Institutionen för fysik, kemi och biologi

Master Thesis

Stability Of Bulk Cubic Silicon Carbide (3C-SiC) On Off Oriented Hexagonal Silicon (4H-SiC) Substrate

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Abstract

Different polytypes in Silicon Carbide give a very wide range of applications in electronic devices and the graphene growth. To improve the performance of the electronic device, high purity material and large size domain of SiC are required. The growth method, which is called fast sublimation growth process (FSGP), for SiC on low off-axis substrates was introduced to grow cubic SiC on the hexagonal SiC substrate. The growth mechanisms were discussed.

The aim is to get the optimal and favorable growth criteria for the high quality bulk-like cubic SiC, which can be used as the seed to grow the homopolytypic growth of 3C-SiC and is also useful for graphene growth. Several series were done to get the transition critical temperature and how the parameters, including the growth temperature, growth time, growth rate, graphite plate, cutting off-axis angle, the thickness of the layer, influence the as-grown epilayer. The target is to get the 3C-SiC covering all the substrate with good quality and big domain.

Optical Microscopy was used for studying the morphology of the as-grown epilayers. In each image, different kinds of defect and the condition of the domains on the cubic SiC epilayer were studied. Furthermore, Atomic Force Microscopy was utilized in characterization to know the step-bunching effect in the growth. Also, average step heights and the terrace width of the as-grown layer were calculated and analyzed.

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CONTENTS

1. Introduction

Silicon Carbide (SiC) is a wide bandgap semiconductor material, which consists of silicon-carbon atoms with whole covalent bonds. There are lots of fascinating properties with this wide band gap material, like a high blocking voltage, high-saturated electron velocity, higher thermal conductivity. These are useful since they can improve performance in the electronic devices. Today the most development is related to high-reliability, high-efficiency power electronics.

However, high purity material and large size domain of SiC are still difficult to fabricate, so substrates are still expensive in mass-production. We have not really clearly understood all mechanisms like how to stabilize different polytypes, even though the commercial productions show full polytype domination while details of physics of stability are not fully known. Hence, to develop a better quality of the material and also to stabilize the processing using epitaxial growth in each polytype, studies of polytype stability are very important steps for the SiC-based substrate or device development. We still need more information about ways to grow SiC, how to stabilize the process, and also to further understand the unique properties for each of the polytype.

In addition, to elaborate on the future possibilities, SiC can also be an excellent substrate for other materials. Nowadays, there are several techniques to fabricate monolayer and multilayer graphene on SiC in large size. But some of them still have some difficulties to overcome. For example, in exfoliation of graphite, the largest flake you can get is still too small for many industrial purposes. Other techniques are employed for graphene like chemical vapor deposition (CVD) for graphene on metal substrate, and also the chemical reduction of graphite oxide [1].

Unlike the case for graphene on metal, the graphene grown on the silicon carbide is basically a layer that is on a semiconductor substrate. It is an advantage that the semiconductor can be a part of the device and there is no need to transfer for device processing. The substrates also have useful electronic properties. Therefore, recently there are large interests in the techniques that grow graphene on different kinds of polytypes of SiC substrates, for example 4H-SiC and 6H-SiC, by using sublimation process. Since 4H-SiC and 6H-SiC have a hexagonal structure and show a good performance for the graphene growth, the most studies have been on these polytypes. However, compared to the hexagonal substrate, the cubic substrate has not been as much studied. Until now there is no commercial cubic SiC as substrate. Also, the techniques to grow a good crystalline quality of 3C-SiC bulk grown material have not matured enough to make substrates be used in active device structures. The most important reason why we are interested in the cubic substrate growing is that 3C-SiC has some different properties from the 4H-SiC or 6H-SiC, which can result in modified or different structure of graphene. For example, 3C-SiC has the higher saturated electron velocity and isotropic physical properties [2]. These in combination with graphene properties can make interesting structures compared with graphene on hexagonal SiC. Furthermore, the fascinating biocompatibility of 3C-SiC may offer the chance that the material can be used in the new generation of advanced biomedical devices. In another potential application, 3C-SiC can be an excellent solar cell material and as pure substrates. Also, graphene may be used for contact layers on such solar cells made on 3C-SiC substrate [3].

There are already some fundamental observations of graphene growth SiC polytypes. For example, when comparing graphene on hexagonal and cubic SiC, it has been shown that there are strong relations between the roughness of the substrate, SiC step heights and the quality of the morphology of grown graphene in 6H, 4H and 3C-SiC. In general, a lower substrate surface roughness results in more uniform step bunching with a lower distribution of step height, this will bring the better quality of graphene grown on 3C-SiC [1]. These reflect that if 3C-SiC would be available as substrate, there would be various new scientific findings to expect. Therefore, to stabilize and get the high crystalline quality domain of 3C-SiC substrates are vital factors for obtaining new scientific findings.

Generally, the thesis is about the studies of 3C formation and stability on off-axis 4H-SiC. It can be divided into three main parts. The first part is mainly studying step bunching at initial 4C-SiC formation on 4H-SiC at different growth temperatures and growth times by using different low off-axis angle 4H-SiC substrates to understand more details about the nucleation in the central step region. The second part concerns the stability of the cubic bulk-like materials up to 2 - 3 mm thickness, and compares 3C-SiC crystal stability by dominating mechanism at the edge or stepped central region. The third part is to discuss about the relation between the 3C-SiC epilayer expansion and different kinds of growth conditions on the 4H-SiC off-axis substrate.

The main part is related to growth, and some characterization methods, which are Sony Kaiser RAI optical microscope (OM) and Atomic Force Microscope (AFM) Dimension 3100 from Digital instruments, were used.

2. Silicon Carbide (SiC)

Crystal structure

Silicon carbide is consisting of a tetrahedral of carbon and silicon atoms with strong bonds between Si and C atoms in the crystal lattice. Each C atom is surrounded by four Si atoms. Similarly, each Si atom is surrounded by four C atoms. Because of the strong bonds, SiC is a very hard and strong material. It has a good resistance to chemicals and radiation. The advantageous properties at high temperatures make SiC as one of the most suitable materials for electronic devices.

The SiC has over 200 different crystalline structures, which are called polytypes. The most common polytypes of SiC are 3C-SiC, 4H-SiC, 6H-SiC and 15R-SiC, which are cubic (C), hexagonal (H) and rhombohedron (R) structure, respectively. As Fig. 1 shows, the height of the unit cell is different in the polytypes and there is a slight difference in the stacking sequences, denoted as ABC in the figure, along the c-axis direction that makes up the different polytypes and influence variations in electronic properties. The cubic crystal structure, which is the only cubic polytype, is denoted 3C-SiC, and also referred to as β-SiC. The other common polytypes are referred as α-SiC.

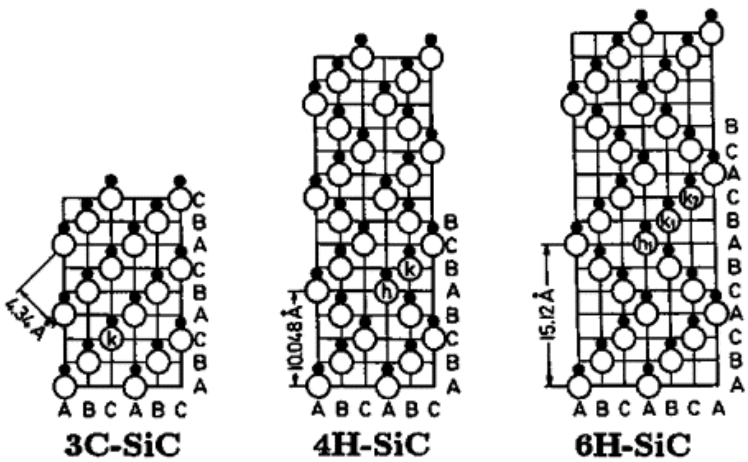


Fig. 1. SiC polytype for plane ($11\bar{2}0$) of the three most common SiC polytypes. h and k denote the hexagonal and cubic sites in the lattice, respectively. Open and full circles stand for the silicon and carbon atoms, respectively. The length is the height of the unit cell c [4].

Properties

Different polytypes have slightly different properties, which make the applications of SiC wider and more flexible. Table. 1 shows the comparison of some important properties between the three most common SiC polytypes.

	Lattice constant [Å]	Band gap E_g [ev]	Relative dielectric constant ε_S	Breakdown Field E_B at $N_D = 10^{17} cm^{-3}$ [MV/cm]	Thermal Conductivity κ [W/cm K]	Electron Mobility μ_n at $N_D = 10^{16} cm^{-3}$ [cm^2/Vs]	Saturated Electron Velocity ν [10^7cm/s]	Maximum operating temperature [°C]
3C-SiC	a=c=4.359	2.4	9.72	>1.5	3.2	800	2.5	1240
4H-SiC	a=3.073 c=5.048	3.26	9.7	‖c-axis: 3	4.9	⊥caxis:800 ‖c-axis:900	2	1240
6H-SiC	a=3.08 c=15.11	3.03	9.66	⊥c-axis:>1 ‖c-axis:3.2	4.9	⊥c-axis:400 ‖c-axis:60	2	1240

Table 1. Comparison of some important properties between three most common SiC polytypes, which are 3C-SiC, 4H-SiC, and 6H-SiC [5][6].

Generally, SiC polytypes have fairly similar (wide) bandgap and electric field, which results in a high electric breakdown field and higher breakdown voltage in the electronic device. In addition, the operating temperature of devices using conventional semiconductors is limited by the bandgap, difference between the number of the intrinsic carrier and the intentional doping concentration. The wide bandgap in SiC can reduce the intrinsic carrier concentration so the device can operate at higher temperatures [7]. Furthermore, SiC has a high saturated electron drift velocity, which can enable devices to switch at higher frequency. The outstanding properties of SiC can easily fit to the demand in electronic device applications and also make the device operate under the high temperatures and the high power conditions surroundings. However, the challenge is in the growth and stability of material to reduce the defects that limit these outstanding properties.

3. Growth mechanisms and Defects

a. Nucleation mechanism

In order to grow a good quality crystalline epilayer, the nucleation and crystal growth mechanism play important roles during the growth process. The growth mechanism depends on several growth conditions and factors. For example, supersaturation level, Si/C ratio and the type of the substrate [8]. These are two nucleation mechanisms that influence during the cubic SiC formation and growth.

In general, the hexagonal SiC epilayers are forms by step-flow growth on off-axis substrates, and the cubic SiC epilayers forms on hexagonal SiC via two-dimensional nucleation and subsequent step-flow, which will be discussed below [9].

Two-Dimensional (2D) Nucleation Island growth

Two-dimensional nucleation is the main growth mechanism of the cubic SiC. It mostly starts at the large areas of a flat terrace, the place where there is a lower density of steps. The step widths increase with a lower angle of cutting off-axis of the substrate. Therefore, the 2D nucleation has higher probability to appear on lower off-axis substrates [10].

