

Department of Physics, Chemistry and Biology

Master's Thesis

Optical properties of free standing cubic silicon carbide

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Linköping, January 1, 2015

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1

Introduction

SiC is a semiconductor which has attracted interest in research and industry since the 19:th century, when it was first fabricated and used as an abrasive [1]. SiC has been found to be a very stable material. It exhibits a high chemical inertness [2], and is currently commonly used in high power, temperature and radiation applications due to its ability to survive in such environments [3].

SiC is a material which exists in a large number of different polytypes, the most common of which are hexagonal, cubic and rhombohedral. The work described in this thesis deals with the only cubic polytype, denoted 3C. This is one of the structurally most simple polytypes. Compared to the hexagonal counterparts 4H and 6H, the 3C polytype has for a long time been difficult to fabricate in good quality and large volume, and is therefore less studied than the hexagonal types. Recently a method of fabricating good quality free standing material has been reported, using sublimation epitaxy [4]. The method, called fast sublimation growth process (*FSGP*), has been used to grow the samples used in the work described in this thesis. With this method it is possible to grow free standing 3C SiC, which means that the material can exist on its own, without a substrate.

Cubic SiC has many interesting material properties, which have given rise to several proposals of applications for 3C. Some of these proposed applications are not possible with the hexagonal polytypes, but are unique for the cubic polytype. One such application is the use of boron doped 3C in an impurity photovoltaic solar cell (*IPV*). This is suitable for 3C due to its band gap size together with the binding energy of boron as an acceptor in the material, which is almost ideal for photovoltaic cell material. This would give a significant increase in photovoltaic cell efficiency compared to the currently commercially available alternatives [5][Current efficiencies citation needed]. Another proposed application of 3C is as a photo-electrode in a photoelectrochemical cell used for water splitting [6, 7], where solar energy is used in the decomposition of water into hydrogen and oxygen gas.

This thesis describes the growth and optical characterization of free standing cu-

bic silicon carbide (*SiC*). The focus is on characterization of the optical properties of 3C-SiC. This is done using absorption spectroscopy, photoluminescence spectroscopy, Chapter 2 gives an introduction to silicon carbide, its structure and properties. Chapter 3 describes the process of growing the material. Both growth of undoped and boron doped material is described here. In chapter 4 a description of the different characterization methods is given, together with a theoretical description of what the measurements can tell about material properties. Chapter 5 describes how the experiments were done and chapter 6 describes the results obtained from the experiments. The results are discussed in chapter 7. Chapters 8 and 9 discuss what has been learned about the material and how the work should be continued in the future.

2

An introduction to silicon carbide

This chapter describes the properties of SiC which are relevant to this thesis. Section 2.1 describes the atomic arrangement in the material, and some different arrangements are discussed. Section 2.2 discusses the energy band structure of 3C-SiC. Finally section 2.3 describes the mechanism and some effects of doping in 3C-SiC.

2.1 Crystal structure

Silicon carbide is a crystalline material consisting of silicon and carbon atoms. The crystalline nature of the material means that the atoms are arranged in a periodically repeating structure called a *lattice*. For given chemical elements there may be several different ways to arrange the atoms in a lattice, i.e. different chemical compounds of the same types of atoms. This is called *polytypism*, where the different lattice structures are called *polytypes* of the material. SiC has a large number of different polytypes - there are more than 250 known polytypes of SiC [8]. The different polytypes can be described as different stacking orders of layers of atoms [9]. Figure 2.1 shows one such layer. The depicted layer is the (111) surface. Here each circle symbolizes one carbon and one silicon atom, displaced a small distance from each other. This pair of atoms is called the *base* of the crystal.

The marked hexagon in figure 2.1 marks an area of the crystal plane which can be used to define the different polytypes, where the polytypes are defined by the placement of the base atoms in this area in different layers. Figure 2.2 shows these stacking sequences for three of the most common polytypes. The depicted orders of the layers refers to one period in the periodic structure. The names for the different polytypes are stated at the top of the figure. The digit in the name refers to the number of layers of a period and the letter denotes the crystal symmetry. The letter *H* denotes the hexagonal polytypes, whereas *C* stands for the cubic polytype. Another common polytype is the 6H-SiC, which thus is a hexagonal structure with a period of

