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Homo-epitaxial growth on low-angle off cut 4H-SiC substrate

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Abstract. The growth of 4H-SiC epilayers on 1.28° off-cut substrates is reported in this study and comparison when using standard 4° and 8° off-cut substrates is added. Growth at high temperature is needed for the polytype stability, whereas low C/Si is requested to decrease both triangular defects density and roughness of the grown surface. An in-situ etching with Si rich ambient allows the growth of epilayers with specular surface. The formation of Si droplets can be observed on the grown surfaces when lowering the growth temperature and appears first for the high off-cut angle.

Introduction.

A solution to avoid degradation problem of the bipolar devices when using 8° or even 4 ° off-cut materials is the use of nominally on-axis substrate which also reduces the production cost since the loss of material when slicing the wafers from the ingot will be lower. However, even if the problem of polytype instability with formation of 3C inclusions has been overcome and 100% 4H homoepitaxy has been demonstrated, the main drawback is related to the growth mechanism: spiral growth around screw dislocation is involved together with step flow growth in the regions where a very small off-cut angle exists and slightly higher than typically 0.3°, as reported in a recent book chapter [1]. This influences first the morphology with high roughness which increases with the thickness of the layer; steps as high as 7 µm have been observed on 100 µm thick epilayer. Secondly large in-homogeneous distribution of minority carriers lifetime related to the growth mechanism has been observed [2]. Even if stable bipolar diodes without observation of degradation have been processed [3], the processing using on-axis material needs additional steps which reduce its yield.

Thus the solution to avoid the propagation of the basal plane dislocations which are the origin of the bipolar devices degradation when using standard off-cut material and to get smoother surface than with nominally on-axis substrate is probably the use of very low off-cut angle substrate, as recently proposed [4-6], for which only step flow growth will appear. When using off-angle as low as 1° or just below it was necessary to lower the growth temperature below 1550 °C and the C/Si ratio to 0.5 to get epilayers with good morphology without triangular defects and step bunching [6]. We can notice that the background level of the net doping concentrations is not mentioned in these reports [4, 6].

The main goal of this study is to investigate the growth on 1.28° off-cut substrate with standard chemistry.

Experimental details.

The 4H-SiC epilayers were grown in a hot-wall chemical vapor deposition reactor. Hydrogen was used as carrier gas; small amount of Ar, typically 5% of the H₂ flow, was added to favor and/or increase the homogeneity of the growth area. The growth takes place in a graphite susceptor coated with TaC which can be heated to about 1650 °C. For standard chemistry silane (SiH₄) and a light hydrocarbon as propane (C₃H₈) or ethylene (C₂H₄) was used. We present here results using a relatively low growth rate (8 µm/h) with Si/H₂ = 0.038%. Rotation was applied to the substrate even if only small pieces (12x12 mm²) were used in the present study. The growth on 1.28° off-cut

Fig. 1: AFM images of $10 \times 10 \mu\text{m}^2$ area acquired on $15 \mu\text{m}$ thick epilayers grown on a) 4° and b) 1.28° off-cut substrates within 2 hours (note that the scale for the roughness is twice higher for b) than a)) and c) and d) surface morphology as observed with (NDICO) microscopy for the b) sample showing a large triangle defect.

substrate is compared with growth using other types of substrate, mainly other off-cut angles such as 8° or 4° off-cut, all off-angle being towards the $[11\text{-}20]$ direction. The substrate off angle was confirmed with XRD diffraction and small variation over the 75 mm diameter wafer was typically observed with less than 0.1° .

Nomarski differential interference contrast optical (NDICO) microscopy is used to investigate the morphology of the layer as a first characterization technique. Using atomic force microscopy (AFM) in a tapping mode regime the roughness of the surface is extracted in root mean square (RMS) value. To evaluate the stability of the polytype or visualize polytype inclusion in the 4H-SiC layers UV emission imaging is used; the sample placed in a nitrogen bath at 77 K is illuminated with UV excitation. The thickness of the layers is measured using reflectance technique in a Fourier Transform Infra-Red spectrometer. Net doping concentration is determined at room temperature using Hg probe capacitance-voltage measurement and photoluminescence at low temperature completes the characterization.

Experimental results.

The optimal conditions for the growth on 4° off-cut substrate were found to be in this reactor as: temperature close to 1630°C , $\text{C/Si} = 0.9$ which led to typical RMS value of 0.24 nm for $10 \times 10 \mu\text{m}^2$ area (Fig.1.a) and unobservable step bunching with optical microscope. However using the same growth conditions, step bunching was observed for the epilayer grown on 1.28° off-cut substrate with $\text{RMS} = 2.6 \text{ nm}$. Narrow steps are observed with width less than 200 nm and step height typically 8 nm (Fig.1.b).

