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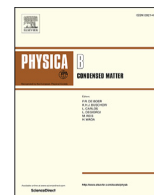

Defect and interface analyses of non-stoichiometric *n*-type GaSb thin films grown on Ge(100) substrates by rapid thermal annealing

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ABSTRACT

In this study, $Ga_{0.6}Sb_{0.4}$ thin films were grown on quartz and Ge(100) 1° off-axis substrates by RF magnetron sputtering at 500 °C. $Ga_{0.6}Sb_{0.4}$/Ge(100) shows *n*-type conductivity at room temperature (RT) and *p*-type conductivity at low temperatures, whereas undoped GaSb thin films exhibit *p*-type conductivity, irrespective of their growth methods and conditions. Their electrical properties were determined by rapid thermal annealing, which revealed that $Ga_{0.6}Sb_{0.4}$/Ge(100) contains two types of acceptors and two types of donors. The acceptors are considered to be Ga_{Sb} and electrically active sites on dislocations originating at the $Ga_{0.6}Sb_{0.4}$/Ge(100) interface, while donors are believed to be Ga_i and electrically active sites originating at the $Ga_{0.6}Sb_{0.4}$/Ge(100) interface. In these acceptors and donors, the shallow donor concentration is higher than the shallow acceptor concentration, and the shallow donor level is deeper than the shallow acceptor level. Thus, we concluded that $Ga_{0.6}Sb_{0.4}$/Ge(100) shows *n*-type conductivity at RT due to electrically active sites originating at the $Ga_{0.6}Sb_{0.4}$/Ge(100) interface and native defects originating from excess Ga.

1. Introduction

GaSb is a III-V semiconductor with high electron mobility, a high saturation velocity, and a band gap of 0.73 eV at 300 K [1]. Its electrical properties are suitable for high-speed electronic devices, and its relatively narrow band gap promotes efficient operation of optical devices at long wavelengths. Due to these superior properties, GaSb-based materials have been extensively researched for application in various electronic devices including medical equipment [2–5].

Controlling conduction mechanisms is essential in semiconductor devices. Undoped GaSb, grown using various methods and conditions, exhibits *p*-type conductivity due to the charge states of native defects [1]. To obtain a deeper understanding of its defect formation mechanism, the electronic structures and native defect formation energies of GaSb have been theoretically studied using ab initio methods [6–8]. These studies led to the following conclusions: the important native defects in GaSb are Ga antisites (Ga_{Sb}), which act as acceptors, Ga interstitials (Ga_i), which act as donors, and Sb antisites (Sb_{Ga}), which exhibit a neutral charge state. Since Ga_{Sb} has the lowest formation energy of these, undoped GaSb has always been a *p*-type conductor. During the last decade, the defect concentration and the cause of the *p*-type conductivity in GaSb has been extensively studied experimentally [9–13]. The Ga_{Sb} and the Ga vacancy (V_{Ga}) are both found to be present in GaSb and contribute to the *p*-type conductivity. The Ga_i and the Sb_{Ga}, although defects with a low calculated formation energy in bulk GaSb, has not been detected through measurements.

In our previous study, undoped GaSb thin films with *n*-type conductivity were obtained using a Ge(100) 1° off-axis substrate [14]. The off-axis substrate was adopted to suppress the formation of antisite defects. Simultaneously, GaSb thin films were grown under Ga-rich conditions. These conditions accelerated Ga_i formation while suppressing Ga_{Sb} formation. However, the behavior of these native defects and the GaSb/Ge(100) interface in non-stoichiometric GaSb thin films on Ge(100) 1° off-axis substrates remains poorly understood.

This study aims to clarify the behavior of the native defects and the interface of the non-stoichiometric GaSb layer and Ge(100) 1° off-axis substrate in *n*-type GaSb/Ge(100), in comparison with those in GaSb/quartz, via rapid thermal annealing (RTA). RTA is a post-annealing method that can suppress the diffusion of impurities [15] and can be expected to prevent further diffusion of Ga, Sb, and Ge into GaSb thin films from the substrate. Therefore, using this process, the effect of solid-phase growth can be easily evaluated in non-stoichiometric GaSb thin films on both quartz and Ge(100) 1° off-axis substrates.

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2. Experimental procedure

2.1. Growth and annealing conditions of GaSb thin films

GaSb thin films were prepared on quartz and Ge(100) 1° off-axis substrates by RF magnetron sputtering in an HSR-351L system (Shimadzu Emit Co., Ltd.). The off direction of the Ge(100) 1° off-axis substrate was [110]. The source material was a 2-inch diameter GaSb disk of 99.9% purity (Kojundo Chemical Laboratory Co., Ltd.). Sputtering was performed in an Ar atmosphere at 0.5 Pa for 30 min at a growth temperature of 500 °C. The GaSb thin films were subjected to RTA in a N_2 atmosphere. After preheating to 120 °C, RTA was performed for 14, 16, and 17 s using a MILA-5000 heating lamp (Advance Riko, Inc.); the power was then turned off. The peak temperatures after irradiation for the above times were 475 °C, 575 °C, and 615 °C, respectively.