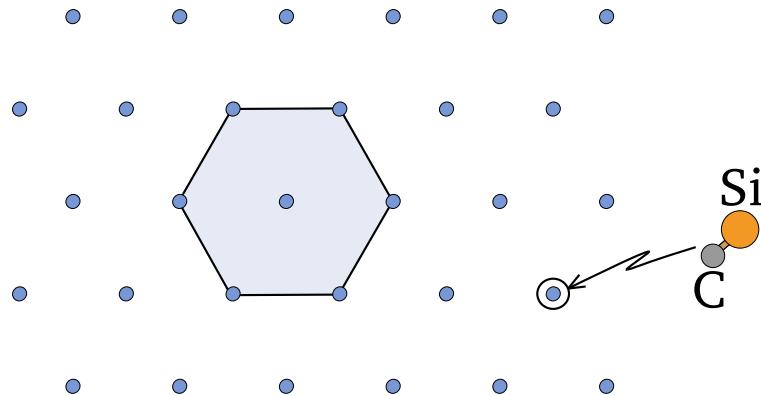


Figure 2.1: Atomic arrangement of the atoms in each (111) layer. Here each sphere corresponds to one carbon and one silicon atom, as shown by the arrow.

six base layers. It should be noted that the 2H structure is the wurtzite structure and the 3C is the zincblende structure. The unit cell of the zincblende, or 3C, crystal has a lattice constant of $a = 4.35 \text{ \AA}$ [10].

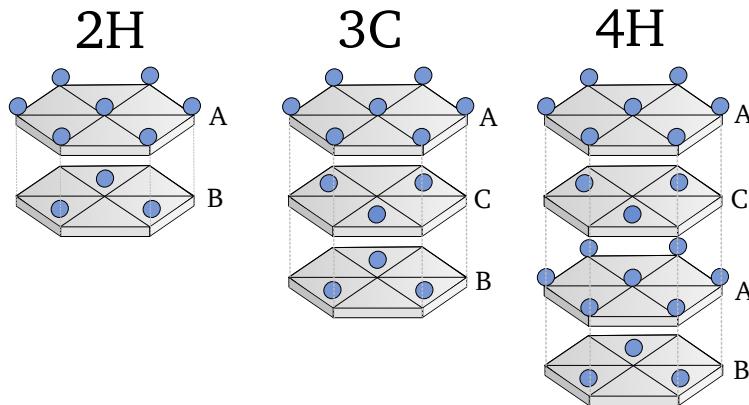


Figure 2.2: Stacking order for the three most simple polytypes.

2.2 Band structure

In SiC the atoms are bonded together with covalent bonds [Citation Zumdahl: Chemistry?] into a crystal. When the atoms are bonded together, the energy levels for the electrons in the material are defined by the material and lattice structure. This means that all materials have characteristic energy levels. When many atoms are bound together, as is the case in crystals, the discrete energy levels for the electrons in the atoms merge together to form continuous bands, called energy bands.

The band structure for 3C-SiC is shown as a band diagram in figure 2.3. In this figure, each curve describes allowed energies for the electrons, and the spaces in between the curves are not allowed. The x-axis shows different points in reciprocal space, or k -space. The marked points are specific points in the first Brillouin zone of k -space, and the intermediate intervals form straight lines between the points. This figure has been obtained using the approximate method of pseudopotentials, as described in [11]. Here the zero point energy has been chosen to be at the Γ -point in k -space.

The grayed area in the figure is the *band gap* of the material, since there are no allowed electronic states in this area. The band with lower energy than the band gap is called the valence band, and the band with higher energy is called the conduction band. 3C, as is characteristic for semiconductors, has the Fermi level in the middle of the band gap. This means that at the temperature of 0 K, all electrons will be in the valence band, and the conduction band will be unoccupied. When the conduction band is unoccupied the material will not be able to conduct electricity. If the temperature is raised above 0 K there will be some occupation of the conduction band and some electrical conduction will be able to occur. With a higher temperature there will be more electrons in the conduction band and thus a higher conductivity of the material.

