CHAPTER 2

SILICON CARBIDE TECHNOLOGY: BULK AND EPITAXIAL GROWTH

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INTRODUCTION

SiC is a wide bandgap material with well-recognized potential for high-power, high-temperature electronics. The fundamental parameters of SiC material are very attractive for the fabrication of semiconductor devices with superior characteristics for military and industrial needs in the aircraft and space electronics, nuclear power, automotive, and power utility industries. Silicon carbide devices will meet commercial and military needs for high current and high voltage devices, integrated circuits for power transmission and distribution systems, and other types of advanced electrical equipment and machinery. Because of their fundamental material parameters, SiC devices have been predicted to have a higher breakdown voltage (at the same doping level) and to operate at a higher forward current density than Si devices. The unique physical properties of SiC include a large energy bandgap, high thermal conductivity, and a high electric breakdown field. Recently, because of significant progress in SiC technology, these predictions have become a reality. SiC rectifiers with blocking voltages of a few kilovolts and devices operating at a forward current density of up to 1 kA/cm^2 have been fabricated (Park 1998; Pensl et al. 1998; Baliga 1998; Weitzel and Moore 1998; Itoh and Matsunami 1997; Chelnokov et al. 1997). SiC *p-n* junctions have been shown to operate at 500°C. However, SiC devices are still in the R&D stage, not in production. Factors that are limiting the performance of SiC devices relate to the material quality of SiC substrates and their epitaxial structures. This chapter summarizes recent results in SiC bulk and epitaxial growth in Japan and the United States.

6H-SiC and 4H-SiC Bulk Crystal Growth

Silicon carbide substrates are key elements in the development of SiC electronics. Compared to other wide bandgap semiconductors, the availability of SiC substrates for homoepitaxy is a big advantage. Because of the phase equilibria in the SiC material system (specifically the material sublimes before it melts), the most popular bulk growth techniques are based on physical vapor transport. These techniques were initially developed in the late 1950s and were modified and introduced for production in the early 1980s. Although sublimation techniques are relatively easy to implement at the high growth temperatures required, these processes are difficult to control, particularly over large substrate areas. Three types of processes have been used for growing bulk SiC commercially:

1. the Acheson process (Acheson 1892)

2. the Lely process (Lely 1995)

3. the modified Lely process (Tairov and Tsvetkov 1978; Tairov and Tsvetkov 1981)

Many university and commercial research teams have adopted the latest methods. The university teams include Kyoto University (Itoh 1995), Kyoto Institute of Technology (Takanaka et al. 1996), Howard University (Shields et al. 1993), and Carnegie Mellon University (Heydemann et al. 1998). The companies include Nippon Steel (Takahashi et al. 1995; Takahashi et al. 1997), Toyota Central R&D Laboratories Inc. (Sugiyama et al. 1996), SiCrystal (SiCrystal n.d.), Westinghouse Corp. (Barrett et al. 1993, now a part of Northrop Grumman Corp.), Cree Research, Inc. (Glass et al. 1997), Advanced Technology Materials Inc. (Buchan et al. 1994), and Sterling Semiconductor (Sterling Semiconductor n.d.).

Physical vapor growth is accomplished through the sublimation of a SiC source placed in the hot zone of the growth furnace and the subsequent mass transport of the vapor species to the seed crystal located in a cooler region of the furnace (Glass et al. 1997; Dmitriev and Spencer 1998; Hofman et al. 1998; Nishino 1995; Konstantinov 1995). Single crystal SiC material is formed from deposition of the supersaturated vapor species. Source materials may be composed of SiC powder, Si and C powders mechanically mixed, or crystalline SiC. In this technique, growth proceeds (usually along the c-axis) by vapor transport of C- and Si-bearing species from the source or carbon species from the graphite walls. For a typical 6H- and 4H-SiC bulk sublimation growth process, the SiC source temperature is 2100-2400°C (Barrett et al. 1992), growth pressure is less than 20 Torr, and the temperature gradient between source and seed ranges from 20-35°C/cm. Typical growth rates for the bulk growth of SiC are in the range of 0.5-5 mm/hr. Currently the maximum commercially available 4H- and 6H-SiC crystals are 50 mm in diameter (see Table 2.1) (Cree Research n.d.). Three-inch boules of SiC were demonstrated (Brandt et al. 1996), and record 80 mm diameter SiC wafers were reported (Maksimov et al. 1998). Other polytypes of silicon carbide (i.e., 3C, 2H, 15R, 33R) are not commercially available.

