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# TEMPERATURE DEPENDENCE OF GaSb OVERGROWTH OF TUNGSTEN ON GaSb (001) SUBSTRATES USING MOVPE

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**Abstract— We demonstrate GaSb overgrowth over tungsten patterns and that selective area epitaxy is achievable in the W/GaSb system. By controlling the facet growth at low temperatures, it is possible to embed a metal grating in a thin layer.**

*Keywords—SAE, GaSb, lateral growth, overgrowth, tungsten*

## I. INTRODUCTION

Antimonide-based III/V semiconductors are a very promising class of materials with many applications. Gallium Antimonide (GaSb), in particular, is interesting because it is lattice matched to InAs [1], which are candidates for long wavelength ( $>2 \mu\text{m}$ ) infrared (IR) applications [2,3]. In addition, GaSb has the highest hole mobility among the III/V's, making it a candidate for p-type III/V electronic devices.

For integration of electronic devices on a chip, electric isolation is required. Semi-insulating (SI) GaSb substrates are currently not available. Buried tungsten (W) could constitute a barrier to n-type GaSb, and effectively remove leakage current into the substrate. This effect has previously been shown for overgrowth of W on GaAs [2]. Another use of overgrown W is as a permeable gate in resonant tunneling transistors [4]. Overgrowth studies are required for this approach, as epitaxial overgrowth depends on the crystallographic direction and it varies with different growth parameters. In this work, the epitaxial GaSb overgrowth on W patterns by Metal Organic Vapor Phase Epitaxy (MOVPE) has been studied to get an understanding of the overgrowth mechanism in this material.

## II. EXPERIMENTAL PROCEDURE

We studied the growth of GaSb on patterned substrates and in particular investigated the influence of temperature on the GaSb overgrowth. GaSb growth was performed on "Epi-ready" GaSb (001)-substrates with 20-nm-thick tungsten (W) patterns. The patterns were defined by electron beam lithography (EBL) using polymethyl methacrylate (PMMA) as positive resist. After EBL

exposure, the pattern was developed for 90 s in a methyl isobutyl ketone (MIBK):isopropanol solution. Resist and oxide residues were removed by subsequent plasma cleaning and HCl:H<sub>2</sub>O (1:1) etch. W was evaporated onto the patterned surface and lift-off of the resist was then performed followed by final plasma cleaning. Prior to growth, each sample was etched in 37 % HCl for 3 minutes and rinsed in pure 2-propanol for 3 min, to reduce the native oxide thickness. The samples were then immediately transferred into an inert atmosphere and then into the reactor. Before actual growth started, the samples were annealed at 575 °C under H<sub>2</sub> flow for 10 min to fully remove any surface oxide. No group V stabilization was used during deoxidation. The sources used for growing GaSb were triethylgallium (TEG) and trimethylantimony (TMSb). During growth the TEG mole fraction was kept at  $1.88 \cdot 10^{-4}$ .

To study the effect of temperature, a series of samples were grown keeping a constant V/III-ratio of 1.4, while changing the growth temperature. The growth time was 20 min for all samples. The samples were evaluated using high-resolution SEM and AFM. The patterns used were arrays of 100-nm-wide lines with different spacing oriented along the [110]- and [1-10]-directions, full and hollow circles 2-20  $\mu\text{m}$  in diameter, as well as concentric circles. The growth was performed in a standard MOVPE setup (AIX200/4), with a horizontal double-walled quartz reactor. The reactor pressure was 100 mbar and hydrogen was used as carrier gas at a total flow of 7.5 l/min during growth. The substrate holder was rotated at around 1 rotation per second during all growth runs

## III. RESULTS

The growth time of GaSb was about 20 min with 3 different temperatures: 575, 600, and 625°C. The first effect of the temperature is a change in the growth rate. While samples at 575 and 600 °C had a similar vertical growth rate of 0.75  $\mu\text{m}/\text{h}$ , the 625 °C sample had a lower vertical growth rate of 0.4  $\mu\text{m}/\text{h}$ . This can be explained by the increased desorption rate of the growth species from the surface at the highest growth temperature. Also, it is observed that the growth rate decreases at higher temperature. After measuring the diameter of the grown GaSb inside the circle along the {110}- and {1-10}-directions (Fig. 1) it was found that the

lateral growth rates are uniform for patterns oriented in the main crystallographic directions ( $\{011\}$ ,  $\{0-11\}$ , and  $\{101\}$ ), which indicates an isotropic diffusion coefficient for the adatoms. In contrast, a larger lateral overgrowth was observed for patterns oriented off the main crystallographic directions.