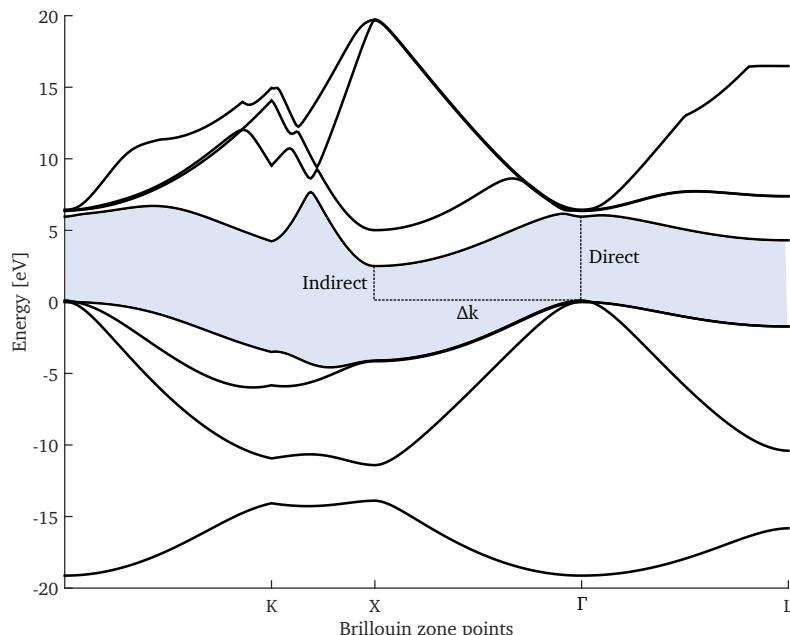


Figure 2.3: The band diagram of SiC at ambient conditions for different positions in the Brillouin zone. The indirect and direct band gaps are marked in the figure. The marked area is the band gap. The energy levels have been obtained using the pseudopotential method, as described in [11].

In the figure 2.3 it can be seen that the smallest energy difference between the valence and conduction bands is between the Γ -point in the valence band and the X-point in the conduction band. This difference is called the *band gap*. The difference between the Γ - and X-points is called the indirect band gap, while the difference between the values at the Γ -point is called the direct band gap, as illustrated in the figure. These values are given in table 2.1. The value for the indirect band gap at room temperature is taken from the simulation, whereas the latter values are taken from literature.

Table 2.1: Values for the indirect and direct band gap energies. The indirect values are given both for room temperature and low temperature, as this will be of value later in the thesis.

$E_{g,\text{indirect}}$ (300 K)	$E_{g,\text{indirect}}$ (2 K)	$E_{g,\text{direct}}$ (300 K)
2.36 eV	2.42 eV [10]	6.00 eV [12]

When an electron makes the indirect transition from the valence to the conduction band the energy is increased by E_g , and the k-value is changed, as indicated by Δk in figure 2.3. This change of k-value implies a change in momentum of the electron, since k and momentum p is related by

$$p = \hbar k,$$

where \hbar is Dirac's constant. This is of importance when studying interaction between the material and light. Photons are capable of providing the energy needed to transit from the valence to the conduction band, but cannot provide the change of momentum needed. The law of conservation of momentum requires the total momentum of the system to be preserved during the transitions. The extra momentum can however come from interaction with phonons.

The phonon energy spectrum has four ground modes, since there are two different kinds of atoms in the material. There are the acoustic and optical phonon modes. Theoretical calculations of the phonon dispersion curves for 3C have been done by Karch et al. [13]. They show the four modes: TA, LA, TO, LO, and give the wavelength for each mode. The transition in k-space for the indirect transition is between the Γ - and the X-points, hence it is of interest to know which phonon wavelengths and energies this corresponds to. Table 2.2 shows these computed wavelengths and the corresponding energies, which have been calculated using the fact that

$$E_{\text{meV}} = \frac{hc(\frac{1}{\lambda_{\text{cm}}})}{e} \times 10^5,$$

where h is Planck's constant, c is the speed of light and e is the elementary charge.

Table 2.2: Inverted wavelengths and energies for the Γ -X transition. Values for λ from [13].

Mode	$\lambda^{-1} [\text{cm}^{-1}]$	E [meV]
TA	368	45.6
LA	637	79.0
TO	760	94.3
LO	829	102.9

2.3 Doping in 3C

Doping is a way to change the electronic structure of a material by substituting some of the native atoms with a foreign element. In the case of SiC this means that either silicon or carbon is replaced by some other element. How the band structure of the material is changed depends on which material is introduced to the crystal. By choosing the dopants it is possible to tailor the band structure to create various new properties of the material.