Table 2.1

Size of SiC Bulk Crystals

Firm	Demonstration	Production
Cree Research	3 inches	2 inches
Northrop Grumman	3 inches	NA
ATMI/Sterling	3 inches	1 3/8 inch
Nippon Steel	2 inches	1 inch

Structural Defects in Bulk SiC Crystals

There has been significant progress in the quality of material produced by seeded sublimation technology. Dislocation density and micropipe density in SiC bulk crystals grown by the modified Lely method currently range from 10^4-10^5 cm^{-2} and 10–10^2 cm^{-2}, respectively. Note that for original Lely crystals, these values are 10-100 cm^{-2} and 0 cm^{-2}, respectively. Micropipes are defects unique to the growth of SiC. These micropipes, which are physical holes, can travel large distances in the crystal lattice. Micropipes have been shown to be "killer" defects if they intersect with the active regions of a device (Neudeck 1998). For small size wafers (35 mm in diameter), Cree Research has reported a micropipe density of 0.7 /cm^2 (Tsvetkov et al. 1996). Nippon Steel has reported micropipe density reduction from more than 1000 cm^{-2} down to 2 cm^{-2} for 1-in diameter R&D material (Takahashi et al. 1996; Ohtani 1998). The SiC project at Nippon Steel targets a micropipe density of less than 1 cm^{-2} for 2-in diameter wafers (Ohtani 1998). Northrop Grumman has also reported a significant reduction of micropipe density (a micropipe density of 2 cm^{-2} 35 mm wafers has been achieved). Tsvetkov et al. (1996) have summarized the possible mechanisms of micropipe formation. Recently, TDI Inc. has developed a micropipe filling technology (TDI n.d.; Rendakova et al. 1998a; Rendakova et al. 1998b). This technology has resulted in 41 mm diameter SiC epitaxial wafers with micropipe densities of less than 0.5 cm^{-2}, and in the first commercial 2-in SiC epitaxial wafers with micropipe densities of less than 10 cm^{-2} (Dmitriev et al. 1998). Table 2.2 shows the various micropipe densities in SiC wafers by manufacturer.

Table 2.2.
Micropipe Density in SiC Wafers (cm^{-2})

Firm	**Commercial wafers**		**Best R&D wafers**	
	Standard Grade	Select Grade	Diam. < 2-inch	Diam. 2-inch
Cree Research	>101-200	31 – 101*	0.7	26
ATMI	~100		12	
Northrop Grumman	NA		2	
TDI (epi wafers^{+})	<20		<0.5	5
Nippon Steel			2	50

*Cree is also selling Low Micropipe Grade SiC wafers with micropipe density less than 30 cm^{-2}.
+Note that these are epitaxial wafers, not bulk crystals.

Note that the micropipe problem becomes more severe for large SiC boules. Each step toward larger SiC wafers has resulted in a significant increase in micropipe density. Thus, the 3-in SiC wafer, when commercially available, will also likely suffer from high micropipe density.

Other types of crystal defects found in sublimation-grown SiC crystals include basal plane tubes, cracks, foreign polytype inclusions, and crystal domains. The crystal quality of the boules of SiC also suffers from problems common to all semiconductors, i.e., impurities, dislocations, and point defects. During the panel’s site visits, Japanese experts repeatedly emphasized that the poor quality of SiC substrate materials is the main limiting factor for the creation and production of SiC devices. It is important to note that there are no published data on defect structure and defect density for either insulating or highly doped SiC substrates.

Electrical Characteristics of Bulk SiC Crystals

Nitrogen usually contaminates undoped SiC bulk material, producing *n*-type conductivity. The typical background level of electron concentration for undoped SiC crystals grown by modified Lely method is 10^{16} -10^{17} cm^{-3}. Hobgood et al. have reported high purity undoped SiC crystals with room temperature resistivity from 10^{2} to 10^{3} ohm cm (Hobgood et al. 1995). These crystals have *p*-type conductivity with background carrier concentrations of 10^{15} cm^{-3} due to residual B impurities.

