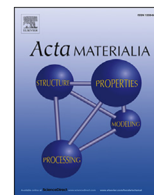
Acta Materialia 143 (2018) 121–129

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat



Full length article

On the origin of threading dislocations during epitaxial growth of III-Sb on Si(001): A comprehensive transmission electron tomography and microscopy study

Michael Niehle [a, *], Jean-Baptiste Rodriguez [b, c], Laurent Cerutti [b, c], Eric Tournié [b, c], Achim Trampert [a]

[a] *Paul-Drude-Institut für Festkörperphysik, Hausvogteiplatz 5-7, 10117 Berlin, Germany*
[b] *Université Montpellier, IES, UMR 5214, F-34000 Montpellier, France*
[c] *CNRS, IES, UMR 5214, F-34000 Montpellier, France*

A R T I C L E I N F O

Article history:
Received 8 June 2017
Received in revised form
22 September 2017
Accepted 24 September 2017
Available online 26 September 2017

Keywords:
Dislocation structure
Antiphase boundary
Tomography
Transmission electron microscopy
Semiconductor heterostructures

A B S T R A C T

Electron tomography and complementary (scanning) transmission electron microscopy (STEM) are applied to investigate the origin of threading dislocations in the large lattice misfit, heteroepitaxial system of III-Sb on vicinal Si(001). Buried AlSb islands of the initial wetting layer are revealed at the interface toward the substrate in the three-dimensionally reconstructed data. Locations of island coalescence are retrieved from the tomogram. Complementary (S)TEM measurements reveal the location of threading dislocations and the presence of antiphase boundaries at the same specimen area. The number density of threading dislocations emanating from the interface and their distribution are unexpected. It is shown that the presence of threading dislocations is not simply correlated to sites of AlSb-islands coalescence or to the film closure during the transition from a 3D to a 2D growth. Moreover, an interaction with antiphase boundaries is suggested by the presented observations. Consequently, the contemporary notion of threading dislocation formation is refined and, eventually, it is suggested that measures to avoid antiphase domains and such to reduce threading dislocations have to be balanced for future strategies to epitaxially grow sphalerite structure III-V semiconductors on Si or Ge.

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1. Introduction

The group III-antimonide compound semiconductors attract attention due to their opto-/electronic properties. They are relevant for the application in infrared (IR) devices as, for instance, lasers and detectors [1,2]. Their integration into mature silicon technology is desirable according to economic aspects. The epitaxial fabrication of antimonide based IR lasers on silicon has been demonstrated [3,4]. The large lattice mismatch between Si and AlSb/GaSb of about 13% is effectively relieved by the introduction of a misfit dislocation (MFD) network located directly at the interface [5]. The MFDs are perfect 90° (Lomer type) dislocations which are sessile in the face-centred cubic lattice structure of III-Sb. Rocher et al. [6] proposed a mechanism for the MFD nucleation during the initial 3D growth mode. They form directly at the intersection of the laterally advancing growth front and the substrate. These perfect edge dislocations are most efficient for the strain relaxation. In contrast, MFDs have to nucleate at the sample surface as half loops in case of layer-by-layer grown heterostructures [7]. The loops extend and glide toward the heterostructure interface leaving threading dislocation (TD) segments within the epitaxial film. These glissile dislocations are of 60° type and less efficient in strain relaxation [6]. The occasional reaction of two complementary 60° dislocations results in a Lomer dislocation (see e.g. Refs. [8,9]) but leaves four threading segments. The detrimental impact of these segments on opto-/electronic device properties [10] demands their reduction or suppression. The aforementioned relaxation mechanism is beneficial because, at first instance, it circumvents the introduction of TDs into the functional antimonide layer stack. On the other hand, the formation of MFD networks happens uncorrelated for each initial island. Consequently, current models discuss the origin of TDs in large lattice misfit epitaxy of III-V materials on Si with regard to the coalescence of islands and the simultaneous linkage of MFD networks [11,12].

* Corresponding author.
E-mail address: niehle@pdi-berlin.de (M. Niehle).

https://doi.org/10.1016/j.actamat.2017.09.055
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The growth of III-V material with sphalerite structure on (001) substrates with diamond structure (Si, Ge) entails the formation of antiphase boundaries due to the presence of single atomic surface steps [13]. In fact, the term antiphase boundary has been predominantly used in the respective semiconductor community for almost five decades [14] although the neighbouring domains are crystallographically related to each other by a polarity inversion. Most authors stick to the term antiphase boundary (e.g. Williams and Carter [15]) and give reason for its use [16] while others insist on the crystallographically correct term inversion domain boundary as defined, for instance, in Ref. [17]. In the following, the term antiphase boundary (APB) is applied. This planar crystal defect arises during the epitaxial growth of material with polar axes on a non-polar substrate due to single atomic surface steps [13]. It occurs independent of the above discussed effect of lattice mismatch as seen in the case of GaP on Si(001) [18] exhibiting an approximate lattice match. A common route to reduce the number of antiphase domains is based on the application of vicinal Si(001) wafers. A wafer miscut of $2^\circ - 5^\circ$ results in the predominant formation of double atomic surface steps [19]. As a consequence, the formed terraces exhibit the same (1×2) surface reconstruction. The III-V layers will nucleate in the same domain type [13] if the first atomic layer on the substrate is composed of exclusively one chemical element type. Practically, a residual number of antiphase domains remains. In our recent study of the 3D arrangement of TDs [20], the strong interaction of dislocations with APBs has been elaborated. Therefore, the discussion on the formation and distribution of TDs should regard the presence of APBs as well. Eventually, the control of material properties requires the understanding of the complex interaction of crystal defects.

