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Effect of process parameters on dislocation density in thick 4H-SiC epitaxial layers grown by chloride-based CVD on 4° off-axis substrates

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Abstract: The effect of process parameters such as growth temperature, C/Si ratio, etching time, and Si/H₂ ratio on dislocation density was investigated by performing KOH etching on 100 μm thick epitaxial layers grown on 4° off axis 4H-SiC substrates at various growth conditions by a chemical vapor deposition (CVD) process using a chloride-based chemistry to achieve growth rates exceeding 100 μm/h. We observe that the growth temperature and the growth rate have no significant influence on the dislocation density in the grown epitaxial layers. A low C/Si ratio increases the density of threading screw dislocations (TSD) markedly. The basal plane dislocation (BPD) density was reduced by using a proper in-situ etch prior to growth.

Introduction

The dislocation density in a 4H-SiC single crystal wafer is high ($> 10^3 \text{ cm}^{-2}$) [1], and the replication of dislocations from the wafer to the epitaxial layer is a significant problem for the fabrication of SiC based devices. The propagation of basal plane dislocations (BPD) from the substrate into the epitaxial layers is known to cause a drift of the forward voltage in bipolar devices during operation [2, 3]. The majority of the BPDs in the substrate are converted to threading edge dislocations (TED) in the epitaxial layer, but some BPD propagate as BPD from the substrate into the epitaxial layer [1, 4]. This occurs since the elastic energy of TED per unit growth thickness of the epitaxial layers is less than that of the BPD [5]. Reports confirm that the use of 4° off-angle substrates leads to a further reduction of BPDs in the epitaxial layers compared to 8° off-angle substrates [6, 7]. Furthermore, it has been shown that the density of BPD in the epitaxial layers has been reduced using molten KOH etching [8] or in situ growth interrupts [9]. In a previous study [10], we investigated and optimize a chloride-based CVD growth process for thick, $> 100 \text{ μm}$, epitaxial layers of 4H-SiC on 4° off-axis (0001) Si-face substrates at growth rates $> 100 \text{ μm/h}$ with the aim to minimize the amount of structural defects and roughness of the epitaxial layers by tuning the process. Here, we study the effect of growth conditions on dislocation density by decorating the dislocations using KOH etching.

Experimental

Growth experiments were performed in a horizontal flow hot wall chemical vapor deposition reactor without rotation of the substrate. All substrates in this study were approximately $1.5 \times 1.5 \text{ cm}^2$ samples cut from one chemo-mechanically polished 4 inch 4H-SiC wafer with a 4° off-cut towards the [11-20] direction. For all experiments, the growth pressure and growth time were kept constant at 100 mbar and 1 hour, respectively. Palladium membrane purified H₂ was used as carrier gas at a flow rate of 50 l/min. SiH₄ (silane) + C₂H₄ (ethylene) + HCl were used as precursors with a Si/H₂ ratio of 0.25%. The growth temperature was varied between 1550 – 1625 °C and the C/Si ratio was varied between 0.4 and 1.1 by changing the C₂H₄ flow. The Cl/Si ratio was varied between 5 and 8 by changing the HCl flow. For surface preparation, a HCl flow of 300 ml/min was added to the H₂ flow when the temperature in the growth chamber was 1175 °C, the HCl flow was kept constant during the temperature ramp up to the growth temperature and followed by etching at

growth temperature between 0 – 24 min, after which the precursors were gradually introduced to the gas mixture. The precursor flows are ramped up from low flows to the growth process flows during a few minutes, maintaining the C/Si and Cl/Si ratios. No intentional dopants were added in this study. In order to estimate and compare dislocation densities in the epitaxial layers grown under different growth conditions, the epitaxial layers and substrates were etched in molten potassium hydroxide (KOH) at 500 °C for 3 minutes to decorate dislocations on the epitaxial layers surface. The shape and distribution of etch pits were studied by Nomarski differential interference contrast microscopy (NDIC). The counted areas did not include parts of the samples within 2 mm from the edge.

Results and Discussion

The density of BPD, TED and threading screw dislocations (TSD) in the substrates were found to be $4 - 5 \times 10^3$, $2 - 3 \times 10^3$, and $0.9 - 1 \times 10^3 \text{ cm}^{-2}$ respectively. The BPD density was higher than the density of the TED and TSD. Figs. 1(a) and (b) show 200 times magnified optical microscope images of KOH-etched substrate surface and epitaxial layers surface, respectively. Large hexagonal, small hexagonal and conical etch pits are related to TSD, TED and BPD respectively. Most of the BPDs were converted to TED in the grown epitaxial layers for all growth conditions, this is consistent with the results of Ref.4.

Fig. 1. Optical microscope images at 200 X magnification of molten KOH-etched (a) 4° off-cut substrate 4H-SiC; (b) epitaxial layers grown at a temperature of 1575 °C, Cl/Si=5, growth time=1 hour, Si/H₂ = 0.25 %, and C/Si = 0.9 .

