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Observation of the stacking faults in In.53Ga.47As by electron channeling contrast imaging

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Abstract:	The observation and interpretation of Frank stacking faults, Shockley stacking faults, Lomer dislocations and 60° misfit dislocations which have similar line shapes in the (001) In.53Ga.47As crystalline surface are performed with the electron channeling contrast imaging (ECCI) technique. In order to minimize the backscattered electron (BSE) contrast resulted from the surface morphology, a relatively flat region is first selected and compared with an atomic force microscopy (AFM) image and then, secondly, examining ECCI with TEM-like invisibility criteria. By orthogonally choosing the diffraction vector $g \perp$ between (220) and (2-20), misfit dislocations seem to be always visible but partially faint in the $g \perp$ parallel to line direction on the surface. In respect of the image contrast, Frank stacking faults and Lomer dislocations are likely to be completely invisible for parallel $g \parallel$. The criteria are further confirmed by cross-sectional TEM analysis, which shows a preferred homogeneous surface nucleation.
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GADEST 2019 special issue

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1 Observation of the stacking faults in In_{0.53}Ga_{0.47}As by electron channeling 2 contrast imaging

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13 **Keywords:** electron channeling contrast imaging (ECCI), InGaAs, heteroepitaxy, misfit
14 dislocation, stacking fault, atomic force microscopy (AFM)

15 Abstract

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28 nucleation.

1. Introduction

One of the most fundamental methods for evaluating the quality of crystalline semiconducting material is to quantify and identify the type of extended defects. This method is particularly important for complementary metal-oxide-semiconductor (CMOS) technology which tends to implement III/V heterostructures on the Si substrate^[1] to reduce the production cost and to provide more functionality. One of the major issues in heterostructures are the various extended defects caused by the lattice and thermal mismatch during heteroepitaxy^[2]. For over decades, many techniques have been used for carrying out defects' analysis, e.g. transmission electron microscopy (TEM), X-ray diffractometer (XRD)^[3], cathodoluminescence (CL)^[4], selective defect etching (SDE)^[5] and electron-beam-induced current (EBIC)^[6], each technique having its well-known advantages and disadvantages^[7]. Among other, TEM is the most widely used due to the ability to clearly distinguish various types of defects via two-beam analysis with excellent spatial resolution. However, the relatively time-consuming and destructive sample preparation confines the application to a limited number of samples, and a small analyzed area (typically ~ a few μm^2) makes TEM relatively difficult for measuring samples with a defect density $< 10^7 \text{ cm}^{-2}$.

By contrast, Electron Channeling Contrast Imaging (ECCI) is a non-destructive electron microscopy technique with many similarities to TEM. ECCI makes use of the electron diffraction phenomenon inside crystalline materials to image the near surface crystalline defects (e.g. threading dislocations (TD), stacking faults (SF)). In scanning electron microscopy (SEM), if an electron beam scans a large area of the specimen surface at low magnification, the intensity of backscattered electrons (BSE) will change rapidly near Bragg angles due to electron channeling^[8], thus generating the so-called electron channeling bands (ECB) visualized as dark lines on the top of BSE images. Each ECB represents a specific atomic plane corresponding to its Bragg angle, so a set of ECBs can further form an electron channeling pattern (ECP) (e.g. Figure 1), which can be indexed according to the

crystalline directions of the specimen normal to the surface. At Bragg angles, the BSE intensity has a higher sensitivity to the lattice plane bending around crystalline defects and is thus greatly modulated to reveal the defects as either bright dots or lines with a black-white contrast change. Furthermore, the well-known invisibility criteria ($\vec{g} \cdot \vec{b} = 0$ and $\vec{g} \cdot \vec{b} \times \vec{u} = 0$, where $\vec{b}$, $\vec{u}$ and $\vec{g}$ are the Burgers vector, the dislocation line element and diffraction vector, respectively) for defect analysis in TEM are proved applicable in ECCI [9]. Likewise, the invisibility criterion for a stacking fault is $\vec{g} \cdot \vec{R} = n$, where $\vec{R}$ and n are the displacement vector and an integer number. It allows the determination of the defects' Burgers vector by acquiring a group of ECCIs with carefully selected diffraction vectors. In addition, the defect contrast reversal through selection of anti-parallel diffraction vectors is also repeatedly reported for defect analysis [10-12].