Normally, when atoms from the sublimed source arrive at the terrace surface, the atom can either become adatoms that diffuse along the surface to the step edge to form the step flow growth or desorb back to the ambient. In general, the atoms nucleate at the surface at an energetically favored position or a thermodynamically favorable position. For example, these can be steps, defects or islands on the terrace [11]. When the first diffusing adatoms can not find any spot of lower energy or do not have enough energy to go forward, the nucleation site will be decided on the terrace and start to nucleate at the point. The following incoming adatoms will start to nucleate just beside the nucleation site to accumulate and form an island. The expanding island may continue to grow until it meets another extending island on the terrace. In the next step, a new nucleation site on the top of this layer may form, and a second layer of the adatoms will continue to expand.

The initial nucleation is a very important step during the growth. This is when the most common defects, for example twin boundaries and stacking faults, in 3C-SiC form. Also, the stacking faults are formed at the initial growth from the two-dimensional nucleation. The order of stacking sequence, which decides the polytype, and the distribution of the stacking faults are strongly dependent on the growth condition [12].

A higher supersaturation level is a result due to more incoming sublimation atoms, so the number of the nucleation cendters on the terrace increase. However, in order to lower the defects that accumulate at the domain boundaries, the fewer number of nucleation centers are preferred to form large domain.

Step-Flow Growth

In the homoepitaxial growth on off-oriented substrates, the step-flow growth nucleation does not occur on terraces but at the step edge. The step flow growth direction is the same as the off-orientation direction $[11\bar{2}0]$ in all the series done in the thesis. The initial homoepitaxial nucleation at the steps controls the polytypes and also increases the crystalline similarity between the epilayer and the substrate.

However, there are several problems that can arise when there is step-flow growth. The most common one is step bunching, which will be discussed in details later on, and the others are defects that are forming and uneven nucleation of polytype.

b. Defects

There are several kinds of defects formed during the growth of the SiC epilayer. As mentioned before, most of the defects were formed at the initial stage of the growth process. These defects normally degrade the performance of the application of the electronic device because of the lower crystal quality. The common defects like dislocations (screw and edge in hexagonal SiC), micropipes, stacking faults, double positioning boundaries and step bunching will be mentioned below.

Dislocations

Dislocation is a very common line defect within the crystal lattice. The dislocations are introduced in order to reduce the inner stress existing in the lattice, which result from the difference in different polytypes and lattice constant, or between two different orientations in domains while growing. There are two types of dislocations. The edge dislocation, where the burgers vector is perpendicular to the line of dislocation, and the screw dislocation, where the burgers vector is parallel to the line of dislocation. In most cases, the mixed combination of edge dislocation and screw dislocation exist in the crystal lattice.

Micropipes

The most common defects that have present in the hexagonal SiC are the “micropipes”. It's a hole with the propagation along the crystal growth direction <0001>, and from substrate these may penetrate into the epilayer. As can be seen in the Fig. 2, two nucleation sites have four basal planes layers with the expanding direction parallel to the basal planes. The expanding of the basal planes is supposed to happen within the same equivalent level to form a single crystal. However, in the real case, there will be some mismatched-coalescence happening during the growth, which results in imperfections or disturbances. Depending on the nucleation sites, there will be different levels of mismatched coalescence. Furthermore, if there are more nucleation sites, these mismatched coalescences will result in the dislocation at the center of these nucleation centers. The subsequent growth will follow the pattern and make the dislocated center to become a “micropipe”. [13].

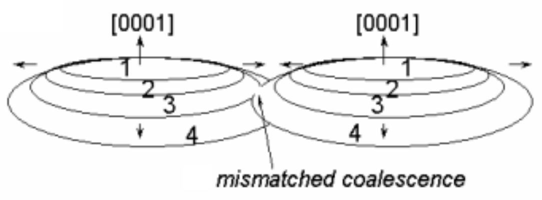


Fig. 2. The mismatched-coalescence during growth with the crystal growth direction [0001] being between two nucleation sites[13].

Stacking faults

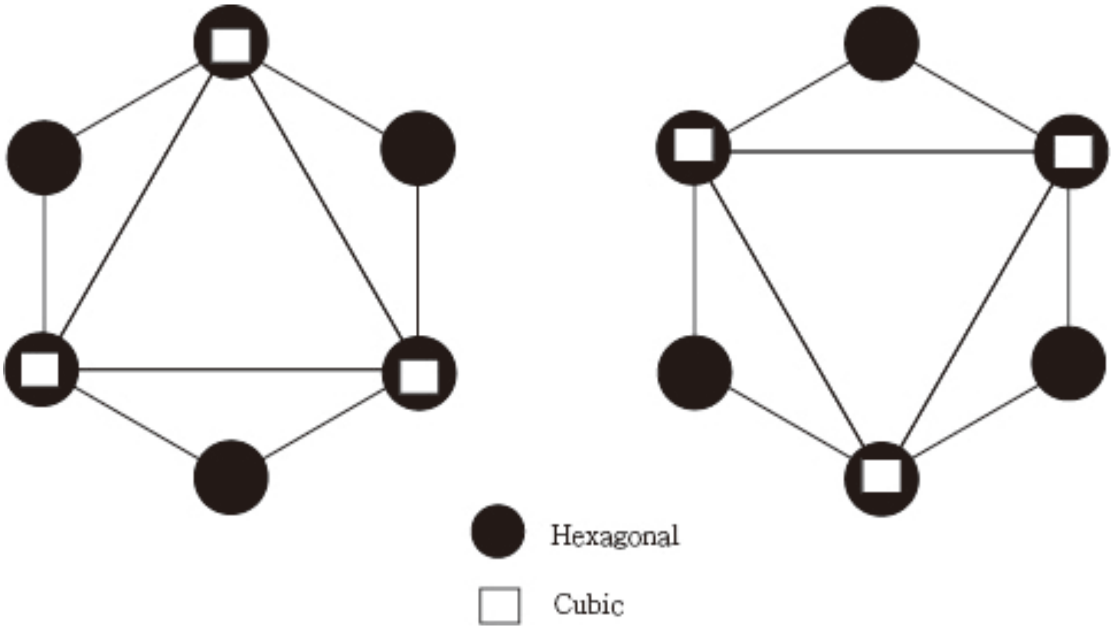
Stacking faults are defects, which are formed at dislocated and interrupted places along the c axis in the crystal lattice, where the stacking sequence should be repeated. Similar to the micropipe formation, the mismatched coalescences and stacking sequence disturbance cause the stacking faults.

The energy, which is needed to form the stacking faults, is lower when growing the cubic SiC [14]. In cubic SiC, unlike the other polytypes, the energy will release to form many stacking faults.

Double Positioning Boundaries (Twin domain) (DPB)

It is a common defect that appears uniquely in the 3C SiC structure due to the 2D-nucleation [15]. The two domains, which have different orientations, but the same cubic structure, are called “twins”. In between the domains there will be twin boundaries [16].

Because of the two possibilities of the stacking sequence, either ABCABC or ACBACB, and different orientations in 3C-SiC, the domain with different stacking sequences cannot merge together when domain are expanding. As one can see in Fig. 3, the diffraction patterns for the cubic, where the atoms are located on (111) plane that has nucleated on the hexagonal substrate, can have two possibilities. Within these two possibilities, during growth, there will be some growth and size limitation between two domains. Then the defects will form at the boundaries as they merge. These boundaries between the domains are named as "Double positioning boundaries". Normally in the off-axis case, the domains can be easily recognized in 3C-SiC by the shaped of triangle with an angular difference. Between two different orientations of triangular domains, the double positioning boundaries will form at the boundaries.



Fig, 3. Two possibilities of the cubic nucleate on the hexagonal substrate. There is 60 degrees orientation different within these two possibilities. The circles are the diffraction pattern of hexagonal and the squares are the cubic.

Step Bunching

Step bunching is a phenomenon which commonly is happening during the growth of SiC. It appears in order to lower the surface energy. Different polytypes and terraces within the crystals have different surface free energy.

When the atomic terrace steps coalescence, there is a rearrangement of the surface. The atomic steps can coalesce since the different surface energies make the lateral speed of the steps different. The small steps (microsteps) catch up other small steps to become a big step that is a so-called “macrostep”. The reconstructing processes not only change the terrace width and step height, but also influence the performance of the electronic devices by influencing the doping and structural quality. Generally, the step-bunching result in a higher surface roughness, which lowers the uniformity of the surface and reduce the epilayer quality. Heavy step-bunching can be observed on epitaxial layers, for example in growth on 4-degree off-axis Si-face substrates [17][18].

4. Growth Techniques & Affecting Factors

Growth techniques

Nowadays, the most common growth is chemical vapor deposition (CVD). Although the epitaxial layers grown using on CVD have shown a good quality and polytype stability, the growth rate, which is around 10 µm/hr , is too low for growth of thick layers. In comparison, the growth rate of FSGP can be up to 1 mm/hr [8], not only can the method achieve good quality of thick layers, but potentially it may also fit the growth of bulk like material, which can be used as SiC seeds [19].

Fast Sublimation Growth Process (FSGP)

The fast sublimation growth process (FSGP) provides a very high growth rate and it is useful since it can grow thick layers. In this thesis, all the SiC epilayer series were grown by FSGP.

In FSGP, there are mainly two parts of the system, which are a heating system and pumping system that are connected to the quartz tube acting as a growth chamber inside which a crucible is placed. The actual growth occurs in the crucible. In these two systems, the RF-generator and the turbo pump are used, respectively.

In the heating system, there are coils outside the tube . A current through the coils will create magnetic fields, which in turn will be heating up the crucible. A cylindrical graphite growth crucible consisting of a bottom piece and a lid was mounted inside thermally insulating graphite foam. Between the crucible and the tube, graphite-insulating foam was added to thermally isolate the system [8]. At the top of the graphite foam, a hole was made for a pyrometer to detect the temperature of crucible. There is difference in the temperature between the inside of the crucible and what is measured by the pyrometer since it measures on the top of the crucible.

Inside the crucible, the sandwich-like layers arrangement was put as the Fig. 4 shows below. A polycrystalline SiC source and a 4H-SiC substrate were separated by 1 mm from each other with a graphite spacer. When heated by the RF coil, the crucible will produce a temperature gradient. This is used to provide a sublimation of the source and the sublimed material is then deposited on the cooler SiC substrate. The temperature gradient is designed using the coils position in respect to the crucible position, and also the geometry of the bottom piece of the crucible and the lid. Normally , the measured temperature by the pyrometer is around 1750℃ to 1950 ℃. At the bottom of the crucible, a C-getter was used to control the important growth parameter Si/C ratio, which will be mentioned in more details later.

In the pumping system, the lowest pressure that the turbo pump can get is around 10^{-5}mbar, which is nearly vacuum. This is the ambient used in the series in the thesis.

