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Growth of Defect-Free 3C-SiC on 4H- and 6H-SiC Mesas Using Step-Free Surface Heteroepitaxy

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Abstract: A new growth process, herein named step-free surface heteroepitaxy, has achieved 3C-SiC films completely free of double positioning boundaries and stacking faults on 4H-SiC and 6H-SiC substrate mesas. The process is based upon the initial 2-dimensional nucleation and lateral expansion of a single island of 3C-SiC on a 4H- or 6H-SiC mesa surface that is completely free of bilayer surface steps. Our experimental results indicate that substrate-epilayer in-plane lattice mismatch ($\Delta a/a$ = 0.0854% for 3C/4H) is at least partially relieved parallel to the interface in the initial bilayers of the heterofilm, producing an at least partially relaxed 3C-SiC film without dislocations that undesirably thread through the thickness of the epilayer. This result should enable realization of improved 3C-SiC devices.

Introduction

For many years researchers have attempted to grow 3C-SiC heteroepitaxial films on Si and 4H/6H-SiC substrates. However, all previous efforts resulted in 3C-SiC with extended crystal defects including double-positioning boundaries (DPB's) and/or stacking faults (SF's). The presence of these defects in high densities is believed responsible for the poor electrical performance of 3C-SiC devices fabricated in these films, relative to the superior electrical performance of homoepitaxial 4H/6H-SiC devices. The role of surface steps in causing defects in 3C-SiC epitaxial films has been reported previously [1]. The formation of SiC mesa surfaces as large 0.2 x 0.2 mm completely free of a single atomic step was recently reported [2]. Step-free surfaces have been proposed as ideal for realizing greatly improved heteroepitaxial growth of 3C-SiC and III-N films with much lower dislocation densities [2-4]. The ability to reproducibly obtain 3C-SiC heteroepitaxial films free of extended crystal defects could enable advantageous new electrical devices to be realized. This paper reports the achievement of 3C-SiC heteroepitaxial films completely free of observed extended crystal defects using a new growth process we have termed "step-free surface heteroepitaxy".

Experimental

As described in [2], step-free surfaces are produced on commercially purchased on-axis 4H- or 6H-SiC wafers by first dry etching trench patterns into the wafer surface to form an array of isolated growth mesas. Pure stepflow epitaxial growth, carried out under conditions that suppress two-dimensional (2D) terrace nucleation, is then used to grow all initial surface steps on top of the mesa over to the edge of the mesa, leaving behind a top mesa surface that is completely free of atomic steps. However as reported in [2], the high density of screw dislocations (SD's) limited the yield and size of step free mesas attained on commercial SiC substrates. Mesas that initially contain SD

defects cannot be flattened due to the continual spiral of new growth steps that emanate from screw dislocations during epitaxial growth.

A half-dozen 3C-SiC heteroepitaxial growth runs were carried out separately on 4H- and 6H-SiC wafer pieces with pre-growth mesa patterns etched into them as described in [2]. The major findings of these experiments can be summarized by examining results from SD-free 4H-SiC mesas that underwent three different 3C-SiC heteroepitaxial growth recipes. Following 15 minutes of homoepitaxial growth at 1620 °C described above and in [2] to produce step-free mesas, the in-situ growth temperature was lowered as described in Table I to facilitate initial 2D nucleation of 3C-SiC on the large basal plane surface [5]. Growth temperatures were measured by pyrometry to an absolute accuracy of ±30 °C, with relative temperature decreases shown in Table I accurate to within ±10 °C. Samples A and B were grown for an additional 70 and 60 minutes (respectively) following the 5-minute ramps, while sample C growth was concluded at the end of its 60-minute temperature ramp. After epitaxial growth, samples were dry oxidized for 5 hours at 1150 °C to color-map polytype and reveal DPB and SF defects [6].