An important factor for how the band structure is changed is the number of valence electrons in the introduced element, compared to the element it replaces. Silicon and carbon both have four valence electrons, creating four bonds to its neighbouring atoms in the crystal. The electrons are strongly bound to the atomic nuclei, requiring energy corresponding to the band gap to free one electron. If one silicon or one carbon atom is replaced by an element with five valence electrons however, the additional electron is not as strongly bound to the nucleus and can easily release an electron to the conduction band. This is called a *donor* atom. Similarly if an atom with only three valence electrons is introduced to the crystal, it can readily bind an electron from the valence band, creating an electron deficiency, or *hole* in the valence band. This is called an *acceptor* atom. Figure 2.4 shows a simplified band diagram with a donor and an acceptor level. The donor level donates an electron to the conduction band by supplying the energy E_D to the electron. The acceptor accepts an electron from the valence band by supplying the energy E_A . The energy required to supply the material with one carrier from a dopant level is called the *binding energy* (or *ionization energy*) of the dopant.

2.3.1 Donors

Since silicon and carbon both have four valence electrons, a donor atom in SiC must have five or more electrons. One common donor is nitrogen, which has five valence electrons. This means that each added N-atom can supply the conduction band with one electron. A material with more donors than acceptors is said to be an *n-type* material. Freitas et al. have measured the binding energy of nitrogen in 3C, and found it to be 54 meV [14]. This means that the lowest N-level is 54 meV below the conduction band.

N-doping can be achieved intentionally by fabricating the material in a nitrogen

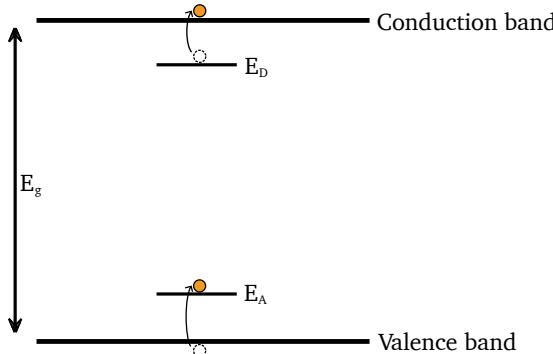


Figure 2.4: Two new energy levels are introduced by doping.

atmosphere, but nitrogen always present in FSGP-grown material even at dynamic vacuum conditions [15]. This means that material grown by this method is generally n-type. The n-type material can also be created by other elements in group five. Reports of doping with As, P and Sb exist, but are far less common than N-doping [16].

2.3.2 Acceptors

Acceptor atoms need to have fewer valence electrons than silicon and carbon, which is why group three elements are the most common. These include boron and aluminum, which have the binding energies 257 meV [14] and 735 meV [5] respectively. A material with more acceptors than donors is said to be *p-type*. Table 2.3 summarizes the different dopant energy levels.

Table 2.3: Binding energies for some of the most common SiC dopants.

Element	Dopant type	$E_{D/A}$ [meV]	Reference
N	Donor	57	[14]
B	Acceptor	735	[5]
Al	Acceptor	257	[14]

The B-doping energy level is of particular interest for photovoltaic applications, where the binding energy is ideal for the impurity photovoltaic solar cell. This is described in more detail in chapter ??.

There are several ways to create doped materials. It can be done by ion implantation, where ions are accelerated to high energies and then made to collide with the undoped material [16]. It can also be done as the material is grown, by including the doping element in the ambient. The first method has several advantages: it can

be done with good control over doping density and it is possible to select only certain areas of the material to dope. The latter method is done *in situ*, so it requires no additional equipment. It is also not as prone to create defects as the ion implantation method is, where annealing is often required after implantation. In the work described in this thesis, the method of doping is to include the doping material during the growth. This method is described in more detail in chapter 3.

During doping some of the native atoms are replaced. This will have effects on the quality of the produced material. Different elements have different atomic radii, so replacing one atom by one of a different element will create some strain in the material. This may lead to defects in the material.

3

Growth technique

SiC can be fabricated by several different techniques, and the different techniques are advantageous for different polytypes and applications. Some of the most common methods are *sublimation epitaxy*, *liquid phase epitaxy (LPE)*, *chemical vapour deposition (CVD)* and *physical vapour deposition (PVD)* [17].