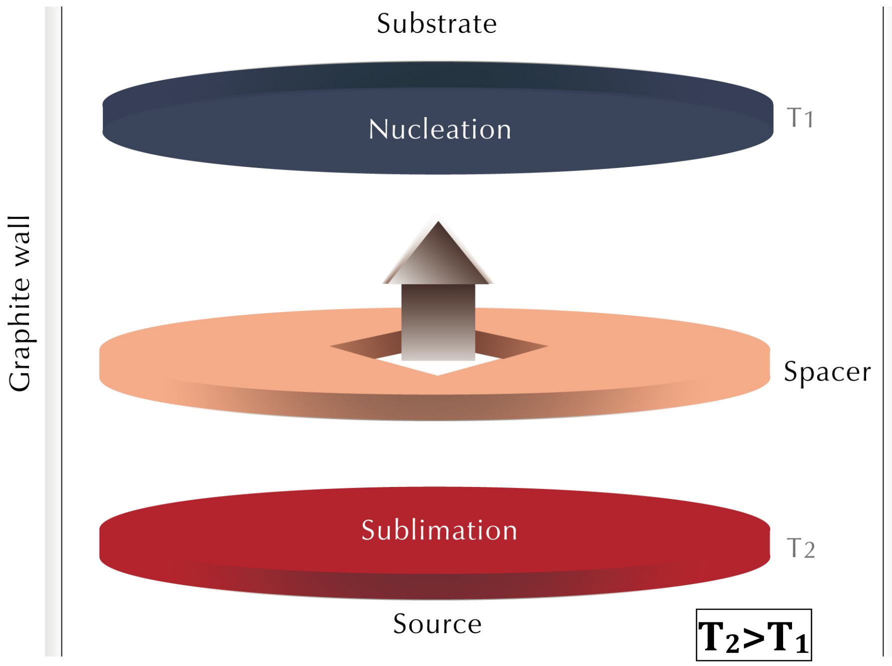


Fig. 4. The schematic layout of the sandwich-like arrangement inside the graphite crucible. The temperature gradient difference make the sublimation happened.

Affecting factors

There are lots of factors on substrate that affect the polytype formation, i.e. the morphology, the orientation of the surface (given by the angle when cutting). In addition, the growth conditions also play important roles on the polytype formation, i.e. the growth temperature, the growth time, the graphite geometry, the gas ambient, vapor composition (Si/C ratio), supersaturation at the growth front and the temperature gradient [20]. However, the Si/C ratio and supersaturation level are both influenced by the temperature and the temperature ramp-up rate. Therefore, these factors are all related and interact with each other to have some impact on the growth process. In the following section, these affecting factors and how they influence the growth conditions, experimentally as well as theoretically, will be discussed.

Growth temperature

The growth temperature is very important parameter in the growth, because it influences the process in many different ways. As mentioned before, the growth temperature is around 1750℃ to 1950℃, but it depends on what kind of polytype is required. For example, the formation of 3C-SiC needs higher growth rate, or higher temperature, than 4H-SiC to form an island on the terrace. This is also linked with the layer thickness. The adatom’s diffusion length will be longer so that the adatoms can move further on the terrace and reach a step to form a step flow growth when there is a higher temperature. However, the higher temperature also gives more energy so that adatoms can diffuse. It results in a higher possibility of an island to grow on a terrace if they have appeared to form the cubic SiC [21]. Therefore, there are several different factors that interact with each other and influence the growth. But, without exception, the heating-up rate should be slow and smooth. A sudden change of the heating-up rate will definitely raise the possibility to form defects and lower the epilayer’s quality. Generally, in FSGP when the temperature is increased 50℃, the growth rate is doubled.

Graphite plate

The graphite should be put on the top of the substrate. The graphite plate may influence the growth rate but most important is to prevent the substrate backside from subliming at a high growth temperature. It is particularly useful when growing thicker bulk like layers.

The growth rate will be influenced because the graphite plate increase the mass of the crucible's lid, which will have a larger volume to heat up and affect the temperature gradient within the crucible.

In the thesis, some of the series were with graphite and some without. The difference between adding graphite plate or not will be discussed.

Si/C ratio

In the growing process, when SiC sublimes, there will be some common constituents like Si, Si_2C, SiC_2. They have different activation energy and vapor pressures so they have different percentage when doing the mass transport. Silicon has a higher vapor pressure so it will sublimate fast and have the higher percentage in the ambient. Since there are so many components made by graphite in this lab set up, there could be excess carbon in the reactor and which will result in the phenomenon called "graphitization" [19]. Graphitization means that the surface of source is covered with carbon, which will lower the growth rate and block the sublimation to continue the growth. Therefore, to balance the ratio between Si and C it is very important to consider during the growth. Either one can introduce more silicon or decrease the carbon to control the balance of the Si/C ratio.

It has been shown that the higher of Si/C ratio have more close to the condition of nucleation of 3C-SiC because it is easier to get the inclusion to form the 3C island on a terrace at a high Si/C ratio [10].

To absorb the excess carbon, typically a tantalum foil is used as a C-getter by formation of TaC [22]. Tantalum C-getter has to be changed every ten times after use since its ability to absorb carbon is getting lower when it has been used many times. In our case, tantalum plate has been placed at the bottom beneath the source.

Substrates

There are different kinds of polytypes that can be used to grow the SiC epilayer. The difference stacking sequence in each polytypes substrate strongly related to the initial growth. The orientation, the cut off-axis angle are also the main factors to influence the initial growth. The cutting off-axis angles were usually chosen around low angles area with cut toward some specific direction, like [$11\bar{2}0$]. Comparing to the on-axis substrates, the 3C–SiC nucleate on off-axis substrate at even lower growth temperature since it does to need to compete with step-flow growth and it is easier to create inclusion 3C islands by two-dimensional growth [9]. Similarly, on a substrate, which have lower cutting off-axis angle, it is easier to form 3C-SiC by using the combination of two dimensional initial nucleation and extension by step flow growth. In the thesis, two different cutting off-axis angles, which are 2 degrees and 4 degrees, of 4H-SiC substrates were used and discussed.

5. Characterization Techniques

After the growth of epilayer, several characterized techniques have been used for studies of the surface morphology and the step structure. This is typically done in order to know how the growth parameters influence the quality of the epilayer and thereby to modify growth conditions, and in the long run improve the electronic devices. In this thesis, all samples and the morphology in each series have been characterized using the optical microscopy. Furthermore, some samples and their steps structures in the surface morphology have been compared by using atomic force microscopy (AFM).

Optical Microscopy

Optical microscopy with Nomarski differential Interference Contrast was used for the characterization of the morphology of the epilayers. The setup is like shown in Fig. 5. There are two modes in the microscopy; these are reflected light mode and transmitted light mode. A prism is used for splitting the incident polarized beam of light into two polarized beams by a distance equal to the resolution of the objective lens. These two beams continue travelling in slightly different directions until the beams hit the surface of the epilayer. In the reflected light mode, after the reflection on the surface, the beams will go through the prism again and recombine together. The morphology of the surface gives different optical path for the beams, which make the path to have a slightly different shift in distance, and an interference effect. After passing through the second polarizer above the upper beam-combining prism, our eyes can distinguish the depth and the morphology of the surface by different intensity and color [23]. The optical path differences in beams let the amplitude variations seem different in brightness and even small variations can thereby be observed in comparison to conventional microscopes.

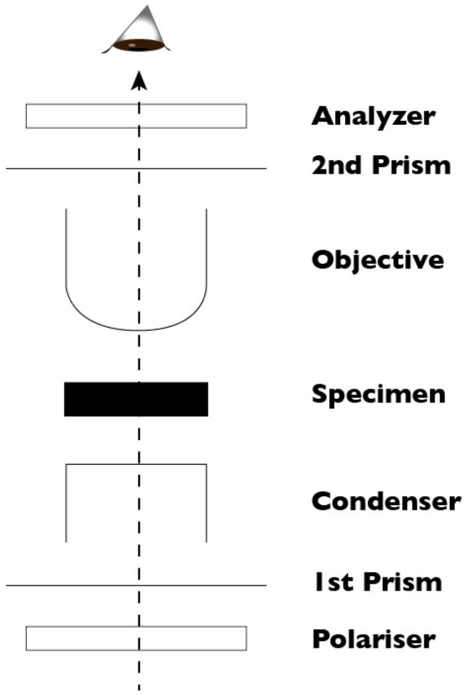


Fig. 5. The set up of the Optical microscopy with Nomarski differential Interference Contrast.

Transmitted light mode is used when the specimen is transparent. In transmitted light the illuminating light is passing through a polarizer located beneath the condenser and shown through the specimen by the condenser.

In transmitted light mode, the yellow color of 3C-SiC is clearly seen and useful to detect the polytype inclusions in comparison to hexagonal SiC. Thereby the locations, which have 3C-SiC on the surface of the epilayers, can be recognized, along with double positioning boundaries, where the other polytypes are transparent or greenish or brownish.
There are 4 different magnifications that were used, which are 50X, 200X, 400X and 1000X.

Atomic Force Microscopy (AFM)

The Atomic Force Microscopy provides a good way to observe the morphology of the surface in more details than in optical microscopy. It is non-destructive, high resolution, and only needs a simple pre-treatment of the sample. The AFM can scan the surface in an atomic level, which is in Ångström range.

The basic principle of the AFM is the interaction between the tip, which is really sharp at the end of the tip, on the cantilever and the sample surface. There are three modes in the AFM, which are contact mode, non-contact mode and tapping mode. In the thesis, all the characterizations of AFM were using the tapping mode.

By using the tunneling current and the Van der Waals forces, in the tapping mode the tip is not in contact with the sample. It oscillates at its resonant frequency and taps lightly on the sample when processing the scan. Constant oscillation amplitude is maintained between the tip and the sample, and then the information of the surface is recorded by the detector to reflect how the morphology of the surface is depicted. The tip is scanning at x-y axis line by line by the piezoelectric holder and acquires the 3D-images of the surface. There is only a very minor damage of the sample when using the tapping mode. The important information from AFM used in the thesis are the step height and the terrace width of the terraces, which help us to know more about the terrace formed by the off-axis substrate in which the 3C-SiC islands appear in the 4H-SiC epilayers that contain steps. This gives a deeper understanding about the step bunching and the step flow growth mechanisms in relation to stability and formation of 3C-SiC [19].

The disadvantages of the AFM are that the scan speed is slow, compared to conventional microscope; AFM can only observe small part area of the sample. The limiting factors of the AFM are the sharpness of the tip and the resolution of the detector [16].

6. Experimental Details

All samples in the series in this thesis were grown by using the method of fast sublimation growth process (FSGP), which has shown to be suitable in order to get thicker epilayers and high growth rate to fit the criteria of studying bulk like material by formation of 3C-SiC. The following show the process step by step and some details in the process.

