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Characterization of bulk <111> 3C-SiC single crystals grown on 4H-SiC by the CF-PVT method

L. Latu-Romain^{1a}, D. Chaussende^{1b}, C. Balloud², S. Juillaguet²,
L. Rapenne¹, E. Pernot¹, J. Camassel², M. Pons¹ and R. Madar¹

¹ INPGrenoble-CNRS, BP 46, 38402 Saint Martin d'Hères, France

² Groupe d'Etude des Semiconducteurs, Université des Sciences et Techniques du Languedoc,
34095, Montpellier Cedex 5, France

E-mail: ^alaurence.laturomain@ltpcm.inpg.fr, ^bdidier.chaussende@inpg.fr

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Abstract. Because of the formation of DPB (Double Positioning Boundary) when starting from a hexagonal <0001> seed, DPB-free 3C-SiC single crystals have never been reported up to now. In a recent work we showed that, using adapted nucleation conditions, one could grow thick 3C-SiC single crystal almost free of DPB [1]. In this work we present the results of a multi-scale investigation of such crystals. Using birefringence microscopy, EBSD and HR-TEM, we find evidence of a continuous improvement of the crystal quality with increasing thickness in the most defected area, at the sample periphery. On the contrary, in the large DPB-free area, the SF density remains rather constant from the interface to the surface. The LTPL spectra collected at 5K on the upper part of samples present a nice resolution of multiple bound exciton features (up to $m=5$) which clearly shows the high (electronic) quality of our 3C-SiC material.

Introduction

3C (or β) SiC is the only known cubic SiC polytype. Because of its excellent intrinsic properties, 3C-SiC should be more and more considered in the forthcoming years. For instance the cubic lattice isotropy, associated with a high mobility of electrons, makes it a good candidate for n-type inversion channel MOSFET applications [2]. In a totally different application area, <100>-oriented 3C-SiC can also be used as seed for the growth of cubic III-nitrides [3]. Unfortunately, the only “bulk-like” material available (free-standing <100>-oriented 3C-SiC layers grown from Si substrates) is still of poor structural and electrical quality [4]. As a consequence the growth of, strictly speaking, bulk 3C-SiC single crystal has never been reported up to now.

To by pass the lack of 3C-SiC seed, the most realistic way is to grow <111>-oriented 3C-SiC layers on a hexagonal <0001> seed. This avoids the problems of lattice mismatch and differential thermal expansion. However, in this case, one encounters the formation of Double Positioning Boundaries (DPBs). In a previous work, a detailed high temperature study of 3C-SiC nucleation on a 6H-SiC seed was presented [5] which showed that, using adapted nucleation conditions, thick single crystals nearly free of DPB could be grown [1]. In this work, we investigate the structural and optical properties of such bulk-like 3C-SiC material in order to characterize its suitability for microelectronic device applications.

Experiments

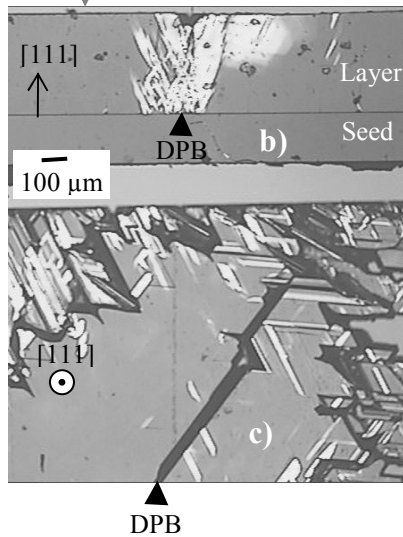
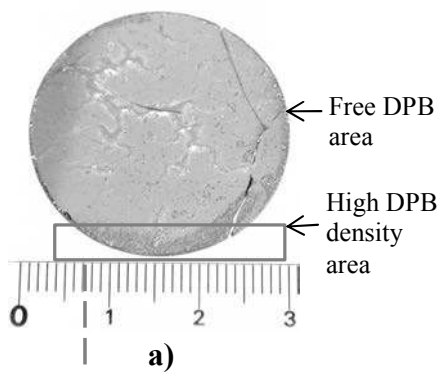
The 3C-SiC sample used in this work was grown in a graphite crucible using the CF-PVT (Continuous Feed-Physical Vapour Transport) method. Briefly, this process can be assimilated to sublimation fed by high temperature CVD through a highly porous medium [6]. The seed was a 30

