Characterization of protrusions and stacking faults in 3C-SiC grown by sublimation epitaxy using 3C-SiC-on-Si seeding layers

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Abstract

In this article, sublimation growth of 3C-SiC on 3C-SiC-on-Si seeding layers was evaluated by characterizing the densities of protrusions and stacking faults (SF). Both defects are among the most critical concerning the growth process and the realization of high quality material for device applications. By variation of growth parameters like temperature, growth rate and 3C-SiC-thickness we conducted a series of experiments and characterized these layers by optical microscopy and KOH etching. The protrusion density is predetermined by the seeding layers and was kept at a constant level, whereas a decrease of SF-density was observed with increasing layer thickness during subsequent sublimation growth steps. Therefore, in the case of Sublimation Epitaxy (SE) it has been found appropriate to distinguish between defects that can be reduced during SE and defects that are merely reproduced from the seeding material during sublimation growth. Furthermore, a weak trend towards a decrease of SF-density with increasing growth temperature was observed. The findings in this work demonstrates the potential of SE in growing thick and high-quality 3C-SiC layers if sufficiently good seeding layers were available. Copyright © 2017 VBRI Press.

Keywords: 3C-SiC, sublimation epitaxy, single crystalline, protrusions, stacking faults.

Introduction

Cubic silicon carbide (3C-SiC) is supposed to be a promising candidate for power electronics in the medium voltage range between 600 V and 1200 V [**1, 2**] that could fill the gap between silicon- and 4H-SiC-based applications. 3C-SiC shows the highest electron mobility and saturation drift velocity [**3, 4**] while the band gap (2.3 eV [5]) is the lowest among all SiC polytypes. In contrast to the situation for 4H- and 6H-SiC, trap states at the 3C-SiC/SiO_2-interface are within the conduction band representing a major advantage for MOS-devices [**1, 2**].

However, the cubic polytype of SiC shows various technological challenges regarding the growth-process and the lowering of defect-densities that hinder the implementation of reliable devices. Since Nishino et al. [**6**] introduced a multi-step Chemical Vapor Deposition (CVD) growth process for 3C-SiC heteroepitaxy on Si-substrates in 1982, a variety of approaches has been proposed aiming to improve the quality of cubic silicon carbide. Promising concepts include the growth on patterned Si-Substrates [**7–10**], the Switch-Back-Epitaxy [**11**] and the use of various precursors with varying C/Si ratios, just to mention a few of them.

From an economic point of few, heteroepitaxy on Si-substrates provides the greatest potential for cost reduction and scale up of wafer-size [**12**] and therefore outperforms epitaxy on hexagonal SiC-substrates. However, notwithstanding the promising results that have already been achieved with heteroepitaxy on Si, the 3C-SiC/Si-system still suffers from high defect densities. A large mismatch in lattice parameters (19.7 % at room temperature [**13, 14**]) and in thermal expansion coefficients (~ 23 % at deposition temperatures and 8 % at room temperature [**15, 16**]) between the two materials results in the incorporation of misfit dislocations [**17**], planar defects [**18**] and the occurrence of stress fields. The latter can even lead to a bending of the whole wafer, also referred to as “wafer-bow” [**19, 20**].

High growth temperatures and low growth-rates are supposed to be beneficial for reducing defects in 3C-SiC [**21**]. Heteroepitaxy on Si-substrates is limited to growth-temperatures below the melting point of Silicon at around 1414 °C. Using free-standing 3C-SiC, after the Si-substrate is removed, allows growth temperatures significantly higher. However, the predominantly employed CVD-methods for growing 3C suffer from an intrinsic drawback. At typical growth temperatures between 1550 °C and 1700 °C on free-standing 3C-SiC, surface diffusion limits the growth rate to values of approximately 100 µm/h [**22**]. Even though, low growth rates are in accordance with decreasing defect densities, later on, high growth rates are desired to increase the thickness when growth is performed on high-quality 3C-SiC-films.

High growth-temperatures as well as high growth rates can be achieved in the Physical Vapor Transport (PVT) [**23, 24**] method that is used for the realization of state-of-the-art SiC boules of the hexagonal polytypes (4H and 6H). However, this bulk-growth-method cannot simply be applied for 3C-SiC as certain growth-conditions are required, with respect to gas-phase composition, temperature-gradients and super-saturation. The merger of the PVT-method and the Sublimation Sandwich Method (SSM) [**25**] provides the necessary adaptations and led to the development of the Sublimation Epitaxy (SE) [**26–28**]. Both, high growth-temperatures and high growth rates can be achieved in SE [**26, 29**].