The effect of growth temperature on dislocations density was observed in the 1550 – 1625 °C range with C/Si fixed at 0.9, etching time at 12 min, growth time of 1 hour and Cl/Si at 5. For all growth temperatures, the growth rate was around 105 μm/h. The dislocation densities for various growth temperatures are plotted in Fig. 2. The density of TSD did not depend on the growth temperatures and the density of TED increased somewhat at higher temperatures. The density of BPD decreased at 1625 °C to about 100 cm⁻². The density of BPDs was about 300-400 cm⁻² at the growth temperature of 1550, 1575 and 1600 °C. At the highest temperature (1625 °C), severe step bunching with 150-950 nm wide terraces and 5-6 nm step height were formed. Ha et al. [1] suggested that in the case of a surface covered with macro-steps, the interaction between macro-steps and basal plane dislocations is governing the dislocation conversion, thus explaining the reduction of BPD at 1625 °C.

Fig. 2. Effect of the growth temperature on the densities of BPD, TED and TSD, for 4H-SiC epitaxial layers with C/Si = 0.9, Cl/Si = 5, Si/H₂ = 0.25 %, growth time = 1 hour and growth rate = 105 $\mu\text{m}/\text{h}$.

Fig. 3. Effect of the C/Si on the densities of BPD, TED and TSD, for 4H-SiC epitaxial layers with growth temperature = 1575 °C, Cl/Si = 5, Si/H₂ = 0.25 %, growth time = 1 hour. Open circles show the RMS surface roughness on a 20×20 μm^2 area.

In order to reduce the formation of step bunching and structural defects, a growth temperature of 1575 °C was chosen for the following experiments. The effect of the C/Si ratio on the dislocation density was studied in a set of experiments with a fixed Cl/Si of 5, etching time at growth temperature of 12 min, with the C/Si ratio varied between 0.4 and 1.1 at growth temperatures of 1575 °C. Fig. 3 shows the dislocations densities for various C/Si ratios. The density of TSD increased markedly at C/Si < 0.8. Kamata et al. [11] reported that the probability of micropipe dissociation into threading screw dislocations is very high at low C/Si ratio. The density of TED was almost unchanged for various C/Si ratios. The density of BPD was about 250-400 cm^{-2} at C/Si of 1.1, 1, 0.9 and 0.8. No BPDs were observed at C/Si of 0.7 and 0.6. The BPD density was as low as 50 cm^{-2} for C/Si of 0.5 and 0.4. For C/Si ratios of 0.7 and 0.6, severe step bunching with 58-102 nm and 25-82 nm high steps respectively was observed while the step height decreased to 15-17 nm and 11-13 nm for C/Si ratio of 0.5 and 0.4, respectively. It has been reported that severe step bunching may be formed at very low or high C/Si ratios due to the presence of Si or C clusters on the terraces [12]. At lower C/Si the growth rate is much lower which results in a reduced step bunching [10]. This suggests that the dislocation conversion might be connected to the increased height of the steps. This is consistent with the results of Ref. 1 where it was shown that in the case of a surface covered with macro-steps, the interaction between macro-steps and basal plane dislocations is governing the dislocation conversion.

The best morphology was obtained at C/Si ratio of 0.8 and 1575 °C. At this condition, the etching time at growth temperature was 12 minutes. The effect of the etching time at growth temperature on dislocations density was observed in a set of experiments with fixed C/Si of 0.8, Cl/Si of 5 and growth temperature of 1575 °C, the etching time was varied between 0-24 minutes. The best condition was obtained with etching time of 18 minutes. The BPD density was then reduced to below 10 cm^{-2} . As mentioned above, the BPD density was 250 cm^{-2} when the etching time at growth temperature was 12 minutes. It has previously been reported [8] that the creation of BPD etch pits on the substrate surface after molten KOH etching, enhanced the conversion of BPD into TED during epitaxial growth, this effect also increases as the dimension of the BPD etch pits on the substrates became larger. It can thus be speculated that as the etching time increased, the BPD etch pits on the substrates became larger which facilitated the BPD to TED conversion. Etching time of 0, 6 and 24 min results in layers with very high structural- defect density and very bad morphology which makes it impossible to count the etch pits of the dislocations.

The impact of the growth rate on dislocation density was studied in a set of experiments at different growth rates with C/Si at 0.8 and 18 minutes etching time at growth temperature of 1575 °C. The growth rate was controlled by varying the Si/H₂ ratio between 0.15 and 0.25% rendering growth rates between 65 and 105 µm/h. The densities of BPD, TED and TSD were not found to be dependent on the growth rate. It has previously been reported for the non-chlorinated growth chemistry that the BPD density can both increase [4] and decrease [13] with increase in growth rate.

Summary

In this work we have studied the influence of process parameters such as growth temperature, C/Si ratio, etching time, and growth rate on the dislocation density in 4H-SiC epitaxial layers grown on 4° off-axis substrates. We have observed that the growth temperature and the growth rate have no significant influence on the dislocations density in the grown epitaxial layers. At low C/Si ratio the density of TSD increased markedly. The in situ etching prior to growth was found to have a great effect on reducing the BPD density. On the epitaxial layers having very high step height no BPDs were observed.

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