In heteroepitaxial III-V ternary compounds with a zinc-blende structure, the strain relaxation occurs primarily by a 2D network of 60° misfit dislocations (MD) in the {111} <110> slip system. Among the MDs, two like-sign asymmetric dislocations can be distinguished based on the direction of the half-plane and the missing atoms in the most distorted core [13]. By using the notation of Alexander et al. [14], an As core glide set dislocation $As_{(g)}$ and Ga (or In) core shuffle set dislocation $Ga_{(s)}$ and $In_{(s)}$ are identified as α type misfit dislocation (α MD), and vice versa $Ga_{(g)}$ (or $In_{(g)}$) and $As_{(s)}$ are β type misfit dislocations (β MD). Because of the opposite edge component of the two dislocations, the first one tends to relieve tensile stress and the other to relax compressive stress, while the preferred orthogonal array of both dislocations parallel to the (001) surface is also changed with tensile/compressive stress [15]: α MD is likely to yield MD lines in [110] direction under tensile stress and in [1-10] direction under compressive stress. By contrast, β MD results in [1-10] under tensile stress and in [110] under compressive stress. In addition, the shuffle set is believed to be sessile in III-V heterostructure [16][17], and a mobile 60° misfit dislocation is

generally understood to belong to the glide set. Hence, only glide set 60° MD are considered in this work, i.e. $\text{As}_{(g)}$, $\text{Ga}_{(g)}$ and $\text{In}_{(g)}$.

Stacking faults (SF) are frequently observed in heteroepitaxial III-V compounds as a common planar defect. Especially in selective area heteroepitaxial growth with shallow trench isolation (STI) [2][18] which can efficiently “trap” $\{111\}$ defects in trenches and significantly reduce the dislocation density, stacking faults seem to be a major defect [2]. In general, SF could form primarily by either dissociating a glide set 60° MD into a 30° and a 90° Shockley partial dislocation binding an intrinsic stacking faults, or by directly nucleating intrinsic (or extrinsic) stacking faults involving a Frank partial dislocation with the diffusion of vacancies, interstitials or impurities. The first have a displacement vector $\vec{R}$ in $a/6 \langle 112 \rangle$ and have the possibility to glide. On the contrary, the latter have an $a/3 \langle 111 \rangle$ displacement vector and are believed to be sessile.

The aim of this paper is to distinguish between 60° MDs, Lomer dislocations and SFs by ECCI which have similar line shapes on the (001) surface in zinc-blende structures. With the help of TEM-like invisibility criteria, the direction of the different defects can be determined by changing the diffraction vector $\vec{g}$ orthogonally. Besides, the cross-hatching pattern generated from the strained field of buried misfit dislocation at the heterointerface [19] will result in the contrast of BSE image due to the edge effect of surface morphology. In order to minimize this contrast, a relative flat region (the root mean squared roughness < 0.5 nm) will be selected by comparing the image from atomic force microscopy (AFM) which have high sensitivity on z-axis (Height). Therefore, the ECCI of MDs reported in this paper is a relatively pure “channeling” image of the MDs gliding to the near surface.

2. Experimental details

Three blanket samples, having the same structure but different Indium content of 150 nm p+ $\text{In}_{.53}\text{Ga}_{.47}\text{As}$ ($\sim 10^{18} \text{ cm}^{-3}$) / 850 nm n- $\text{In}_{.53}\text{Ga}_{.47}\text{As}$ ($\sim 2 \times 10^{16} \text{ cm}^{-3}$) / 550 nm n+ $\text{In}_x\text{Ga}_{1-x}\text{As}$.

$x\text{As}(\sim 2 \times 10^{19} \text{ cm}^{-3})$ /2" semi-insulating (SI) InP substrate (001), are grown by Metal-Organic Vapor Phase Epitaxy (MOVPE) [20]. The growth-induced extended defects are generated intentionally by the lattice mismatch of the n^+ layer with different indium content x and propagated to the surface of p^+ $\text{In}_{.53}\text{Ga}_{.47}\text{As}$ of interest that is analyzed by ECCI. Depending on the indium content x , the Ga-rich samples are labelled as P_In53 (lattice match), P_In47 and P_In40, respectively. Except these Ga-rich samples, an In-rich sample with same $p^+/n^-/n^+/SI$ -InP layout but with indium component 76% in full layer is marked as P_all_In76. The extended defect density of the n^+ layer and n^- layer (up to 750nm from the junction toward the n^+ layer) is estimated by the rocking curve of high-resolution X-ray diffraction (HRXRD) and deep level transient spectroscopy (DLTS) [20], showing a range between 1×10^5 and $1 \times 10^8 \text{ cm}^{-2}$.