mm diameter on-axis 4H-SiC substrate, with an “Epi-ready” Si-face prepared by NOVASiC StepSiC™ polishing. The seed was first heated up to 1875°C under a high silicon partial pressure, with a heating ramp of 12.5°C/min. A slower rate (10°C/min) was next used to raise the seed up to the growth temperature (1950°C). The polycrystalline SiC source was elaborated in-situ, from 50 sccm of tetramethylsilane (TMS) diluted in argon. The TMS injection started from 1850°C at 2 Torr. With such conditions, a 0.4 mm thick 3C-SiC layer was grown at 68 μm/h.

Both structural and optical investigations were carried out at different scales. They ranged from large areas (3*3 mm²) EBSD (Electron Back Scattering Diffraction) maps to nanometre scale HR-TEM (High Resolution- TEM) investigations. Low Temperature PhotoLuminescence spectra, collected at 5K, were used to complement the data.

Results and discussion

Roughly three main kinds of extended defects should be observed.



-Double Positioning Boundaries. As described by Kong et al. [7], DPBs are a special case of twin boundaries which comes from the two possibilities of orienting a threefold cubic 3C-SiC axis on a sixfold hexagonal basis. From several 3x3 mm² EBSD maps collected on the as-grown surface, we find that the top part of sample is free of DPB and free of hexagonal inclusions. The sample is pure 3C-SiC, with a single domain size of ~ 30 mm diameter. However, at the sample periphery, a small (defective) area still contains a high density of DPBs (Fig.1a,b,c). On the in-plane view, the DPBs appear as trenches which separates two twinned 3C-SiC domains and the cross-section view shows that the DPBs start from the seed to layer interface. This is consistent with the idea that the DPBs are due to nucleation steps.

In Fig.2 we show two cross-sectional, cross-polarised, optical microscope views: a) close to the seed and b) close to the surface. In Fig.2-a, the bottom arrows indicate 2 DPBs starting from the layer-seed interface, while the top arrow points out the end of the DPB for a layer thickness of 150 μm. Fig.2-b is almost DPB free.

Fig. 1: a) 30 mm diameter 3C-SiC thick layer nearly free of DPB. Cross-polarized optical microscopy in the high DPB density area. DPBs are observed b) in cross-section and c) in planar view geometries.

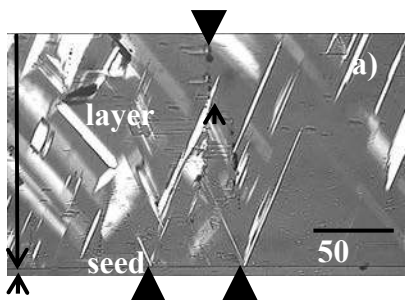


Fig. 2: Two examples of cross-polarised optical microscope views: a) close to the seed ; b) close to the surface.

-Micro-twins (MTs). Due to their twin nature, such micro-domains shift the light polarization. They are then easily observed under cross-polarized transmitted light microscopy (Fig.1 and Fig.2). MTs lie along the $\{111\}$ planes and appear as linear or trapezoidal shapes, depending on the direction of observation. These two figures clearly evidence that most of the MTs are related to the presence of DPBs. It is believed that DPBs are highly uncoherent grain-boundaries and the high energy stored is (in part) relaxed by the high density of MTs. Far from DPBs, the MT density is very low, as for example to the left and to the right of the DBP in Fig.1b,c. After elimination of the DPBs with thickness, the MT density is also very low (see Fig.2b taken close to the surface).

-Stacking Faults (SFs). According to their size and their low stress field, SFs cannot be observed by birefringence microscopy. TEM specimens were then prepared in the DPB free area. Even if no extended defects could be observed by birefringence microscopy at the interface, the estimated SF density was of the order of $1 \times 10^4 \text{ cm}^{-1}$ within the first $2 \mu\text{m}$. The 3C/4H interface is thus more appropriate to the growth of 3C-SiC films than the 3C-SiC/Si one (in which the defects density is close to 10^{12} cm^{-2}). However, the SFs density close to the surface is still similar to the value reported close to the interface, and then similar to the value reported close to the surface of Hoya wafers [8].