In this work, protrusions and stacking faults in sublimation grown 3C-SiC on 3C-SiC-on-Si seeding layers are characterized. Both, protrusions and stacking faults, can be considered as two of the most critical defects concerning the growth-process and the realization of high quality material. While stacking faults are electrically active, however expected to be partially eliminated during growth, the density of protrusions seems to remain the same once present in the 3C-SiC-layer.

Experimental

In this work, growth of 3C-SiC on (100)-oriented 3C-SiC-on-Si seeding-layers was conducted. For this purpose, a transfer-process [**30**] was developed in our group that enables the use of CVD-grown 3C-SiC as seeds for the Sublimation Epitaxy. In a first step, heteroepitaxially grown 3C-SiC-on-Si layers were cut into pieces of 15x15 mm^2. Wet chemical etching in a solution of hydrofluoric acid (HF), nitric acid (HNO_3) and water (H_2O) with a mixing ratio of 1:1:0.5 was used to remove the original Si-substrate. In the next step, the 50 µm thick free-standing 3C-SiC films were transferred onto a poly-crystalline silicon carbide carrier using a carbon glue for attachment. The produced seeding-stack **(Fig. 1a)** increases the mechanical stability of the seed and prevents an evaporation of the backside during the actual growth process.

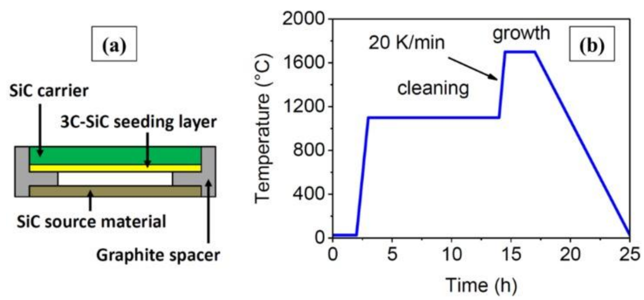

Fig. 1. (a) Simplified growth-cell for Sublimation Epitaxy with source material, spacer and seeding-stack consisting of carrier and seeding-layer; (b) Schematic of a typical growth process for sublimation epitaxy.

The growth runs were performed in a 2-inch SE-growth cell **(Fig. 1a)** that is realized in an inductively heated standard PVT-reactor. In this work, square-shaped samples with an edge-length of 12 mm and circular samples with a diameter of 12.7 mm were grown. Size and shape of the samples are determined by the design of the graphite-spacer that sets the distance between the source material (bottom) and the seeding-layer (top) to 1 mm **(Fig. 1a)**. Tantalum (Ta) acting as a carbon getter was placed into the growth cell in order to reach a Si-rich gas phase [**31**]. A simple schematic of a typical growth process is depicted in **Fig. 1b.** Holding the temperature at approximately 1100 °C for 12 hours at vacuum conditions serves as a cleaning step in order to remove adsorbates. A temperature ramp of approximately 20 K/min [**32**] up to growth-temperature is applied to achieve high supersaturation during ramp-up and initial growth. A pressure of 10^{-2} to 10^{-3} mbar and a temperature between 1600 °C and 1800 °C are set for the growth-step. By variation of growth parameters like temperature and growth time a series of experiments were conducted growing layers of 3C-SiC with various thickness.

Following the growth experiments, oxidation of the grown samples was performed at 800 °C to get rid of the carbon glue as well as the carrier and to remove the graphitized surface layer. Afterwards, the clean free-standing 3C-SiC films were characterized. The thickness of the CVD-seeding-layer and the total thickness of the 3C-SiC was determined as average value by measuring the thickness at nine positions uniformly distributed over the whole sample surface. Optical microscopy was used to evaluate the surface morphology and to determine the density of protrusions. Furthermore, KOH etching was performed at 530 °C for 5 min in order to make the stacking faults visible at the sample surface. The setup for this etching step can be found in [**33, 34**]. Measuring the total length of SFs within a defined surface area allows the calculation of the linear stacking fault density in cm^{-1}.