ECCI measurements are carried out by using an Apreo lab SEM system from Thermo Fisher Scientific, equipped with an annular solid-state BSE detector mounted below the pole piece. The specimen can be aligned with the primary beam (optical axis) with an accuracy of 0.1° through a combination of tilting and rotation of the sample stage, to realize the fine control of the diffraction conditions. In this study, the used beam voltage is either 5 keV or 20 keV, and the current is 0.8 nA at the diffraction vectors $\vec{g}$ of (220) and (2-20) as shown in Figure 1. The default working distance is 5 mm and the inner and outer diameter of the annular detector is 1.5 mm and 8 mm, respectively.

AFM images are performed on a Bruker dimension edge Atomic Force Microscope in tapping mode. The tip is made of silicon with a 7nm radius sharpened tetrahedral shape. The resolution is 512×512 pixels per image and the scan rate is 0.5Hz, giving a lateral resolution of $\sim 0.1 \mu\text{m}$ in $50 \times 50 \mu\text{m}^2$ and $\sim 10 \text{ nm}$ in $5 \times 5 \mu\text{m}^2$ AFM images.

After capping with SoC and Pt, TEM lamella are prepared along the (1-10) direction by dual beam FIB/SEM Helios 450 HP system with a beam energy of 5kV. Then the high resolution (HR) TEM and bright field (BF) TEM are both performed on Titan³ G2 60-300 (FEI)

with a beam energy of 120kV, the latter uses 2-beam conditions (220) to visualize the strain field around lattice defects.

3. Results

ECCI at $\vec{g} = (220)$ with different beam voltage for the three samples are shown in Figure 2 (f)-(k) and compared to the morphology from AFM in Figure 2 (a)-(e). In the AFM image, many cross-hatch lines in the [1-10] direction originating from α MD^{[15][21]} can be observed in Figure 2 (b)-(d) and additional defects called micro-crack^{[22][23]} (dark grooves in [110] direction in Figure 2 (c)) are recognized in sample P_In40 only. A detailed interpretation can be found elsewhere^[20]. Although the AFM image fairly well reveals the defects by morphology variation, by selecting a relative flat region (the square region in Figure 2 (d) and (e)), ECCI could further distinguish more defects from dark or bright contrast straight lines in the [1-10] direction. This kind of defects which is neither visualized by AFM nor originating from surface roughness have the same origin as α MD as will be discussed below. Besides, by the higher beam voltage (20kV) in ECCI, the terraces on the surface disappear because the larger interaction volume of BSE results in a higher detection depth.

To further determine the orientation of the defects, two orthogonal diffraction vectors $\vec{g} = (220)$ and $\vec{g} = (2-20)$ are applied on the same area (Figure 3). It clearly shows that the contrast of the line defects in the [1-10] direction (vertical lines in the graph) becomes relatively obscure when rotating the diffraction vector to a parallel direction $\vec{g} = (2-20)$ (i.e. Figure 3 (a)(b) compare to (d)(e)). On the other hand, if zooming in a smaller area in sample P_In40, another line defect is found in the opposite direction [110] (horizontal lines indicated by arrows with yellow color) in Figure 3 (g). In contrast to the previous defect, this kind of defect tends to be completely invisible when rotating the diffraction vector to a parallel direction $\vec{g} = (220)$ (i.e. Figure 3 (g) compare to (c)).

