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Quality Aspects for the Production of SiC Bulk Crystals

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Abstract. For several years the major focus of material issues in SiC substrates was laid on the reduction of macroscopic defects like polytype inclusions, low angle grain boundaries and micropipes. Since then significant improvements have been achieved and micropipe densities could be reduced to values below 1 cm^{-2}. Nevertheless the fabrication of high quality substrates at high volume and low cost is still challenging. Therefore preconditions for reproducible process and quality control will be discussed. Since it is obvious that dislocations are the main reason for degradation in power devices the prevailing attention has also been shifted to that field of material research. Intense studies were utilized on dislocation and stacking fault formation during sublimation growth. For this reason we systematically varied crucial parameters of the crystal growth process and applied several specific characterization methods, e.g. KOH-defect-etching, electron microscopy and optical microscopy, to evaluate resulting material properties. The investigations were accompanied by failure analysis on devices of the Schottky-type. We found out that for the improvement of substrate quality emphasis has to be laid on the reduction of thermo-elastic stress in the growing crystal. The results of numerical calculations enabled us to derive moderate growth conditions with reduced temperature gradients and correspondingly low defect concentration.

Introduction

The main application for silicon carbide is still its use as a substrate for GaN-based optoelectronic devices, which were already produced on 2"-wafers of the 6H-modification and elevated micropipe densities several years ago. Meanwhile micropipe densities for the modifications 6H and 4H have been substantially decreased and diameters of 3 inch and larger were established as state of the art also for the production of semi-insulating SiC-substrates. Thereby the development of GaN-based high frequency devices was promoted and is already approaching production level. Based on the significant improvement in recent years also the originally intended use of SiC-substrates for SiC-based power devices is raising to considerable volumes. However the realization of the proposed market expansion rates requires further reduction of defects. Micropipes need to be eliminated and dislocation densities, especially basal plane dislocations, have to be significantly reduced. In this paper we will discuss fundamental aspects for the production of SiC-substrates with reproducible low micropipe densities as well as principles of dislocation generation.

Experimental

Silicon carbide crystals of both modifications 6H and 4H were grown applying the physical vapour transport method (PVT), as described elsewhere [1, 2, 3]. During optimization basic parameters like temperature, pressure, powder properties and crucible geometry were varied. As the inner growth chamber is not optical accessible for pyrometric temperature measurements, we performed additional numerical simulations to calculate the thermal field inside the crucible and the stress distribution inside the growing crystal with the software Virtual Reactor. Before characterization the crystals were processed to wafers by conventional semiconductor preparation techniques. The crystalline quality and the stress distribution in the substrates were routinely checked by stress birefringence images while the micropipe density was determined by a combination of optical microscope mappings and analysis of selected etch pictures. The latter were obtained by etching in molten KOH (520°C, 10 min). For the studies on Schottky structures also electron microscopy (SEM, SEM-CL) was executed.

Results and Discussion

Optimization Procedure. Contrary to melt growth processes where perfect seeds with diameters below cm-scale are used and increased by several times during initial stages of growth, the seed diameter in silicon carbide PVT-growth is similar compared to the growing crystal and therefore various defects like micropipes, low angle grain boundaries or high dislocation density areas are transmitted from the seed into the bulk crystal. A prerequisite for quality enhancement is the development of conditions causing decreasing defect densities during growth. Implying a high reliability of this process conditions, defects can then be reduced by repeated growth and seed selection. Commonly this procedure is quickened by the implementation of a new seed qualities, produced under special conditions, e.g. lateral growth [4, 5, 6], or processes with moderate growth conditions [7, 8]. Importance has to be attached to a fundamental understanding of defect formation in order to maintain low defect densities if the growth process has to be changed according to other demands, e.g. diameter increase, reduction of new defects or cost reduction.

Micropipes (MPs): Characterization. MPs are hollow defects running parallel the crystallographic *c*-axis and can be described as screw dislocations with large burgers vectors [9]. As they show a typical contrast in optical transmission microscopy the average MP-densities were commonly measured by manual counting. With decreasing MP-densities statistical point tests were no longer useful and automatic counting procedures were developed. Recent studies show that there are still shortcomings in the comparability between different work groups and different characterization techniques [10]. We identified this problem as fundamental for the further quality development and therefore put strong efforts in the improvement of measurement reliability. By comparing destructive, e.g. etching, and non-destructive methods, e.g. light microscopy or scanning electron microscopy, we were able to define guidelines for the determination of reliable MP-densities and distributions [11].

