obviously, a linear relationship exists between responsivity and applied voltage when other factors remain the same, i.e., photoconductive gain. In Fig. 7(a), the slopes of the voltage dependent photocurrent curves decreased with increasing temperature. The photocurrent measured at 1.0 V also decreased with increasing temperature as shown in Fig. 7(b). The decrease in the photocurrent was attributed to a shorter minority carrier lifetime (τ) and a lower carrier mobility (μ) at higher temperature [39]. Above 200 K, the photocurrent became undetectable due to the high dark current. The 300 K transmission spectrum of the InSb layer was measured from 3 to 10 μm using FTIR. In Fig. 7(c), a cutoff wavelength of $\sim 7.2 \mu\text{m}$ was observed, which is consistent with the 300 K bandgap of InSb (0.17 eV).

4. Conclusion

In summary, the growth of a 0.8 μm InSb layer on Si via a 250-nm-thick AlSb/GaSb intermediate buffer by a MBE system and infrared photoconductors based on this heteroepitaxy structure is demonstrated. The lattice mismatch between InSb and Si is accommodated via the IMF array at the GaSb/Si interface and misfit dislocations at the InSb/AlSb interface. Introducing the Si delta doping three times at 30 nm, 40 nm, and 50 nm above the GaSb/Si interface is an effective approach to reduce the density of threading dislocation in the GaSb layer on Si and the optimal concentration is $5 \times 10^{12} \text{ cm}^{-2}$. The degree of relaxation in the InSb layer is nearly 100%. 300 K electron mobility and

concentration are $22,300 \text{ cm}^2/\text{Vs}$ and $3.17 \times 10^{16} \text{ cm}^{-3}$, respectively. The InSb photoconductor on Si exhibited a photoconductive gain behavior under a broad-spectrum infrared light source. Thus, InSb on Si via the AlSb/GaSb intermediate buffer provides a platform for the further development of InSb-based devices on Si.

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