CHAPTER 4

SiC GROWTH TECHNOLOGY IN EUROPE

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INTRODUCTION

This chapter gives a review of SiC growth technology and then discusses the results of the TTEC panel review of European growth technology.

SIC BULK CRYSTAL GROWTH

Silicon carbide substrates are the key elements in the development of SiC electronics. Because of the phase equilibria in the Si and C materials 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 fifties and have been modified and introduced for production in the early eighties. 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.

Bulk Growth by Physical Vapor Transport

Physical vapor growth is accomplished by 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 a cooler region of the furnace. 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. The vapor transport is performed in either a vacuum or gas ambient. Single crystal growth can be realized either seeded or unseeded. Typical temperature and pressure ranges for SiC sublimation growth are 1600 to 2700°C and 10^{-6} to 20 torr, respectively. Usually, the lower temperatures are employed for sublimation epitaxial SiC growth, while bulk SiC growth is performed at the higher temperatures.

Modified Lely Process

Growth of SiC boules is possible using the modified, or seeded Lely method, also called the Tairov - Tsvetkov method (Tairov & Tsvetkov 1978; Tairov & Tsvetkov 1981) or physical vapor transport technique. In this technique, a seed crystal of SiC is introduced into the Lely chamber, and growth proceeds (usually along the **c**-axis) by vapor transport of carbon and silicon 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 to 35 °C/cm. Currently the maximum commercially available 4H and 6H-SiC crystals are 2” in diameter, however record 4" boules of SiC have been recently demonstrated. Sublimation growth is usually performed on {0001} (Si or C face) of either 6H-SiC or 4H-

SiC seeds. Growth on faces perpendicular to the (0001) basal plane has also been reported (Takahash, et al. 1994) in an effort to reduce micropipe defects.

Vapor phase equilibria in SiC

The equilibrium gas species over SiC have been measured by Drowart et. al. (1958; 1960) and Behrens and Rinehard (1979). The principal molecular species are Si, SiC_2, and Si_2C. In the Si-C vapor system, carbon is transported by the Si bearing compounds. The vapor pressure of all the major species are related to the pressure of Si. In thermal equilibrium, the vapor equilibria over SiC has only one degree of freedom. If any of the species are specified by the temperature, source powder composition and/or crucible wall interactions the remaining species are uniquely determined. Because Si bearing species are the most prevalent, it is usually convenient to discuss the vapor composition in terms of Si partial vapor pressure. Using the experimental data together with thermodynamic calculations, the relationship between the partial pressure of Si and that of Si_2C and SiC_2 for the SiC-C vapor equilibria is given by Eqns 1 and 2 (Glass, et al. 1997):

$$P_{Si_2C} = 2.85 x 10^{2} e^{(-1.79 x 10^{4}/T)} x P_{Si} \quad (1)$$

$$P_{SiC_2} = 9.41 x 10^{28} e^{(-14.35 x 10^{4}/T)} \frac{1}{P_{Si}} \quad (2)$$

The partial pressure of the vapor species varies dramatically between the SiC- C equilibrium and the SiC-Si equilibrium. However, due to the large amount of carbon present in the growth chamber of most systems, the vapor interactions with the graphite walls of the crucible usually will cause the system to operate close to the SiC-C part of the curve. Since the composition of the vapor has such a large allowed existence region, the composition of the powder has important consequences for the vapor composition and the growth rate. Source graphitization is also an important problem for bulk growth. Graphitization occurs under growth conditions when it is possible to preferentially lose Si and obtain a carbon layer over the source which prevents further sublimation of the source. This has been treated in detail by Karpov et. al. (1995).

Growth Rate

Typical growth rates for the bulk growth of SiC are in the range of 0.5-5 mm/hr.

Structural defects

There has been significant progress in the quality of material produced by seeded sublimation technology (Hobgood, et al. 1993; Tsvetkov, et al. 1995). Dislocation density and micropipe density in SiC bulk crystals grown by the modified Lely method currently range from 10^3 to 10^4 cm^{-2}. Other types of crystal defects found in sublimation grown SiC crystals include basal plane tubes, cracks, and crystal domains (Tuominen, et al. 1994). However, micropipes are defects unique to the growth of SiC. These micropipes which are physical holes can travel large distances in the crystal. It was shown that micropipes are "killer" defects if they intersect the active regions of a device (Neudeck & Powell 1994). The density of the micropipes could be correlated with domains in the crystals (Glass, et al. 1997). These domains are thought to be related to growth spirals which are nucleated and interact. The relationship between micropores and growth spirals was originally suggested by Frank (1951), and later papers on the subject by Krishna et. al. (1985) have expanded on this theme. More recently, x-ray studies by Fazi et. al. (1993) also has suggested this mechanism. There are, however, other views as to the origin of the micropipe defects. Some have observed that these defects are nucleated at the seed. Other investigators suggest that impurities, inclusions (or Si droplets) are responsible for micropipe formation. Support of alternate views of micropipe formation comes

from the experimental observation that many of these defects are nucleated in the bulk of the materials and are not continuous. It is generally conceded that micropipes are the most important current obstacle to the production of high quality SiC devices. The possible mechanisms of micropipe formation are summarized by Tsvetkov et. al. (1995).

Recently, x-ray and TEM techniques have been applied to the study of the quality of bulk SiC (Barrett, et al. 1993). Lely platelets as well as substrates grown by the modified Lely method have been investigated. In general, the results of various investigations suggest that Lely crystals exhibit higher material quality. The full width at a half maximum (FWHM) of the x-ray rocking curve is ~7-10 arc sec for Lely platelets, while for modified Lely crystals this value ranges from 20-100 arc sec. For Lely crystals, a single x-ray diffraction peak is observed for each x-ray reflection. Conversely for modified Lely crystals several peaks are observed indicating a mosaicity in the material.