Both Nippon Steel and Cree Research have produced *n*-type SiC crystals with carrier concentrations up to 10^{20} cm^{-3} using nitrogen doping (Onoue et al. 1996; Onoue et al. 1998; Cree Research n.d.). The minimum reported resistivities for 6H-SiC and 4H-SiC bulk crystals are 0.0016 ohm cm and 0.0028 ohm cm, respectively. Sanyo Electric and Cree Research have obtained *p*-type SiC crystals using Al doping (Nakata et al. 1989; Cree Research n.d.). For bulk *p*-type SiC, Cree Research reported the highest carrier concentrations: up to 10^{20} cm^{-3}.

Semi-insulating 6H-SiC crystals have been produced using V doping (Hobgood et al. 1995). Cree Research has also reported semi-insulating 4H-SiC crystals with comparable resistivities (Cree Research n.d.). Only Cree Research fabricates semi-insulating 4H-SiC wafers for production. There are no reports on the development of insulating SiC in Japan.

Surface Preparation

A major characteristic of any substrate material is the quality of its surface. Due to its high chemical stability and hardness, surface treatment of a SiC substrate is a very difficult task. The panel was told repeatedly that Nippon Steel has made great progress in this area and that SiC wafers fabricated at Nippon Steel have high surface quality.

3C-SiC BULK GROWTH BY PHYSICAL VAPOR TRANSPORT

The 3C polytype has been considered metastable and therefore difficult or impossible to grow in bulk form. However, if the quality of the 3C seed material is high, its tendency to transform to 6H during the growth is much less. Some success has been obtained in the growth of bulk 3C-SiC crystals using the modified Lely technique. Bulk 3C-SiC crystals have been grown at Howard University (Jayatirtha and Spencer 1998; Jayatirtha and Spencer 1996), Kyoto Institute of Technology (Yoshikawa et al. 1996), Sharp Corporation (Furukawa et al. 1993), and Kyoto University (Nishino et al. 1996).

SiC EPITAXIAL GROWTH

In order to improve the quality of bulk material and to produce complicated device structures, epitaxial techniques are necessary. Chemical vapor deposition (CVD) is presently the most widely used epitaxial technique for the growth of SiC device structures.

CVD Homoepitaxial Growth of 6H-SiC and 4H-SiC

Homoepitaxial CVD growth of SiC has been reported for 6H, 4H, and 3C polytypes of SiC while heteroepitaxy of 3C-SiC has been reported on AlN, sapphire, Si, 6H-SiC, and 15R-SiC substrates (Matsunami and Kimoto 1997; Nishino 1995; Larkin 1997).

Kyoto University has developed the homoepitaxial growth of α-SiC (6H-SiC, 4H-SiC, 21R-SiC) by CVD using off-oriented SiC substrates (Itoh et al. 1994). This technique is called step-controlled epitaxy because the growth process is determined by the lateral growth rate of the terraces. The growth rate, substrate misorientation, and growth temperature determine whether growth will occur via a step-controlled mechanism. If the growth is step-controlled, the epitaxial layer will replicate the stacking order of the substrate. Several researchers have discussed the growth mechanism for SiC homoepitaxial CVD layers (Kimoto et al. 1994; Hong et al. 1995). Kimoto and Matsunami (1995) have investigated nucleation processes during SiC CVD growth, and analyzed the surface kinetics of adatoms in the CVD growth of SiC. They based this analysis on the Burton-Cabrera-Frank theory (Kimoto and Matsunami 1994).

Details of several epitaxial growth processes have been published. Researchers from North Carolina State University described a growth process employing the SiH_4-C_2H_4-H_2 gas system (Wang and Davis 1991). In the growth procedure developed at NASA Lewis Research Center for 6H-SiC and 4H-SiC CVD, the samples are initially etched by HCl at 1350°C prior to growth (Powell et al. 1991). The initial HCl purge reduces the density of surface defects in the resultant SiC layers.

The surfaces of SiC epitaxial layers can contain large numbers of imperfections. Surface defects observed in SiC CVD layers are growth pits, polytype inclusions (which sometimes appear as triangular features), macro-steps (often referred to as step bunching), and micropipes. Some of these defects are relatively large (tens of microns), while others have an average size less than 1 micron.

