

## Bulk II

### **Lateral Enlargement of Silicon Carbide Crystals**

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### **“Insitu synthesis” of Source Material from Elemental Si and C during SiC PVT Growth Process and Characterization Using Digital X-ray Imaging**

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### **The Development of 4H-SiC{03-38} Wafers**

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## Lateral enlargement of silicon carbide crystals

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Large seeds with low defect density are important for bulk growth of silicon carbide (SiC). At present, several kinds of crystallographic imperfections exist in SiC crystals. Many of these defects are inherited from the seed crystal. Micropipes and threading dislocations are defects that are continued from the seed crystal into the growing bulk crystal. These defects are normally observed when the growth proceeds in the *c*-axis direction,  $\langle 0001 \rangle$ .

In this work crystals have been grown laterally along the *a*-axis direction in a modified sublimation growth system. The idea was to have a small crystal with few defects and enlarge the crystal in the *a*-axis direction without increasing the number of micropipes and other threading defects. The system was optimized for lateral growth concerning susceptor, pressure and temperature gradient. Simulation of the susceptor design was made to optimize the conditions for lateral growth. The growth mechanism for the lateral growth is different compared with that along the  $\langle 0001 \rangle$  direction. No screw dislocations are formed in this case. The growth of the crystal proceeds in a hexagonal habit independent of the shape of the seed crystal depending on different growth velocity in different directions.

On-axis 6H-SiC Lely and 4H-SiC modified-Lely crystals with 8° off-cut were used as seed crystals. Due to a remaining small vertical temperature gradient in the center of the susceptor, growth also occurred on top of the seed crystal at the center of the seed. The ratio between *c*-axis growth (normal) and *a*-axis growth (lateral) was around 1/20 in the center. Depending on the type of seed, the polytype of the grown material can be different in different growth direction (*c*- and *a*-direction). The lateral enlargement reached 6 mm, which is limited by the susceptor design. Growth temperature varied between 2320 and 2420 depending on seed crystal and the growth rate between 1.2-4.0 mm/hour. The polytype of the laterally grown 4H-material is stable in a certain temperature range according to PL measurements. The grown crystals have been studied concerning morphology and crystalline structure. The grown crystals have been investigated by high-resolution X-ray diffraction and synchrotron topography. The results show that this growth technique makes it possible to make enlargements of crystals without increasing the number of micropipes. The mechanism of the lateral growth will be discussed.

Figure 1 shows a demonstration of a laterally grown part of a crystal and the overgrowth of the crystal. Region *A* is the laterally grown part of the crystal, *B* is the overgrowth of the laterally grown part of the crystal. Particles on the surface interrupting the step-flow growth are marked with arrows. Figure 2 shows a section transmission topograph of the laterally grown part of the crystal and the interface with the seed crystal. The bands with different contrast seen in the topograph are due to strain probably caused by bending of the crystal. No micropipes are seen in the topograph.

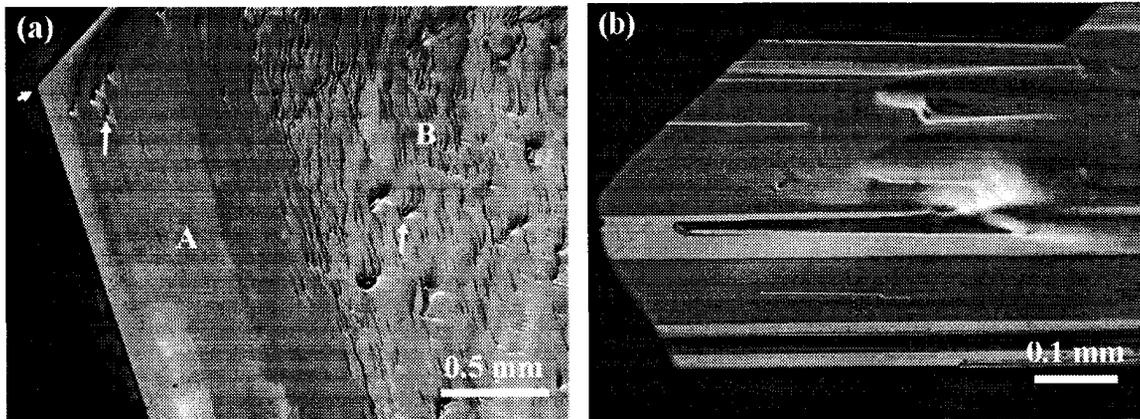


Fig. 1 (a) Region *A* shows the laterally grown part of the crystal, *B* shows the overgrowth of the laterally grown part of the crystal. Particles on the surface interrupting the step-flow growth are marked with arrows; (b) image taken at edge of laterally grown part (indicated by arrow in figure a).