All three processes nucleated 3C-SiC on the vast majority of step-free mesas. The thickness of the 3C-SiC films shown in Figs. 1 and 2 is close to 2 µm. Steps observed by atomic force microscope (AFM) on these films are 0.25 nm, the height of a single Si-C bilayer. Fig. 1 shows the highest quality 3C-SiC layer produced on sample A. As expected, the heteroepitaxial nucleation of 3C-SiC on the step-free 4H-SiC mesa surface successfully eliminated DPB defects [3,7,8]. However, an abundance of SF defects are easily observable in Fig. 1, indicating that atomic-scale steps are not the only source of extended defects in the 3C-SiC heteroepitaxial film. Fig. 2 shows a typical SD-free mesa from sample B (0.2 mm x 0.2 mm) in which nucleation and growth of 3C-SiC took place following the formation of a step-free surface. There are no observable SF or DPB defects in Fig. 2, or for over 80% of SD-free mesas over most of the wafer. The defect-free yield for SD-free mesas on sample C was near 60%. Low substrate SD density yielded a few defect-free 0.4 mm x 0.4 mm 3C-SiC films on other samples in the study. Defect-free 3C-SiC was also achieved on 6H-SiC samples.

Table I: Experimental 3C-SiC Initial Nucleation Processes

Sample	Temperature Profile (from 1620 °C)	SF Density
A	Ramp down 190 °C over 5 min.	$> 10^4/cm^2$
B	Ramp down 120 °C over 5 min.	0
C	Ramp down 190 °C over 60 min.	0

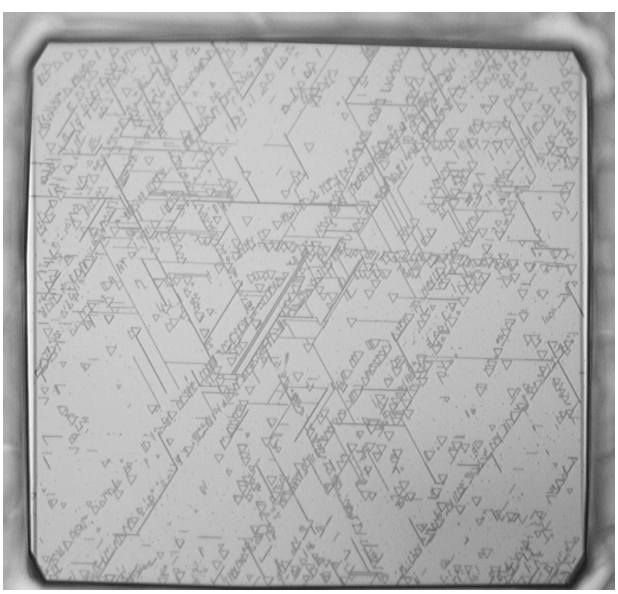

Fig. 1: Best 3C-SiC layer grown on 0.2 mm x 0.2 mm SD-free mesa on Sample A following oxidation to reveal SF defects.

Fig. 2: Typical 3C-SiC layer grown on 0.2 mm x 0.2 mm SD-free mesa on Sample B following oxidation to reveal SF defects.

Discussion

Detailed comparison of the processes (Table I), results (Figs. 1 & 2), and prior understanding indicates that low initial rate of 2D nucleation of the first bilayers of 3C-SiC is necessary in order to realize defect-free 3C-SiC layers on the step-free mesas. Based upon prior growth system experience, we believe the 5-minute linear ramps are long enough to avoid direct thermal and chemical transient effects in the study. The temperature/time profile at the initial nucleation of 3C is the only difference in the entire growth process between SF-plagued sample A and defect-free sample B. Previous works have shown that probability (and rate) of 2D terrace nucleation of 3C-SiC increases as growth temperature decreases due to decreased adatom surface mobility [5]. Therefore, compared to the 5-minute 120 °C temperature decrease that nucleated the defect-free 3C of sample B (Fig. 2), the 5-minute temperature decrease of 190 °C effectively subjects sample A to a faster transition into higher rate of 2D terrace nucleation at the initiation of 3C-SiC heteroepitaxial growth. However, the 3C nucleation profile of defect-free sample C has the same starting and ending temperatures (i.e., starting and ending nucleation rates) as the defective sample A, but the transition between the two takes place over a much longer time period. Therefore, the low nucleation temperature (i.e., higher nucleation rate) cannot be the sole cause of the SF's observed in sample A. Instead, the rapid transition to a high 2D nucleation rate is surmised to cause the abundance of stacking faults in the sample A 3C-SiC film. The sample C process slowly sweeps 3C nucleation rate, from near-zero (at 1620 °C), through both sample B (defect-free) and sample A (defective) 3C nucleation conditions. Therefore, the defect-free nature of sample C bears witness to the fact that after a certain thickness of 3C-SiC film is grown (by sample B conditions), further defect-free growth of the 3C-SiC film can be maintained at higher 2D nucleation rates (sample A conditions) unacceptable for the initial nucleation of 3C-SiC on the step-free 4H-SiC surface.