Electrical characteristics

Undoped SiC bulk material usually is contaminated by nitrogen which produces n-type conductivity. The typical background level of electron concentration at room temperature for undoped SiC crystals grown by modified Lely method is 10^{16} -10^{17} cm^{-3}. High purity undoped SiC crystals with room temperature resistivity from 10^2 to 10^3 cm have been reported by Hobgood et. al. (1993). These crystals had p-type conductivity with a background carrier concentration of 10^{15} cm^{-3} due to residual boron impurities.

N-type SiC crystals with carrier concentrations up to 10^{20} cm^{-3} were produced using nitrogen doping (Onoue, et al. 1995). The minimum reported resistivity for 6H-SiC and 4H-SiC bulk crystals are 1.6 m cm and 2.8 m cm, respectively (Glass, et al. 1997). No information on defect density in highly doped n-type material is available. P-type SiC crystals with carrier concentrations up to 10^{20} cm^{-3} were obtained using aluminum doping (Glass, et al. 1997). Information about Al doping during bulk SiC growth is limited. No information on defect density in highly doped p-type material is available.

Semi-insulating 6H-SiC crystals were produced using vanadium doping (Hobgood, et al. 1995). The amount of vanadium soluble in the material is limited by the precipitation of vanadium silicide. The material resistivity at room temperature can be estimated by high temperature resistivity measurements and was determined to be in the range of 10^{15} cm. Semi-insulating 4H-SiC crystals with comparable resistivities have also been reported (Tsvetkov, et al. 1995; Glass, et al. 1997).

Bulk growth by chemical vapor deposition

6H-SiC crystals have been grown by high temperature CVD with growth rates of 0.5 mm/hr (Kordina, et al. 1996). This technique is a direct adaptation of the CVD epitaxial technique for growth of SiC. The control of the Si/C ratio in such a system is excellent compared with sublimation growth and additional results have been presented in the recent SiC conference.

Bulk Growth from the Liquid Phase

Crystal growth from melt-solutions is widely used for many semiconductor materials. Liquid phase growth of SiC has not been considered promising based on results obtained during the early stage of SiC development. Two main objections to liquid phase technology are low solubility of SiC in the Si melt (which limits the growth rate), and inclusions incorporated in grown crystals due to parasitic phase formation. On the other hand, it has been shown that high quality SiC can be grown from melt-solutions (Nelson. Et al. 1966; Marshall 1969; Wolff, et al. 1969): undoped cubic SiC crystals grown from a silicon melt at ~1500°C exhibited a mobility of 980 cm^2/V s and carrier concentration of $4x10^{16}$ cm^{-3} (300 K). Significant progress has also been made in understanding the nature of SiC growth from liquids (Dmitriev 1995; Yakimova, et al. 1995). It has been shown that high quality SiC epitaxial layers with no micropipes and low dislocation density can be grown from a Si melt (Rendakova, et al. 1997). Epitaxial layers of 6H- and 4H-SiC were grown from Si melts on 35 mm diameter substrates indicating the possibility of liquid phase growth of large

crystals. It was also found that the solubility of SiC in Si melt does not severely limit the growth rate, and a SiC growth rate of about ~0.2 mm/hr has been obtained at 1650°C.

SIC EPITAXIAL GROWTH

In order to improve the quality of bulk material and produce complicated device structures, epitaxial techniques are necessary. As with other semiconductor material systems, liquid phase epitaxial techniques and CVD were used early in the development of SiC to produce device structures. Although the material produced by LPE was of high quality, difficulties with molten Si (used as the melt) prompted the development of vapor phase techniques such as sublimation epitaxy and CVD. Chemical vapor deposition is presently the most widely used epitaxial technique for growth of SiC device structures.

Chemical Vapor Deposition

Chemical vapor deposition is a growth process in which gaseous compounds are transported to the substrate surface where chemical reactions occur resulting in formation and growth of the desired material. The growth of 6H-SiC layers on 6H-SiC {0001} substrates by CVD in the temperature range from 1500 to 1850°C have been reported since the sixties (Campbell & Chu 1966; Jennings, et al. 1966; Minagawa & Gatos 1971; Wessels, et al. 1974; Matsunami, et al. 1975; Muench, et al. 1975; Muench & Pfaffeneder 1976; Nishino, et al. 1978). A significant lowering of the growth temperature and improvement of material quality has been achieved by using substrates which are mis-oriented a few degrees off the {0001} plane toward the $<11\bar{2}0>$ direction. This growth on mis-oriented substrates has been termed "step controlled epitaxy" and has the added advantage of stabilizing the polytype structure.

A review of CVD growth of SiC has been published by Nishino (1995) and Larkin (1997). Growth temperatures for typical SiC CVD processes range from 1200-1800°C while growth pressures vary from 100-760 torr.

Growth Equipment

Three types of growth apparatus are used for SiC CVD: 1) cold-wall horizontal atmospheric pressure reactors have been used for many years with results reported by Siemens AG (Karmann, et al. 1993), Kyoto University (Matsunami 1992), NASA Lewis Research Center (Mandel 1962). A commercial horizontal water-cooled reactor for SiC CVD operating in the pressure range from 10 to 1000 mbar was developed by AIXTRON GmbH (Sculte, et al. 1994). This system uses low pressure and a specially designed inner sleeve to maintain laminar gas flow. 2) hot-wall horizontal atmospheric pressure reactors have been designed at Linkoping University (Kordina, et al. 1993). Still another hot wall horizontal reactor design was proposed by the Industrial Microelectronic Center in Sweden (Nordell, et al. 1995). 3) cold-wall vertical low pressure reactors (Feng, et al. 1996) have been built commercially by EMCORE Corp. and used at Howard University and Siemens AG (Rupp, et al. 1995). In these reactors, laminar flow is obtained by a high speed rotating disk (in conjunction with high gas flow velocities) which produces a pumping action.