The goal when growing freestanding material is to have high quality material of large volume. Up to this point in time, researchers have had more success fabricating high quality 4H and 6H material compared to 3C [3]. The hexagonal polytypes are commonly fabricated using PVD. This method has not been widely adapted to 3C growth however, which is often attributed to the fact that this method uses homoepitaxy, and there are no 3C seeds widely available. Historically in the growth of 3C using CVD, there has been a trade-off between crystal quality and growth rate. Nishino et al. reported in 1983 a growth rate of approximately 2.5 microns per hour with the CVD technique for 3C growth [18]. This rate is much too low to create free standing material. The growth rate for CVD growth has been improved since this time, and in 2002 Nagasawa et al. reported a rate of 40 $\mu\text{m}/\text{h}$ [19], which makes it possible to fabricate free standing 3C.

Another growth method is sublimation epitaxy, which was demonstrated by Lely in 1955 [20]. In this method material is transferred between a source material and a substrate using sublimation. Recently there have been reports of high quality cubic SiC fabricated by a type of sublimation epitaxy called *fast sublimation growth process (FSGP)*. Growth rates as high as 500 $\mu\text{m}/\text{h}$ have been reported using this method, while still growing good quality material [4]. This high growth rate is obviously ideal for growth of free standing (and even bulk) material. This chapter describes in detail the sublimation method in general and FSGP in particular. FSGP is the method used to obtain the results in this thesis.

3.1 About sublimation and nucleation

Sublimation is the phase transition where a material transitions between solid and gas, without the intermediate liquid phase. At normal conditions SiC does not melt, but rather it sublimes. For SiC to melt a pressure of 100,000 atm and a temperature of at least 3200 °C is required - at atmospheric pressure SiC can only sublime [21]. Sublimation of a SiC source material will give a vapour consisting of different molecules consisting of silicon and carbon, for example Si, Si₂C, SiC₂ etc. The composition and vapour pressure of the gas can be controlled using parameters such as temperature and pressure of the ambient.

If a substrate is placed in the silicon-carbon vapour, some of the material will be adsorbed on the surface of the substrate, in the form of *adatoms*. When many such atoms are adsorbed at the surface they bind together, forming a *nucleus* of crystal growth. In a sublimation growth setup, a source of SiC is heated, initiating the sublimation. The vapour then travels to an appropriate substrate, by the means of a heat gradient. Material is adsorbed on the substrate which forms the grown crystal. Figure 3.1 shows a schematic of this process.

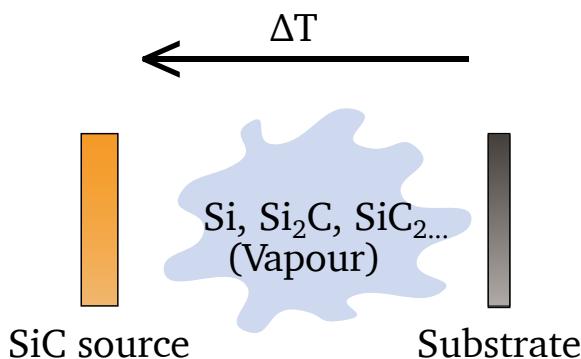


Figure 3.1: A vapour containing silicon and carbon molecules is created by sublimation of a SiC source. The vapour is adsorbed at the substrate.

As the material is adsorbed at the substrate the growth begins. The crystal growth is governed by the free energy of the whole system. A decrease in free energy works to promote the growth. In crystal growth, a decrease in free energy is an increase in chemical potential μ , meaning that crystal growth is driven by an increase in chemical potential. The system is made up by the vapour and the crystal phases, hence a change in chemical potential is given by

$$\Delta\mu = \mu_v - \mu_c,$$

where μ_v is the chemical potential for the vapour and μ_c is for the crystal. For growth from vapour phase, the chemical potential difference between vapour and crystal is dependent on the *vapour pressure* and the *saturated vapour pressure* through the formula

$$\Delta\mu = k_B T \log(p/p_e),$$

where p is the vapour pressure and p_e is the saturated counterpart. Defining the *supersaturation*, σ , as

$$\sigma = \frac{p - p_e}{p_e}$$

thus

$$\Delta\mu = k_B T \log(\sigma + 1).$$

Taking the first order Taylor expansion this becomes

$$\Delta\mu \approx k_B T \sigma.$$

It is concluded that the change in chemical potential is directly proportional to the supersaturation of the system. An increase in supersaturation will hence promote growth and increase growth rate.