Micropipes (MPs): Reduction. The generation of MPs is often related to macroscopic defects like modification changes [12], or secondary phase inclusions [13], but can also be caused by improper seed attachment [14]. On the other hand MPs can be eliminated by moderate growth conditions [7], e. g. low axial temperature gradients [8]. Therefore low MP-densities can only be realized if formation is minimized while the temperature field is kept moderate. We reduced temperature gradients in our growth process, optimized the seed attachment and performed fundamental investigations on the influence of powder and crucible material properties on the formation of phase transitions. Based on the latter we were able to adjust the specifications of raw materials. Improvement of process control additionally enhanced the reproducibility of growth conditions and allowed us to produce 3 inch substrates with MP-values below 1 cm^{-2} for 6H- and 10 cm^{-2} for 4H-substrates respectively even for high volume output [Fig. 1]. While a standard 6H-substrate [Fig. 1, left] shows single statistically distributed MPs indicating a process limited MP-density, a 4H-wafer [Fig. 1,right] taken from the actual 3”-production contains large areas free of MPs and some localized MP-clusters, which have their origin mainly in the seed.

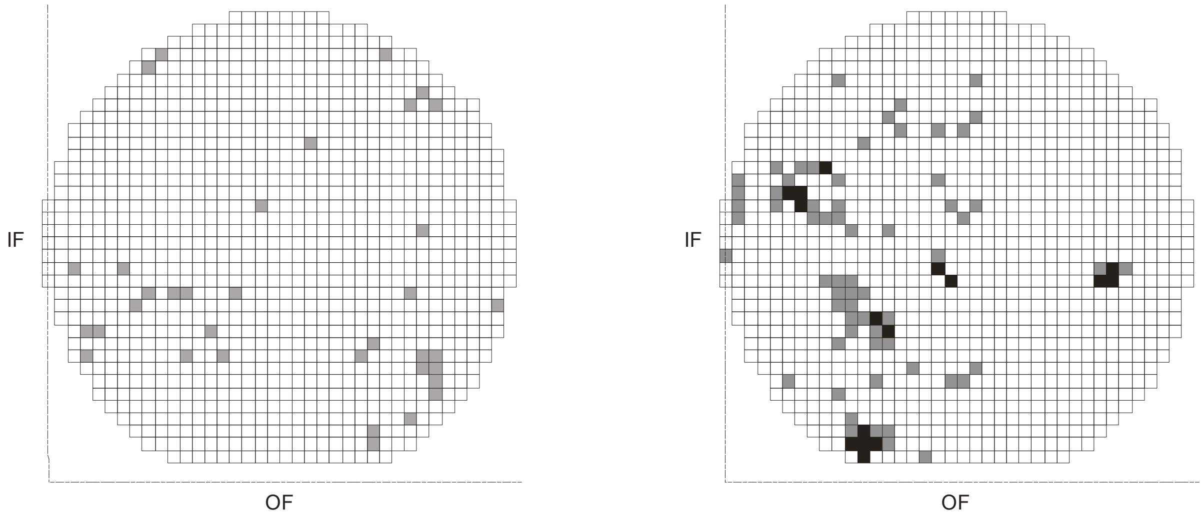

Fig. 1. Automatically captured full scan micropipe mappings. The shade of grey of the fields indicates the number of MPs inside the squares (white: zero, grey: 1-2, black: >2). Left: 3 inch 6H substrate with an overall MP-density of 0.9 cm^{-2}. Right: 3 inch 4H substrate with an overall MP-density of 4 cm^{-2}.

Low Angle Grain Boundaries (LAGB) Bright contrasts in stress birefringence images show disorders of the crystal lattice in the investigated substrates caused by stress, LAGBs or MPs. LAGBs are generally caused by strong growth disturbances like modification changes and their healing by repeated growth is difficult. A stress birefringence picture of a 4H-3"-substrate [Fig. 2, left] shows disturbed areas which originate mainly from the seed and can be correlated to the micropipe clusters shown in the previous section [Fig. 1, right]. In order to eliminate these non-usable areas we are continuously developing new seed generations with reduced low angle grain boundaries and MPs for our 3 inch production lines. A 2"-4H intermediate stage with enhanced quality is shown below [Fig. 2, right].