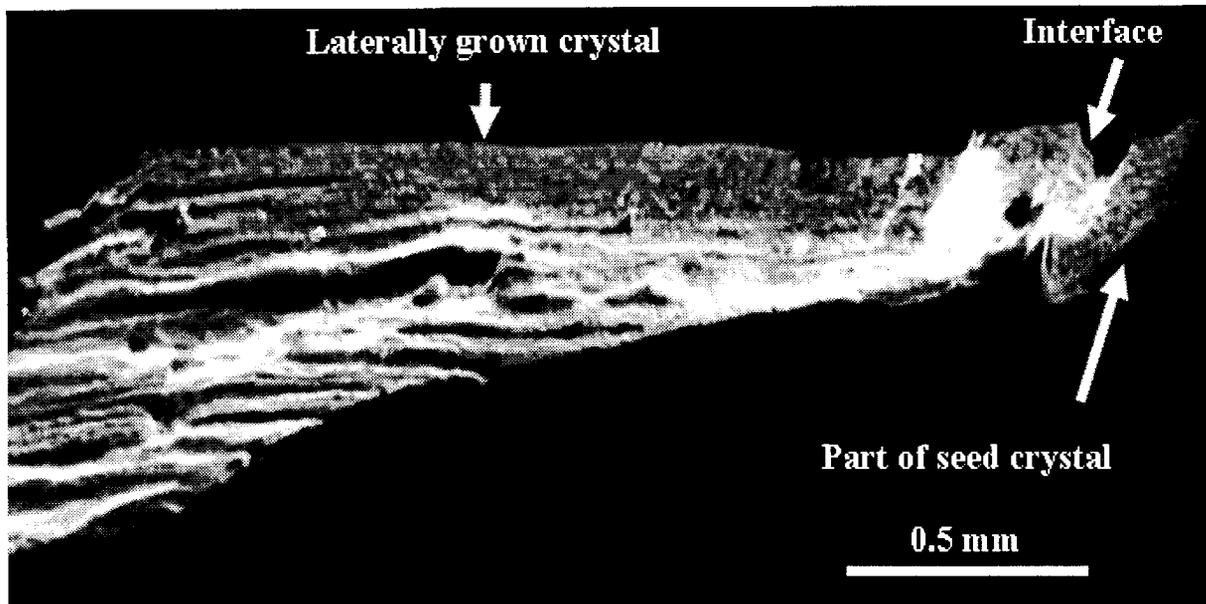


Fig. 2 Section transmission topograph of the laterally grown part and the interface with the seed crystal. The bands with different contrast seen in the topograph are due to strain probably caused by bending of the crystal. No micropipes are seen in the topograph.

## “Insitu synthesis” of source material from elemental Si and C during SiC PVT growth process and characterization using digital x-ray imaging

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Today, SiC single crystals for commercial applications are grown by the physical vapor transport (PVT) method using SiC powder as source material, the latter being synthesized prior the real growth process during an extra technological step. In order to save one process step we have investigated the possibility to combine both, the SiC powder synthesis from elemental Si and C and the SiC crystal growth process into one technological step. First of all we had to prevent the SiC seed from raising SiC powder grains due to highly exothermal reactions during SiC synthesis from elemental Si and C (midrange temperatures, i.e. 1450°C). Secondly we had to guarantee comparable SiC crystal growth conditions (elevated temperatures, i.e. 2200°C) as in the original two step counterpart, i.e. adjust the same SiC powder grain size, etc.. In order to address these questions we used a recently developed digital x-ray imaging technique [1,2] which allowed an online visualization of the ongoing processes and an identification of problems at the time when they occur.

The insitu synthesis of the SiC source material was carried out at the same temperature (i.e. 1600°C) and for the same holding time as in the conventional two step PVT process. It turned out to be mandatory to prevent the SiC seed from the exothermal synthesis process. For this purpose