Supersaturation is also a measure of how the crystal is formed. SiC can grow in several ways. One such way is called *spiral growth*. In this growth method there are steps which originate from one point on the surface. As atoms attach to these steps, they move around the center point. This gives a spiral shape on the surface. Another way of growth is *2D nucleation*. This is when atoms are adsorbed to a flat surface, and the adatoms bind to each other creating a 2D nucleus on the surface. Additional atoms will attach to this nucleus as they are adsorbed to the surface. The value of supersaturation governs which of these two methods is present.

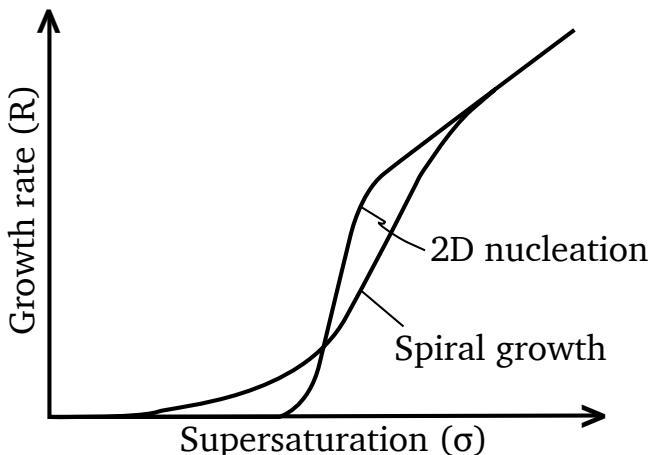


Figure 3.2: The growth rate for different modes vary depending on supersaturation.

Figure 3.2 shows the general relationship between the growth rate (R) and the supersaturation (σ). It should be noted that at a certain value of σ the growth rate of 2D growth is higher than that of spiral growth. At σ higher than this value the 2D growth will dominate over spiral growth.

3.2 The fast sublimation growth process

The fast sublimation growth process is setup as shown in figure 3.3. An insulating graphite foam is placed inside a quartz tube, which is surrounded by a copper coil (shown as a cutaway in the figure). The copper coil is connected to an RF-generator capable of creating a current in the coil. The foam cylinder contains a graphite crucible, which will couple with the magnetic field created by the current in the copper coil. This will heat the crucible. To measure the temperature of the crucible there is a pyrometer fixed above the quartz tube (not shown in the figure), which can measure the temperature of the crucible through a small hole at the top of the insulating foam.

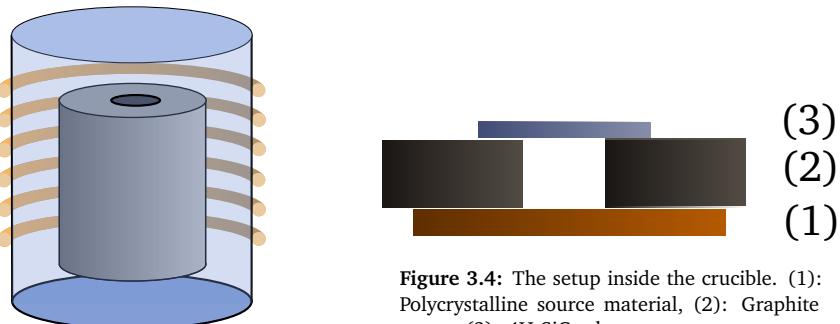


Figure 3.4: The setup inside the crucible. (1): Polycrystalline source material, (2): Graphite spacer, (3): 4H-SiC substrate.

Figure 3.3: An outside view of the reactor setup of the FSGP method. A copper coil surrounds a quartz tube containing an insulating graphite foam.

The graphite crucible contains the components of the sublimation process, as shown in figure 3.4. At the bottom of the crucible is a source material, which will sublime away during the growth. When growing free standing 3C-SiC, a polycrystalline SiC wafer piece is used as source material. The source and the substrate are separated by a graphite spacer, which is a disc with a hole in it where the vapour can flow freely. The size of the spacer and the hole can be varied depending on the desired size of the grown sample. The substrate can consist of different materials, generally 4H-, 6H-SiC or Si. There is a significant mismatch of the lattice constants and of the thermal expansion coefficients of Si and 3C-SiC. This is not favourable for high quality grown material. It is instead favourable to grow 3C-SiC on one of the hexagonal polytypes of SiC. Wafers if both 4H- and 6H-SiC are commercially available and can thus readily be used as substrates in growth. When the wafers are cut, they can be cut at different angles, i.e. in different crystal planes. It has been shown that cutting along the (0001) plane gives a worse quality of the grown material than cutting slightly off from this plane [22]. Using the FSGP technique it has been shown that the best quality 3C-SiC is grown when using 4° off-axis cut substrate. This means that the cut is made 4° from the (0001) direction towards the <1120> direction on a 4H-SiC substrate [4].