High temperatures in SiC epitaxial reactors can be obtained with either resistive or rf heating. In most systems the susceptor is made from graphite. Because of the reaction between graphite and H_2 at temperatures in excess of 1300°C a thin SiC coating layer has been used by many researchers. Unintentional incorporation of contaminants from the susceptor during SiC CVD was studied by Karmann and coworkers (Karmann, et al. 1995). In these experiments, 6H-SiC layers were grown in an atmospheric pressure reactor at 1390°C with a growth rate of 0.7 m/hr. Uncoated and SiC coated (100 - 120 m thick) graphite susceptors were used for comparison. For the uncoated susceptors, the layers were found to be contaminated with aluminum, boron and nitrogen. Conversely, using a SiC coated graphite susceptor in the same system, SiC layers with concentration N_d - N_a of $4x10^{15}$ cm^{-3} could be grown. In a low pressure vertical reactor with high speed substrate rotation, SiC with background concentrations in 10^{14} cm^{-3} range was demonstrated without use of SiC-coated parts (Rupp, et al. 1995). This low amount of contamination is attributed to the favorable gas flow patterns generated in this reactor

Temperature measurement is a major equipment issue for CVD growth at high temperatures. During growth, substrate temperature is usually measured by an optical pyrometer calibrated by melting of Si or Ge. The temperature of the susceptor typically is found to be 50 - 100°C higher than that of the SiC substrate. Power settings are often used to calibrate the substrate temperature.

Precursors and Reaction Chemistry

A number of precursors have been used for the growth of silicon carbide. For transport of the Si species the most popular choice is SiH_4 (Powell, et al. 1987), but Si_2H_6 (Nishino & Saraie 1989a) and $SiCl_4$ (Muench & Pfaffeneder 1976) have also been used. For growth of Si, the hydrocarbon species most reported is C_3H_8. However, there are also reports of SiC growth using C_2H_2 (Liaw & Davis 1985), CH_3Cl (Ikoma, et al. 1991), CH_4 (Rai-Choudhury & Formigoni 1969), CCl_4, C_7H_8, or C_6H_{14} as carbon sources. In addition to the use of individual gas species, single precursors have also been investigated, among them CH_3SiCl_3 (Nishino & Sarate 1989b) and $(CH_3)_2SiCl_2$ (Rai-Choudhury & Formigoni 1969).

Gas-phase equilibrium calculations were reported for Si-H-C gas mixtures (Allendorf 1993) in the temperature range used in CVD growth of SiC. Modeling of the gas phase chemical processes which occur during growth as a function of distance from the heated susceptor was reported by Stinespring and Wormhoudt (Stinespring & Wormhoudt 1988). The results of the modeling study shows that the injected C species are not fully decomposed to equilibrium values at the growth interface. Gas-phase reactions in SiC CVD growth using the SiH_4-C_3H_8-H_2 gas system were experimentally investigated by Hong et. al. (1993) using a microcavity technique. The microcavity study suggested 1) that multiple species contribute to the film growth in the system, and 2) possible precursors in the chamber contain SiH_2 (a gas-phase intermediate derived from SiH_4), and another species containing Si and C derived from SiH_2 and C_3H_8. Under certain experimental conditions Si droplets and graphite inclusions have been observed in SiC epitaxy. Modeling of these Si-droplets and graphite inclusions formation in the CVD process was done (Karpov, et al. 1995) considering several homogeneous and heterogeneous chemical reactions.

Growth Rate

Increasing the growth rate in SiC epitaxy is important because of the demands of SiC power device structures. A typical base layer in a power thyristor can be as thick as 100 m. Since growth rates of SiC films vary from 0.1 to 6 m/hr for a growth temperature of 1500°C, the minimum growth time for a single structure can be as long as 15 hours. Growth under normal conditions is determined by the diffusive transport of the Si species through the stagnant layer, although control of the growth by carbon species has been observed. Since the growth rate of 6 mm/hr is too low for layers greater than 10-12 m there are several studies in progress on increasing the CVD growth rate. Growth rates of 500 m/hr were obtained by high temperature CVD (1800 to 2300°C) (Kordina, et al. 1996) with a maximum crystal thickness of 2 mm. This result looks very promising for growth of thick layers (>100 m) for high power SiC devices, but many questions about this technique, including possible material contamination at high temperature, remain unanswered.

The temperature dependence of SiC CVD growth rate has been investigated by several researchers. If the growth rate is determined by surface kinetics, the activation energy of the growth is expected to be relatively large. If on the other hand the growth is transport limited, the observed activation energy is expected to be small. For the $SiCl_4$-CCl_4-He gas system, an activation energy of 20 kcal/mol was reported for growth on well-oriented substrates (Jennings, et al. 1966). An activation energy of 22 kcal/mol was obtained for SiC growth on the $(000\bar{1})$C face of well-oriented substrates for the C_3H_8-SiH_4-H_2 gas mixture (Wessels, et al. 1974), however the same investigators observed a complicated temperature dependence on well-oriented (0001)Si faces. The complicated temperature dependence of growth rate on the Si face was explained using a growth model in which the growth rate is limited by the adsorption-desorption of reactants at the growth interface. For step-controlled epitaxy an activation energy of SiC growth rate was measured to be 3.0 kcal/mol (Kimoto, et al. 1993). The layers were grown at atmospheric pressure in the temperature range

from 1200 to 1500°C on 6H-SiC {0001} substrates (with 6° off-orientation) using SiH_4-C_3H_8-H_2 precursors. The small value of activation energy is attributed to the fact that the growth in step-controlled epitaxy is mass transport limited and the temperature dependence is due diffusion through the stagnant layer.