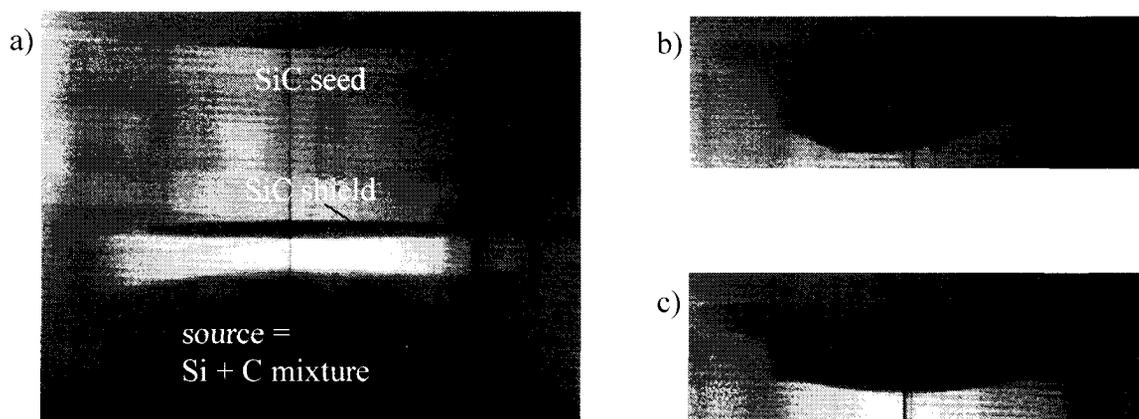


Figure 1. X-ray image of PVT crucible interior showing (a) the SiC seed, source material (mixture of high purity Si and C, and SiC shield between source and seed prior the growth process, (b) crystal growth interface with a broken SiC wafer shield after insitu Si + C synthesis process and (c) crystal growth interface using a modified, stable shield.

a SiC wafer was mounted as shield between the Si+C mixture (=source material) and the SiC seed (see Fig. 1a). Using this minor modification to the conventional growth procedure, 6H SiC single crystals with 40mm in diameter were grown. The strength of the underlying synthesis driving force became evident when the SiC shield broke one time at the Si+C synthesis temperature. In this case a rather inhomogeneous SiC sublimation occurred resulting in a highly non-uniform crystal shape (Fig. 1b). However, after modifying the shield in Fig 1a, homogeneous growth conditions were achieved leading to an uniform and flat growth interface (Fig. 1c) and low overall defect density comparable to the conventional SiC growth process (micropipe density  $<200\text{cm}^{-2}$ , high polytype stability, etc.).

The analysis of the x-ray images showed that the growth rate in the initial time (first 5h...10h of 72h) was smaller than in comparison to the convention PVT process with SiC powder as source material. It turned out that the smooth surface of the SiC shield limited the initial SiC sublimation. In the case of SiC powder, a large effective surface leads to a higher sublimation rate. After about 5h...10h both sources (SiC powder and Si+C mixture) developed a needle like surface morphology which serves as an optimized sublimation interface. The latter was supported by the SiC PVT growth experiment in which only a part of the SiC source surface was shielded by a smooth SiC wafer (Fig. 1b). In this case the crystal growth interface showed the above described non-uniformity, i.e. lower growth rate in the case of a smooth surface. Once a needle like structure was formed, typical (conventional) growth rates of about  $250\mu\text{m/h}$  were reached.

We will demonstrate the successful application of the insitu synthesis of the SiC source material prior the real crystal growth process. We will discuss the arising problems and solutions like the application of a SiC wafer shield by showing several x-ray images which were taken online during different growth runs. Finally we will address fundamental aspects of the sublimation kinetics arising from the analysis of our experimental x-ray imaging data.

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## Full Si wafer conversion into bulk 3C-SiC

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If obtained in a sufficiently good crystalline quality, cubic silicon carbide (3C-SiC) would be the most attractive SiC polytype. It exhibits superior electronic properties with respect to 4H-SiC and could be used as substrate for  $\text{Ga}_{1-x}\text{Al}_x\text{N}$  growth. This would be true in the nitride cubic or hexagonal phases depending only on the [001] or [111] orientation of the cubic substrate. Half of the production of hexagonal SiC is used as substrates for nitride optoelectronics, and this application is far less demanding for residual doping than electronic devices. As a consequence, producing large area 3C-SiC substrates appears of very much interest.

3C-SiC is usually grown by CVD on Si wafers [1]. It can be obtained at much lower temperature than hexagonal SiC (and in much larger substrate dimensions). However crystal quality is poor, due to the large mismatch of lattice parameters and thermal expansion coefficients between Si and SiC. The effect is even worse in the  $\langle 111 \rangle$  direction which corresponds to a more compact stacking.

To overpass this problem, we investigated the effect of completely converting a Si wafer into bulk SiC. The principle of the transformation is the following one [2]. A first SiC layer is grown by CVD on top of a Si wafer. Then the layer is put downwards to serve at the same time as a substrate and a crucible. After heating over the melting point of Si some propane is introduced and, provided a convenient temperature gradient exists, all Si converts into SiC by LPE (Liquid Phase Epitaxy) on the CVD SiC seed. No Si remains in contact with SiC and the overall stress is expected to release. All layers obtained in this way were light yellow and transparent, with thickness of 40 to 100  $\mu\text{m}$ .