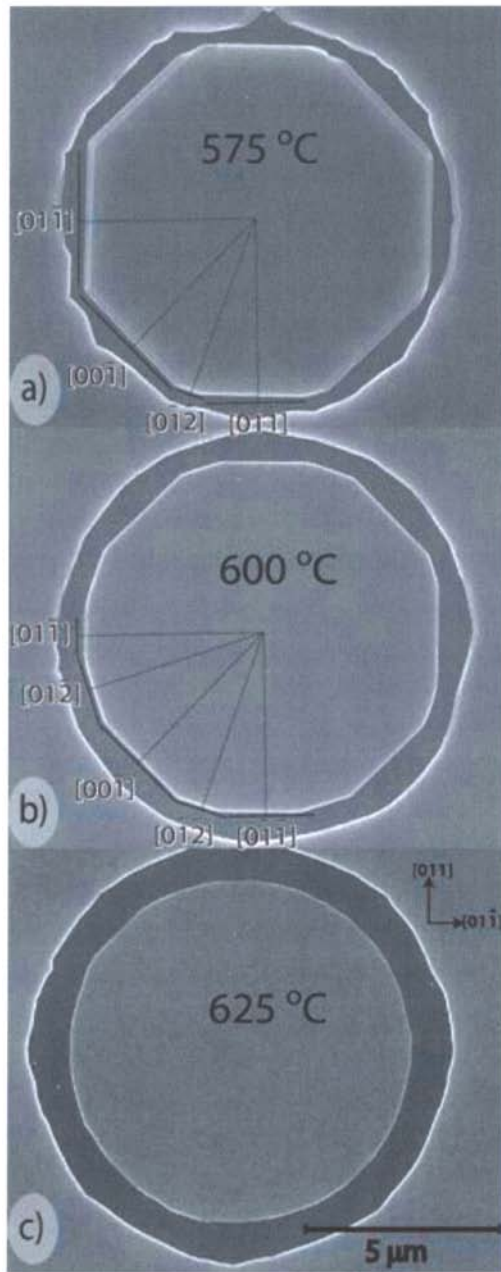


Figure 1. Temperature effect on the selective epitaxy over  $1\ \mu\text{m}$  wide W rings: a)  $575\ ^\circ\text{C}$  b)  $600\ ^\circ\text{C}$  c)  $625\ ^\circ\text{C}$

At the lowest growth temperature, the GaSb grown in the center of the W ring is mostly limited by the family of  $\{011\}$ -planes (Fig. 1a). Increasing the temperature to  $600\ ^\circ\text{C}$ ,

leads to the appearance of additional planes with facets orientated in the  $[01-2]$ -directions (Fig. 1b). At the highest temperature investigated, no clear facets can be identified, as can be seen by the circular shape of the overgrown area (Fig. 1c). The temperature dependence on the plane formation is interpreted in the scheme of the Wulff theory. The principle relates the chemical potential to the geometrical distribution of facets with different surface energies under thermodynamic equilibrium. According to this principle, a larger chemical potential allows the formation of surfaces with more surface energy, thus promoting the formation of multiple facets.

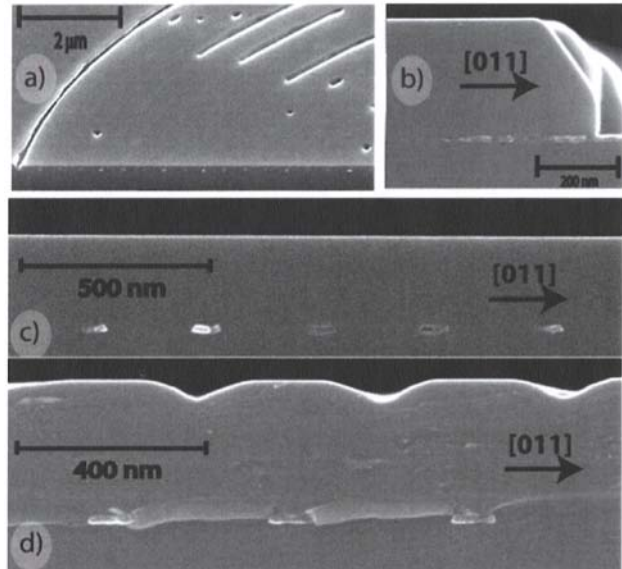


Figure 2. SEM image of the cross-section of the circular W grating overgrown by the GaSb layer. Cross-sections were taken along the  $[011]$  cleavage plane a) The tilted ( $52^\circ$ ) SEM image over the cross-section, showing fully overgrown W lines. Due to the penetration depth of the electron beam W lines are visible through the GaSb layer. b) SEM image illustrating lateral growth over the W ring. c) SEM image of the void free cross-section of GaSb overgrowth, taken over the same location as a) but without tilt.

By depositing GaSb over patterns with narrow W lines, it is possible to conceal the pattern completely by the GaSb layer, as shown in Fig. 2. In fact, full overgrowth was observed at all temperatures over all circular gratings, consisting of thin ( $\sim 100\ \text{nm}$  wide) W lines (not shown). The growth front above the grating was planar for growth temperatures of  $600$  and  $625\ ^\circ\text{C}$ , while the lowest temperature showed small grooves (Fig. 2d), forming over the W lines. Fig. 2a shows a SEM image of the cross-section of a circular grating with a cross-section in the  $[011]$ -plane. No voids were observed above the overgrown W-lines (Fig. 2c) at neither growth temperature. Void free overgrowth was published before [6], but the surface was not planar. The overgrown film is planarized because lateral growth exceeds the vertical growth rate, as illustrated in Fig. 2b.