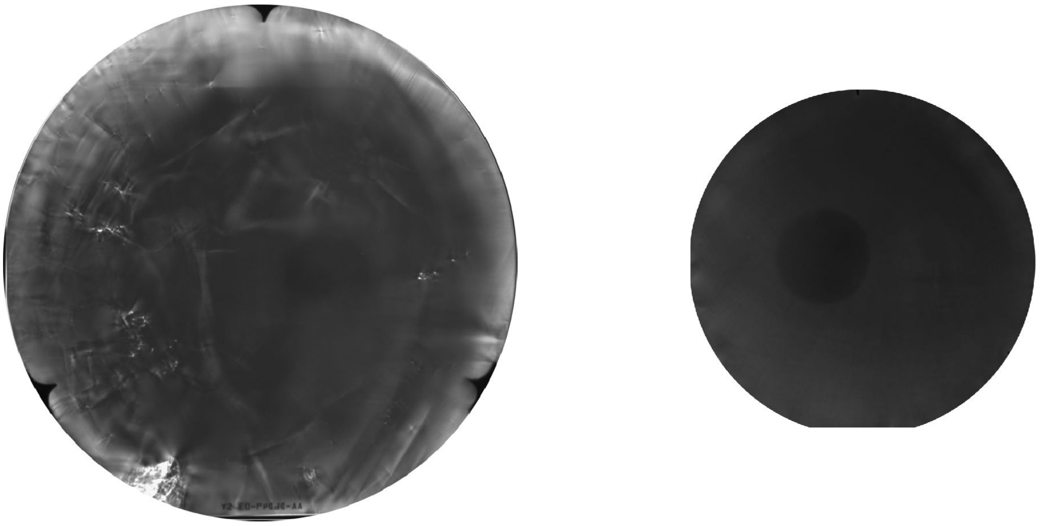

Fig. 2. Stress birefringence images. Left: 3" 4H substrate with current production quality. Right: Next generation 2" 4H substrate with enhanced crystalline quality (*) and without micropipes (confirmed by full scan measurement with optical microscope and KOH-etching). *Although the exposure time is increased the contrast is clearly weaker.

Basal Plane Dislocations (BPDs). Since a long time MPs are known to lead to failures in power and high frequency devices and therefore densities below 1 cm^{-2} are targeted in substrates for these applications. The influence of dislocations on the other hand is not similarly obvious as they lead to more subtle effects like reduced device performance and long term degradation. But since it is evident that the degradation of forward operation characteristics of bipolar devices is related to BPDs and staking faults (SF) [15] the focus of material development has shifted to this type of defect. In our work we focused on the influence of dislocations on unipolar devices, fabricated simple Schottky-test structures on 4H wafers and compared device properties with the defect structure in the underlying substrate areas. In SEM-CL images [Fig. 3, middle] of devices with electric performance below average we found CL-contrast bands aligned in $<1\bar{1}00>$ which we were able to correlate with arrays of BPDs [Fig. 3, left] after removing the epitaxial layer and etching in KOH.

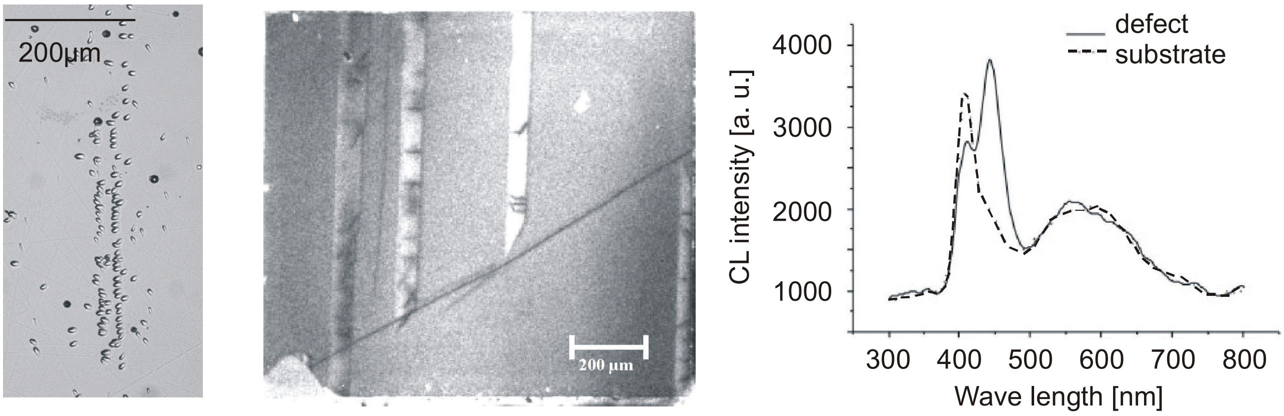

Fig. 3. Left: 4H wafer surface after etching in molten KOH, showing arrays of oval etch pits correlated with basal plane dislocations. Middle: SEM-CL image of a Schottky-test structure on a 4H wafer after ion etching. The bright lines indicate areas with changed CL-wave length. Right: spectral measurement of the test structure with peak maxima at 445 nm for characteristic bright areas and 400 nm for undisturbed substrate.