In Fig. 1, we show the results of a cross sectional TEM investigation. The dark part is the CVD layer. This indicates a large dislocation density. Obviously, in the LPE region, the dislocation density reduces. In Fig. 2, a plan view TEM shows no dislocation in the LPE layer which means that the dislocation density is of the order of (or lower than)  $10^6 \text{ cm}^{-2}$ . This is 2 to 3 orders of magnitude below the standard CVD part. The same figure shows that the density of stacking faults remains high (in the range of  $5 \times 10^8 \text{ cm}^{-2}$ , which is not different from the CVD layers). This means that the stacking faults are very difficult to control in 3C SiC while the density of dislocations can be lowered thanks to the combined effects of LPE, lack of external stress and increased thickness.



Fig 1: TEM micrograph of the cross section of LPE SiC/CVD SiC

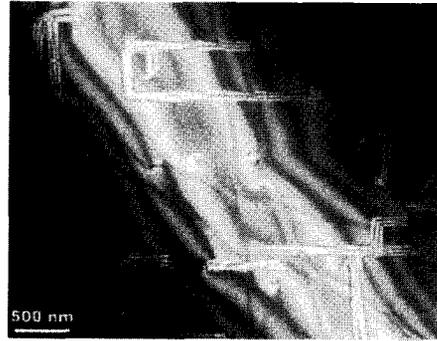


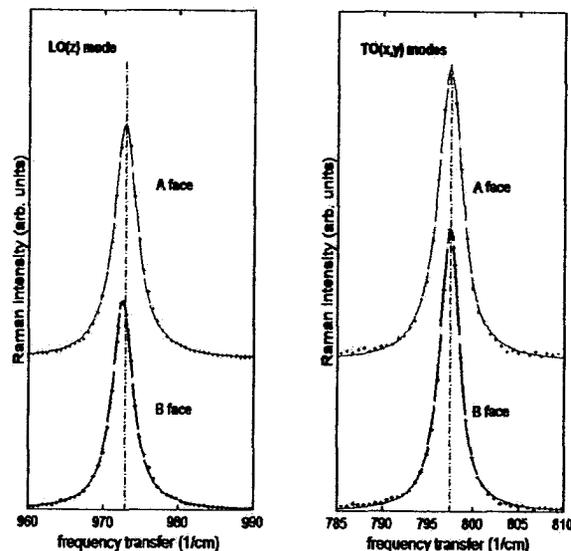
Fig 2: TEM micrograph of the plan view of LPE SiC/CVD SiC

A comparison of Raman spectra collected on the two different faces is shown in Fig.3 for the longitudinal and transversal modes, respectively. Concerning the longitudinal (Z-like) modes, the position on the B face is  $972.6 \text{ cm}^{-1}$  slightly shifted with respect to the A face. Notice that both lines remain symmetric. When fitted with simple Lorentzian forms (solid lines) they give a width (FWHM) of  $3.7$  and  $3.3 \text{ cm}^{-1}$  for the A and B faces, respectively.

The line shapes of the TO mode are more asymmetric and could not be fitted using a simple Lorentzian shape. To explain this asymmetry, we have to address the phonon-disorder interaction [3]. Because it results in a slower drop of the low-frequency wing in comparison with the high-frequency wing of the line, the line shape depends on the geometry of the defects.

The defects can have a point-like form, a line form (like dislocations, for instance) or a plane form (stacking faults, micro-crystal boundaries, etc...). In our case the best fit was achieved using a theory which includes the phonon interaction with plane defects. The position and width ( $797.5$  and  $3.16 \text{ cm}^{-1}$  for the A face and  $797.4$  and  $2.51 \text{ cm}^{-1}$  for the B face, respectively) are intermediate between the two extreme values obtained in a previous study of strain and strain-relaxation at the SiC/Si interface [3] and we conclude to that the strain relaxation is still not complete.

Fig. 3: Raman spectra collected on both sides of a LPE 3C-SiC layer obtained as described in the text.