The influence of the V/III-ratio has been studied at  $600\ ^\circ\text{C}$ , as more facets are available at this temperature and growth rate is not strongly affected by the temperature (as observed at  $625\ ^\circ\text{C}$ ). Figure 3 shows ring structures, partly

overgrown by GaSb. The same lateral facets were found around rings with varying V/III-ratios. Structures were measured by AFM to evaluate the growth rate. The grown layer thicknesses were determined to be 90, 120 and 180 nm at V/III-ratios of 0.8, 1.4, and 2.8, respectively. Complete epitaxial overgrowth above the patterns was observed for all V/III-ratios.

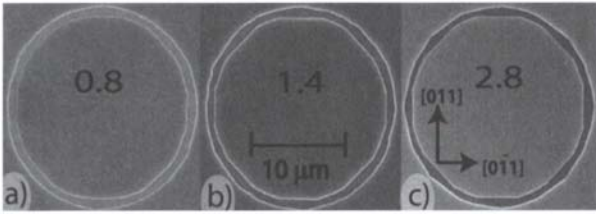


Figure 3. V/III ratio effect on the selective epitaxy over 1 μm wide W rings: a) V/III=0.8 b) V/III=1.4 c) V/III=2.8

Careful investigations with AFM revealed an interesting effect related to shadowing near the tungsten pattern (Fig. 4a). Apparently, the deposition of the GaSb is affected by the presence of the narrow tungsten line and the thickness of the deposited material is reduced, as shown in Fig. 4b. The effect is observed at higher V/III ratios as well (Fig. 4c and 4d). Also, this effect has a clear directional dependence with the largest influence near the tungsten pattern. Locally, a 25% reduction in layer thickness was observed. All samples investigated showed the same effect, although at different magnitude. Generally, the direction of the shadowing was found to be independent from particular crystallographic directions of the substrate and may thus be related to the local growth conditions. The high V/III-ratio sample also showed an increase in the material quality in the areas affected by the shadowing. That V/III-ratio is on the higher side of growth window, and the finding suggests that the effect may be attributed to local changes in the V/III-ratio. Furthermore, our investigation showed that the growth rate reduces if the V/III-ratio is reduced in agreement with Haywood et al. [7]. This further suggests a local reduction of the V/III-ratio around the W pattern. Finally, a reference run with GaAs was performed in the same reactor. As expected, the GaAs sample did not show the same effect as the GaAs is less sensitive to the V/III-ratio [8].

#### IV. CONCLUSIONS

High quality GaSb epitaxial overgrowth over W was achieved via selective area growth by MOVPE at various temperatures. The facet formation during partial overgrowth shows a clear temperature dependence, and our studies reveal a complex facet formation. We found that deposition of a thin film (90 nm) is sufficient to achieve full planarization of the growth front, in the temperature range of 550 to 625°C and with V/III-ratios between 0.8 and 2.8.

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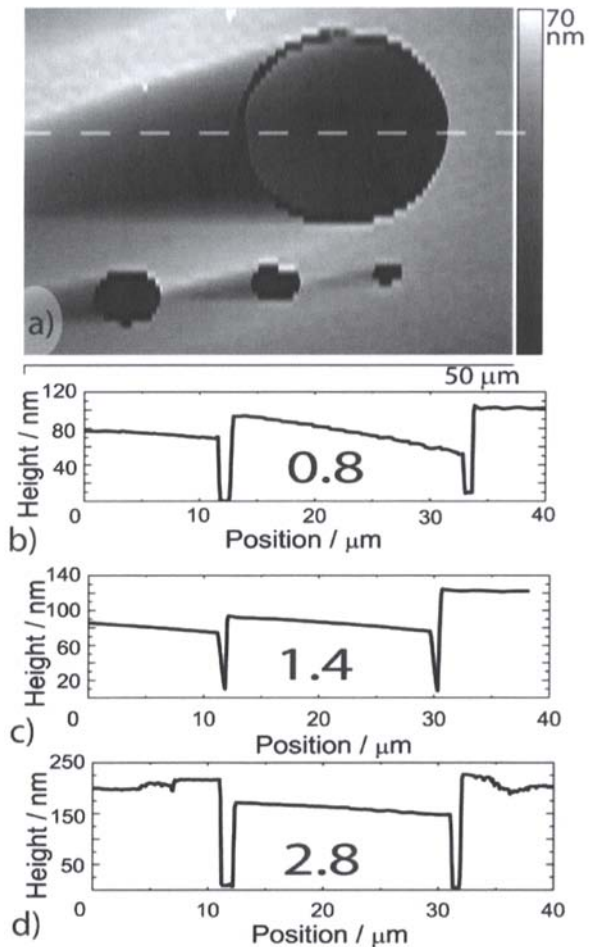


Figure 4. Shadowing of the growth rate at  $T_G = 600^\circ\text{C}$ , V/III = 1.4 a) AFM scan around the tungsten ring; Line scans over the ring at the location marked by a line in a at b) V/III-ratio = 0.8 c) V/III ratio = 1.4 d) V/III-ratio = 2.8

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