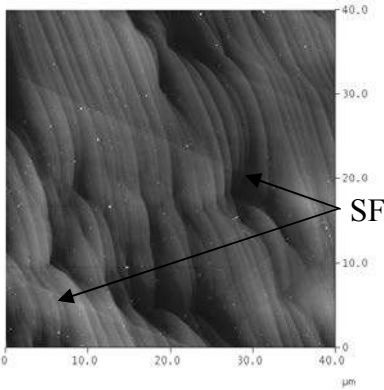


Fig. 3: As-grown morphology observed by AFM. Scanned area = $40 \times 40 \mu\text{m}^2$.

On Fig.3, the straight lines which cross the growth front are due to the emergence of a SF at the sample surface. Note that this SF does not affect the step flow growth.

Optical investigations

In Fig. 4 we show an example of a low temperature luminescence spectra collected at 5K. Excitation was provided by the 244 nm line of a FreD (Frequency Doubled) 20 mW Argon-ion laser. Detection was done using a cooled CCD camera.

On every spectra we resolve the usual NBE (Near Band Edge) features found in low temperature, high quality, 3C-SiC recombination spectra [9]. They have been labelled ZPL (Zero Phonon Line) and TA, LA, TO and LO-phonon replicas (with index 1 because, in this case, there is only one exciton bound to the recombination centre). We also resolve numerous extra features. First is the weak (intrinsic) I_{TA} line which comes from the radiative recombination of a free exciton with emission of a TA (46 meV) phonon. It gives for the 3C-SiC excitonic bandgap at 5K a value:

$$E_g(X) = 2.389 \text{ eV},$$

which is in excellent agreement with the result of Ref. [10]. More interesting are the companions lines of the main ($m=1$) NBE exciton features. They are Multiple Bound Exciton Complexes (MBEC) which have been labelled $m=2$ to 5 to indicate the number of electrons and holes which participate in the final (excitonic) molecule.

These multiple bound exciton features are a specific signature of the low temperature

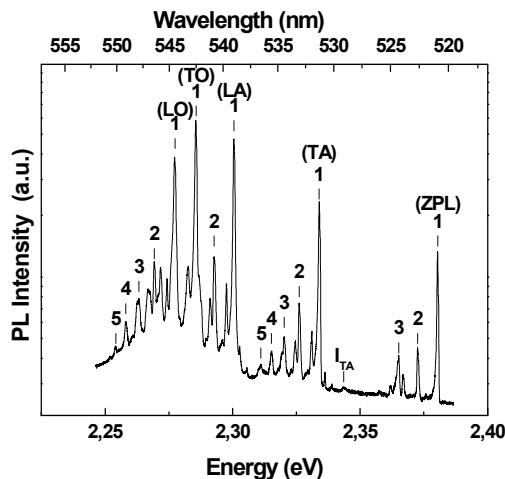


Fig. 4: Example of the LTPL spectra collected at 5K. Notice the presence of MBEC, up to $m=5$. This appears only on high quality 3C-SiC material.

luminescence spectra of high quality 3C-SiC single crystals. They have only been reported in a few works (see, for instance, Ref.[11] and references therein) performed on a few specific samples. Basically, before they can be observed three conditions must be fulfilled. First, one needs a high enough excitation intensity in order to create more than one single exciton per recombination centres. This makes the LTPL spectrum excitation-density dependant. Second, one needs a low density of recombination centre (i.e. a high quality material) to fulfil more easily condition #1. Third, one needs rather long carrier lifetime (i.e. again a high quality material) to give a chance to the extra-excitons ($m=2, 3\dots5$) to bind and not to recombine directly as intrinsic luminescence lines, like the I_{TA} feature in Fig.4.

Conclusion

Bulk (111) 3C-SiC single crystals have been grown on 4H-SiC substrates by the CF-PVT method. With adapted nucleation conditions, the 30mm-diameter samples were almost free of DPB. The main extended defects were micro-twins and stacking faults. At the sample periphery, a higher density of defects correlates with the presence of DPBs but, even in these defected areas, large improvements are found vs thickness. In the DPB free areas, the SF density remains constant from bottom to top but close to the density measured on Hoya material. The LTPL spectra collected at 5K in the DPB-free area presents a nice resolution of near band edge excitonic features (with MBEC resolution up to $m=5$). This evidences the high (electronic) quality of our 3C-SiC material and constitutes a most promising result for the development of high quality 3C-SiC wafers.

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