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Effect of temperature gradient on microstructure and properties of GaSb crystals grown with Bridgman method

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Abstract

GaSb crystal ingots were grown with vertical Bridgman method. The effects of temperature gradient on the structure and properties of GaSb crystals were investigated. When the temperature gradient increased from 5 to 7 °C cm⁻¹, the crystallinity of the ingot improved, the dislocation density decreased by 55%, from 3928 to 1785 cm⁻²; the carrier mobility increased by 29.6%, from 868 to 1125 cm² V⁻¹ · s⁻¹; the resistivity decreased 50.6%, from 12.45 to 6.332 × 10⁻³ Ω · cm; the infrared transmission increased from 27% to 32%. When the temperature gradient increased from 7 to 9 °C cm⁻¹, the crystallinity of the ingot deteriorated obviously, the dislocation density increased 4.38 times, from 3928 to 9609 cm⁻²; the carrier mobility decreased by 52.4%, from 1125 to 738 cm² V⁻¹ · s⁻¹; the resistivity increased 6.2 times, from 6.332 to 23.94 × 10⁻³ Ω · cm; the infrared transmission decreased from 32% to 25%.

1. Introduction

Gallium antimony (GaSb), a binary compound of group III-V, is an excellent photoelectric crystalline material, which has attracted extensive attention of researchers. In recent years, with the vigorous development of optical fiber communication technology, the demand for new devices is increasing and the performance requirements are getting higher and higher [1, 2]. In order to reduce the transmission loss in optical fiber communication, the long wavelength light is always used as far as possible, increasing from 0.8 to 1.55 μm or even to 2–4 μm. Accordingly, the light emitter, light amplifier, light receiver, and so on also need to use longer light waves. GaSb lattice constants can match with those of ternary and quaternary solid solution alloys of various III-V materials as substrates. The spectral range of these materials almost covers 0.8 to 1.55 μm or even to 2–4 μm, which can meet the requirements of optical fiber communication [3, 4]. In addition, GaSb-based superlattices can be used to fabricate detectors with longer wavelength (8–14 μm) using their interband absorption characteristics [5]. And GaSb materials are also widely used in solar cells [6] and light emitting diodes [7].

Vacancies and dislocations are the main defects in GaSb crystals. There have been some works on the preparation of high quality GaSb crystals. Garandent et al prepared p-type GaSb ingots by vertical gradient solidification [8]. The vacancy concentration is 10¹⁷ to 10¹⁸ cm⁻³ at room temperature. Kondo et al have grown GaSb crystal by liquid-sealed Czochralski method [9]. The results show that NaCl-KCl coating can significantly reduce the dislocation density in the crystal. Ge et al [10] prepared GaSb ingots under microgravity. They thought that the melt did not contact the crucible wall directly under microgravity, there was neither thermal convection nor thermal stress, so the dislocation density of the crystal decreased significantly. Yu et al [11] also prepared GaSb crystals with liquid-sealed Czochralski method. The effects of two liquid-sealed agents (KCl + LiCl) and B₂O₃ on the quality of GaSb crystals were compared. It was found that the crystal with the latter had lower dislocation density and higher crystal quality. Although the quality of GaSb crystal has been significantly improved, it is still difficult to meet the requirements of quality and quantity from the epitaxial devices.

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Bridgman process is one of the most widely used methods for bulk single crystal preparation, in which the furnace temperature gradient is one of the main factors affecting crystal quality. Liu et al [12] calculated the effects of furnace temperature gradient on the morphology of solid-liquid interface and the segregation of composition in crystal. The results show that the temperature gradient obviously affects the shape and intensity of the flow field at the front of the solid-liquid interface, and the interface curvature decreases with the increase of the temperature gradient of the furnace. Yao et al [13] studied the effects of temperature gradient on the shape of solid-liquid interface and the growth rate of CaF₂ crystal by finite element method. The results show that the convexity of solid-liquid interface decreases with the increase of temperature gradient to a certain extent. Liu et al [14] investigated the effect of temperature gradient on PbI₂ crystal growth. The results show that when the temperature gradient is small, the melt decomposition of PbI₂ is rather serious. When the temperature gradient rises to 5 K/cm, the melt decomposition is alleviated, and the crystal composition deviation from stoichiometric ratio is weakened.