Homoepitaxy of -SiC

Homoepitaxial growth of -SiC (6H-SiC, 4H-SiC, 21R-SiC) by CVD has been advanced at Kyoto University using off-oriented SiC substrates (Itoh, et al. 1994; Hong, et al. 1995). 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 mis-orientation and growth temperature determine if growth will occur via the step controlled mechanism. If the growth is step controlled, the epilayer will replicate the stacking order of the substrate. The growth mechanism for SiC homoepitaxial CVD has been discussed by a number of researchers (Kimoto, et al. 1993; Kimoto & Matsunami 1993; Hong, et al. 1995). Nucleation processes during SiC CVD growth were investigated by Kimoto and Matsunami (1993) and the surface kinetics of adatoms in CVD growth of SiC were analyzed based on Burton-Cabrera-Frank theory (Kimoto & Matsunami 1994).

Typical growth of epitaxial 6H-SiC is performed on wafers which are mis-orientated 3.5° toward the <11$\bar{2}$0> direction. The growth temperatures are about 1500°C. 6H-SiC is usually grown with large amounts of H2 in the chamber, gas ratios of 1:1000 (SiH4+C3H8):H2 are usual. 4H-SiC layers are typically grown (the growth is performed at temperatures somewhat higher than those used for 6H growth) on 5-8° off-oriented 4H-SiC substrates using the same gas system. The (0001)Si face 4H substrates are misoriented toward the <11$\bar{2}$0> direction. A typical growth rate is about 2.5 m/hr.

At high growth temperatures (1500°C or greater) used for 6H-SiC and 4H-SiC deposition, a wide range of Si/C ratios have been used (Rupp, et al. 1995b). The Si/C ratio affects not only the growth rate and crystal quality but also the dopant incorporation. A major problem in the epitazial growth of SiC is the unintentional incorporation of C in the gas phase which comes from various parts of the reactor. This unintentional C is transported to the substrate via reaction with H2 in the gas stream and the subsequent formation of hydrocarbons makes it difficult to maintain a proper Si/C ratio.

Details of several epitaxial growth processes have been published. A growth process employing the SiH4-C2H4-H2 gas system was described by researchers from North Carolina State University (Wang & Davis 1991). In this process, SiC substrates were initially heated to the growth temperature (1350-1600°C) for 10 minutes in H2 flow to clean the surface. In the growth procedure developed at NASA Lewis Research Center (Powell, et al. 1995) for 6H-SiC and 4H-SiC CVD, the samples are initially etched by HCl at 1350°C prior to the growth. The initial HCl purge reduces the density of surface defects in resulting SiC layers. The importance of pre-growth treatment of the substrates was emphasized in this study. A high resolution x-ray diffraction study on 6H-SiC layers grown by CVD (Bakin, et al. 1995) also demonstrated the importance of pre-growth treatment. The layers in the study were grown in the temperature range from 1500 to 1600°C with the growth rate of 2 to 2.5 m/hr using a C/Si ratio of 2.5 to 3. Prior to the growth, the substrates were etched in H2 gas flow at 1500-1600°C for 10 - 30 min. A 9 arcsec full width at the half maximum x-ray rocking curve was obtained for layers grown on substrates having a FWHM of 72 arcsec. Without H2 etching, the FWHM for the layer was often observed to be larger than the FWHM of the substrate.

All the CVD processes discussed up to this point were developed for SiC deposition on polar {0001} (C or Si face) substrates. Epitaxial growth of SiC has also been investigated on the two nonpolar crystal planes, namely, the (10$\bar{1}$0) and (1$\bar{2}$10) planes (Burk, et al. 1993).

Structural Properties

The surface of SiC epitaxial layers can contain a large number 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 one micron.

Attempts to understand the nature of surface defects in SiC CVD layers have appeared in several recent studies (Burk, et al. 1996; Powell, et al. 1995; Powell, et al. 1995b; Powell, et al. 1996; Kimoto, et al. 1993d; Rupp, et al. 1995; Rupp, et al. 1995b). A large number of factors influence the production and density of surface defects. These include substrate characteristics (orientation, face polarity, tilt angle, 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 Si/C ratio, growth rate, and growth termination procedure. The best reported results thus achieved indicate surface defect densities of 10^3 cm^{-2}. It is noteworthy that this value corresponds to the density of unknown defects in SiC pn structures which appear to cause pre-mature junction breakdown (Chelnokov, et al. 1996). Investigation of surface defects in 6H-SiC and 4H-SiC layers have shown that the morphological defect density varies widely from run to run.

Growth pits are a common morphological defect observed in the growth of SiC. The relationship between growth pits on the epitaxial layer and surface imperfections in the starting substrates has been studied by Powell et. al. (Powell, et al. 1995). It was concluded that the main factor in the formation of growth pits is the polishing and preparation of the substrate rather than bulk defects such as micropipes and dislocations. This conclusion has lead to improved substrate polishing techniques such as the use of colloidal silica for chemo-mechanical polishing (Powell, et al. 1997).

Step bunching (which is the combination of atomic steps on the surface to form large macro-steps) often occurs in the CVD growth of SiC. Results on the initial investigations of step bunching on 6H-SiC epitaxy (which enhances terrace nucleation) was discussed by Kong et. al. (1988). Step bunching in 6H- and 4H-SiC growth by step-controlled CVD on mis-oriented {0001} substrates was investigated by AFM and TEM techniques (Kimoto, et al. 1993d). In this study, the SiC {0001} substrates were mis-oriented 3-5° toward the <$11\bar{2}0$> irection. It was observed that epitaxial growth on (0001)Si face yielded macrosteps with an average terrace width of 280 nm and an average step height of 3 nm. On the ($000\bar{1}$)C face, the surface was relatively flat and no microsteps were observed. On the (0001)Si faces, 3 bilayer-height steps were the most dominant type of step seen using 6H-SiC samples while 4 bilayer-height steps predominated on 4H-SiC samples. Step bunching as a function of tilt angle (0.1-3.5°) was studied by Powell et. al. (1995b) with a conclusion that step bunching in epitaxial growth can actually be reduced at higher substrate mis-orientations. A TEM study as well as a qualitative model of the step bunching phenomena was reported by Chein et. al. (1994). In their model, Chein and co-workers show that variations in the surface energies of the different steps which comprise the 6H unit cell are responsible for the different lateral growth velocities and consequently the step bunching.