Results and discussion

On the sample surface, pyramid-shaped protrusion defects can be observed **(Fig. 2).** The origin of these defects can be assigned to the nucleation step during the initial growth of 3C-SiC-on-Si in the CVD process [**35**]. These protrusions are classified as twins featuring the {111} planes. Hence, the angle between a pyramid-face and the corresponding {100} plane is forming an angle of 54.7° as depicted in **Fig. 2c.** Protrusions predominantly appear as sharp structures on the sample surface surrounded by surface areas with comparatively smooth morphology **(see Fig. 2a). Fig. 2b** shows the surface of a CVD-3C-SiC seeding layer and the same specimen section after Sublimation Epitaxy. All protrusions on the seed-surface are highlighted by red circles (**Fig. 2b** top) and numbered consecutively. It is demonstrated that the same defects appear on the surface of the SE-layer with increased size **(Fig. 2b bottom)**. However, no new defects have been created and no defects have been eliminated during sublimation growth. These finding indicates that the predetermined distribution and density of protrusions on the seeding layer is not influenced by Sublimation Epitaxy**. Fig. 2c** illustrates a schematic of the growing pyramid-shaped protrusions with increasing 3C-SiC-thickness during sublimation growth. This schematic can be compared with the structures in **Fig. 2b**.

Starting at the former Si/3C-SiC-interface of the heteroepitaxial CVD-layer, the protrusions grow and can be observed at the surface of the CVD-layer with an edge-length x_1. During subsequent sublimation growth the protrusions continue to grow and appear with increased size and an edge-length x_2 at the surface of the homoepitaxial 3C. A simple mathematical approach was used as rough estimation of the SE-thickness using the edge-length of the protrusions as depicted in **Fig. 2b** and **Fig. 2c.** The mathematical relation is given as:

$$h_{SE} = \frac{1}{2}\,(x_2 - x_1)\tan\alpha$$

In **Fig. 2** the edge-lengths of a single protrusion are noted with $x_1 = 43$ µm and $x_2 = 253$ µm. Inserting these values to the above mentioned mathematical approach reveals a layer thickness h_{SE} of approximately 148 µm for the 3C-SiC film in **Fig. 2b**. This result is in good agreement with the average thickness of the sample shown in **Fig. 2b,** using the entire sample surface for determination. The total thickness (including the 50 µm seeding-layer) was calculated as (212 ± 13) µm. As the CVD-grown layer is homogeneous, the thickness of the SE-layer is assumed to be (162 ± 13) µm. Applying this rough estimation to any other clearly identifiable protrusion defect would give equivalent results. This finding indicates that pyramid-shaped protrusions continuously grow as depicted in the schematic in **Fig. 2c.**

Analysis of the protrusion-densities in large sections of the sample-surfaces were performed to evaluate the sublimation growth process with respect to this type of macro defect. Comparison of the protrusion density at the seeding layers and at the SE-layers resulted in the percentage change. This change was examined as a function of 3C-SiC-thickness **(Fig. 3)** as well as the growth rate **(**Error! Reference source not found.**).**

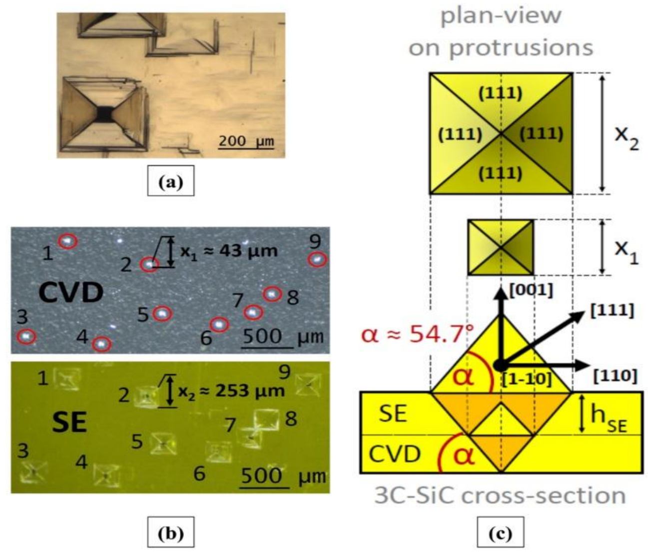

Fig. 2. (a) Pyramid-shaped protrusion defect on top of a (227 ± 8) µm thick 3C-SiC layer (including 50 µm seeding-layer) grown by Sublimation Epitaxy; (b) Comparison of the sample surface with protrusion defects before and after sublimation growth. On the 50 µm thick CVD-seeding layer the protrusions are small. After Sublimation Epitaxy (SE) the protrusions in the same sample area have grown and the total thickness is (212 ± 13) µm; (c) Illustration of the growing protrusion defects with increasing 3C-SiC thickness during SE-growth. In (b) and (c) the edge-lengths of protrusions on the seeding layer and after SE-growth are denoted with x_1 and x_2, respectively.