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# The Effect of Nitrogen on Crystal Growth of SiC on $\{11\bar{2}0\}$ Substrate

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## 1. Introduction

High quality crystals have been grown by Chemical Vapor Deposition (CVD) on vicinal (0001) Si-plane, however, channel mobility of MOSFET on this plane has been miserably low. Recently, high channel mobility on  $\{11\bar{2}0\}$  was reported [1], so crystal growth on  $\{11\bar{2}0\}$  substrate is strongly focused. Nevertheless, SiC boules have been conventionally grown on  $\{0001\}$ , and growth mechanism on  $\{11\bar{2}0\}$  have not known well. Nitrogen doped "buffer layer" is introduced into the interface between substrate and epilayer to grow high quality epilayer on  $\{11\bar{2}0\}$  substrates in case of CVD [2]. However, the effect of nitrogen on crystal growth have not discussed well. Crystal growth on  $\{11\bar{2}0\}$  substrates were conducted and the effect of nitrogen was discussed.

## 2. Experiment

Sublimation growth was achieved using a quartz tube reactor with a water-cooled jacket.  $\{11\bar{2}0\}$  substrates were cut from the boule previously grown on  $\{0001\}$ , and they were put on the lid of graphite crucible. Abrasive SiC powder was charged into the crucible as a source material. The temperatures at the top and the bottom of the crucible were monitored by optical pyrometers, and they were kept at approximately 2200°C and 2500°C, respectively. Several crystals were grown under argon atmosphere and the others were grown under nitrogen atmosphere. The growth pressure was approximately 30 Torr. Surface morphology of grown crystals were observed by optical microscope, scanning electron microscope (SEM) and atomic force microscope (AFM).

## 3. Results and Discussion

### 3.1 Crystal Growth of 6H-SiC on $\{11\bar{2}0\}$ Substrate

Surface morphology of the crystal grown by sublimation method on  $\{11\bar{2}0\}$  substrates have known as smooth [3], and reports concerning about the defects on  $\{11\bar{2}0\}$  is few. However, crystal grown on  $\{11\bar{2}0\}$  substrates contained hollow core defects if growth conditions were not optimized. Hollow core defects penetrated to the  $\{11\bar{2}0\}$  surface and pits were observed on the surface as shown in Figure 1. The pits observed here were also seen on the sidewall of grown crystal on  $\{1\bar{1}00\}$  substrates, namely  $\{11\bar{2}0\}$ . So, these pits could be thought as intrinsic defects on  $\{11\bar{2}0\}$  in the crystal growth of SiC.

From observing the pits on  $\{11\bar{2}0\}$ , we suggest a growth model of SiC on  $\{11\bar{2}0\}$  substrate. On flat  $\{11\bar{2}0\}$ , two-dimensional growth would proceed at optimum growth conditions. However, if substrate surface was not atomically flat or growth conditions were not optimized, surface diffusion length would become shorter and growth mode would change from two-dimensional growth to three-dimensional growth. As three-dimensional islands grow at all over the surface, they would coalesce each other. Hollow core defects would be created at the boundary of coalesced islands, if there was a misalignment between each island. Stacking faults might affect to this misalignment of islands. Though several mechanisms have been reported [3], generation mechanism of stacking faults on  $\{11\bar{2}0\}$  has not been cleared well. We contributed the stacking

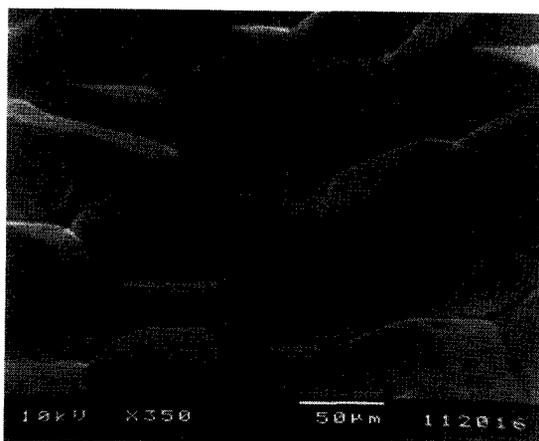
fault generation on  $\{11\bar{2}0\}$  to three-dimensional growth. Stacking fault density should be low on  $\{11\bar{2}0\}$  as long as crystal grows two-dimensionally, because bonding configuration is always uniquely determined on  $\{11\bar{2}0\}$ . In case of three-dimensional growth,  $\{1\bar{1}00\}$  facets would appear on the shoulder of islands because of growth rate anisotropy between  $\langle 1\bar{1}00 \rangle$  and  $\langle 11\bar{2}0 \rangle$ . Stacking faults would be created easier on  $\{1\bar{1}00\}$  by kinetically-induced misarrangement of surface adatoms [3]. Stacking faults could have harmful effect on coalescence of islands. Therefore, crystal growth on  $\{11\bar{2}0\}$  substrate should be proceeded two-dimensionally so as not to increase the incorrect coalescence of islands. Atomically flat surface is thought to be significantly important to keep two-dimensional growth on  $\{11\bar{2}0\}$ .