Typical 4H-SiC and 6H-SiC epitaxial layers also contain dislocations and micropipes. In all cases in which micropipes were observed at an layer/substrate interface, the micropipes originated in the substrate and propagated into the epitaxial layer (Powell, et al. 1993). This result implies that if micropipes in the substrate are eliminated or closed (such as by liquid phase techniques), the epitaxial films will be micropipe free.

Polytype inclusions are a common type of crystalline defect in 6H and 4H-SiC epitaxial layers grown by CVD These polytype inclusions (usually 3C-SiC) are formed due to nucleation on terraces or dislocation sites. It was found that with proper pre-growth surface treatment with HCl etching, 6H-SiC layers without 3C-SiC inclusions can be grown by CVD on {0001} 6H-SiC substrates with small tilt angles (0.1°-0.6°) (Powell, et al. 1991). Conversely if a pre-growth HCl etch at 1375°C for 20 minutes was used, predominantly 6H-SiC growth was obtained. The etching process appears to be effective in removing unintentional 3C nucleation sites on 6H-SiC wafers. However, deviation from the optimal etching conditions lead to 3C-SiC growth on the 6H-SiC substrates. It was also found that 4H-SiC homoepitaxial layers are more susceptible than 6H layers to 3C inclusions (Powell, et al. 1995). The mechanism of cubic SiC nucleation on off-axis 6H and 4H substrates has been investigated by Hallin et. al. (1995). These investigators showed that the 3C-SiC nucleation occurs via the formation of triangular stacking faults at

substrate imperfections. In 6H-SiC, these defects are usually found in on-axis material where the probability for 2-dimensional nucleation of 3C-SiC is increased. In 4H-SiC epitaxial layers, 3C-SiC inclusions having a triangle shape are found even if substrates have a tilt angle of 3.5°. In order to reduce the density of inclusions in 4H material, a 8° tilt angle was found to be necessary. In the 4H-SiC epitaxial layers grown on 8° off-substrates, the 3C-SiC inclusions are almost eliminated.

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

Electrical Properties

Significant progress has been achieved in producing epitaxial layers of 6H and 4H-SiC with superior electrical properties. 6H-SiC epitaxial layers having a low background doping concentration were grown and characterized by Kordina et. al. (1994). It was shown that using propane as a carbon precursor uncompensated SiC layers with donor concentrations less than 10^{15} cm^{-3} may be grown, whereas with methane uncompensated layers can be produced with electron concentrations in the mid 10^{14} cm^{-3} range (however with a slightly worse morphology). The optimal growth temperatures for these films was found to vary between 1550 and 1600°C. At higher temperature, contamination from graphite parts became noticeable and bake-out of the growth system had a significant impact on the background doping. The effect of unintentional hydrogen doping by CVD was studied by Clemen et. al. (1993).

4H-SiC layers with electron concentrations as low as $2x10^{14}$ cm^{-3} were reported (Kordina, et al. 1994; Itoh, et al. 1993; Kimoto, et al. 1995) (to obtain the low impurity concentration, the growth system was pumped for several hours prior to growth according to some of the authors). Electrical and optical measurements on high quality 4H-SiC layers were reported by Kimoto and co-authors (1995). The background doping concentration in the layers was determined to be $3x10^{15}$ - $2x10^{16}$ cm^{-3} and electron mobility in the {0001} basal plane was 600 - 720 cm^2/V s (300 K). Deep level transient spectroscopy (DLTS) measurements on these films showed that the concentration of electron traps was approximately 10^{13} cm^{-3} independent of substrate polarity. Minority carrier lifetimes have been measured on 6H-SiC layers with N_d - N_a ranging from 10^{14} to 10^{17} cm^{-3}. Lifetimes as high as 0.45 s (300 K) have been achieved for thick low doped samples (Kordina, et al. 1995a). However the maximum reported values of minority carrier diffusion length for CVD grown SiC pn structures do not exceed 3 m.

Doping of SiC homoepitaxial layers grown by CVD has been reported in numerous publications. Nitrogen is commonly used as a donor and aluminum is the acceptor of choice. Nitrogen doping has been investigated for several years as a n-type dopant (see for example Rupp, et al. 1995b; Karmann, et al. 1992). Nitrogen doping in these studies produced donor concentrations ranging from 10^{16} to 10^{19} cm^{-3}.

P-type doping has been achieved by using Al as a dopant (see for example Nordell, et al. 1995). Epitaxial layers in this study were grown using SiH_4-C_3H8-H_2-TMA (Trimethylaluminium) precursors at a C/Si ratio of 2.5. The reactor pressure was 800 mbar and growth temperature was 1550°C. A growth rate was about 2 m/hr. The atomic Al concentration in 6H -SiC was controlled from 10^{17} to 10^{21} cm^{-3}. When the Al concentration exceeded $2x10^{20}$ cm^{-3}, impurity banding occurred and the Al acceptors were completely ionized while 1% ionization was observed at lower doping levels (in keeping with the measured Al ionization energy of ~0.25 eV). Schoner and coworkers (1995) found that the Al ionization energy varied with doping concentration as well as the degree of compensation. As expected, at high doping levels crystal quality was degraded.

A greater range of doping control is possible with site-competition epitaxy (Larkin, et al. 1993, 1994; Larkin 1995). 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 nitrogen, phosphorus, aluminum and boron incorporation in 6H-SiC and 4H-SiC films. The layers were grown using

SiH_4-C_3H_8-H_2 gas precursors at 1450°C with a typical growth rate of 3-4 m/hr. Secondary ion mass spectroscopy (SIMS) determined that the Al incorporation increased as the propane flow was increased and also when the silane flow was decreased. It was found that results of site-competition doping control is similar for 6H-SiC and 4H-SiC epitaxial layers grown on (0001)Si faces of the substrates. The effect of surface polarity and the chemistry of the particular impurity on site-competition doping control are discussed by Larkin (1995). Deep-level impurities in SiC pn structures grown by site-competition epitaxy have also been investigated also by Saddow et. al. (1995).