4. Discussion

The tendency of contrast for these two defects can be explained by the invisibility criterion [9][24]. For an overlayer under tensile stress, there are eight primary slip systems which produce misfit dislocations with a positive edge component for (001) crystalline directions [20]. These eight systems [21][25] are listed in Table 1. Because the gliding mobility of α MD is much larger than for β MD [26], α MD tends to elongate fast and then blocks β MD, resulting in a preferred [1-10] anisotropic morphology and asymmetric relaxation originating from α MD in the tensile $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{InP}$ system [15][16],[20-23],[27]. If performing ECCI on this preferred α MD (between Figure 3 (a)(b) and (d)(e)), it should be always visible because $\vec{g} \cdot \vec{b}$ never equals zero for both $\vec{g} = (220)$ and $\vec{g} = (2-20)$ diffraction directions. Nevertheless, the α MD can still become faint when using the parallel $\vec{g} = (2-20)$, which results in $\vec{g} \cdot \vec{b} \times \vec{u} = 0$ and then partially invisible. A similar conclusion is also drawn in the GaP/Si system [28], giving a good agreement with the α MD analysis in Table 1 blue region.

On the other hand, the horizontal defect at [110] in Figure 3 (g) is unlikely to be a 60° MD because of the completely invisible feature while using the parallel $\vec{g} = (220)$. Two possible defects, $\langle 110 \rangle \{001\}$ Lomer dislocations or $\{111\}$ Frank stacking faults listed in Table 1 (orange region), are likely to be the candidates. To further distinguish Frank stacking fault and Lomer dislocation, a $\vec{g} = \{224\}$ should be used. Unfortunately, this diffraction vector is not available in our sample as shown in Figure 1. It should be remarked that glissile stacking faults with two Shockley partials dislocations could not be completely extinguished while using $\vec{g} = (220)$ and $\vec{g} = (2-20)$. This is due to the co-existence of two $\langle 112 \rangle$ displacement vectors, e.g. $1/6 [\bar{1}12] + 1/6 [121] = 1/2 [011]$, giving a non-zero value when $\vec{R}$ is partially $[\bar{1}12]$ and $[121]$. As a result, glissile stacking faults are likely to have a similar behavior as 60° MD while using $\vec{g} = (220)$ and $\vec{g} = (2-20)$.

In addition, another shape of stacking faults are also observed in In-rich sample P_all_In76 of compressive $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{InP}$ system. A trapezoid shape of staking faults are clearly showed in Figure 4, which are already reported [28][29] and modeled by other groups [30]. Based on the report, it is likely to be a glissile stacking faults with two Shockley partials dislocations ended at two sides. Unfortunately, we could not validate the ECCI by orthogonal g vector because of the blurry ECP resulted from super rough morphology ($R_q \sim 20\text{nm}$ based on AFM, not shown in here). The edge of ECB can merely be recognized without clearly distinguishing (220), (2-20) and (040) band.

Several papers [15][22-23][27] have reported that stacking faults, twins, V-shape grooves and cracks seem to be generated by homogeneous nucleation at the (100) surface followed by a Cottrell-Lomer lock and a sessile Frank partial dislocation [16][32] in the tensile $\text{In}_{1-x}\text{Ga}_x\text{As}/\text{InP}$ systems. Coincidentally, we also found out these featured defects from cross-section TEM close to surface. A clear V-shape Cottrell-Lomer lock extended to the surface is shown in Figure 5 with a 90° stair-rod dislocation at its tip and two intrinsic SFs on the side. This defect locked by two 60° Shockley partial dislocations nucleated from the surface is frequently observed in large misfit strain systems [22][31][32]. In addition, a typical twin close to the surface (Figure 6) is also found in our sample, inferring the existence of Frank partial dislocations by considering the twin nucleation mechanism [31]. In our samples, due to the relaxation from preferred α MD gliding in the [1-10] direction, the strain relief for the [110] direction is relatively large. It explains why stacking faults, twins, V-shape grooves and cracks are all likely to nucleate in the [110] direction.

5. Conclusion

Three tensile $\text{In}_{.53}\text{Ga}_{.47}\text{As}/\text{In}_x\text{Ga}_{1-x}\text{As}/\text{InP}$ with different $x=53\%$, 47% and 40% , and one compressive $\text{In}_{.76}\text{Ga}_{.34}\text{As}/\text{InP}$ specimens are analyzed by ECCI and their surface morphology is compared by AFM. Two additional defects which cannot be distinguished in

AFM are observed and characterized. One defect is always visible while using $\vec{g} = (220)$ and $\vec{g} = (2-20)$ and the other tends to be out of contrast when using a $\vec{g}$ vector parallel to the line direction on the surface. Based on the invisibility criteria, the first one is likely to be an α MD and the latter seems to be a Lomer dislocation or Frank stacking fault. A further validation from cross-sectional TEM is also performed, showing a Cottrell-Lomer lock and a twin close to the surface. Based on the mechanism of defect formation, it is concluded that the defects in the $[110]$ direction are created by homogeneous surface generation due to the large misfit strain. Besides, the stacking faults with trapezoid shape are observed in compressive sample, which seems to be the glissile stacking faults with two Shockley partials dislocations ended at two sides based on the literature reports. Finally, useful criteria to distinguish MD, SF and Lomer dislocations are proposed and discussed based on our results.