Sublimation Process

The growth is driven by a temperature gradient. The positioning order of parts, from the bottom to the top of the crucible as a sandwich, in the sublimation growth set-up is Tantalum plate, source, spacers, substrate, graphite plate if needed on substrate backside.

The crucible should be placed inside the chamber carefully so that the sandwich is not moved, and thereafter start pumping until a vacuum of around 10^{-4} to 10^{-5} mbar is reached. After reaching the vacuum level and starting the cooling water, the heating process can start. The heating rate was following the recipe from run card from the previous experience to increase the initial growth ramp up properly to the desired growth temperature. The heating up rate is around 20K/min. When the growth temperature is reached, the growth time is defined to start. During the thesis, the growth took place at different temperature for different time periods. When the growth period is considered to be finished, the cooling starts after switching off the generator. The cool down process can be faster by introducing some Ar to the ambient. The cool down process usually took around one hour so that the sample can be loaded out.

Re-sublimation Process

In the graphite parts there can be some deposition that is unwanted for reusing the graphite parts. There is a need to remove and clean the sublimation deposition that is on the crucible and the spacers using process of re-sublimation. This is needed every time after each growth preventing any undesired influences on the next growth, for example a change of temperature gradients given by deposited SiC.

The temperature was held at around 2000°C for 30 min.

Outgassing Process

Between growth series there is a procedure to have an outgassing process, which is necessary to keep good conditions of the machine and chamber, and mainly the graphite parts. After this typically a reference run is done in case there would be an influence of any contamination, or monitor any unintentional shift of the set-up that could result in the instability or change of the growth rate. Normally, the outgassing is done at a processing temperature of 2000°C for 15 min.

Samples Series List

The following tables list three parts of series done within the thesis. All the sources are 3C-SiC made from the company Mitsui Engineering. The substrates are all 4H-SiC having two different off-axis angles.

Part I

Substrate off-axis angles	Temperature(°C)					
	1700°C	1750°C	1800°C	1850°C	1900°C	1950°C
2 degrees	■	■	■	■■		
4 degrees			■	■	■	■

Table. 2.1. The sample series of 3C stability on 4H-SiC (4 degrees and 2 degrees) off-axis substrates and a growth time is 30 minutes without graphite plate on the backside of substrates.

Part II

Growth Time (min)	Temperature(°C)		
	1850°C	1900°C	1950°C
30 min	■	■	■
45 min			■
60 min			■

Table. 2.2. The sample series of 3C stability on 4H-SiC (2 degrees) off-axis substrates with graphite plate on backside of substrates.

Growth Time (min)	Temperature(°C)			
	1800°C	1850°C	1900°C	1950°C
60min	■	■	■	■
30min				■

Table. 2.3. The sample series of 3C bulk like layers on 4H-SiC (4 degrees) off-axis substrates with graphite plate on backside of the substrate.

Part III

Growth Time (min)	Temperature(°C)				
	1700°C	1800°C	1850°C	1900°C	1950°C
30min	■	■	■	■	■
60min					■

Table. 2.4. The sample series of 3C bulk like layer on 4H-SiC (4 degree) off-axis substrates with graphite plate on the backside of the substrate and a new crucible.

7.Results and Discussions

All the series done in the thesis were listed in the previous section of samples series list (Table 2.1- Table 2.4). The results and discussions of these can be divided into three parts. (I) To find a critical growth parameters for the formation of 3C-SiC on 4 degrees and 2 degrees 4H-SiC substrates, (II) Optimal growth parameters for the stable growth of bulk like 3C epilayers on 4 degrees and 2 degrees 4H-SiC substrates. (III) A model of 3C-SiC lateral enlargement on 4H-SiC off-axis substrate. All the series have been done in the dynamic vacuum, which means that the reactor was constantly pumped out during the entire growth process.
The characterization section includes surface characterization by optical microscope and AFM.

Part I. To find critical growth parameters for the formation of 3C-SiC on 4 degrees and 2 degrees 4H-SiC substrates

Previously, 3C-SiC growth has been performed on 6H-SiC on-axis substrates, or on 6H-SiC with a low off-axis orientation angle. However, it is not clear which polytype is preferable for 3C-SIC growth on off-axis substrates. The purpose of this series is to know the critical temperature, or growth rate, for stabilization of 3C-SiC on off-oriented 4H-SiC substrates. Various temperatures were applied to determine experimentally the critical growth parameters since the growth temperature is the main parameter in FSGP that affects growth rate and supersaturation level. Now two different off-axis angles' substrates are discussed separately.

a. Transition temperatures for 4H/3C transition on 4 degrees 4H-SiC substrate

The result of this series is shown and arranged below in Table 3. The increase in growth temperature resulted in an increased growth rate and larger surface coverage with 3C-SiC. This is shown in Fig, 6. Of which the cases 6(b) and 6(c) are discussed later. In the optical microscope, or even visible to the eye, the 3C-SiC looks yellow and the 4H looks transparent. Therefore, the expansion of the 3C coverage can easily be distinguished, either by eyes, or using the transmitted light mode in

OM for more details. Based on experimental results, the formation of 3C-SiC is more favorable at higher temperatures while lower temperatures are more favorable for the 4H-SiC homoepitaxial growth.

By increasing the growth temperature, the growth rate increases and the supersaturation on step terraces also increases. Therefore, at higher supersaturation level the probability to form 2D nucleation of 3C-SiC on step terraces increases.

<u>4 deg-4H-SiC substrate</u>

Growth Temperature (˚C)	1800	1850	1900	1950
Thickness (μm)	46.6	155	177	365
Growth Rate (μm/h)	93.2	310	344	730
Growth Time (min)	30	30	30	30

Table. 3. Growth temperatures for exploring cubic SiC on 4 degrees 4H-SiC substrates.

In Fig, 6(a), which is the sample grown at the lowest temperature, 1800°C, of the series, there are very few cubic domains that have formed on the substrate. The sample is dominated by 4H-SiC. The typical features of 3C-SiC are triangular inclusions to recognize the 3C-SiC in the off-axis substrates as shown at Fig, 7(a) and (b).

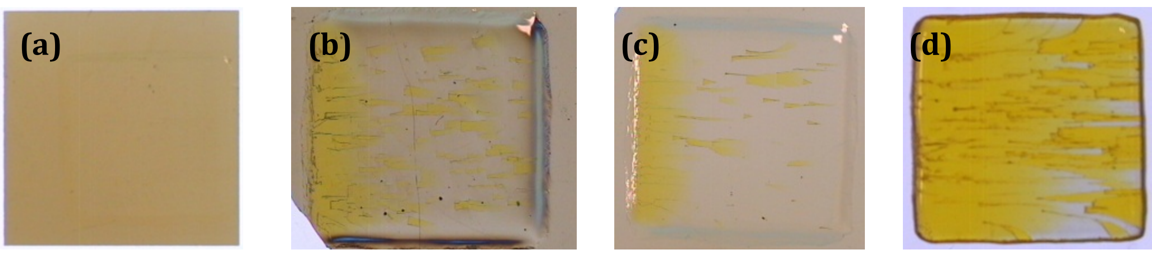


Fig. 6. The 3C coverage on the series of 4 deg-4H-SiC substrate in different temperature.

(a) 1800˚C (b) 1850˚C (c) 1900˚C (d) 1950˚C

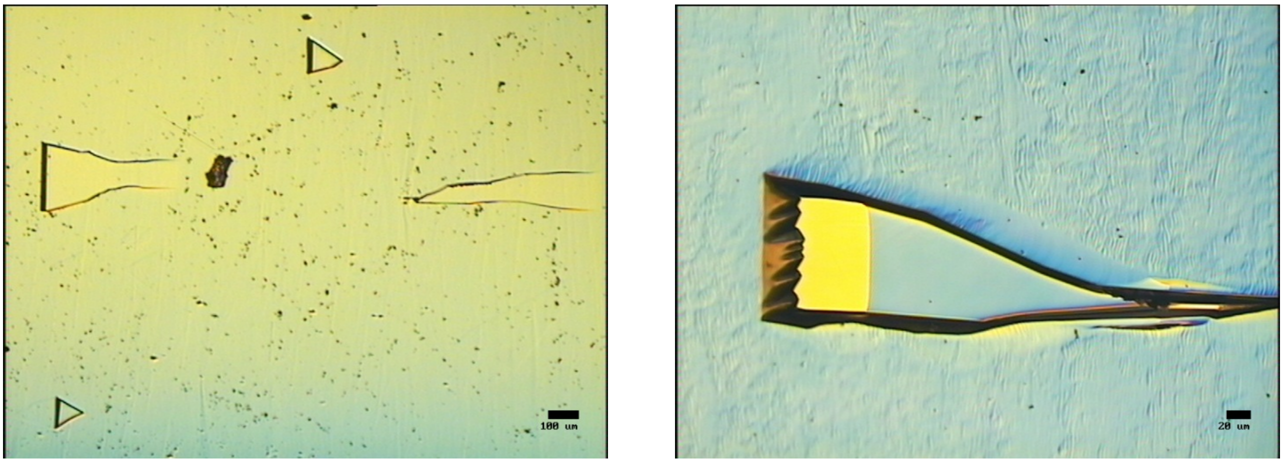
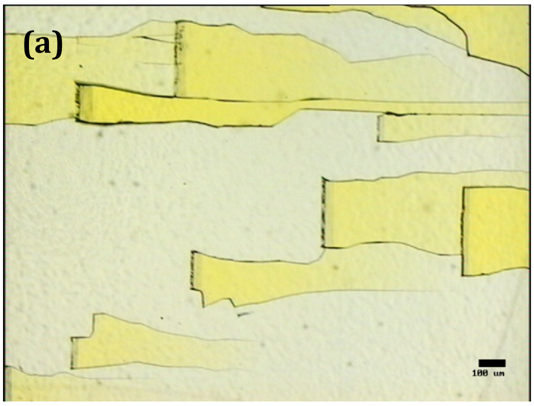


Fig. 7. The 3C triangular inclusion in growth on 4 deg-4H-SiC substrate at (a) 1800°C taken at the 50X enlargement and in reflection mode OM. The scale at the right corner is 100 µm (b) 1900°C taken at the 200X enlargement and in reflection mode OM. The scale at the right corner is 20 µm.

In the Fig, 6(b) and 6(c), as the temperature increases by steps of 50°C, the coverage of 3C is expected to be higher. There are several different factors that influence the growth, not only the temperature, but the supersaturation, substrate condition and number of defects etc. Due to a higher density of surface defects in sample shown in Fig 6(b), the number of 3C-SiC inclusions in the center of the epilayer is higher than in the sample shown in Fig 6(c). Surface defects disturb the step flow growth and create a long terrace, which forms at the defect position and extends along the step flow growth direction (Fig. 7). Therefore, the difference between the 6(b) and 6(c) may not so obvious even the temperature was increased by 50°C. Also, the growth rates are similar and thus suggest that Ta foil or any other geometry has influenced growth. In any case, there is obviously a transition region in the temperature regime and this was used for the further studies of following series. Most important is to find the conditions for full coverage of 3C-SiC, and thereby a high growth temperature was preferred in further runs.