Impurity memory effects on dopant concentration profiles in 4H and 6H-SiC were investigated by SIMS (Nordell, et al. 1997). It was found that dopants were absorbed by the reactor walls and re-evaporated after the dopant precursor flow was switched off. These memory effects limit the doping control range to about three orders of magnitude for aluminum, and two orders of magnitude for boron. The dynamic range for Al doping was increased up to five orders of magnitude by controlling the Si/C ratio and using HCl etching during the 10 min growth interruption after gas switching. For boron, a dynamic range of more than three orders of magnitude was obtained. Doping spikes at the substrate/layer interface were also reduced by an in situ HCl etch (Burk, et al. 1996).

Liquid Phase Epitaxy

SiC LPE growth takes place from a supersaturated solution of Si and C in a melt solvent. The main feature of LPE is that the growing films are in equilibrium with the liquid phase. The endpoints of the process are determined by the phase diagrams for Si, C and the solvent material. Liquid phase epitaxy was used early in the development of SiC technology. Growth from Si melt as well as alloy melts were demonstrated. LPE has been performed in graphite boats, by vertical dipping and by a novel levitation process called container-free epitaxy. Doping of SiC was accomplished in LPE over a large range of concentrations. Due to the difficulty in control of surface morphology, LPE techniques have lost ground in favor of CVD approaches. However, recently discovered unique properties of LPE such as micropipe closing (Yakimova, et al. 1995; Rendakova, et al. 1997) and the ability to produce very heavily doped p-type films (>$5x10^{20}$ cm^{-3}) (Rendakova, et al. 1997) may secure this technology a future role. The usual melt for SiC LPE is silicon, but alternative materials like Sn, Ge, Ga, and their mixtures are also used for SiC LPE (see Dmitriev 1995 for a review).

Sublimation Epitaxy

The mechanism and principals of SiC sublimation epitaxy are similar to those for bulk SiC sublimation growth discussed at the beginning of this chapter. However, sublimation epitaxy is usually performed at lower temperatures with smaller growth rates and for shorter time periods than bulk SiC sublimation growth. Growth of epitaxial SiC using the sublimation process had been the subject of many early investigations (for early work, the reader is refereed to the Proceedings of 1st, 2nd, and 3rd International Conferences on Silicon Carbide0. The breakthrough in sublimation epitaxial technology was achieved with the development of "sublimation sandwich method" by Vodakov and Mokhov (1970). They employed a nearly flat source positioned close to the substrate and performed the growth under near-equilibrium conditions. This method allowed for the vapor equilibrium to be constant over the substrate. The "sublimation sandwich method" made it possible to grow high quality SiC layers in the temperature range 1600 - 2100°C (Vodakov, et al. 1979; Mokhov, et al. 1992; Tairov, et al. 1976; Anikin, et al. 1992). An excellent review on SiC sublimation epitaxy was written by Konstantinov (1996).

ION IMPLANTATION IN SIC

Ion implantation currently is the only alternative to doping during growth and is widely used in SiC device fabrication technology. For a review of ion implantation in silicon carbide see Davis, et al. 1991; Ivanov & Chelnokov 1992; and Wongchotigul 1995. Ion implantation has been used for: 1) pn junction formation (Anikin, et al. 1984; Ramungul, et al. 1995; Shenoy & Baliga 1995; Xie, et al. 1995; Wang, et al. 1995; Palmour, et al. 1995), 2) light emitting diode fabrication (Gusev, et al. 1983), 3) highly doped contact layers

(Spieb, et al. 1995; Slater, et al. 1995), (4) field effect transistors channels (Alok & Baliga 1995; Slater, et al. 1995) and (5) device isolation and termination (Nadella & Capono 1997).

Since the sixties, many elements have been implanted into silicon carbide (Al, B, Ga, In, Tl, N, P, Sb, Be, Bi, Kr, Ar, Er, Si, C) (Leith, et al. 1967; Dunlap & Marsh 1969) although, the most commonly used are Al, B, and N. Ion implanted dopant activation is achieved by thermal annealing using resistively or rf heated furnaces (Gardner, et al. 1997). Excimer laser activation has also been reported (Ahmed, et al. 1995). Simulation of implantation profiles in SiC and their comparison with experimental results has been performed (Ahmed, et al. 1995; Pan, et al. 1997).

N-type doping by ion implantation has been developed into a production process. Annealing of nitrogen implantation has in general resulted in low residual damage due to the small size of the nitrogen atom. Nitrogen ion implantation is usually performed in 6H-SiC at elevated temperatures. Annealing at 1500 and 1600°C for 15 min performed in SiC crucibles results in Reserford back scattering yields at the virgin crystal level indicating a good recovery of the crystalline quality. Recently, activation processes of high-dose (3.8×10^{15} and 7.1×10^{15} cm^{-2}) nitrogen implants into 6H-SiC has been investigated (Pan, et al. 1997). It was shown that low resistivity of the implanted material can be obtained after long time (2000 min) anneals at 900°C.

