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Thermodynamic evolution of antiphase boundaries in GaP/Si epilayers evidenced by advanced X-ray scattering

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ABSTRACT

We have investigated quantitatively anti-phase domains (APD) structural properties in 20 nm GaP/Si epilayers grown by molecular beam epitaxy, using fast, robust and non-destructive analysis methods. These analyses, including atomic force microscopy and X-ray diffraction, are applied to samples grown by various molecular beam epitaxy growth modes. Roughness, lateral crystallite size of the epilayer, ratio of antiphase domains and their relationship are discussed. It is shown that both these analysis methods are useful to clarify the physical mechanisms occurring during the heterogeneous growth. Low temperature migration enhanced epitaxy is found to guarantee smoother surface than conventional molecular beam epitaxy. Effect of annealing temperature on antiphase boundaries (APBs) thermodynamics is discussed. The modification of the thermodynamic equilibrium through a thermal activation of APBs motion is expected to play an important role in the dynamic evolution of surfaces during thermal annealing and growth.

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1. Introduction

Many recent research developments focused on the ability to develop photonics on silicon. While a large part of optoelectronic integrated circuits elemental components have already been demonstrated, efficient and reliable laser source on silicon is still challenging [1]. The heterogeneous epitaxy of III–V semiconductors on silicon has been first proposed in metamorphic systems; the lattice mismatch between the compound semiconductors (GaAs, InP) and Si, results however in a large density of misfit defects leading to a crippling density of threading dislocations [2,3]. The coherent growth of III–V semi-conductors has then been proposed, based on both the initial deposition of GaP (lattice mismatch ∼0.36%) and the subsequent overgrowth of the diluted-nitride GaPN compound lattice-matched to Si, the lattice matching being realised with an appropriate adjustment of the N content (2.2% assuming Vegard's law) [4–8].

Then, a large variety of light emitters, based on diluted-nitride alloys or quantum nanostructures can be grown on the GaPN/GaP/Si platform [9–14]. Recent research developments have led to the development of optically pumped laser based on diluted-nitrides quantum wells (QW) that is GaAsPN/GaPN/Si [15]. However, the development of electrical injection through the GaP/Si interface or through GaP front-contacts in these devices is still limited by the GaP/Si originating defects, such as micro-twins (MTs) (planar defects related to the 180° rotation of the GaP upon [1 1 1], [1 1 −1], [1 −1 1] and [−1 1 1] axes directions), APDs (defects originating from the growth of a polar crystal on non-polar crystal), or stacking faults (SF) [4,16]. Different strategies have been developed to overcome these problems, such as growth on a misoriented substrate for APDs elimination [6,17,18], alternated growth techniques like migration enhanced epitaxy (MEE) which aim to limit the density of planar defects originating from island-like nucleation [4,6–8,19], and/or multi-temperature growth sequences, including annealing processes which are assumed to improve the overall structural quality [6,7,20,21]. One of the main issues in these studies is related to the structural characterisation of defects, especially for APDs, where dark field TEM images provide only a local information [4,6,22–24]. In-situ reflectance has been proposed for the monitoring of APDs during the growth [25].

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X-ray diffraction (XRD) has long ago demonstrated capabilities for APD detection in bulk material but more recently in the case of epitaxial thin layers (see for instance [21,26]). As a non-destructive and global technique; XRD is complementary to TEM since it does not require sample preparation and provides a larger statistical averaging over the sample. The XRD method described in Ref. [2] has been also applied to the GaP/Si materials system [8,21].

In this paper, we present quantitative APD detection using X-ray scattering on 20 nm GaP epilayers deposited on 4° misoriented Si substrate toward [1 1 0], by several molecular beam epitaxy (MBE) growth modes. A fast and non-destructive method is applied to characterise the crystalline quality of epilayers, based on a complementary combination of both advanced X-ray scattering and atomic force microscopy (AFM) images analysis. Special emphasis is given on the effect of annealing on the GaP/Si samples. Qualitative and quantitative information on the APD shape as well as antiphase boundaries (APBs) thermodynamics during the growth, are extracted from this analysis.

2. Experiment

2.1. Growth of GaP/Si

Samples under investigation are grown in a Riber Compact 21 Solid Source MBE chamber. In this work, (0 0 1)-oriented Si substrates, intentionally offcut of 4° toward the [1 1 0] direction are used, which is expected to prevent from APD generation [5]. Before being introduced into the chamber, the Si substrate is carefully chemically cleaned using the so-called conventional Radio Corporation of America (RCA) procedure [21]. Samples are then transferred to the growth chamber and heated at 900 °C during 10 min. The temperature is then decreased down to 450 °C under P_2 flux. The growth of 20 nm GaP is then performed at this temperature with a growth rate of 0.2 ML/s and a V/III beam equivalent pressure (BEP) ratio equals to 4. Such a low temperature is expected to prevent the generated defects to migrate from the GaP/Si interface to the volume [5]. Three different growth schemes were implemented for comparison. The first one named "MBE" refers to the very conventional MBE growth mode, using a phosphorus excess during all the growth sequence. This kind of growth is typically used in previously developed GaP-based devices [12,27]. The second growth mode, referred in the following as the "MEE" one [4], begins with the deposition of 2 ML of GaP to suppress the melting-back effect of Ga droplets [28]. Then the MEE process starts with an alternation of 5 s deposition of gallium and phosphorus at the surface. Each MEE cycle is separated by a 15 s growth interruption in order to let the atoms migrate. The number of Ga atoms deposited per MEE cycle has been determined to reach a complete GaP monolayer coverage of the surface. The third growth scheme is similar to the previous one, but instead of lowering the temperature down to room temperature after the MEE sequence, post-growth annealing is performed under phosphorus during 10 min at 600 °C. This annealed-MEE sample will be referred as a-MEE in the following.