### 3.2 Crystal Growth on $\{11\bar{2}0\}$ Substrate under Nitrogen Atmosphere

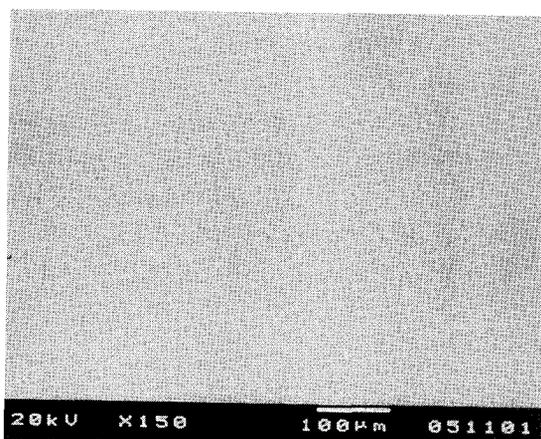
Similar treatment as CVD, that is introducing the nitrogen doped buffer layer into the interface between substrate and grown crystal, might be also useful for sublimation growth to grow high quality crystals on  $\{11\bar{2}0\}$  substrate. To investigate the effect of nitrogen, sublimation growth on  $\{11\bar{2}0\}$  substrates were carried out under nitrogen atmosphere for total growth duration. Another growth conditions were same as the growth under argon atmosphere. Significant improvement of surface morphology was achieved as shown in Figure 2. Pits were not observed on the surface.

The effect of nitrogen in CVD growth has been considered as the reduction of lattice mismatch caused by doping difference between substrate and epitaxial layer [2]. In spite of small doping difference between substrate and grown crystal, nitrogen effected a lot to improve the crystal quality even in sublimation growth. Growth mode seemed to be changed from three-dimensional growth to two-dimensional growth by growing the crystal under nitrogen atmosphere. So, besides reducing the lattice mismatch, the role of nitrogen seemed to be flattening the surface and inactivating defects on the surface. Silicon nitride might be created from place to place on  $\{11\bar{2}0\}$  surface. Selective growth using self-constructed silicon nitride mask might affect as slowing down the growth toward  $\langle 11\bar{2}0 \rangle$  and flattening the surface.

In addition, lowered C/Si ratio by using tantalum improved the surface morphology in the growth under both argon and nitrogen atmosphere. Lowered C/Si ratio might be contributed to increase the effective adsorption of nitrogen on  $\{11\bar{2}0\}$ .



**Figure 1.**  
SEM image of the pits observed on  $\{11\bar{2}0\}$ .



**Figure 2.**  
SEM image of flat surface grown on  $\{11\bar{2}0\}$  substrate under nitrogen atmosphere.

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### QuaSiC Smart-Cut® substrates for SiC high power devices

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Keywords : 4H SiC, polycrystalline SiC, tungsten silicide, wafer bonding, Smart-Cut®, epitaxy,

The development of SiC based electronic power devices, such as Schottky diodes, is today limited by both the availability and price of high quality low resistivity 4H SiC wafers.

One way to overcome cost and availability limitations is to propose an alternative material source suited to power electronic devices. The generic nature of the Smart-Cut® process, based on proton implantation and wafer bonding, is now recognized through successful demonstrations of Si [1], SiC [2], InP [3] and GaAs [4] thin film transfers. In the particular case of SiC, previous works have been first focused on the multiple transfer of high quality SiC thin layers onto dissimilar substrates, mainly such as silicon and polycrystalline SiC wafers, via oxide layers, for the fabrication and characterization of SiCOI substrates (SiC On Insulator) [5]. In these studies main results have concerned the control of the electrical properties of the transferred thin film.

One possible material solution for the SiC technology for power electronics is to transfer several times thin layers cut from a very high quality SiC substrate (low micropipe and dislocation densities) onto a lower cost substrate such as polycrystalline SiC or a lower crystal quality SiC substrate. This technology, as demonstrated for SOI wafers (Silicon On Insulator), is scalable to larger substrate diameters. This is particularly interesting as SiC wafers are shifting towards 4 inch diameter.