One of the current problems in ion implantation technology is activation of the p-type dopants. Because Al is a large atom, higher annealing temperatures and times are required to produce device quality p-type layers. Amorphization and re-crystallization of Al-implanted 6H-SiC were investigated (Pan, et al. 1997). Ion implantation was performed in n-type 6H-SiC in the temperature range from room temperature to 1000°C. Al ions were implanted with a dose ranged from 5×10^{13} to 5×10^{16} cm^{-2} and implant energies of 180 and 360 keV into 6H-SiC epitaxial layers having doping concentrations of 1×10^{16} cm^{-3}. After implantation, the samples were annealed between 800 and 1600°C for 30 min in an Ar flow. It was shown that density of defects induced by implantation decreased exponentially with implantation temperature. However residual defects were detected even after annealing at 1600°C. At 1600°C anneal temperature, sublimation of SiC was observed. Carrier concentration and mobility were found to be independent of the implantation temperature and the carrier concentrations at room temperature was measured to be about 5% of implanted dopant. The measured hole mobility was less than 1 cm^2/Vs (300 K). Annealing p-type implants at lower temperatures has proven ineffective. For example, annealing at 1400°C resulted in hole concentration of only 5×10^{17} cm^{-3} (Rao, et al. 1995). Also C or Si co-implantation also did not improve Al activation efficiency (Rao, et al. 1995). Experimental data on ion implantation in other than 6H polytype are limited.

RESEARCH ON SIC GROWTH AND PROCESSING IN EUROPE

In this section the results of the TTEC panel trip to Europe will be discussed. In this chapter the efforts on SiC growth and processing will be highlighted

Wide Band Gap Efforts in France

The SiC efforts in France are centered in several universities, Laboratoire d'Electronique de Technologie et d'Instrumentation (LETI), and Thompson CSF. Leti is perhaps the largest French research institute with over 950 people. LETI consists of five departments and several programs. LETI is particularly known for it's contribution to wafer bonding technology. At LETI the Smart Cut ® process was described. It was pointed out that the quality of Si bonded using the Smart Cut ® process is similar to that of the starting material. This technology is applicable not only to Si but also to GaAs-based materials and SiC. The current status of SiC growth research in France is that crystal growth is in the research stage and development has just started for wafers. SiC epitaxy activity is primarily in LETI and in approximately 10 universities.

The funding sources for the effort include: CNRS, industry, and defense programs. SiC projects are divided into national programs and European programs. The two national programs in SiC include "Saut Technologique", which is a three-year program (funded by the government and coordinated by industry, with

industrial partners contributing some funding as well). The European SiC program has a goal to achieve 3-inch wafers with no micropipes in three and to achieve a low defect density on two-inch wafers (less than 10 micropipes per cm^2). Under the auspices of this project, the French teamed with a Swedish-Finnish company, Okmetic. More detailed goals include cheaper and bigger crystals, a lower defect density, and the development of SiC fabrication process compatible with a silicon line. The bulk SiC material grown at LETI was described. The boule diameter was 50 mm. However, the material quality was inferior at the edges. The effort at LETI also includes the simulation of crystal growth. Results of ion implantation investigations were also discussed. The best results achieved at LETI include 95 mohm cm, $p = 10^{19}$ cm^{-3}, 14% activation for 10^{21} cm^{-3} of Al acceptors.

Wide Bandgap Efforts in Germany

The SiC research efforts in Germany is dominated by the work at Erlangen University and Siemens Corporation. Both institutions have a long history of contribution to SiC technology. In Siemens the main effort is at Siemens Erlangen, although some work is also being done in the coporate research laboratory. Two spinoff institutions were discussed. The first, Infineon Technologies, will be responsible for semiconductor components. Not much information was available on the second institution, Frielectronics, but there was speculation that this organization could do bulk crystal growth. In Seimens Erlangan the focus of the work is on power switching electronics. Over the last few years investigators in Erlangan have made significant contributions to the understanding of epitaxial growth technology (see the technology discussion above). In the last few years they have advanced their understanding of the growth and fabrication processes. The investigators in Seimens have reported detailed studies of the reliability and failure mechanisms of their high voltage diodes. Their understanding of these mechanisms has put them in the position to go into high volume production of SiC diodes. This would be the first high volume production of a SiC product (discounting the early effort on SiC LED's). In addition to the work on optimization of their 6H and 4H processes, they have demonstrated the advantage of using 15R polytype for MOSFET, instead of 4H polytype. The reason for this is that there are apparently few localized trap states near the conduction band edge in 15 R-SiC and, consequently, a much higher inversion electron mobility can be achieved than in 4 H-SiC (33 vs. 0.4 cm^2/V-s). Besides, while 6 H-SiC also has similar inversion electron mobility values, the anistropy in bulk mobilities is much less in 15 R-SiC than in 6H-SiC. The Siemens researchers are working with Erlangan University to characterize the 15R polytype.

The University of Erlangan has two separate departments with active SiC programs. The first is the Department of Material Science and Engineering. This department forms part of the Technische Fakultat (Faculty of Engineering Sciences). Conversely the second group visited was a part of the traditional Physics Department of the University. In the Department of Materials Science most of the work in wide bandgap technology is done in the Electronic Engineering Materials group. The TTEC group received a detailed presentation on the research for the bulk growth of SiC. Most of this effort is supported by a research grant from Bavaria. This research grant ~ $1 million/year has also supported SiCrystal (a startup wafer company) in their effort to commercialize bulk crystal growth in Germany. SiCrystal was present and gave a brief overview of the progress and direction of the company. In the Materials Science Deptment there are efforts to understand the basic thermodynamics of bulk crystal growth and defect formation. Of special interest, micropipes and their formation and research results on formation mechanisms were presented. These topics are investigated experimentally in a sublimation reactor, which is currently growing 1.5" SiC boules. Theoretical modeling of heat transfer is closely linked to this effort. Another technique for the production of bulk SiC is Liquid Phase Growth. The group in Erlangen and another group in Russia are unique in their efforts to investigate the possibility of large area growth of SiC by this technique. The Erlangen group is producing SiC at 2100°C and 150 torr of pressure. Under these conditions the growth rate is .6 mm/hr. The potential advantages of this technique include improved mass transfer and easier scaling.