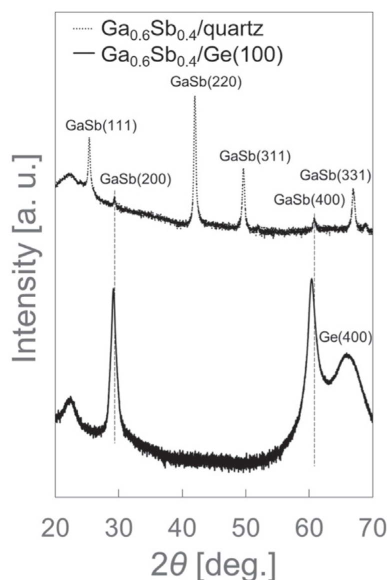

Fig. 1. XRD patterns of $Ga_{0.6}Sb_{0.4}$ thin films on quartz and Ge(100) 1° off-axis substrates. The broken line indicates $Ga_{0.6}Sb_{0.4}$/quartz and the solid line indicates $Ga_{0.6}Sb_{0.4}$/Ge(100). The broad diffraction peak around 20°–25° is attributed to the quartz substrate holder.

2.2. Characterization

The film thicknesses and compositions were determined by X-ray fluorescence (XRF; XRF-1800, Shimadzu), and the surface morphologies were observed by dynamic mode atomic force microscopy (AFM; NaioAFM, Nanosurf) using an anti-vibration table (Isostage, Nanosurf). The crystal structures were evaluated by X-ray diffraction (XRD; RINT-2000, Rigaku) using $CuK\alpha_1$ radiation and microscopic Raman scattering spectroscopy (JRS-SYSTEM 2000, JEOL). Raman scattering was collected in a back-scattering geometry at room temperature (RT) with no polarization; a He-Ne laser (632.8 nm) was used for excitation and the spot size on the sample was $<10\,\mu m$. The measurement conditions of Raman spectrum were as follows; exposure time: 10 s, measurement number: 10 times. Moreover, Hall-effect measurements (HL5500PC, Nanometrics) were performed in the van der Pauw configuration to analyze the electrical properties, considering the depletion layers at the surface and interface [16]. The electrical properties were measured at an impressed current of 5.0 mA for an impressed voltage of 20 mV because the properties of the Ge(100) 1° off-axis substrate should be distinguishable in the Hall-effect measurements of GaSb/Ge(100) [14].

3. Results and discussion

3.1. Properties of as-grown GaSb thin films

The Ga content x of Ga_xSb_{1-x} thin films on both quartz and Ge(100) 1° off-axis substrates is 0.6. The film thicknesses of $Ga_{0.6}Sb_{0.4}$/quartz and $Ga_{0.6}Sb_{0.4}$/Ge(100) are 543 and 569 nm, respectively. The thin film compositions become Ga-rich because Sb, which has higher vapor pressure than that of Ga, is re-evaporated from the substrate. The crystal orientation of $Ga_{0.6}Sb_{0.4}$/Ge(100) differs significantly from that of $Ga_{0.6}Sb_{0.4}$/quartz (Fig. 1), although these non-stoichiometric GaSb thin films are considered to maintain the zinc blende structure in view of the lattice constant. $Ga_{0.6}Sb_{0.4}$/Ge(100) grows epitaxially along the plane of the substrate, whereas $Ga_{0.6}Sb_{0.4}$/quartz forms a polycrystal. The diffraction peaks (GaSb(200) and GaSb(400)) of $Ga_{0.6}Sb_{0.4}$/Ge(100) shift toward lower angles compared with those of $Ga_{0.6}Sb_{0.4}$/quartz. The shift to lower angles indicates that the lattice constant in the surface-normal direction increased, suggesting that compressive strain is induced in $Ga_{0.6}Sb_{0.4}$/Ge(100) due to the difference between the lattice constants of GaSb and Ge ($a_{GaSb} = 6.10$ Å and $a_{Ge} = 5.66$ Å). Fig. 2 shows the surface morphologies of $Ga_{0.6}Sb_{0.4}$/quartz and $Ga_{0.6}Sb_{0.4}$/Ge(100).

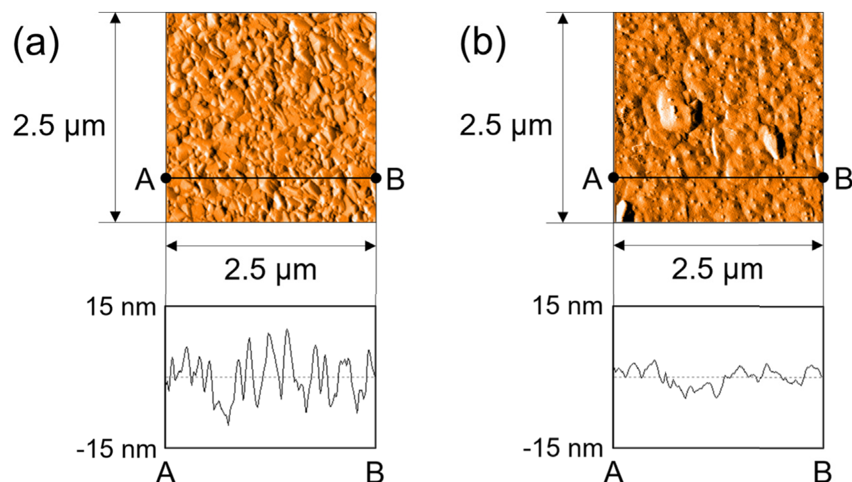

Fig. 2. AFM measurements of $Ga_{0.6}Sb_{0.4}$ thin films grown on (a) quartz substrate and (b) Ge(100) 1° off-axis substrate. The upper figure shows the surface morphology, and the lower figure shows the height profile between A and B in the upper figure.

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