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Figure 1 (a) A typical electron channeling pattern (ECP) with the (001) zone axis parallel to the primary beam from $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ (P_In53) and (b) a schematic correspondingly indexed ECP according to the crystalline directions of the specimen. The red cross marked in ECP are two g vectors used in this paper.

Figure 2 Two different scanning size AFM images are on the left hand-side (a)-(e) and ECCI $\vec{g} = (220)$ with beam voltage 20 kV and 5 kV are on the right-hand side (f)-(k) for three different samples. R_q is the root mean squared roughness in the unit of nm. ECCI is performed in the relatively flat region inside the white squares of (d) and (e)

Figure 3 ECCI of sample P_In47 and P_In40 with two orthogonal $\vec{g}$ vectors. Two different defects are found at $[1-10]$ and $[110]$ line direction, respectively. The one in the $[1-10]$ direction (vertical lines) tends to be faint (but still in contrast) while $\vec{g}$ is parallel to its line direction ((d) and (e)). The other in $[110]$ (horizontal lines indicated by arrows with yellow color in (g)) is completely out of contrast in (c).

Figure 4 The ECCI of sample P_all_In76 with g vector (220) or (2-20). Many threading dislocations are recognized by white-black dots. Two stacking faults with trapezoid shape, probably consist of a glissile stacking faults and two Shockley partials dislocations ended at two sides, can be observed in the yellow square.

Figure 5 (a) HRTEM images of sample P_In40, (b) is the frequency-filtered image of (a) by inverse fast Fourier transformation (FFT). A clear Cottrell-Lomer lock is shown with a 90° dislocation at its tip and intrinsic SFs on two sides.

Figure 6 (a) the TEM bright field (220) image and (c) is the frequency-filtered image of (b) by inverse fast Fourier transformation. Two twin boundaries are shown with white lines and the mirror lattice points are indicated with red and yellow color.

Type of dislocation and stacking fault	Line direction parallel to the (001) surface	Burgers vector $\vec{b} (MD) \times (a/2)$ Displacement vector $\vec{R} (SF) \times (a/3)$	Slip plane	Invisibility with $\vec{g} = (220)$	Invisibility with $\vec{g} = (2\bar{2}0)$
β 60°=Ga _(g) (tensile stress)	[110]	[011]	(1 $\bar{1}$ 1)	– (weak)	–
		[$\bar{1}$ 01]	(1 $\bar{1}$ 1)	– (weak)	–
		[01 $\bar{1}$]	($\bar{1}$ 11)	– (weak)	–
		[$\bar{1}$ 0 $\bar{1}$]	($\bar{1}$ 11)	– (weak)	–
α 60°=As _(g) (tensile stress)	[1 $\bar{1}$ 0]	[01 $\bar{1}$]	(111)	–	– (weak)
		[10 $\bar{1}$]	(111)	–	– (weak)
		[101]	($\bar{1}\bar{1}$ 1)	–	– (weak)
		[011]	($\bar{1}\bar{1}$ 1)	–	– (weak)
Frank stacking fault	[110]	[1 $\bar{1}$ 1]	(1 $\bar{1}$ 1)	○	–
		[$\bar{1}$ 11]	($\bar{1}$ 11)	○	–
	[1 $\bar{1}$ 0]	[111]	(111)	–	○
		[$\bar{1}$ 11]	($\bar{1}$ 11)	–	○
Lomer dislocation	[110]	[1 $\bar{1}$ 0]	(001)	○	–
	[1 $\bar{1}$ 0]	[110]	(001)	–	○

Table 1 The Burgers vector, line direction on the (001) surface and slip plane for specific defects. The results of the invisibility criteria is examined based on $\vec{g} \cdot \vec{b}$ and $\vec{g} \cdot \vec{b} \times \vec{u}$, and marked by – (visible) and ○ (invisible).