Heteroepitaxial Growth of InSb Films on V-Grooved Si(001) Substrate

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V-shaped grooves were prepared by patterning of line and space (LS) using photolithography and BHF etching, and anisotropic etching using hot KOH solution on patterned 100 nm-SiO$_2$/Si(001) substrate. The V-shaped grooves consist of two (111) planes. The width of grooves was varied from 2 to 10 µm while keeping intervals ((001) planes with 1 µm width) between grooves. The heteroepitaxial growth of InSb films on the V-grooved Si(001) substrate was carried out by using a two-step growth procedure in an ultra-high vacuum chamber. The samples were characterized by X-ray diffraction (XRD) and scanning electron micrograph as a function of the space width of LS. From comparison of the XRD pattern of InSb films grown on Si(001) substrate with and without the V-grooves, we found that InSb crystals were heteroepitaxially grown not only on the (111) surface of the V-shaped grooves but also on the narrow (at least <3 µm) (001) surfaces. [DOI: 10.1380/ejssnt.2009.669]

Keywords: Molecular beam epitaxy; Indium antimonide; X-ray diffraction; Si(001); V-shaped grooves

I. INTRODUCTION

Due to its excellent electronic characteristics such as the highest electron mobility of about 78,000 cm$^2$/Vs and the saturation velocity of 5×10$^7$ m/s at 300 K, InSb has attracted a great deal of attention as a candidate material for ultra-high speed and very low power device application [1]. The fabrication of InSb-based devices on Si substrate is very important from a view point of integration with Si-LSI. However, InSb epitaxial film on the Si substrate is very difficult to achieve because of the large lattice mismatch problem [8, 9]. We have attention to the surface reconstruction induced by In and Sb atoms on the Si substrate at the initial stage of the growth of InSb films [5–7], and found that use of InSb bi-layer (Si(111)-2×2-InSb) is good candidate to solve the lattice mismatch problem [8, 9].

We have reported the heteroepitaxial growth of 30°-rotated InSb films on Si(111) substrate using InSb bi-layer. The InSb bi-layer is prepared on Si(111) substrate by adsorption of 1 monolayer (ML) Sb atoms onto In-induced surface reconstruction such as $\sqrt{3}\times\sqrt{3}$ In, 2×2-In and $\sqrt{7}\times\sqrt{3}$ In. Due to this rotation, the large lattice mismatch between InSb and Si is nominally decrease to about 3.3%. The InSb films grown on the InSb bi-layer, prepared via $\sqrt{7}\times\sqrt{3}$ In surface reconstruction, have good crystal quality and high electron mobility of about 16,800 cm$^2$/Vs at RT. However, this growth method via InSb bi-layer is difficult to apply on the Si(001) surface, because of lack of the suitable surface reconstruction to prepare the InSb bi-layer. Even if it can be applied, and InSb films rotate 45° with respect to Si, the compressive strain of about 19.3% only changes into the tensile strain of about 18.9%.

One of the solution to apply our growth method to Si(001) surface may be the formation of the (111) surfaces (V-grooves) onto Si(001) surface by anisotropic etching with KOH solution. If the InSb films grow heteroepitaxially on the (111) surfaces, the (001) planes of the InSb film face toward the normal direction of Si(001) substrate. In this paper, we report the heteroepitaxial growth of InSb films on Si(001) substrate with the V-shaped grooves, which composed from two (111) surfaces. Taking into account of the device fabrication, the film must have high crystal quality and smooth surface. To obtain the smooth surface in growth on V-shaped grooves, the LS width has to be narrower. We also investigated the space width dependence of the crystal quality.