2.2. Surface morphology and crystalline quality

Surface morphology of the three samples is characterised by AFM measurements in tapping mode giving access to roughness and surface typical shape. With regards to the structural properties, conventional structural characterisations are not well-adapted, due to their inefficiency to detect APDs. Characterisation methods for the epilayers heterogeneously grown usually include dark field transmission electron microscopy (TEM) [8,29], crystallographic etching [30], or even in situ reflectance monitoring [31]. In comparison, analysis based on X-ray scattering is quick, non-destructive and can provide global information for the epitaxial layer. As shown long ago for $AuCu_3$ alloys and more recently for (Al, Ga)N epitaxial layers, XRD can be very sensitive to APD [26,32]. When considering that an APD consists only in a domain with substitution of both the Ga and P positions compared to crystal cells of the surrounding main phase, the scattered intensity in the vicinity of weak reflections $\left|A_{\text{Total}}(S)\right|^2$ is given by:

$$\left|A_{\text{Total}}(S)\right|^2 \approx (F_{\text{Total}}^2(S) + 4F_{\text{APD}}^2(S) - 4F_{\text{Total}}(S)F_{\text{APD}}(S))(f_{\text{Ga}}(S) - f_P(S))^2 \quad (1)$$

where $f_{\text{Ga}}(S)$ and $f_p(S)$ are the atomic scattering factors, $F_{\text{Total}}(S)$ is the form factor of the whole layer (main phase and APD) and $F_{\text{APD}}(S)$ the APD form factor. It becomes clear that the APD form factor remains hidden on strong reflections ((0 0 4) for instance) and brightly appears (4 times larger than the contribution of a void of the same shape) on weak ones ((0 0 2) and (0 0 6) for instance):

Reciprocal space mappings (RSM) are used to determine the strain status and the structural quality. They have been measured on a Bruker AXS D8 advance diffractometer equipped with a Lynx Eye linear detector. Iso-contour and grey scale levels of the intensity are then plotted and transverse scans are extracted. The Cu K_α wavelength (λ = 0.15418 nm) has been used with a focusing reflection incident beam monochromator and a 0.1 mm divergence slit providing an incident beam divergence equals to 0.028° (integral breadth of the silicon substrate (0 0 4) reflexion). The detection resolution, determined by the linear detector channel width, is equal to 0.016°.

Fig. 1(a) describes the geometry of the 4°-off Si substrate crystal. The direction along the mean surface is referred as $\vec{X}_\parallel$ and the growth direction as $\vec{X}_\perp$ in the real space. Thus, the (0 0 l) crystal plane family is about 4° misoriented with respect to $\vec{X}_\perp$. In the following, we will refer to the reciprocal space coordinates ($\vec{S}_\parallel$, $\vec{S}_\perp$) where $\vec{S}_\parallel$ and $\vec{S}_\perp$ are respectively along the mean surface (toward $\vec{X}_\parallel$) and the growth direction (toward $\vec{X}_\perp$).

$$\left\|\vec{S}_\parallel\right\| = \left\|\vec{k}_i - \vec{k}_r\right\| \times \sin\psi; \quad \left\|\vec{S}_\perp\right\| = \left\|\vec{k}_i - \vec{k}_r\right\| \times \cos\psi \quad (2)$$

$\vec{k}_i$ and $\vec{k}_r$ are the reduced scattering vectors along respectively the incident and reflected directions, with $\left\|\vec{k}_i\right\| = \left\|\vec{k}_r\right\| = 1/\lambda$. ψ is the angle between $(\vec{k}_i - \vec{k}_r)$ and $\vec{X}_\perp$ (growth direction). From Fig. 1(b), one can easily understand that both ω–scans (rotation of the sample around the ω axis, while keeping the detector at a fixed 2θ position) and $\omega/2\theta$ scans, that is basically $\theta/2\theta$ scans with the angle of incidence ω different from θ due to the 4°-miscut, (represented by arrows) are not performed along $\vec{S}_\parallel$ and $\vec{S}_\perp$ because of the substrate misorientation and consequently are not adapted to the problem. $\vec{S}_\parallel$ transverse scans have thus been extracted from RSM. The parameter extraction from these curves (peak position, width, area and integral breadth) is performed using pseudo-Voigt fitting functions.

3. Results and discussion

3.1. Surface morphology

Fig. 2(a) presents the 1 μm² AFM image of a 4°-misoriented silicon substrate, which has received both RCA preparation and thermal treatment (900 °C during 10 min). The measurement was performed just after the sample was carried out of the chamber, in order to avoid silicon oxide surface perturbation. Fig. 2(b–d) presents the 1 μm² AFM images for GaP/Si samples grown by different growth modes on similarly prepared silicon surfaces. Surface morphology is characterised by the roughness root mean square (rms). When looking at the initial silicon surface in Fig. 2(a), the flatness of the surface reaches a rms of 0.2 nm (experimental limit

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