Kozhemyakin et al [15] believed that a smaller temperature gradient could reduce the convection velocity in the melt and inhibit the generation of fringes in the crystal. Wu et al [16] suggested that a larger temperature gradient could effectively inhibit the visible inclusions formed by component supercooling. Trukhanov et al [17] reported that a smaller temperature gradient could significantly reduce the dislocation density in Ge crystals. Ren et al [18] studied the effect of temperature gradient on the growth process of silicon single crystal. The results show that with the increase of temperature gradient, the solute diffusion boundary layer decreases significantly, which effectively reduces the segregation of impurities.

In the process of melt solidification, the larger temperature gradient in the furnace provides a greater supercooling degree, and at the same time leads to a larger thermal stress in the ingot, which may lead to more dislocations and even micro-cracks, especially for those materials with very low stacking fault energy near the melting point. When the temperature gradient is small, the crystal growth interface tends to lose its stability due to the constitutional supercooling, and the crystal growth power is insufficient. Therefore, it is very important to select the appropriate temperature gradient for the crystal growth process.

Herein, GaSb ingots were grown by vertical Bridgman method. The effect of temperature gradient on the structure and properties of GaSb crystals was investigated.

### 2. Experimental details

#### 2.1. Crystal growth

GaSb crystals were grown by vertical Bridgman method, in which high purity quartz crucible was used, of which inner wall was coated a carbon film to avoid adhesion between the melt and the crucible at high temperature, so as to improve the quality of crystal. Firstly, Ga (99.9999%, Shanghai Gallium New Materials Technology Co., Ltd, Shanghai, China) and Sb (99.9999%, Zhongnuo New Material Beijing Technology Co., Ltd, Beijing, China) were loaded into the quartz crucible after coating according to the chemical ratio, and the crucible was sealed when vacuum was pumped to 10⁻³ Pa. Then the crucible was put into a swing synthesis furnace to synthesize GaSb polycrystalline material. The synthesis temperature was 762 °C and the reaction time lasted for 20 h. After the polycrystalline material synthesis experiment, the polycrystalline ingot was removed from the synthetic crucible and broken into small pieces, which then were loaded into the crystal growth quartz crucible sealed at vacuum 10⁻³ Pa. Next, the crucible was put into the Bridgman crystal growth furnace to grow the crystal. There were three heaters in the furnace, dividing the furnace chamber into three zones, high temperature zone, temperature transition zone (temperature gradient zone), and low temperature zone. The distance between 1# heater and 2# heater was 10 cm, and there was a temperature transition zone of 20 cm between 2# heater and 3# heater, shown as in figure 1(a). The temperature of the high temperature zone (1# heater and 2# heater) of crystal growth furnace was set to 762 °C and the temperature of the low temperature zone (3# heater) of crystal growth furnace was set to 662 °C, 622 °C and 542 °C, respectively. Therefore, the calculated temperature gradient inside the furnace chamber was 5 °C cm⁻¹, 7 °C cm⁻¹ and 9 °C cm⁻¹, respectively. Finally, we used thermocouples to measure the temperature at various locations inside the furnace chamber, and got the temperature curves, shown as in figure 1(b). The measurement results of the temperature gradient in the temperature transition zone were roughly consistent with the calculated results.

At beginning, the crucible was placed in the high temperature zone of the furnace. After the furnace temperature reached the set temperature for 36 h, the crucible began to move down at a speed of 1.0 mm h⁻¹. After passing through the gradient zone, the crucible reached the low temperature zone and the crystal growth process ended. The as grown ingots are shown in figure 2.
2.2. Characterization

The ingot was cut into 2 mm thickness wafers with a diamond wire cutting machine along the direction perpendicular to the crystal growth axis. A precision lapping and polishing machine (UNIPOL-1502, Shenyang Kejing Equipment Co., Ltd, China) was used to polish the wafers. The crystal structure and crystallinity of the powder samples were analyzed with x-ray diffractometer (XRD; D8 Advance, Bruker AXS, Germany, Cu-Kα ray, wavelength $\lambda = 0.15405 \text{ nm}$, $2\theta = 20 \sim 80^\circ$, scanning speed $20 \degree \text{ min}^{-1}$). An inverted metallographic microscope (MDS-D, Chongqing Auto Optical Instrument Co., Ltd, China) was used to observe the surface morphology of the wafer and calculated the dislocation density, which was characterized by the etching pit method. The ratio of corrosive agent was HNO₃: HF: H₂O = 1:1:1. The etch pit density (EPD) formula is as follows:

$$EPD = \frac{N \times M_i}{S}$$  \hspace{1cm} (1)