We propose that low initial nucleation rates, consistent with processes B and C, produce defect-free 3C-SiC films because coalescence of multiple expanding initial 3C-SiC islands on the 4H surface is avoided. In particular, we hypothesize that the low initial nucleation rate enables 3C-SiC heteroepitaxial growth to initiate from a single 3C-SiC island, which subsequently expands laterally via stepflow to cover the entire mesa before nucleation of a second 3C-SiC island can occur elsewhere on the 4H mesa surface. We believe this eliminates defects associated with lateral coalescence of multiple 3C-SiC islands expanding laterally on a step-free 4H-SiC mesa. These observations are the basis of a new crystal growth process for carrying out lattice mismatched heteroepitaxy we have named "step-free surface heteroepitaxy", because the heteroepitaxy must be initiated on a step-free surface.

The in-plane lattice parameter (a) of SiC decreases as a function of polytype hexagonality, and previously reported 3C-SiC and 4H-SiC bulk lattice parameters predict fully relaxed $\Delta a/a$ = 0.0854% ($a_{3C} > a_{4H}$) [9,10]. Preliminary High Resolution Triple-Axis X-ray Diffraction (HRTXD) measurements were conducted on one of the 3C/4H samples not listed in Table I [8]. The HRTXD setup measured areas larger than the isolated mesas, so the measurements also contained information from material grown in trenches between mesas. The results were consistent with previous measurements of 3C-SiC films on hexagonal SiC substrates in that partial lattice mismatch relaxation is indicated [11]. It is well known that lattice-mismatched heteroepitaxy is based upon film atoms growing in pseudomorphic alignment with substrate atoms until strain is relieved by dislocation generation as the film reaches a critical thickness [12]. We conducted preliminary calculations of critical thickness for a strained pseudomorphic 3C-SiC layer on 4H-SiC (using <111> planes for strain relief) that resulted in values ranging from 0.07 to 0.12 µm. This range is significantly thinner than the defect-free film depicted in Fig. 2.

The fact that no dislocations propagate to the top of the Fig. 2 heteroepilayer implies that dislocations associated with lattice mismatch relief do not thread through the epilayer thickness. This fact is remarkable when one considers the ease with which dislocations usually propagate

along the close-packed planes of SiC, three of which in 3C-SiC are <111> planes that crosscut the epilayer while the fourth equivalent <111> plane is parallel to the 3C/4H interface [13]. We propose that the mismatch relief disorder is confined along close-packed planes parallel to the heterointerface. We believe that the lack of interface steps and the unconstrained nature of the mesa edge are keys to confinement of mismatch relief to interface-parallel basal planes during step-free surface heteroepitaxy.

The experimental results show that the low-nucleation rate (i.e., single-island) mode of growth is only needed for the initial stages of 3C-SiC film growth. Multiple islands nucleated near the end of the sample C growth coalesce without defects. This implies that lattice mismatch in the upper layers of the 3C film is smaller than in the lower layers of the film, due to strain relief during growth. If the complete relaxation of lattice mismatch has not occurred by this stage of growth, the 3C layer will accumulate strain as it grows thicker, until the partially relaxed critical layer thickness is reached, after which additional strain relief should occur via additional dislocation generation [12]. Further measurements to better ascertain the degree of relaxation at various stages of film growth have been initiated. Studies of thicker 3C-SiC heterofilms and prototype devices are planned. Likewise, experimental growth of III-N films by step-free surface heteroepitaxy will also be undertaken in an effort to realize predicted improved material quality [2,4].

Conclusion

A new growth process, herein named step-free surface heteroepitaxy, has achieved 3C-SiC films completely free of DPB and SF defects on step-free 4H-SiC and 6H-SiC substrate mesas. Our experimental results indicate that substrate-epilayer in-plane lattice mismatch is at least partially relieved parallel to the interface in the initial bilayers of the heterofilm without dislocations that undesirably thread through the thickness of the epilayer. We conclude by pointing out that further growth and characterization experiments are planned to better elucidate the full nature and limits of the step-free surface heteroepitaxy process that we have developed for growing defect-free 3C-SiC films on 4H- and 6H-SiC substrates.

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