In this work, we present the last developments of this technology for the demonstration of vertical truly conducting SiC based substrates. We have particularly studied the development of structures such as monocrystalline 4H SiC thin film onto CVD polycrystalline SiC substrates. Wafer bonding between mono and polycrystalline SiC wafers with refractory and conductive tungsten silicide based bonding layers has been developed. This bonding layer has been chosen regarding physical considerations such as thermodynamical equilibrium with SiC, refractory behavior and ability to form ohmic contacts with SiC. This has led to the demonstration of SiC thin film transfer onto poly and mono SiC substrates using the Smart-Cut® technology (QuaSiC substrates) (Figure 1). Successful CVD epitaxial regrowth using standard bulk conditions have also been demonstrated onto QuaSiC substrates.

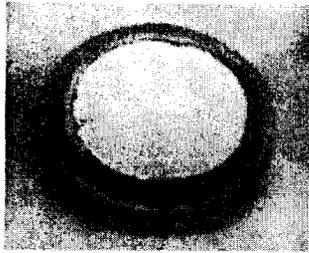


Figure 1 : 50.8 mm QuaSiC substrate obtained with the Smart-Cut® technology

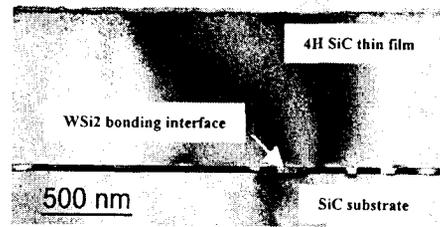


Figure 2 : TEM cross section of a QuaSiC substrate without epi regrowth

Both QuaSiC substrates with or without epitaxial regrowth have been physically and electrically characterized. TEM cross sections (Figure 2) have been performed for the investigation of the quality of the bonding layer as well as the crystalline transferred SiC thin film. TEM cross sections of QuaSiC substrates with epitaxial regrowth will be also presented and analysed regarding the quality of the different layers and interfaces.

Low temperature photoluminescence spectra show that the quality of the SiC epitaxial layer grown onto QuaSiC substrates is similar to epilayers grown on SiC bulk substrates (Figure 3).

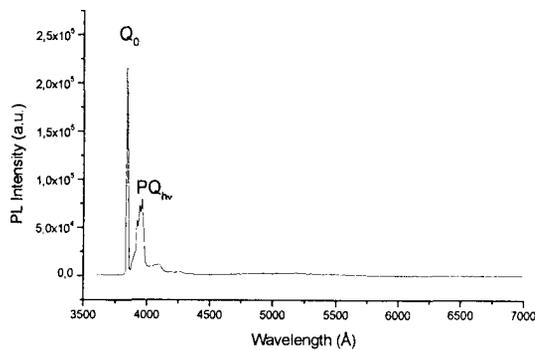


Figure 3 : LTPL of an epitaxial regrowth on QuaSiC substrate.

Indeed, the photoluminescence is dominated by near band edge (NBE) emission with no evidence of deeper energy band associated with impurities or defects. This well known NBE emission for n-type 4H SiC is associated with nitrogen bound exciton luminescence ( $Q_0$ ) and corresponding phonon replicas ( $PQ_{hv}$ ). Finally, the capability of the interface bonding layer to conduct high current flow has been checked using  $I(V)$  measurements on specific patterns. The comparison of electrical characteristics between transferred and non transferred structures has been carried out. Encouraging results have been obtained and will be detailed in this paper.

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# The development of 4H-SiC {03-38} wafers

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## 1. Introduction

Silicon carbide, which are known as the wide-gap-semiconductor, is expected for applications of the power devices such as the diodes or the power MOS FETs. However, in the crystal growth of SiC, the defects, such as the micropipe and the stacking fault, propagate along the growth direction and appear on the surface of ingots. Therefore, the development of high-quality wafer was very difficult.

We developed the crystal grown along  $\langle 03-38 \rangle$  direction which inclines at c axis by  $54.7^\circ$  as shown Fig.1, to make the defects propagated diagonally, and so as not to allow the defects to reach the front surface of the ingots (Fig.2). We report the result of the crystal growth and the characteristics of the crystals.