Where $N$ is the magnification factor of the microscope, $M_i$ is the number of corrosion pits in the field of view, and $S$ is the area of the view field. A Hall Effect tester (HMS-3000, Ecopia, Korea, electric current $I = 2 \text{ mA}$, sample thickness $D = 1.7 \text{ mm}$, magnetic field intensity $B = 1 \text{ T}$, test temperature 298 K) was used to determine the electrical parameters of the wafer, such as carrier mobility and resistivity. The sample was a square wafer with 15 mm edge length. The hardness of wafers was measured with Vickers hardness tester (HVS-50, Yantai Huayin Testing Instrument Co., Ltd, China, with load of 9.8 N and 15 s load time). The infrared transmission spectra of the wafers were recorded with a Fourier transform infrared spectrometer (FTIR; Nicolet 5700, Thermo Electron Scientific Instruments Corp., USA; wafer sample from the middle position of the ingot, scanning speed 6.28
cm$^{-1}$ s$^{-1}$, wave number range 400–4000 cm$^{-1}$). The Raman spectra of the crystals were recorded with a Raman spectrometer (LabRAM HR Evolution, Horiba, Japan; powder sample from the middle position of the ingot, wave number 50–1500 cm$^{-1}$).

3. Results and discussion

3.1. Effect of temperature gradient on crystallinity of GaSb Crystal

Figure 3 shows the effect of temperature gradient on the XRD patterns of GaSb crystals. As shown in the figure, the diffraction peaks of the main crystal planes of the three samples are basically identical and belong to zinc blende structure (PDF #65–2894), which indicates that the temperature gradient cannot affect the basic structure of GaSb crystal. Among the three patterns, when the temperature gradient is 7 °C cm$^{-1}$, the intensity of the diffraction peak on the (111) crystal plane is the sharpest, and its full width at half maximum (FWHM) is the smallest, 62″, followed by that of the sample with the temperature gradient of 5 °C cm$^{-1}$, FWHM = 108″, then followed by that of the sample with the temperature gradient of 9 °C cm$^{-1}$, FWHM = 175″. When the temperature gradient is 7 °C cm$^{-1}$, the intensity of the diffraction peak of (111) crystal plane is about 3.14 times that of the sub-high diffraction peak, and the other two are 1.56 times (5 °C cm$^{-1}$) and 1.08 times (9 °C cm$^{-1}$), respectively.

The intensity of the diffraction peak is related to the crystallinity of the crystal. The sharper the diffraction peak is, i.e., the smaller the FWHM, the stronger the intensity is, the higher the crystallinity is, and the better the crystal quality is [19]. When the temperature gradient is rather small, the driving force of crystal growth is insufficient and the crystallization of ingot is poor. When the temperature gradient is too large, the thermal stress near the crystal growth interface will be high. And there will be many defects in the ingot, such as point defects, dislocations, twins etc.

3.2. Effect of temperature gradient on dislocation density of GaSb crystal

Dislocation is a structural defect inside the crystal, which has a great influence on the physical properties of the crystal. When an etchant is used to etch the wafer surface, because of the high strain energy near the dislocation, the area around the dislocation near the wafer surface will be preferentially eroded to form etching pits. Herein, the density of etching pits is used to represent the dislocation density. Three cross sections perpendicular to the crystal axis were selected for each GaSb ingot, and three places were selected for each cross section to observe the dislocation inside the ingot and calculate the dislocation density. Finally, the average dislocation density of each ingot was calculated.