II. EXPERIMENTAL

All the depositions were carried out in a molecular beam epitaxy (MBE) chamber with a base pressure of about 2×10$^{-8}$ Pa. The substrate with dimensions of about 10×10×0.6 mm$^3$ was cut from a p-type Si(001) substrate with 100 nm-thick SiO$_2$ film. The V-shaped grooves were prepared by following process. First, the line and space (LS) structure along with (110) direction was patterned on 100 nm-SiO$_2$/Si(001) substrate by photolithography and BHF etching. The patterned substrates were then immersed into hot (115°C) KOH solution to form V-shaped grooves by anisotropic etching. The etching time of the Si(001) substrates depends on the space width of the SiO$_2$ LS pattern. The space width of LS patterns was changed from 2 µm to 10 µm. The line width of SiO$_2$ layer of all samples was fixed to 1 µm. This means that a small area of Si(001) surface remains on the substrate. After loading the substrates into the vacuum chamber, they were annealed at 900°C for 20 min to obtain a clean surface. High purity (6N) elemental indium (In) and antimony (Sb) were used as source materials and evaporated from each cell. The substrate temperature ($T_s$) was monitored by an infrared pyrometer. The growth of InSb films was performed by the two-step growth procedure. In this procedure, the $T_s$ was held at 240°C to grow the first InSb layer for 30 min, and then rose to 420°C for the beginning
of the second layer deposition. The deposition time for the second layer was 2 hours. This is the optimal growth condition of InSb films on Si(111) in our laboratory. While elevating \( T_s \) for the second layer growth, the growth was interrupted by closing the shutter. The total film thickness measured by interferometer was about 0.7 \( \mu \)m. For structural analysis, the InSb films were characterized by X-ray diffraction (XRD) using Cu-\( K_{\alpha1} \) radiation. In the XRD measurement, we took two types of 2\( \theta \)/\( \omega \) scan pattern to check the heteroepitaxial growth of InSb films on the (111) and (001) surfaces of the Si(001) substrate. The samples were set on the holder to be the V-shaped grooves in parallel to the incoming X-ray. The degree of heteroepitaxy on the (001) surface is defined as the XRD intensity ratio \((I_{(004)}/\Sigma I_{(hkl)})\) of InSb(004) peak to that of the summation of each InSb(hkl) peak. The XRD intensity ratio \((I_{(111)+(333)}/\Sigma I_{(hkl)})\) of InSb(111) and InSb(333) peak to that of the summation of each InSb(hkl) is also defined as the degree of the heteroepitaxy on the (111) surface. The surface morphology of the samples was observed by using the scanning electron microscopy (SEM).

III. RESULTS AND DISCUSSIONS

Figure 1 shows the XRD (\( \chi=0^\circ \)) patterns of the InSb films grown on the V-grooved Si(001) substrate. Here, the \( \chi \) axis is perpendicular to the \( \omega \) axis, and parallel to incident direction of X-ray at \( \omega=0^\circ \). In this case, the crystal orientation of the films to the growth direction is shown in the patterns. The width of the V-shaped grooves (Spaces) is (a) 2, (b) 3, (c) 5 and (d) 10 \( \mu \)m, respectively. For comparison purpose, the XRD pattern of the sample directly grown on Si(001) substrate is shown in Fig. 1(e). As shown in Fig. 1, the intensity InSb(004) peak is appeared in the samples grown on the V-grooved substrate, indicating preferential growth to the (001) direction. The small InSb peaks may come from the InSb crystals on the line-shaped Si(001) surface. On the other hands, many InSb-related peaks are shown in the sample directly grown on the Si(001) substrate, indicating polycrystalline nature. These enhancement of the intensity of InSb(004) peaks on the V-grooved Si(001) substrate may imply the heteroepitaxial growth of InSb films on the (111) surfaces of V-grooves. However, it remains that the possibility of the heteroepitaxial growth of InSb films on line-shaped Si(001) surface.

To confirm whether the InSb films were grown on the line-shaped Si(001) surface, we prepared two types of LS patterned Si(001) substrate with space width of 3 and 10 \( \mu \)m (300 nm in depth) without V-shaped grooves (i.e. without KOH etching), and tried to grow InSb films on the surface. The line width was fixed to 1 \( \mu \)m. Figure 2 shows the XRD patterns of the InSb films grown on the LS patterned Si(001) substrate without V-shaped grooves. As shown in Fig. 2(a), XRD pattern of the sample grown on the patterned Si(001) substrate with 10 \( \mu \)m-space shows many InSb-related peaks, indicating polycrystalline nature. This pattern is similar to that of the sample directly grown on Si(001) surface (Fig. 1(e)). However, the sample grown on the patterned Si(001) substrate with 3 \( \mu \)m-space shows intense InSb(004) peak, indicating preferential growth in (001) direction. The degree of heteroepitaxy of this sample is about 86%. The areal ratio of the line-shaped Si(001) surface is only 25% for the sample with 3 \( \mu \)m-space. So, these results indicate that the InSb films are preferentially grown in (001) direction not only on the line-shaped Si(001) surface with 1 \( \mu \)m-width but also on the space region which width was limited (at least less than 3 \( \mu \)m). These results are similar to that reported by Li et al. [10]. They reported that the heteroepitaxial growth of high quality GaAs films on the grooves patterned Si(001) substrate with aspect ratio >1. Their aspect ratio trapping (ART) growth method may be able to apply to the growth of InSb films on Si(001) substrate. The details of the heteroepitaxial growth of InSb films grown on the grooves patterned Si(001) substrate without V-shaped grooves will be reported in elsewhere.

The XRD results of the samples grown on the V-grooved Si(001) substrate are summarized in Table I. The full width at half maximum (FWHM) of the InSb(004) peaks is the twice as wide as that of the samples grown without V-shaped grooves.
FIG. 2: XRD patterns (2θ/ω (χ=0°) scan) of the InSb films grown on the patterned Si(001) substrate without V-shaped grooves. The space width of the samples is (a) 10 µm and (b) 3 µm, respectively.