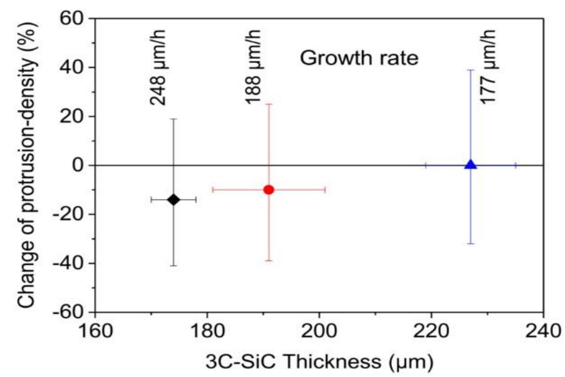

Fig. 3. Percentage change of the protrusion density as a function of total sample thickness. The associated growth rates are indicated at the respective data points.

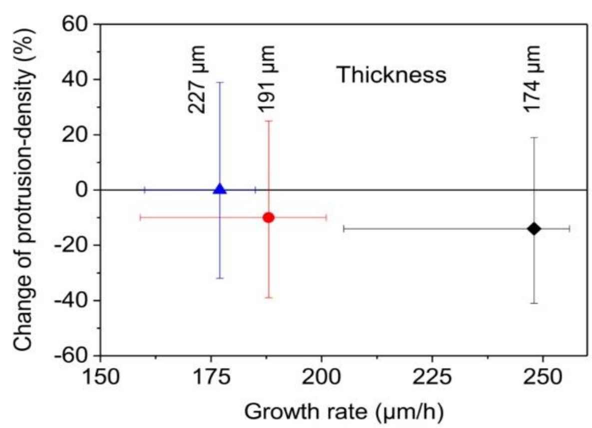

Fig. 4. Percentage change of the protrusion density as a function of growth rate. The associated total thicknesses are indicated at the respective data points.

Three samples with thicknesses between 174 µm and 227 µm are shown. Moreover, the growth rates vary between 177 µm/h and 248 µm/h. Considering both parameters with their associated error-bars, there is no clear trend towards an increase or decrease of the protrusion-density identifiable. These findings are consistent with our observations and indicate that Sublimation Epitaxy just reproduces the 3C-SiC quality that is predetermined by the seeding layers.

Analysis of the stacking fault density was carried out for SE-samples with varying thickness between 155 µm and 286 µm **(Fig. 5)**. The evaluation of the related 50 µm thick seeding layers serves as a reference. The SF-density of the seeding material is 1000 cm^{-1}. Starting from the 3C-SiC seeding layer, three areas can be identified with different trends regarding the SFs. In the transition area between CVD(seed)- and SE-layer an increase of SF-density was found. This result is in line with TEM-analysis (not shown) that revealed a defect-rich section at the CVD/SE-interface. Within the first approximately 125 µm of sublimation growth the SF-density increases and decreases again to a value of more or less the same as the one for the seeding layer. For the subsequent growth beyond approximately 125 µm (total thickness ≈ 175 µm) the SF-density levels slightly below the value of the seed according to the results so far. However, a further decrease of SF-density is expected for thicker 3C-SiC layers.

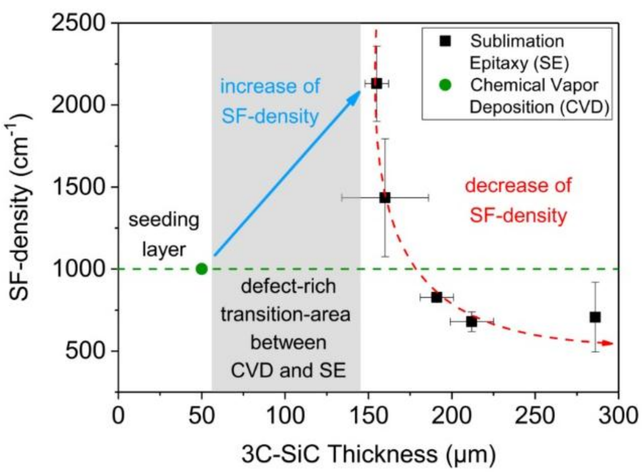

Fig. 5. Stacking fault density plotted versus the total 3C-SiC-thickness of the samples. A strong trend towards a decrease of SF-density with increasing thickness is indicated after the defect density increased within the transition-area.