In general, we can observe two initial growth stages on 4 degrees off-axis substrates. The first one is found at the edge of the samples where the 3C-SiC forms and extends in the down step direction, which is also step-flow growth direction. In the second case the 3C-SiC forms in the central region where the homoeptaxial growth could be expected. In Fig 8(a), the expanding of 3C that has nucleated in the central region is shown using the sample grown at 1850°C. Clearly, there is large 3C-SiC domains but these do only weakly link with each other. Hereby one can expect that there will be deep grain boundaries in this case. In comparison, the nucleation at the edge shows an extension of the domains once the 3C-SiC has formed at the edge. In the transmitted mode, the surface is covered with 3C inclusions with quite large domains, as shown in Fig 8(b).



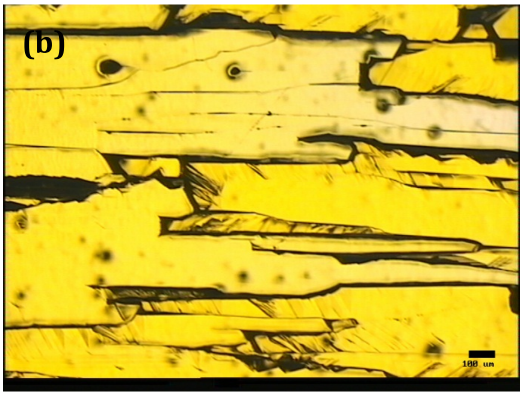


Fig. 8. The expanding of 3C triangular inclusion on 4 deg-4H-SiC substrate at (a) 1850°C with inclusion in the central region; (b) 1950°C taken at the X50 enlargement in transmitted mode OM with large domains that formed at the edge. The scale at the right corner is 100 µm in both images.

To conclude this part, for the epilayers grown on the 4 degrees off axis 4H-SiC substrates, the critical temperature of 4H and the 3C structure is around 1800°C, at which we have a growth rate around 93 µm/h. It indicates that the favorable temperature or the growth rate for the 3C-SiC will be above this temperature (growth rate), so the 3C will start to form and expand. It is dominating at 1950°C, where all the surface is covered by the cubic SiC by 3C-SiC which is formed at the edge and expanding in the down step direction.

b. Critical temperatures for 4H/3C on 2 deg-4H-SiC substrate

In general, the 2 degrees off-axis angle substrates have longer step terraces compared to 4 degrees substrates. This indicates 3C-SiC can form easier and at lower temperature. It can therefore be expected that there is a shift of critical temperature to lower for polytype transition in comparison to 4 degrees substrates. At the same growth conditions there should be a higher probability to form 3C-SiC on 2 degrees substrates.

The growth results are shown in Table 4. Similarly to the 4 degrees case, the surface coverage with 3C-SiC increases by increasing the growth temperature (Fig. 9). Apparently, the homoepitaxial growth is dominating at the lowest growth temperatures. The difference of 9(a) and 9 (b) can been seen more clear in Fig. 10. The triangular 3C domain start to nucleate on the terraces at 1750°C, shown in Fig 10(b), and the 3C domains do not exist at 1700°C as shown in Fig (10(a). The round-hole defects could be some graphite inclusions, but these are not expected to be linked with the 3C-SiC nucleation on terraces.

2 deg-4H-SiC substrate

Growth Temperature (°C)	**1700**	**1750**	**1800**	**1850**
Thickness (μm)	7	22	51	177.5
Growth Rate (μm/h)	14	44	102	255
Growth Time(min)	30	30	30	30

Table. 4. Growth temperatures for growth on 2 degrees off-oriented 4H-SiC substrates.

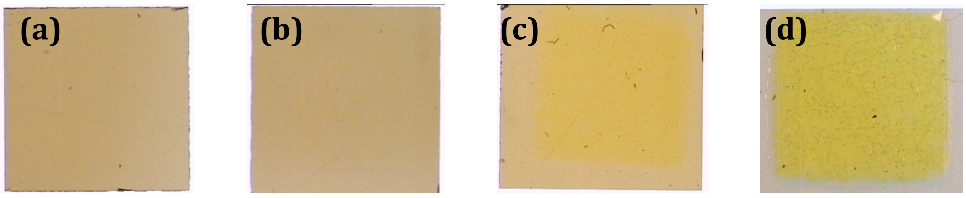


Fig. 9. The 3C coverage on the series of 2 deg-4H-SiC substrates in different temperatures.

(a) 1700°C (b) 1750°C (c) 1800°C (d) 1850°C

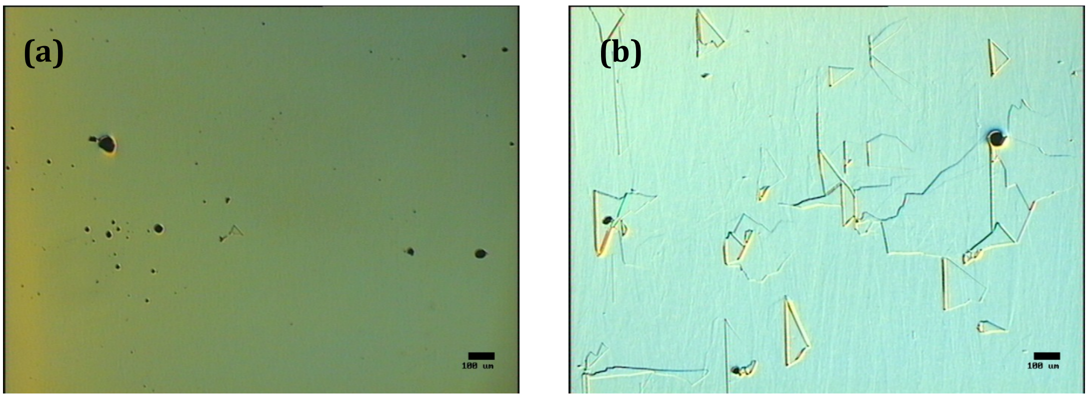


Fig. 10. The 3C domains on the series of 2 degree off-oriented4H-SiC substrates in (a) 1700°C (b) 1750°

taken in the X50 magnification in reflection mode. The scale at the right corner is 100 μm.

For the epilayers grow on the 2 degrees off-axis 4H-SiC substrates, it can be concluded that the critical temperature for 4H/3C transition is between 1700°C-1750°C at which the growth rate is between 14 to 44 μm/h. At temperatures above this range, the 3C-SiC will start to grow and be dominating until it covers all of the substrate, like exemplified of the sample grown at 1850°C depicted in Fig, 11. In conclusion, the growth on 2 degrees off-oriented substrates is dominated by 3C-SiC formation in the central region of the substrate.

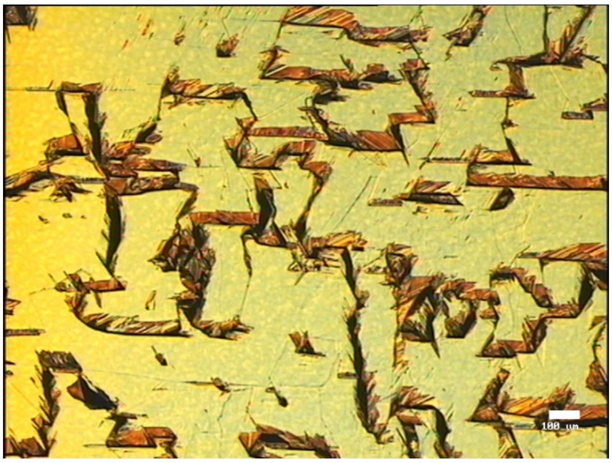


Fig. 11. 3C-SiC start to cover all over the substrate in growth at 1850°C on 2 degree off-oriented4H-SiC substrates. The common defects formed on the boundaries between the domains are clearly shown in the image taken at 50X magnification optical microscopy in reflection mode.

Growth Rate

From the result of above series, it can be seen that generally the growth rate increased with the growth temperature. From previous experience, there is a trend that the growth rate almost doubles by increasing growth temperature by 50°C.

Comparing these two different angles of off-axis substrate, at similar temperatures it is observed that the growth rate is similar. As Fig. 12 shown, in agreement with previous experience that the sublimation of the source is the rate-determining step, we can see that the off-axis cut angle of the substrate is not a factor for influencing the growth rate. On the other hand, the growth rate and growth temperature have a profound influence on the polytype formation of 3C-SiC.

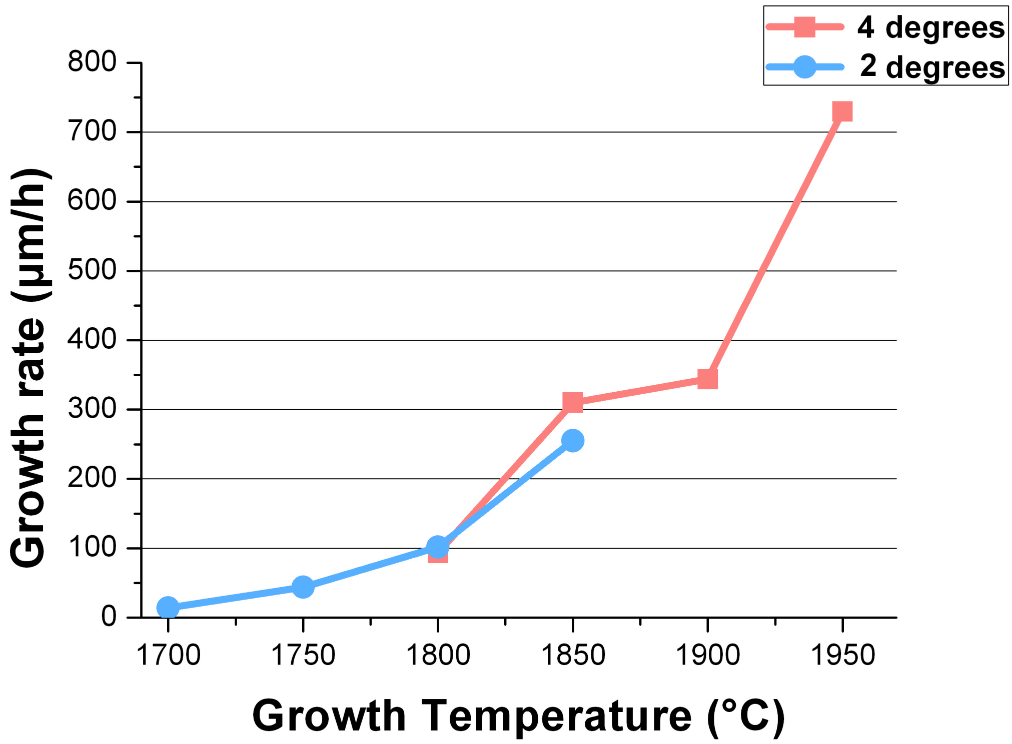


Fig. 12. The comparison of the growth rate between 4 degrees off-oriented 4H-SiC substrate and the 2 degrees off-oriented 4H-SiC substrate in the part I series.