Figure 4 shows the effect of temperature gradient on dislocation density of GaSb crystal. It can be seen from the figure that when the temperature gradient is 5 °C cm$^{-1}$, the distribution of etching pits on the wafer is more complex, and the dislocation density is about 3928 cm$^{-2}$. When the temperature gradient is 7 °C cm$^{-1}$, the number of etching pits on the wafer decreases obviously, and the arrangement of etching pits is more orderly. The dislocation density is about 1785 cm$^{-2}$, lower than that of substrates used by Furlong etc, 2000 cm$^{-2}$ [20]. When the temperature gradient increases to 9 °C cm$^{-1}$, the number of etching pits on the wafer increases remarkably, and the distribution of etching pits becomes disorderly. The dislocation density reaches about 9609...
The temperature gradient has a great influence on dislocation density in ingot [21, 22]. Crystal growth is accompanied by the release of latent heat. When the temperature gradient is small, the latent heat cannot be discharged through the crystal in time, the solid-liquid interface is easy to form constitutional supercooling and lose stability, leading to local large component segregation, inclusions and so on. All these will promote the formation of dislocations. On the other hand, the larger temperature gradient is one of the main causes of thermal stress in ingot [23, 24]. The larger thermal stress makes the lattice slip, resulting in a large number of dislocations in the crystal [25].

In summary, the crystal dislocation density decreased by 55% as the temperature gradient increased from 5 to 7 °C cm⁻¹, while increased 4.38 times as the temperature gradient increased from 7 to 9 °C cm⁻¹.

3.3. Effect of temperature gradient on hardness of GaSb crystal
Hardness refers to the local resistance of solid to invasion of external objects, and it is an important parameter to characterize the quality of crystal materials. Five cross sections perpendicular to the center axis of ingot are selected, and five points on each section are selected for hardness testing, as shown in figure 5.

Figure 6 shows the effect of temperature gradient on the hardness of GaSb wafers. It can be seen that when the temperature gradient is 7 °C cm⁻¹, the hardness value of the ingot is the smallest, about 4.3 GPa; when the temperature gradient is 5 °C cm⁻¹, the hardness of the ingot is between 4.4 and 4.6 GPa; and when the
The temperature gradient is $9 \, ^\circ \text{C/cm}$, the hardness value is the largest, and the maximum value reaches 4.8 GPa. According to the literature, the theoretical Vickers hardness of GaSb crystal is between 4.2 and 4.4 GPa [26]. In addition, by comparing the error bars on the curves in Figure 6, it is clear that when the temperature gradient is $7 \, ^\circ \text{C/cm}$, the discreteness of hardness values is the smallest, that is, the difference of hardness values on the same section is the smallest. This means that the structure of the crystal on the cross section is more uniform, and there is no unwanted crystal grain very probably.

As mentioned above, the driving force of crystal growth is insufficient when the temperature gradient is rather small, and local constitutional supercooling will be easy to occur at the solid-liquid interface, resulting in more unwanted crystal grains on the cross section. Grain boundaries are more serious structural defects than dislocations. This may be the reason for the higher surface hardness of wafers. On the other hand, a larger temperature gradient not only increases the dislocation density in the ingot, but also increases the residual thermal stress. And it is also conducive to the formation of new nuclei near the inner wall of the crucible at the solid-liquid interface, which results in more unwanted crystal grains in the ingot. The pinning effect of grain boundaries and dislocations, as well as the high residual thermal stress, will increase the hardness of wafer surface [27].

### 3.4. Effect of temperature gradient on carrier mobility of GaSb wafers

Figure 7 shows the effect of temperature gradient on carrier mobility of GaSb wafer at room temperature. It can be seen that when the temperature gradient is $7 \, ^\circ \text{C/cm}$, the carrier mobility of the wafer is the highest, the average value is $1125 \, \text{cm}^2/\text{V} \cdot \text{s}$; when the temperature gradient is $5 \, ^\circ \text{C/cm}$, the carrier mobility of the wafer is at the medium level, the average value is $868 \, \text{cm}^2/\text{V} \cdot \text{s}$; and when the temperature gradient is $9 \, ^\circ \text{C/cm}$, the carrier mobility of the wafer is the smallest, the average value is $738 \, \text{cm}^2/\text{V} \cdot \text{s}$. Carrier mobility reflects the average motion rate of carriers per unit electric field intensity and the conductivity of semiconductors. Carrier mobility is mainly affected by the scatterings in crystal, such as lattice vibration scattering, ionized impurity scattering, dislocation scattering, and other scatterings, including equivalent inter-valley scattering, neutral impurity scattering etc. The dislocation density in GaSb crystal exceeds $10^3 \, \text{cm}^{-2}$, so dislocation scattering becomes one of the main factors affecting carrier mobility. With the increase of dislocation number, the probability of dislocation scattering per unit time increases rapidly [28].