## 2. Experiment

Bulk single crystals were grown by the sublimation method. The crucible assembly consisted of the graphite support to which the seed crystal was attached and the graphite crucible containing the source powder. We used the seed 4H-SiC {03-38} crystal which was prepared by slicing diagonally the 4H-SiC {0001}

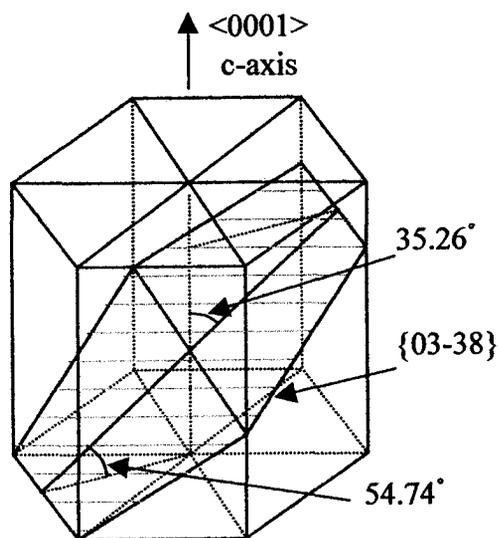


Fig.1 Lattice structure of 4H-SiC

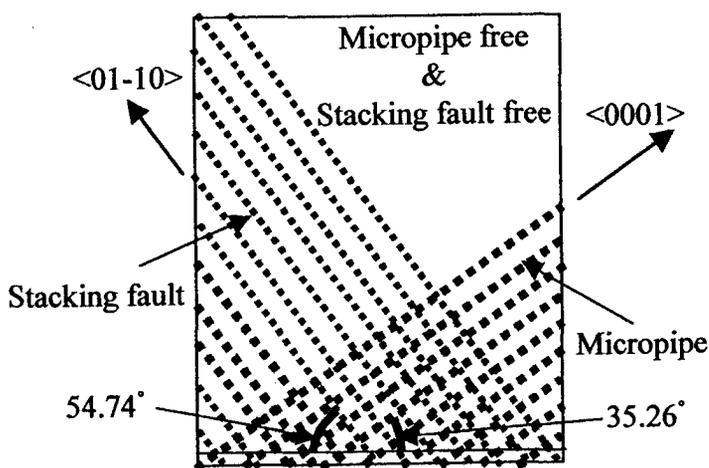


Fig.2 Schematic of defect propagation on crystal growth

ingot. The seed crystal and the source powder in the crucible were heated more than 2000 °C. During the crystal growth, the growth pressure was less than 10kPa. N-doped crystals were prepared by adding controlled amounts of high-purity nitrogen to the inert ambient.

### 3. Result and Discussion

We obtained 4H-SiC {03-38} wafer by slicing the ingots grown along the  $\langle 03-38 \rangle$  direction. We cut the ingots into the 4H-SiC {03-38} wafers. The micropipe densities of the seed crystal and the grown crystal (wafer) were determined by the optical microscopy with Nomarski interference contrast after etching wafers in the molten KOH. The micropipe was not observed on the surface of the grown crystal though there were some micropipes on the surface of the seed crystal (Fig.3). Moreover, when we observed the vertical section of the 4H-SiC {03-38} ingot, the stacking faults propagated diagonally and reached the side of the ingot.

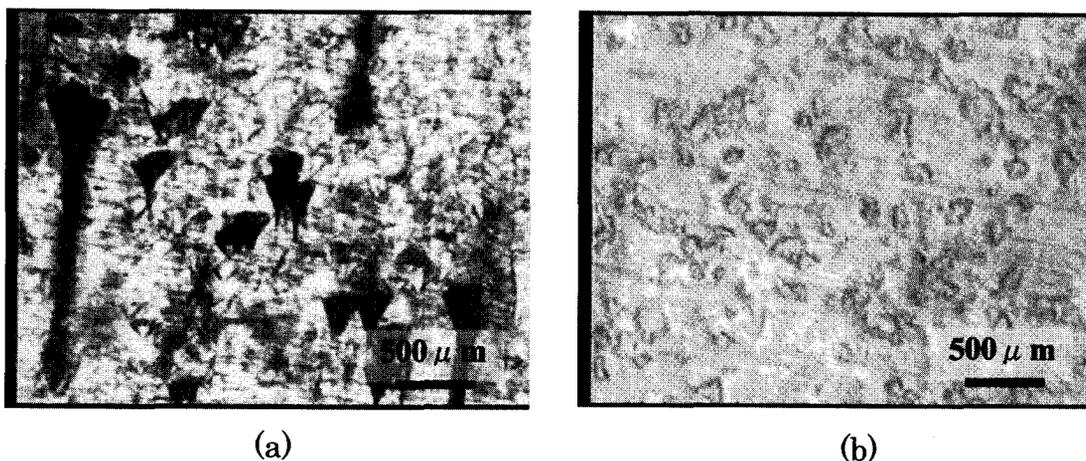


Fig.3 Micrographs of KOH-etched (a)seed (b)wafer

### 3. Summary

When the crystal was grown along the  $\langle 03-38 \rangle$  direction, we confirmed the area of no micropipe and the diagonal propagation of the stacking fault.