Besides the thickness, the growth temperature (measured at the crucible top) was identified as parameter with strong effect on the SF-density. As depicted in **Fig. 6** there is a decrease of SF-density with increasing temperature.

Considering the thickness and the growth rate as well there might also be an influence of the growth rate. In Sublimation Epitaxy, the growth rate is directly connected with the temperature. However, with respect to results from the literature, a decreasing SF-density is predicted for lower growth rates while keeping the temperature constant [21]. This contrasts with the growth rate dependency in **Fig. 6.** Hence, the positive effect that can be observed in **Fig. 6** is attributed to the increase of growth temperature.

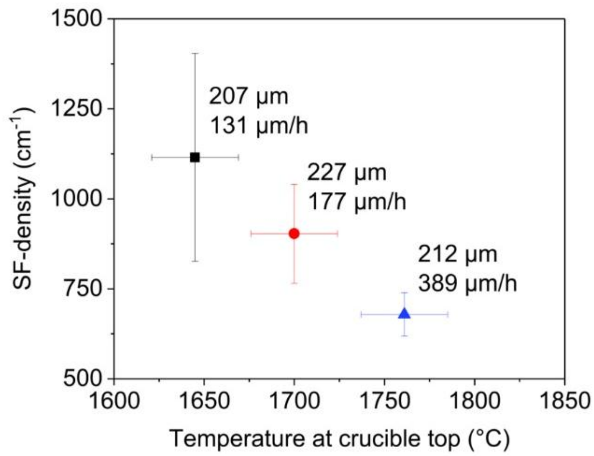

Fig. 6. Stacking fault density plotted versus the growth temperature measured at the crucible top. According to numerical simulations the temperature at the seeding-stack is approximately 50 °C higher compared to the crucible top. Thickness and growth rate of the respective samples are indicated within the diagram. Even though the decrease of SF-density might be an effect of both, the temperature or the growth rate, referring to findings from literature the decrease of SF-density is attributed to growth temperature.

Conclusion

Growth of 3C-SiC on 3C-SiC-on-Si seeding layers was performed. By variation of growth parameters like temperature and growth time we conducted a series of experiments and characterized the grown layers regarding the evolution of defects. In the case of Sublimation Epitaxy, it has been found practicable to distinguish between two types of defects. On the one hand, there are defects that are already present in the seeding-material and which continue to propagate during a subsequent sublimation growth step without a reduction. The pyramid-shaped protrusion defects are an example for this type of defect. The results in this article demonstrate that the density of protrusions is neither influenced by the thickness of the 3C-SiC nor by growth parameters like growth rate and temperature. Hence, during Sublimation Epitaxy the predetermined protrusion density given by the seed is merely reproduced. This finding clearly highlights the necessity of having high-quality seeding layers as starting point for homoepitaxial growth by Sublimation Epitaxy. On the other hand, there are defects, such as stacking faults, which exhibit a significant change in density during subsequent homoepitaxial growth steps. It was shown that there is a strong trend towards a decrease of SF-density with increasing 3C-SiC-thickness of the samples. This finding is in line with relevant literature. However, for sublimation growth on 3C-SiC seeding layers a previous increase of the SF-density within a transition-area was detected, compared to the defect density on the seeding material. The final SF-density was below the value for the seed and is expected to further decrease with increasing 3C-SiC-thickness. Besides thickness, the growth temperature turned out to have an influence on the SF-density. It seems that there is a trend towards a decreasing SF-density with increasing growth temperature.

In conclusion, there are no indications for any negative effects on the 3C-SiC quality due to the sublimation growth. Concerning stacking faults there are even signs suggesting an improvement of 3C-SiC quality. These results demonstrate the potential of sublimation epitaxy in growing thick and high-quality 3C-SiC layers.

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