The average time that carriers experience between two scatterings is the average free time, which decreases with the increase of scattering times. Formula (2) is the relationship between carrier mobility and mean free time.

$$
\mu_p = \frac{q \tau_p}{m_p}
$$

Where $\mu_p$ is the carrier mobility, $q$ is the electric amount of unit charge, $\tau_p$ is the average free time of the hole, and $m_p$ is the effective mass of the hole. It can be seen from the formula that the carrier mobility is proportional to the average free time. When the average free time is large, the carrier mobility is higher, and vice versa. The effect of temperature gradient on carrier mobility coincides with the effect of temperature gradient on dislocation density in section 3.2.
To summarize this section, when the temperature gradient increased from 5 to 7 °C cm⁻¹, the crystal's carrier mobility increased by 29.6%. However, it decreased by 52.4% as the temperature gradient increased from 7 to 9 °C cm⁻¹.

3.5. Effect of temperature gradient on resistivity of GaSb wafers

Figure 8 shows the effect of temperature gradient on the resistivity of GaSb wafers. It can be seen from the figure that the resistivity of wafer is the lowest when the temperature gradient is 7 °C cm⁻¹, the average is 6.332 × 10⁻³ Ω · cm; the resistivity of wafer is at the middle level when the temperature gradient is 5 °C cm⁻¹, the average value is 1.245 × 10⁻² Ω · cm; and the resistivity of wafer is the highest when the temperature gradient is 9 °C cm⁻¹, the average value is 2.394 × 10⁻² Ω · cm. Resistivity is one of the most important characteristic parameters of semiconductor materials and the main parameter for dividing the specifications of semiconductor products. Carrier mobility reflects the movement rate of carriers and the conductivity of materials. Therefore, resistivity and carrier mobility can be combined to analyze. Generally speaking, the resistivity ρ depends on the carrier mobility of the material, and the carrier mobility is inversely proportional to the resistivity [29]. So compared with figure 7, we can see that when the carrier mobility of the material is high, the resistivity is low, and vice versa.

To summary this section: the crystal resistivity decreased 50.6% as the temperature gradient increased from 5 to 7 °C cm⁻¹, from 12.45 to 6.332 × 10⁻³ Ω · cm; it increased 6.2 times as the temperature gradient increased from 7 to 9 °C cm⁻¹, from 6.332 to 23.94 × 10⁻³ Ω · cm.
3.6. Effect of temperature gradient on infrared transmission of GaSb wafers

The infrared transmittance is an important parameter to determine whether a semiconductor single crystal can be used as a substrate material for devices. The crystal quality can be evaluated with the infrared transmittance spectrum of the crystal. Figure 9 shows the effect of temperature gradient on the infrared transmission spectra of GaSb wafers. It can be seen that the infrared transmission of wafer is the highest when the temperature gradient is 7 °C/cm, the average value is 32%; the infrared transmission of wafer is at the middle level when the temperature gradient is 5 °C/cm, the average value is 27%; and the infrared transmission of wafer is the lowest when the temperature gradient is 9 °C/cm, the average value is 25%. The infrared transmission was determined by the following formula [24]:

\[ T = \frac{(1 - R) e^{-ad}}{1 - R e^{-ad}} \]  

Where \( T \) is the infrared transmission, \( R \) is the reflectivity, \( a \) is the absorption coefficient, and \( d \) is the wafer thickness. For GaSb crystals, \( R \) is a constant. Hence, the infrared transmission of GaSb crystal mainly depends on the absorption coefficient \( a \). The incident light is usually absorbed through the following mechanisms in FTIR measurement: (1) intrinsic absorption, (2) impurity absorption, (3) free carrier absorption, (4) charged particle polarization absorption, and (5) lattice absorption [30]. However, within the wavenumber range of 500–4000 cm\(^{-1}\), the intrinsic absorption can be neglected and the impurity absorption is also very weak due to its limited concentration. The charged particle polarization absorption often requires an externally applied electric field. Thus, the infrared light absorption mainly depends on the lattice absorption and free carrier absorptions.