Part II. Optimal growth parameters for the stable growth of bulk-like 3C epilayers on 4 degrees and 2 degrees 4H-SiC substrates

The purpose of this series, in which the growth temperature is maintained at 1950°C to apply a high growth rate, and the growth time is increased from 30 to 60 minutes at this temperature to have extremely thick layers that are bulk like, is to investigate the stability of the 3C-SiC and further explore the optimal criteria for coverage and domain size. In this section we explore how to achieve large and few domains with high coverage of bulk like 3C-SiC. The underlying idea is that if the quality is good then the thickness can be increased by continued growth while maintaining a good quality of the epilayer. In this way we can possibly initiate bulk-like 3C-SiC.

The off-axis angle and the supersaturation level, which are linked to the growth temperature, affect the 3C-SiC domains size. The influence of these parameters was investigated for the growth of thick 3C-SiC layers. A use of higher growth temperature, when the 3C-SiC starts to dominate, can give a higher coverage of 3C-SiC. However, a higher growth temperature also means that there could be more nucleation sites will form during the initial stage of growth. It may lead to a large number of small domains and higher the possibility to get more defects between the domain boundaries. On the other hand, a high growth temperature could also be beneficial for enhanced kinetics that will enlarge the domains.

The factors like temperature, growth time, and the graphite plate placed on top of the substrate were investigated for the growth 3C-SiC bulk like material on 2 degrees and 4 degrees off-axis angle 4H-SiC substrates.

The graphite plates were used in order to diminish backside sublimation of the substrate during the growth at high temperatures at which the substrate actually could sublime away totally. The influence of the graphite plate will also be discussed in this section.

Growth Temperature

Different temperatures were tested to reconfirm the relation of growth temperature and the growth rate is maintained at 1950°C. The result is show in Table 5. As mentioned before, when the temperature is getting higher the growth rate increases. As shown in Fig. 13, the growth rate increases almost twice times with the increase of temperature by 50°C.

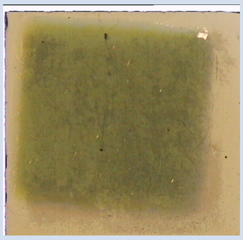
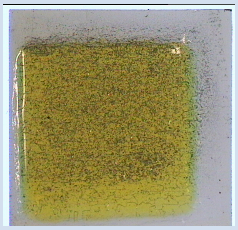
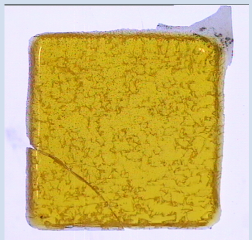
Growth Temperature (°C)	**1850**	**1900**	**1950**
Thickness (µm)	116	244	401
Growth rate (µm/h)	232	488	802
Growth time (min)	30	30	30
Graphite plate	Yes	Yes	Yes
Overview			

Table. 5. The experimental result of how growth temperature influenced the growth rate and polytype formation for growing the cubic bulk like material on 2 degrees 4H-SiC substrate.

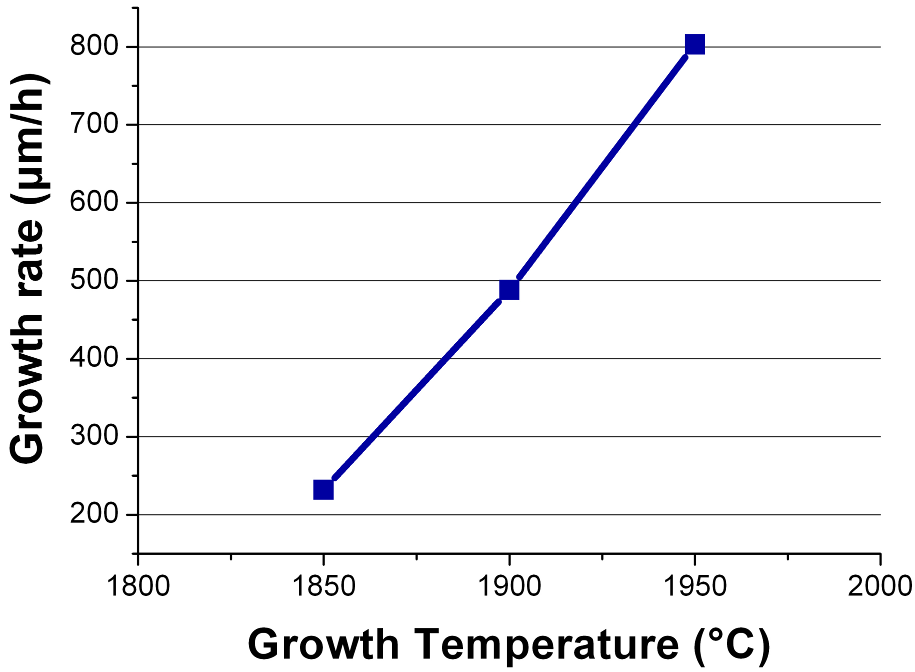


Fig. 13. The growth rate goes double when the temperature increase 50°C as expected in the growth time 30 minutes.

Clearly, there is an increase of 3C-SiC by increasing the temperature. Note that the growth is given by 3C-SiC formation and stability of domains that formed in the central part of the substrate at a temperature of 1950°C which is at a growth rate of 800 µm per hour. This growth rate is similar, or even higher, than the growth rate applied in bulk growth of hexagonal SiC.

Growth Time

In order to grow the epilayer to be more close to bulk like material of 3C-SiC, the growth time was increased to know the influence of growth time. The result is shown in Table 6. However, when increasing the temperature, there might be some problems that arise. Since a longer growth time at a higher temperature is applied, the most serious one is the graphitization of the source, which slows down or terminates the growth by blocking the sublimation of the source. The C atoms cover the surface of the source so that the Si atoms and other species involved in the sublimation could not sublime and move to the substrate. The graphitization of the source may also deteriorate the quality of the epilayer. As a result of graphitization, the growth rate and thickness will change unpredictably, even at the same temperature, as the Fig. 14 shows.

From the Table 6, in the 30 minutes growth time case, there is no graphitization, so the growth rate is at around 800 μm/h as expected. When the time was prolonged to 45 minutes, there was a severe graphitization of the source as seen in Fig. 15, the image taken after the process. Therefore, the growth rate decreased to almost 40% of the normal growth rate. In the 60 minutes growth case, the graphitization of the source was not as serious as in the 45 minutes case, but still occurred and resulted in the growth rate reduction to almost 86% of the normal growth rate. The level of graphitization depends on the substrate, growth conditions and so on. Until now, it is still hard to avoid and predict at very high growth rates.

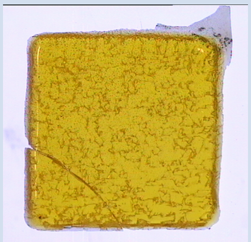
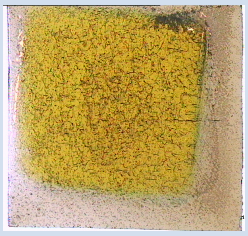
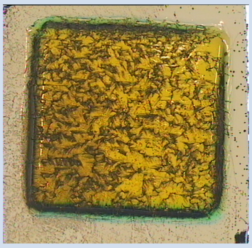
Growth time(min)	**30**	**45**	**60**
Growth Temperature (°C)	1950	1950	1950
Thickness (μm)	401	230	694
Growth rate (μm/h)	802	307	694
Graphite plate	Yes	Yes	Yes
Overview			

Table. 6. Growth time influence on the growth process at the same growth temperature for growing the cubic bulk material on 2 degrees 4H-SiC substrate.

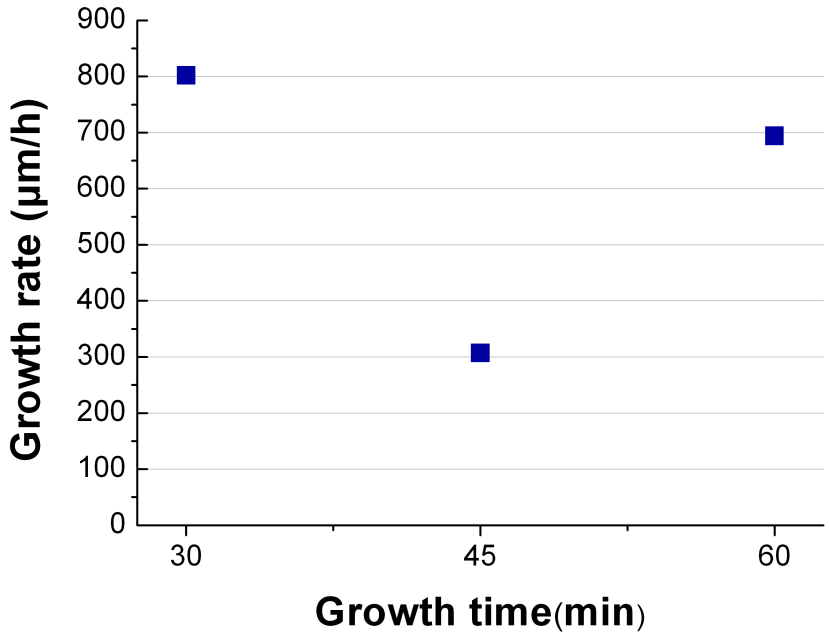


Fig. 14. The growth rate at the same temperature, 1950°C, changed unpredictably when the source started to graphitized.

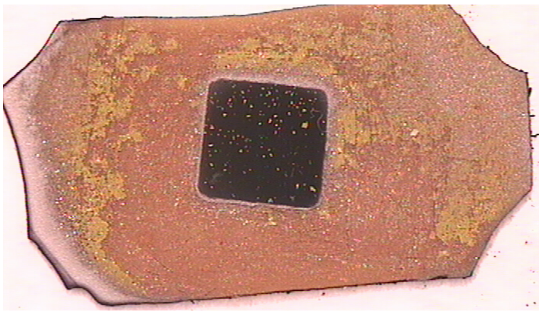


Fig. 15. The source was graphitized for the growth at 1950°C for 45 minutes with graphite plate on the 2 degree off-oriented 4H-SiC substrate

Graphite plate

As mentioned previously, the graphite plates were used to protect the substrate from subliming away during the high temperature growth. Therefore, the series here were tested to know the influence of the graphite plate.

As seen in Table 7, the growth rate went down when the graphite plate was added. This probably occurs because the graphite plate lowers the temperature gradient between the source and the substrate. From the results, the graphite plate lowers the growth rate at the same temperature.