The BPD-line lies within the {0001} basal plane and can be revealed by etching of inclined substrates. 4H substrates are typically off-oriented by small angels towards $<11\bar{2}0>$ and therefore basal plane dislocations appear as oval point-bottomed etch pits. The alignment of the etch pits in arrays along the $<1\bar{1}00>$ direction indicates that the disturbed crystal areas have their main expanse in the basal plane but only small height in c-direction. Due to the existence of the basal slip system a/3 $<11\bar{2}0>$ {0001} and prismatic plane slip system a/3 $<11\bar{2}0>$ $\{1\bar{1}00\}$ it could be supposed that the alignment of BPDs in arrays or bands is caused by slip and cross slip mechanisms [16], which can be activated by plastic deformation. As we observe higher densities of BPD-arrays in the periphery of our crystals we assume that the slip is driven by thermo elastic stress exceeding the level of critical shear stress especially in the outer regions of the crystal and thus generating plastic deformation. According to simulations two strong potentials for the reduction of applied stress were identified. Based upon this findings we reduced the radial temperature gradients in our 4H-3”-process at r/2 by 70% [Fig. 4b] and modified the contact between crystal and growth chamber walls. Stress especially in the outer parts was significantly reduced [Fig. 4 c-e] and dislocation densities especially BPD-arrays were diminished. The best value so far achieved for the sum of both BPD and threading edge dislocations (TED) was 7×10^3 cm^{-2}.

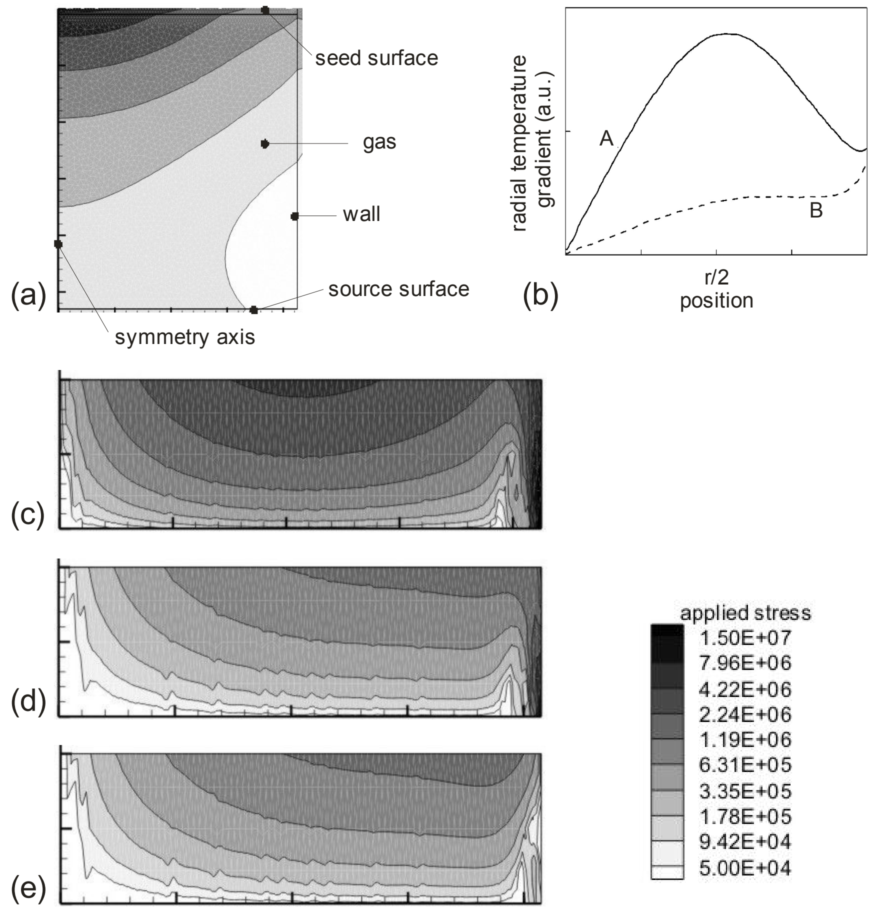

Fig. 4. Results from numerical calculation showing (a) an example of an temperature distribution inside a simplified growth chamber and (b) radial temperature gradients before (A) and after (B) optimization of the thermal field. The resulting applied stress distribution inside a 3” crystal at an early stage of growth is shown (c) before and after (d) optimization of the thermal field, and (e) after enabling unfixed growth at the crystal periphery.

Summary

During the last years we were able to increase the production standards of our SiC-substates for both modifications up to 3 inch and simultaneously reduced micropipe-densities in our production lines down to 1 cm^{-2}. Continuously we work on next generation quality for 4H-substrates without micropipes or low angle grain boundaries. Additionally we intensified our efforts on dislocation reduction and found out that basal plane dislocations can be reduced by optimized growth conditions with lower thermo-elastic stress.

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