In this work, electron tomography is applied to locate the sites of coalescence in the GaSb/AlSb film grown on vicinal Si(001). The presence of TDs is retrieved from the analysis of micrographs that exhibit Moiré contrast. The correlation of both results demonstrates that the mere coalescence of initial islands cannot explain the origin as well as the number and distribution of TDs.

2. Experimental

2.1. Material under investigation

The investigated stack of antimonide based epitaxial layers is grown by molecular beam epitaxy on a vicinal Si(001) wafer. The nominal wafer miscut angle toward the [110] direction is 4°. An overview of the whole sequence of antimonide layers is depicted in Fig. 1. The micrograph shows a cross-sectional specimen imaged along the $[1\bar{1}0]$ direction in the bright-field (BF) scanning transmission electron microscopy (STEM) mode. Layers of different chemical composition are well distinguished. The (Al,Ga)(As,Sb) layers embracing the (In,Ga)(As,Sb) quantum wells (QWs) contain a higher Ga fraction than the Al rich layer below and above the active region. The attention is drawn to dark lines vertically penetrating the epitaxial layer stack. They are assigned to TDs which are traced back to the interface. An AlSb initiation layer of nominally 5 nm thickness is deposited (not resolved in Fig. 1) which allows the growth of smooth GaSb layers [21]. The AlSb forms islands which are the subject of the electron tomographic investigation. It is a merit of the tomographic approach to reveal the buried AlSb islands in their final shape. In that way, a material redistribution during GaSb overgrowth, e.g. similar to the case of InAs on GaAs [22], is excluded. Growth details of similar III-Sb stacks are published elsewhere [3,4].

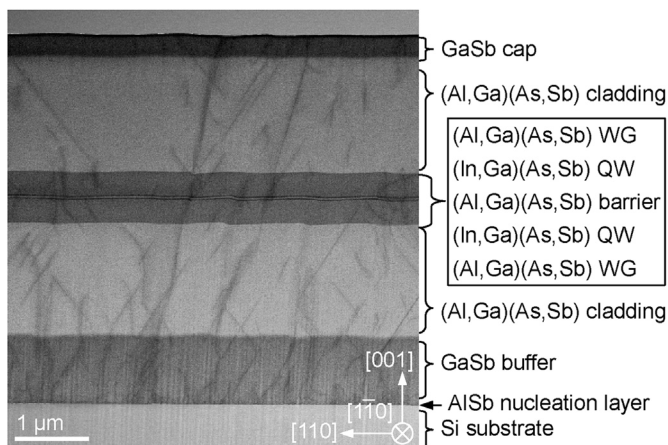

Fig. 1. The BF STEM image of a cross-section through the sample under investigation highlights an overview of the heteroepitaxial stack of antimonide layers and the presence of threading dislocations.

2.2. Tomography specimen preparation

The fabrication of specimens is realized with a dual-beam microscope (JEOL IB4501) comprising a scanning electron microscope (SEM) and a focused ion beam (FIB) in one vacuum chamber. The SEM images in Fig. 2(a) and (b) illustrate the preparation of a specimen that meets the requirements of the tomographic data acquisition and of the target geometry. Electron tomography is ideally carried out on a needle-shaped specimen because it allows to perform the data acquisition over a full rotation without the limitation of increasing specimen thickness at high tilt angles. An as large as possible part of the targeted interface between the substrate (Si) and the antimonide layer (III-Sb) has to be confined in the specimen. Hence, an initial lamella is cut from the cross-section of the heterostructure as shown in Fig. 2(a). This sample orientation enables the extraction of a long stripe of the interface parallel to the later needle axis. The lamella is attached to a micro-manipulator (Kleindiek Nanotechnik GmbH) and transferred to a specimen post suitable for a dedicated TEM tomography holder (Fischione Model 2050). The latter enables the complete rotation of the specimen within the TEM. At this point, it is anticipated that complementary TEM and STEM measurements described below rely on specific crystal orientations. In this respect, the limitation due to the single available tilt axis of the tomography holder has to be remarked. The purposeful, crystallographic orientation of the needle-shaped specimen with respect to the tilt axis is a great benefit of the applied FIB sample preparation in the dual-beam microscope.

After mounting on the specimen post, the lamella is trimmed to form a needle as shown in Fig. 2(b). The last FIB preparation step is performed applying 3 kV acceleration voltage and a circular milling area. The high-angle annular dark-field (HAADF) STEM image in Fig. 2(c) presents the final specimen with the heterostructure interface viewed edge on. Differences between Si and the III-Sb film are observed regarding the remaining amount of material and the surface morphology. In Fig. 2(d), the isosurface representation of the tomographic reconstruction underlines the differently smooth surfaces of both materials. They are indicative for characteristic sputtering and interaction behaviour of the material treated with the Ga ion beam.

2.3. STEM tomography

A JEOL 2100F microscope operated at 200 kV and equipped with

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