Attempts to understand the nature of surface defects in SiC CVD layers have appeared in studies by a NASA research team (Powell et al. 1995; Powell et al. 1996) and at Kyoto University (Kimoto et al. 1995). Many factors influence the production and density of surface defects. These include substrate characteristics (orientation, face polarity, tilt angle, and crystallographic direction of the misorientation), mechanical and chemical treatment of the substrate before the epitaxy, substrate pre-growth treatment in the reactor, and growth conditions such as the Si/C ratio, growth rate, and growth termination procedure. The best results thus achieved have surface defect densities of 10^3 cm^{-2}. It is noteworthy that this value corresponds to the density of unknown defects in SiC *p-n* structures, which appear to cause premature junction breakdown (Chelnokov et al. 1997). Mechanisms of electric breakdown in CVD grown SiC *p-n* junctions are the focus of detailed studies but remain unclear (Neudeck et al. 1998a; Neudeck et al. 1998b; Kimoto et al. 1996).

Although some progress has been made in understanding the nature and cause of structural defects like step bunching and polytype inclusions, the origin and control of many defects in SiC remain to be investigated.

Electrical Properties

Significant progress has been achieved in producing epitaxial layers of 6H- and 4H-SiC with superior electrical properties. 4H-SiC epitaxial layers with electron concentrations as low as $2x10^{14}$ cm^{-3} were reported both in Japan and in the United States (Kimoto et al. 1996; Irvine et al. 1998). Kimoto et al. (1996) reported electrical and optical measurements of high quality 4H-SiC layers. The background doping concentration in the layers is $3x10^{15}$ - $2x10^{16}$ cm^{-3}, and the electron mobility in the {0001} basal plane was 600-720 cm^2/V-sec (300K). Deep level transient spectroscopy measurements on these films show that the concentration of electron traps is approximately 10^{13} cm^{-3} independent of substrate polarity.

Numerous publications have reported doping of SiC homoepitaxial layers grown by CVD. Nitrogen is commonly used as a donor, and aluminum is the acceptor of choice. Nitrogen has been investigated for several years as an *n*-type dopant. Nitrogen doping in these studies produces donor concentrations ranging from 10^{16} to 10^{19} cm^{-3}. *P*-type doping has been achieved by using Al as a dopant.

A greater range of doping control is possible with site-competition epitaxy (Larkin 1997; Larkin et al. 1994; Larkin et al. 1996). The method is based on varying the Si/C ratio within the CVD reactor in order to control the dopant incorporation in SiC during epitaxial growth. Site-competition epitaxy has been used for control of N, P, Al, and B incorporation in 6H-SiC and 4H-SiC epitaxial films on SiC (0001) Si and (000-1) C substrates.

Sublimation Epitaxy

The sublimation sandwich method makes it possible to grow high-quality SiC layers in a wide temperature range (Vodakov et al. 1979). This method provides both low doped and highly doped SiC layers. Thus the SiC growth rate can be controlled in a wide range. Recently, SiC sublimation epitaxy has been used in Japan to grow thick SiC layers for high power device applications (Yoshida et al. 1998; Nishino et al. 1998). In the United States, sublimation epitaxy is not applied for SiC growth.

SUMMARY

- Bulk SiC growth
  - PVT (sublimation) is an accepted technology to grow bulk SiC crystals (United States and Japan).
  - 2-in SiC wafers are in production (United States).
  - 3-in SiC wafers have been demonstrated (United States).
  - Highly doped bulk SiC crystals are demonstrated (United States and Japan).
  - Semi-insulating SiC crystals are in production (United States).
  - The United States is ahead in SiC bulk crystal production.
- Epitaxial SiC growth
  - CVD is an accepted method to grow 6H- and 4H-SiC layers and *p-n* structures (United States and Japan).
  - Low and high doping levels are achieved by SiC CVD technique (United States and Japan).
  - Nature of electrically active defects causing premature breakdown is unknown (research is ongoing in Japan and in the United States).
  - The United States and Japan are about even in R&D on SiC CVD growth.

- 2-in epitaxial layers and *p-n* structures are commercially available (United States). There is no SiC epi-production in Japan.

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