The UV mapping technique shows many triangular defects (Fig.2.a). Some of them detected in the UV map are not observed with NDICO microscopy. When observed with microscopy (Fig.1.c and d) they are generally isosceles-acute with the larger corner of angle typically as 160° decorated with a particle as seen in Fig.1.d. By keeping the growth temperature of 1630°C but lowering the C/Si ratio to 0.85 and 0.8 , the triangular defects vanish (Fig.2.b) but the roughness stays with typical high value as 2.2 nm .

Fig.2: UV map of layers grown on 1.28° off-cut substrate with C/Si ratio of a) 0.9 and b) 0.8 .

Decreasing the growth temperature with few tens of degrees help to decrease the roughness to about 1.8 nm , however, so-called Si droplets can be observed (Fig.3). These are actually a deformation of the surface (see below), since with micro-Raman

Fig.3: NDICO micrograph of the surface morphology of 8 μm thick epilayers grown on a) 1.28°, b) 4° and c) 8° off-cut substrate. The growth was done for 1 hour, at 1590 °C.

only characteristic modes of the 4H-SiC are observed, neither other polytype as 3C nor pure Si modes were detected. These Si droplets are first observed for epilayers grown on the larger off-cut substrates (Fig.3.b and c). On 4° off-cut surfaces when their density is not as high as in Fig.3.b they appear as a decoration of the steps as also observed on 1.28° epilayer surface (Fig.3.a). The size (diameter) of the Si droplets decreases with increasing off-cut angle whereas their density increases. Fig.4 shows a typical AFM view of Si droplets observed on epilayer grown on 1.28° off-cut substrate, on the left part of Fig.4.a alignment of the Si droplets on the steps are visualized. The cross section analysis (Fig.4.b) evaluates depth as high as 14 nm for this case, whereas depths close to 50 nm have been measured for epilayer grown on 4° off-cut substrates. All those layers were grown after etching the substrates at the growth temperature in pure H_2 atmosphere (and small addition of Ar) during 5 minutes. A further decrease of the growth temperature favors the polytype instability: 3C inclusions are observed not only as decoration of the steps but also as large triangle defects.

By changing the conditions for the in-situ etching (from only H_2 ambient to slightly Si rich ambient, $\text{Si}/\text{H}_2=0.006\%$ corresponding to the minimum flow allowed by the system) specular surface is demonstrated without triangular defect or Si droplets and with RMS value of 1.2 nm (see Fig.5). The surface comports large steps (3-4 μm large) still related to the step bunching. To understand the formation of these large steps compared to the narrow steps as seen in Fig.1.b, more carefully AFM investigations have been done as displayed in Fig. 6. Ending of steps are indicated by the arrows and this behavior differs to the slicing or crossover of the steps observed for epilayers grown with sublimation on low off-angle 6H-SiC [7]. This behavior allows progressively

Fig.4: a) AFM image showing Si droplets on 1.28 off-cut layer (same as Fig.3.a) and b) a cross section on horizontal direction of one of the Si droplets.

Fig. 5. AFM images of $10 \times 10 \mu\text{m}^2$ area of epilayers grown on a) 8° off-cut and b) 1.28° off-cut epilayers, respectively, after SiH_4 etching during 5 minutes prior the growth done at 1620 °C and with $\text{C}/\text{Si}=0.8$. (note that the scale for the roughness is twice higher for b)

Fig.6: AFM views of epilayers grown on 1.28° off-cut substrate showing stop of step as indicated by the arrows.

the enlargement of the steps.

Surprisingly epilayers grown at the same time on 4° and 8° off-cut substrates and with the in-situ SiH₄ etching were also free of Si droplet and had a RMS value of 0.5 and 0.3 nm, respectively. Increasing the SiH₄ etching time to 20 min did not change the results. Lowering the C/Si ratio even further is suggested to improve the

roughness for the layer grown on 1.28° off-cut substrates. The main draw-back already observed is regarding the electrical property of the layers: with C/Si=0.8 the residual net carrier concentration is measured to be in the low 10¹⁶ cm⁻³ range whereas for high power applications this should be well below 10¹⁴ cm⁻³. This behavior is confirmed by low temperature photoluminescence where the free exciton was observed with low intensity, the dominant luminescence being associated to the nitrogen donor. However, this approach can be applied for the buffer layers when starting growing the epitaxial device structure whereas the epitaxial growth of the active layer could be done during the same run by increasing the growth rate and the C/Si ratio.

Summary

The growth of epilayer on 1.28° off-cut substrates is reported and compared with the CVD growth on 4° and 8° off-cut substrates. Growth at high temperature is needed to avoid both Si droplets formation and 3C inclusion or decoration of 3C on steps. Low C/Si is an important parameter to control the surface morphology and decrease the roughness of the surface. Mechanism to enlarge the step is reported.

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