The dislocations in a crystal disturb the consistency and periodicity of the lattice, resulting in the lattice mismatch, which leads to the aggravation of the lattice vibration absorption [31]. As can be seen from figure 4, when the temperature gradient was 7 °C/cm, the dislocation density of the crystal was at the lowest level. Hence, the lattice vibration absorption should be the weakest.

The carriers’ concentration could be calculated by the following equations:

\[ \sigma = n q \mu; \quad \rho = \frac{1}{\sigma}; \quad n = \frac{1}{(q \rho \mu)} \]  

where \( \sigma \) is the conductivity, \( \rho \) is the resistivity, \( \mu \) is the carrier mobility, \( q \) is the electric quantity per unit charge, \( n \) is the carrier concentration. According to the average values of the carrier mobility in section 3.4, those of the resistivity in section 3.5, the average carrier concentration is 5.7838, 8.7168, 3.7436 \( \times \) \( 10^{17} \) cm\(^{-3}\), corresponding to the three temperature gradients 5 °C cm\(^{-1}\), 7 °C cm\(^{-1}\), and 9 °C cm\(^{-1}\), respectively. So, the free carrier absorption of the crystal prepared at 7 °C cm\(^{-1}\) should be the strongest. However, the maximum variation of free carrier concentration is 2.33 times, which is far less than the maximum variation of dislocation density 4.38 times. Therefore, the lattice absorption was the dominant factor affecting the infrared transmission of the crystal.

3.7. Effect of temperature gradient on raman spectrum of GaSb crystal

The Raman scattering experiments were carried out demonstrated in backscattering geometry from sample (100) surface with an Ar+ -ion laser. The scattering spectrum is mainly determined by the lattice excitation of the
material and is very sensitive to the lattice vibration [30]. For III-V compound semiconductors with zinc-blende crystal structure, a Raman spectrum in general shows two peaks [32]. The lower-frequency peak corresponds to TO phonons while the higher peak corresponds to LO phonons. In backscattering only LO phonons appear in the (100) backscattering direction, and only TO phonons appear in the (110) direction, while both appear in the (111) direction. Raman spectroscopic technique is the most useful method for the analyses of lattice vibration and interaction with other excitations [26]. Figure 10 displays the effect of temperature gradient on the Raman spectrum of GaSb crystal. Herein, a strong peak at 226 cm$^{-1}$ and a very weak peak at 216 cm$^{-1}$ appear on the spectrum, attributed to LO and TO modes, respectively. With the increase of temperature gradient, there is no obvious change about LO mode but the TO mode shows a trend of decreasing firstly and then increasing, indicating that the defects of crystals decrease first and then increase gradually. The reason is that with the increase of temperature gradient, the dislocation density within the ingot decreases, thus the strong lattice vibration due to the dislocations also decreases first then increases.

4. Conclusions

GaSb crystals were prepared by vertical Bridgman method. The effects of temperature gradient on the structure and properties of GaSb crystals were studied.

1. With the increase of temperature gradient, the crystallization quality of ingot first increases and then decreases. When the temperature gradient is 7 °C cm$^{-1}$, the crystallinity is the best.

2. The dislocation density decreases first and then increases with the increase of temperature gradient. When the temperature gradient is 7 °C cm$^{-1}$, the number of corrosion pits in the ingot decreases, and the dislocation density is the lowest, which is about 1785 cm$^{-2}$.

3. The hardness of the crystal first decreases and then increases with the increase of temperature gradient. When the temperature gradient increases to 7 °C cm$^{-1}$, the hardness of the wafer is the smallest, about 4.3 GPa.

4. The carrier mobility of the crystal increases first and then decreases with the increase of temperature gradient, while the resistivity decreases first and then increases.

5. The infrared transmission of the crystal increases first and then decreases with the increase of temperature, while the variation trend of Raman spectrum is opposite to that of infrared transmittance.

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