TABLE I: FWHM value of InSb(004) and InSb(111) peaks and degree of heteroepitaxy of the InSb films grown on the V-grooved Si(001) substrate with various groove width.

| Width of grooves [µm] | FWHM(χ = 0°) | FWHM(χ = 54.7°) | Degree of heteroepitaxy [%] |
|-----------------------|--------------|------------------|-----------------------------|
| 2                     | 0.163        | 0.435            | 97.0                        |
| 3                     | 0.183        | 0.795            | 95.5                        |
| 5                     | 0.162        | 0.261            | 95.9                        |
| 10                    | 0.169        | 0.325            | 98.9                        |

via InSb bi-layer [8, 9], indicating poor crystalline quality of the films. However, there is no space width dependence in the samples. The degree of the heteroepitaxy over 95% of all samples grown on the V-shaped grooves is quite larger than that of the sample directly grown on the Si(001) substrate. From comparison of the XRD patterns of the samples with (Fig. 1(d)) and without (Fig. 2(a)) V-shaped grooves, this increase of intensity of the InSb(004) peak is evidence of the heteroepitaxial growth of InSb films on the (111) surfaces of V-shaped grooves, especially in the case of the samples with wider space width. However, because the InSb crystals are heteroepitaxially grown on the Si(001) surface with narrower width, effect of the V-shaped grooves for the increase of the intensity of InSb(004) peak decreases with decrease of the space width. The lack of the space width dependence in FWHM of InSb(004) peaks may show that there is no difference of the crystal quality between InSb films grown on line-shaped (001) surface and V-shaped (111) surface.

The intense InSb(004) peaks were appeared in the 2θ/ω scan (χ=0°) patterns, as shown in Fig. 1. To confirm the heteroepitaxial growth of InSb films on the (111) surface of the grooves, we measured the 2θ/ω scan (χ=54.7°) patterns of the InSb films. The angle of 54.7° corresponds to the angle between (111) and (001) surfaces. So, the crystal orientation of the films grown on the (111) surface of the grooves is shown in the patterns. Figure 3 shows the 2θ/ω scan (χ=54.7°) patterns of the InSb films. As shown in Fig. 3, there are only two InSb peaks related with (111) and (333) planes, meaning the heteroepitaxial growth of the InSb films on the (111) surface of the grooves and the line-shaped (001) surfaces. The sample directly grown on Si(001) substrate shows very weak InSb(111) peak, because of a small amount of (001)-oriented crystals.

The FWHM of the InSb(111) peak of the samples with 2 µm and 3 µm-line width is slightly wider than that of the other samples. However, the FWHM of the InSb(004) peak does not show obvious deterioration with decrease in the width of grooves. In the case of 54.7°-tilted scan, the XRD patterns come from one side of (111) planes composing of the V-grooves. Though we compared the XRD patterns of both sides of the (111) surface, there is not obvious difference in the FWHM of the InSb(111) peak. This indicates that there is no difference of crystal quality between both (111) surfaces of V-shaped grooves. The
FIG. 4: SEM image of the InSb film grown on the V-grooved Si(001) substrate with 3 µm space width. For guide to the eyes, the dashed lines are drawn in the image to make clear each (001) and (111) plane.

direct observation of the cross section of the samples using transmission electron micrograph may need to clarify the reason of this broad peak. To grow with high crystal quality and smooth surface, this problem must be cleared.

From the results of XRD measurements, we found that the increase of the InSb(004) peak on the 2θ/ω scan (χ=0°) is caused by InSb films heteroepitaxially grown on the (111) surface of the V-shaped grooves. Furthermore, in the samples with narrower space width, InSb is also grown heteroepitaxially on ⟨001⟩ surfaces. The XRD results also indicate the possibility of heteroepitaxial growth of InSb films without V-shaped grooves.

The typical SEM image of the InSb films grown on the V-grooved Si(001) substrate with 3 µm-space width is shown in Fig. 4. The dashed lines are drawn in the image to make clear each (001) and (111) plane. As shown in Fig. 4, the surface morphology of the InSb film is affected by the unevenness of the surface of the V-grooved Si(001) substrate. Because the depth of the V-grooves of this sample is about 2.1 µm, the V-grooves are not chocked with the 0.7 µm-InSb film. However, the surface of the InSb film on each (001) and (111) plane is relatively smooth. To obtain the flat and (001)-oriented InSb films on V-grooved Si(001) substrate, a narrower LS and/or thicker InSb film is necessary.

IV. CONCLUSIONS

The heteroepitaxial growth of InSb films grown on the V-grooved Si(001) substrate was investigated. The XRD pattern of the InSb films heteroepitaxially grown on the Si(111) substrate with the V-shaped grooves shows the intense InSb(004) peak. This means that the ⟨001⟩ plane of the InSb films faced toward the normal direction of Si(001) substrate, on which it is difficult to grow heteroepitaxial InSb films. The InSb films are heteroepitaxially grown not only on the ⟨111⟩ surface of the V-shaped grooves, but also on the ⟨001⟩ surfaces with narrower width.

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