Growing on on-axis substrates will give many nucleation sites. As these sites

expand and merge with each other there will be many defects at the interfaces. These defects are called *double positioning boundaries (DPB)*. The off-axis substrates have a surface which contains many shallow steps. As the 3C-SiC has nucleated at the edge of the growth area, the nucleation site will expand laterally along the step-flow direction. This growth method is called *lateral enlargement mechanism*, and is the main principle of FSGP after nucleation has occurred. The density of DPBs are greatly decreased in this manner. This process is described in detail by Jokubavicius et al. [4].

As described earlier in this chapter, the growth temperature and pressure are vital parameters to determine growth rate and thus also crystal quality. In FSGP these parameters can easily be controlled during growth. The temperature is controlled by modulating the current from the RF-generator. A temperature ramp up scheme is used to facilitate proper step flow growth. The growth starts by homoepitaxial spiral growth of 4H-SiC on the substrate, until the supersaturation has become large enough for 2D-growth to occur. This is in accordance with figure 3.2. It has been shown that the temperature window for 3C-SiC growth is rather narrow [22], hence proper control of the temperature is vital for good quality material. Another important parameter is the pressure in the reactor during growth. To avoid inclusions of foreign material in the crystal a low pressure during growth is required. Typically a pressure of 10^{-4} to 10^{-5} mbar is used, which is achieved with dynamic vacuum. Some nitrogen inclusions will be unavoidable even at these pressures. A residual N-doping in the order of 10^{16} cm^{-3} has been shown in material grown under these conditions [4], which can be compared to 10^{22} cm^{-3} silicon or carbon atoms in the crystal.

For a good quality crystal to form, the proper ratio between silicon and carbon must be present in the vapour. To avoid the problem with excess of carbon, a *carbon getter material* is often used. This is a material which binds carbon from the vapour, thus reducing the C/Si ratio. One such material is tantalum.

3.2.1 Doping using FSGP

It is possible to grow intentionally doped material using the FSGP method. To do this, the pure SiC polycrystalline source is exchanged for a doped source with the appropriate dopant density. The vapour from the sublimation will then contain the dopant element. Figure 3.5 shows the crucible setup for growth of doped material. Here the substrate contains a 3C-SiC seed before the growth starts. This is to facilitate cubic growth.

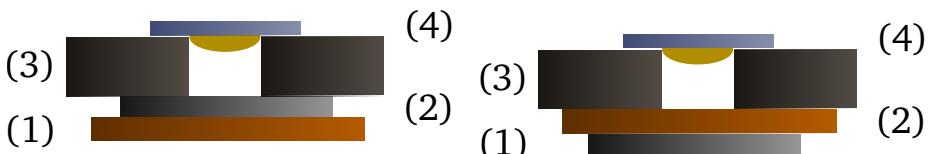


Figure 3.5: The crucible setup when doing doped growth. The source and seed (4) is separated by a graphite spacer (3) from the doped (2) and undoped (1) source material.

Figure 3.6: Indirect growth. Here the doped (1) and undoped (2) source materials have changed places as compared to direct growth.

It is now vital to have the proper ratio between all of the elements in the vapour phase, i.e. silicon, carbon and dopant. In this thesis a new method to possibly alter this ratio is introduced. This method is called *indirect doping*, and is shown in figure 3.6. This differs from the direct doping shown in figure 3.5 in that the doped source is now separated from the substrate by a nominally undoped source layer. If the doped source material is not homogeneous, the indirect method is hoped to give a better grown material, as well as reduce the dopant content in the vapour.

4

Characterization techniques

This chapter will describe the different characterization techniques.

This is a paragraph.

4.1 Absorption measurements

Here will follow a description of how the absorption measurements were done.

5

Experimental setup

Here the setups for the various experiments will be described.

6

Results

This chapter describes the results obtained from the experiments.

7

Discussion

In this chapter I will discuss the results of the work.

8

Conclusion

The conclusions drawn from this work are described here.

9

Future work

In this chapter I will write some of my thought of what is to be done in the future concerning these topics.

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