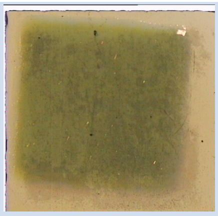
Graphite plate	**No**	**Yes**
Growth Temperature (°C)	1850	1850
Thickness (μm)	177	116
Growth rate (μm/h)	355	232
Growth time(min)	30	30
Overview		

Table. 7. Graphite plate influence the growth for growing the cubic bulk material on 2 degrees off-oriented 4H-SiC substrate.

Comparison of 2 degrees 4H-SiC substrate and 4 degrees 4H-SiC substrate

As discussed before, the off-axis angle is not a deciding factor for the growth rate. However, the off-axis angle influences the morphology, domains size and also the polytypes it formed.

The Domain size and defects

As shown in Fig. 16, the domain size in 4 degrees off-oriented 4H-SiC substrate is much larger than the 2 degrees one. Furthermore, the number of defects in 2 degrees off-oriented 4H-SiC is much higher than in the 4 degrees case.

Because of the cutting angle, in 2 degrees substrates, the number of steps is fewer and the surface is more flat than 4 degrees substrate. This means that there are longer step terraces on 2 degrees substrates, which are preferable for 3C-SiC to form. At the same temperature and the growth rate, the 3C-SiC coverage, and the number of nucleation sites will be higher in 2 degrees substrates. More nucleation sites mean that more small size domains were formed. During the growth some of these domains merge having different rotation on the (0001) plane, the domains scompete and form double positioning boundaries. Such defects severely degrade the crystalline quality.

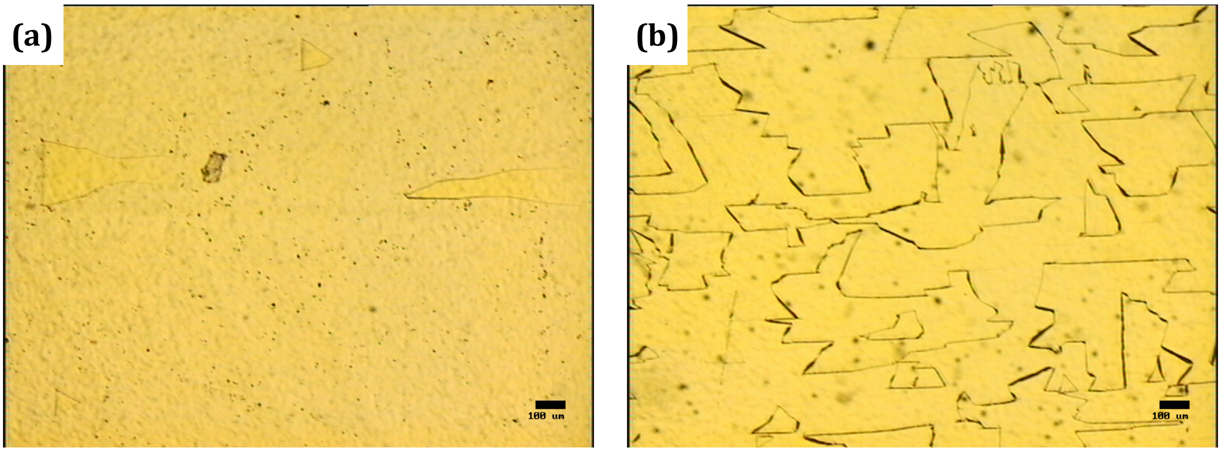


Fig. 16. The comparison between the growth at 1800°C on (a) 4 degrees 4H-SiC substrate and (b) 2 degrees 4H-SiC substrate. The domain size and the number of defect are different due to the off-axis angle being different. The images were taken at 50X in transmission mode in OM. The scale at the right corner is 100 µm.

Part III. A model of 3C-SiC lateral enlargement on 4H-SiC off-axis substrate

A model of 3C-SiC formation on off oriented substrates is suggested. The 4 degrees of 4H-SiC substrates were used in this case. The graphite plate was used to prevent the sublimation of the substrate at high temperature. Four growth temperatures were used in the series, which are 1700°C, 1800°C, 1900°C and 1950°C. The growth time is 30 minutes for all the temperatures, one extra growth time, 60 minutes, at 1950°C was tested to get thicker layer toward a bulk epilayer.

The growth of the 3C-SiC epilayer on 4H-SiC off-axis substrate started from the edge and expanded along the <11$\bar{2}$0> step-flow direction. Three 3C-SiC formation steps were identified: i) formation of a large terrace with a basal plane area at the edge of the sample, ii) 3C-SiC nuclei formation on the basal plane area, iii) lateral enlargement of 3C-SiC domains along the step flow direction [24]. *(Fig 17)*

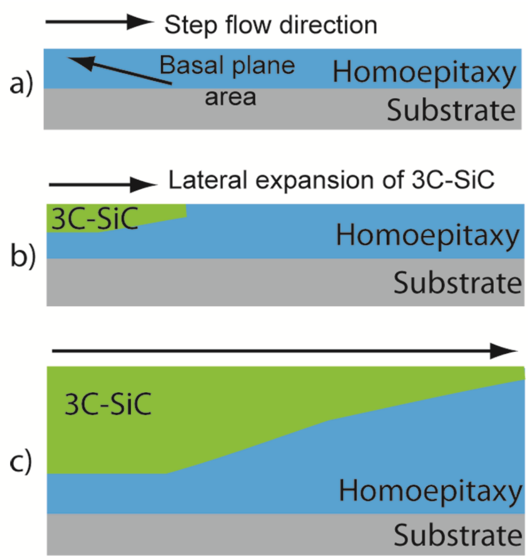


Fig. 17. Three main steps when the 3C-SiC starts to grow on the hexagonal off-axis substrate.(a) formation of a large terrace with a basal plane area at the edge of the sample, b) 3C-SiC nuclei formation on the basal plane area, c) lateral enlargement of 3C-SiC domains along the step flow direction [24].

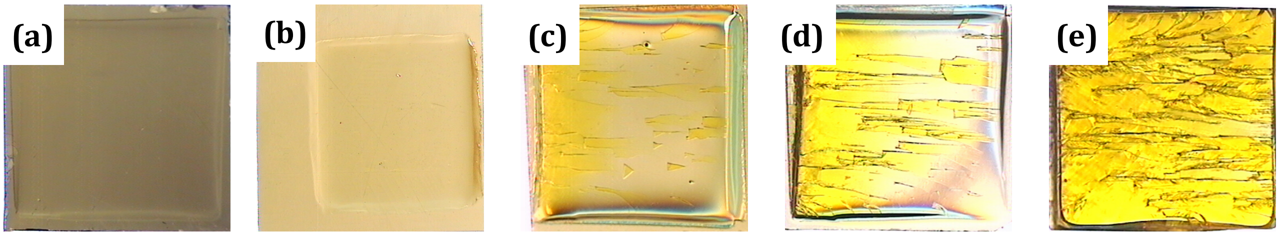


Fig. 18. The overview of the samples which grow at (a) 1700°C, 30min. (b) 1800°C, 30min. (c) 1900°C, 30min. (d) 1950°C, 30min. (e) 1950°C, 60min.

With the temperature increasing, the thickness of the grown-layer increases and the coverage of the 3C-SiC is also expanding along the step-flow direction $\langle 11\bar{2}0 \rangle$. The overview of the samples are shown at Fig.18.The lateral expansion distance of the 3C-SiC, which is X as shown in the Fig. 19, on the epilayer can be estimated by equation (1) if the thickness of the layer is known from the point when the basal plane is formed.

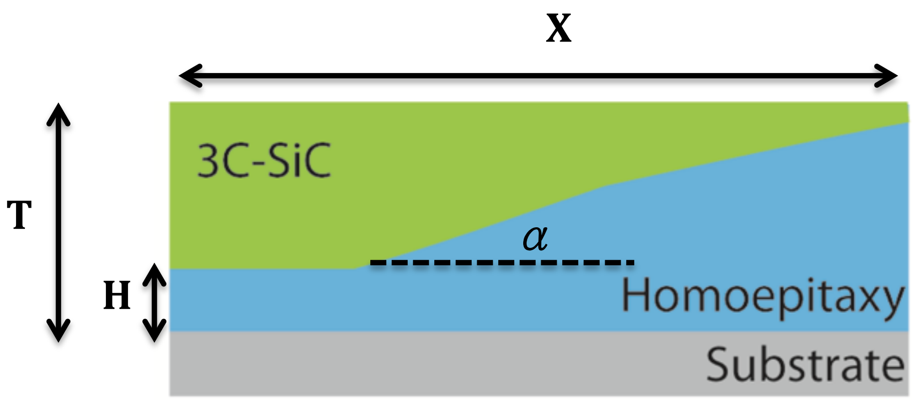


Fig. 19. The cross-sectional view of the grown 3C-SiC epilayer on the substrate.

$$X = \frac{(T - H)}{\tan \alpha} \quad (1),$$

In the formula, *T* represents the total thickness of grown layer, *H* represents the thickness at which basal plane area is formed and *α is* the off-axis angle, which is 4 degrees in the case. The H is around 60 µm in the growth of 3C-SiC on 4 degrees off-axis 4H-SiC substrates with the 3C-SiC lateral enlargement. The results of the series, which correspond to the Equation (1), are shown in Table 9 and Fig. 20. At a growth temperature of 1950°C, the thickness of the bulk like epilayer reach around 700 μm with 3C-SiC covering all the surface as shown in Fig. 20 (e). The thickness of the total grown layer varied from 17 μm to 350 μm at the 30 minutes growth time case, and around 800 μm at the 60 minutes growth times case at 1950°C.

Growth Temperature(°C)	1700	1800	1900	1950	1950
Total Thickness T (μm)	17.5	47.2	260	324.25	618.1
Growth rate (μm/h)	35	94.4	520	648.5	618.1
The theoretical lateral expansion distance X (mm)	0	0	2.86	3.778	7.97
The real lateral expansion distance X (mm)	0	0	2.75	3.72	7.0
Growth time (min)	30	30	30	30	60

Table. 9. The results of the series were compared the lateral expansion distance in each growth condition by the equation (1).