SiCrystal was formed in August 1996. They are currently producing SiC principally for use in LED technology. This material is 2" in diameter. The company estimates that 90% of their market is based on nitride technology. They expect this number to decrease to 50% when manufacture of SiC based devices commences. When the demand for SiC material increases they will start production of 4" SiC.

The other SiC research community is in the Department of Physics. In the Department of Physics there is significant activity on crystal growth and characterization. The specific topics were:

- Bulk Crystal Growth-zero micropipes (6 H), 15 R polytype (in collaboration with Kyoto University)
- Ion Implantation –intrinsic defects, effects of non stociemetry, diffusion (Boron)
- Reconstruction of SiC surface
- MOS interface state density studies-admittance and constant capacitance DLTS

Most of the work is funded by the German Science Foundation. There is however no national program on SiC. The group does participate in a European program, which funds some of the ion implantation work.

Wide Bandgap Research in Sweden

In Sweden the SiC research is conducted in four main locations: Linkoping University, IMC, ABB and the Royal Institute. In Linkoping the TTEC panel visited the Department of Physics and Measurement Technology. The SiC activities in Sweden are funded from several sources:

SiCep

This is a national program on SiC, which provides a total of 18 million SEK of funding for SiC per year. Of that total Linoping receives 8 million SEK.

Jesica

Joint European SiC Activity. This program funds one million SEK/year. This money is split with Okmetic, a wafer growth startup company.

Okmetic

A wafer growth startup company which is partnering with Grenoble to grow semi-insulating and n+ SiC.

ABB

Linkoping is in direct partnership with ABB (a major multi-national company) for development of materials growth processing and basic understanding necessary to fabricate high voltage rectifiers.

Other

Linkoping receives about 3 million SEK from the Swedish Engineering Research Council (TFR), Science Research Council (NFR), Defense Department (FOA).

The emphasis in the Linkoping group was to provide a technology for high voltage devices. To that end they have developed high temperature epi for SiC and are using a variant of this technology for bulk crystal growth. The results for the high temperature epi are impressive. They have obtained a background doping of 2-4 $10^{14}cm^{-3}$ with a growth rate of 40 microns/hr. For the HTCVD (as applied to bulk growth) they have achieved a growth rate .5 - .8 mm/hr. The TTEC Panelists were shown the facilities at Linkoping. All the growth equipment was designed in collaboration with EPIGRISS (a local equipment supplier). After development at Linkoping this equipment is now available on the open market. The Linkoping facilities were about three years old and very impressive. All the equipment was “state of the art” and often brand new. The panel was told that the equipment was purchased with funds from a private foundation as indicated earlier in the report. In addition to the growth effort there is an extensive effort to characterize the defects and impurties in SiC. This characterization is both experimental and theorectical. As a result of this effort the Linkoping can sustainally support ABB in it’s effort to commercialize SiC

IMC is an applied research company that facilitates technology transfer from the research and development stage to prototyping and manufacturing. It has received both industrial (80%) and government (20%) support

to enhance Swedish competitiveness. Recently, it has merged with IOF to form a new company called ACLEO. IMC has mostly performed research in microelectronics and IOF on optoelectronics, the new company has a wide range of interests in both of these technical areas and has more European presence. IMC has locations in Kista and Linkoping. The Linkoping location focuses on interconnect and packaging of electronics and optoelectronics systems.

There are 53 employees in Kista and 27 in Linkoping. The budget for 1998 was 85 million SEK ($10.5 million) and 120 million SEK ($15 million) respectively. It spins off one new company per year on the average. There is a high turnover rate (15-20% per year) with the average age of its employees being 34. IMC utilizes the growth technology developed by EPIGRESS and Linksoping. However IMC has developed novel processing technology. An example of this is the high-temperature sensor research. One approach is to use high-temperature gas-absorbing metal-gate MOSFET. Using a Pd gate, hydrocarbons (CH_x) decompose after adsorption to the Pd and the resulting H^+ ions diffuse to the interface, thereby shifting the barrier height (sensor arrays). The TTEC panel was given a facility tour of the clean room area that is on the ground floor and where all the micro-electronics device processing facilities of three institutions are housed, though separately. The IMC processes a large variety of compound semiconductors for several applications (such as solar cells and optoelectronics). These facilities are very impressive and world-class.

ABB is a large international industrial company that has a major presence in power generation, transmission and distribution. It was instrumental in getting the SiC program started in Sweden in 1988 and has sponsored many programs in developing the SiC infrastructure in Sweden. ABB is very focused in getting SiC devices into power electronics systems. For example, for a 1-5 MW industrial converter system, it is estimated that Si device replacement with SiC can reduce the size by 1/3. Significant improvement in HVDC system is also projected with SiC devices, with the goal of 50 MW plant in 300 m^2 area. The goal is to first implement SiC diodes with Si IGBT, with the implementation of SiC three-terminal switching devices later. Actually, they project that at higher frequencies, the Si IGBT will be thermally limited. At present, there is no GaN work at ABB. In summary, ABB focuses on the system application and leverage of SiC devices and is perhaps most advanced in the world in incorporating SiC devices into power electronics systems.

The SiC work at the Royal Institute of Technology (KTH – Kungl Tekniska Hogskolan a University) was presented, including efforts on the heterojunction SiC HBT with GaN emitter. The SiC/GaN HBT consists of 10^{18} cm^{-3} SiC p-base, over which a n+ GaN emitter was grown by MBE. The p-base thickness can be either optimized for high-voltage power (1 µm) or microwave power (optimized for 2 GHz). The device work is largely supported by SiC EP program of the Swedish government. The heterojunction GaN/SiC diode has been improving, reaching a low value of $I_R \sim 10^{-4}$ A/cm^2 at –50V, which is close to ideal. Contact work on SiC was also presented. TiC formation with $CoSi_2$/Ti bilayer after 900^{oC} anneal leads to a low contact resistivity $^{(10-6}$ Ω-cm^2) on p-type, 10^{19} cm^{-3} doped epi of 6H-SiC.

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