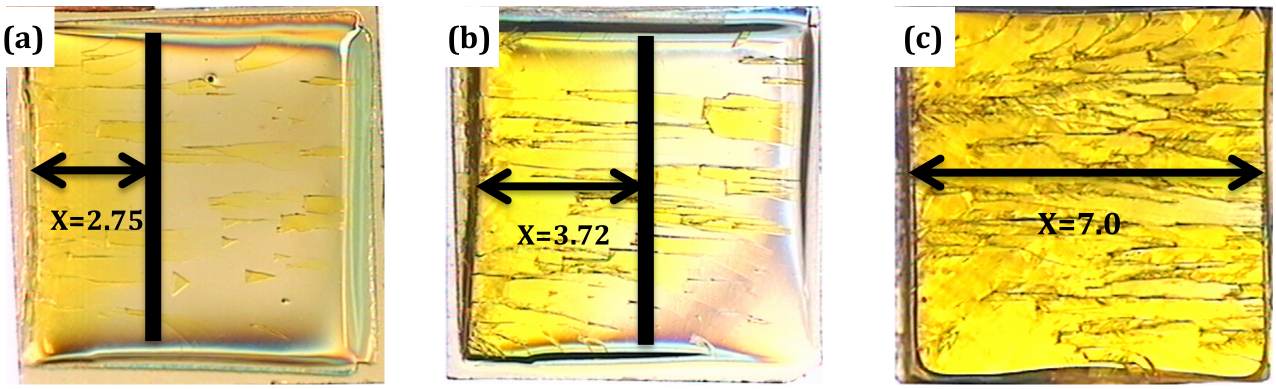


Fig. 20. The expanding of the 3C inclusion from the edge, regardless of the secondary 3C which formed in the center of the sample, at the conditions (a) 1900°C, 30 mins. (b) 1950°C, 30 mins . (c) 1950°C, 60 mins.

Normally, the 3C-SiC was considered to grow preferably on on-axis substrates, which have flat surface preferable for 3C-SiC formation. However, the growth on the on-axis substrate has problems like low quality epilayer and the small domain size of the 3C inclusions. This series shows that 3C can be grown even on substrates having as high as 4 degree off-axis angle and form bulk like layers with few and large domains.

Characterization

Atomic Force Microscopy (AFM)

Two groups of samples, which are 2 degrees off axis angle substrates and 4 degrees off axis angle substrates, were chosen for characterization using Atomic Force Microscopy (AFM). Within these two different off-axis angles, there are samples, which have no or almost none 3C inclusions, and the samples with some initial growth of 3C were chosen. The purpose is to check the terrace width and the step height of the 4H-SiC part without 3C-SiC and compare to the step height of the 4H-SiC part in the one with initial 3C formation. Thus we can have more information about nucleation at the center of samples. Table. 10 shows the conditions and the results of the samples.

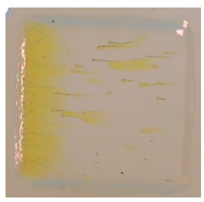
Substrate	**2 degrees SiC**		**4 degrees SiC**	
	No 3C inclusion	**Initial 3C inclusion**	**No 3C inclusion**	**Initial 3C inclusion**
Growth Temperature (°C)	1700	1750	1800	1900
Thickness (µm)	7	22	46.6	177
Growth rate (µm/h)	14	44	93.2	344
Growth time(min)	30	30	30	30
Average Terrace Width(nm)	190	245.9	127.22	153.49
Average Step Height(nm)	6.53	7.89	6.21	6.69
Overview				

Table. 10. The result from AFM measurements for the chosen series with measured average terrace width and step height.

For these four samples, the magnification in 2x2 µm, 5x5 µm, 10x10 µm and 20x20 µm images were taken in AFM. The steps height and the terrace width of 4H-SiC part of epilayer were measured. Around 30 points were measured for each magnification and the average values were calculated.

Average Terrace Width

Comparing two samples in the case of 2 degrees off-axis SiC substrates, as expected, the sample which have the initial growth of 3C-SiC have longer terrace width. The longer terrace width provides a larger flat area on which adatoms can form 3C-SiC inclusion by 2D nucleation. In these two sets of samples with different off-axis surface, the 50 degrees temperature difference caused that the criteria for critical 3C formation to be slightly different. The higher the growth temperature, the higher the level of the supersaturation and the higher the growth rate is. This results in a serious case of step bunching. The growth rate and the supersaturation level are important parameters for the step bunching on low off-axis substrate. The step bunching effect causes the faster growth terrace to merge together with the slower growth terrace, resulting in a longer terrace width. In AFM, the Fig. 21 shows even and straight steps flowing along the step-flow growth direction and with step-bunching. Some uneven and non-straight of the steps are an indication of step bunching due to the different step-flow lateral growth.

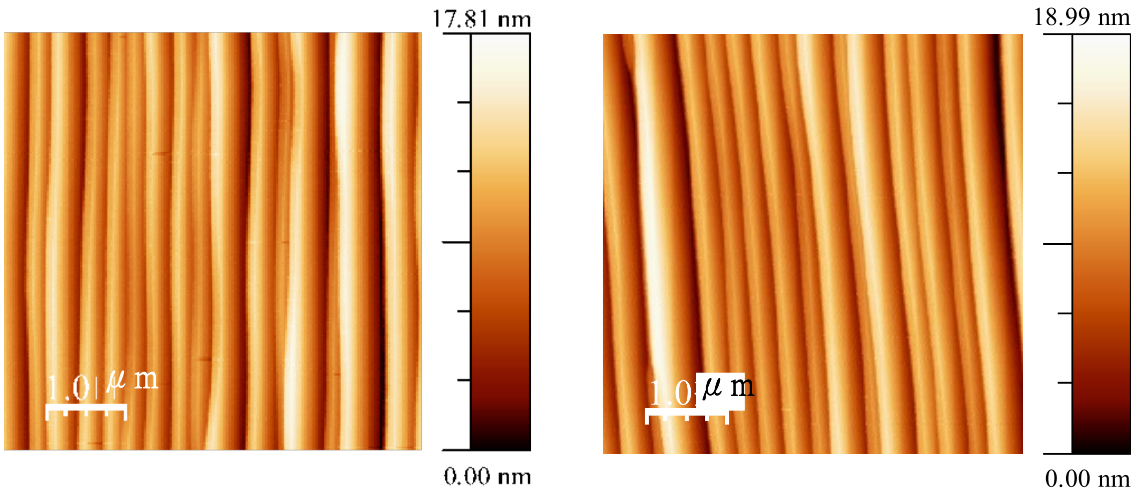


Fig. 21. The AFM pictures show the different terrace width of two samples in 2 degrees off-axis at (a) 1700°C, where there are no 3C inclusions. (b) 1750°C, where the 3C inclusions start to form. The images were taken in AFM at the magnification 5x5 µm. The scale at left corner is 1 µm.

A similar situation happens in the case of 4 degrees off-axis angle substrate, the terraces width are larger in the case where there are more 3C inclusions due to the step-bunching effect.

The relation between the off-axis angle, growth temperature and the average terrace width is shown below in Fig. 22.

When two off-axis angle substrates were compared to each other, because of the geometry of the substrates, the terrace width of 2 degrees off-axis angle substrate is longer than the 4 degrees off-axis angle substrate. The higher the off-axis angle while cutting, the number of the steps is higher and the steps become narrower, so that it is a natural geometry reason that the terraces width are shorter. The tendency is maintained during growth and step-bunching at different growth temperatures.

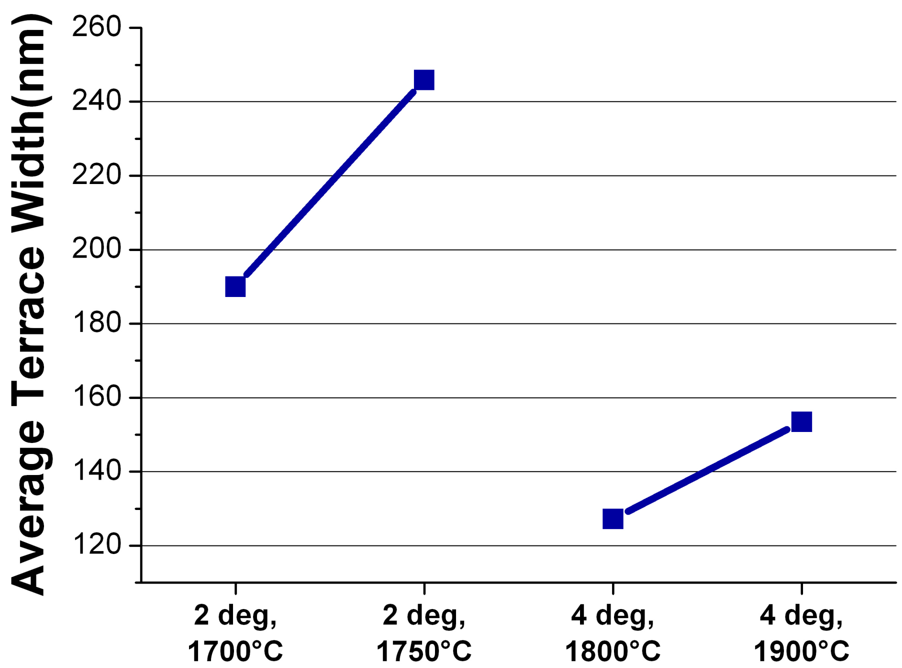


Fig. 22. The relations between the off-axis angle, growth temperature and the average terrace width.

Average Step Height

The relations between the off-axis angle, growth temperature and the average step height are shown in Fig. 23.

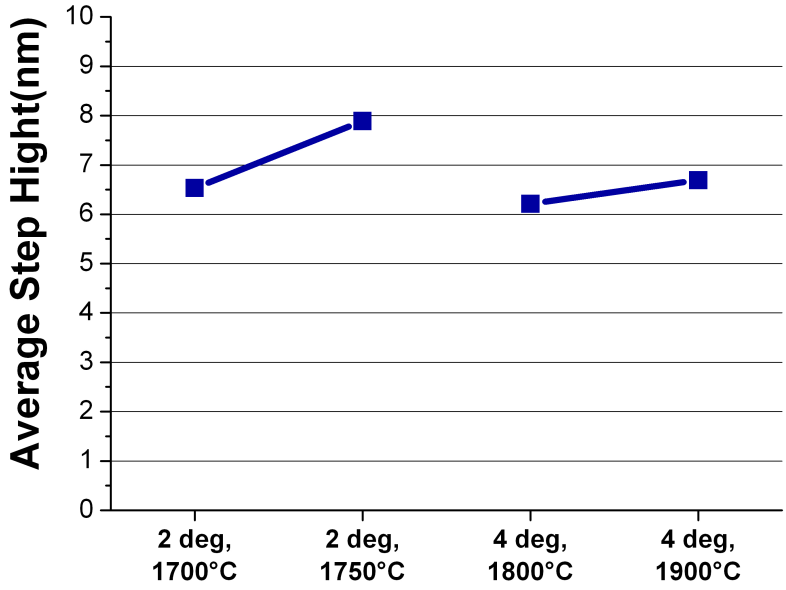


Fig. 23. The relations between the off-axis angle, growth temperature and the average step height.

As the step bunching started to influence the sample in which the 3C-SiC inclusion start to nucleate in both 4 degrees and 2 degrees case, not only the terrace width become longer but the step heights become higher as two or more steps merge together. But in some case the step bunching seems disturbed, as observed in the step with two peaks and uncompleted edge from A point to B point in the epilayer in the morphology-measured figure from AFM shown in Fig. 24.

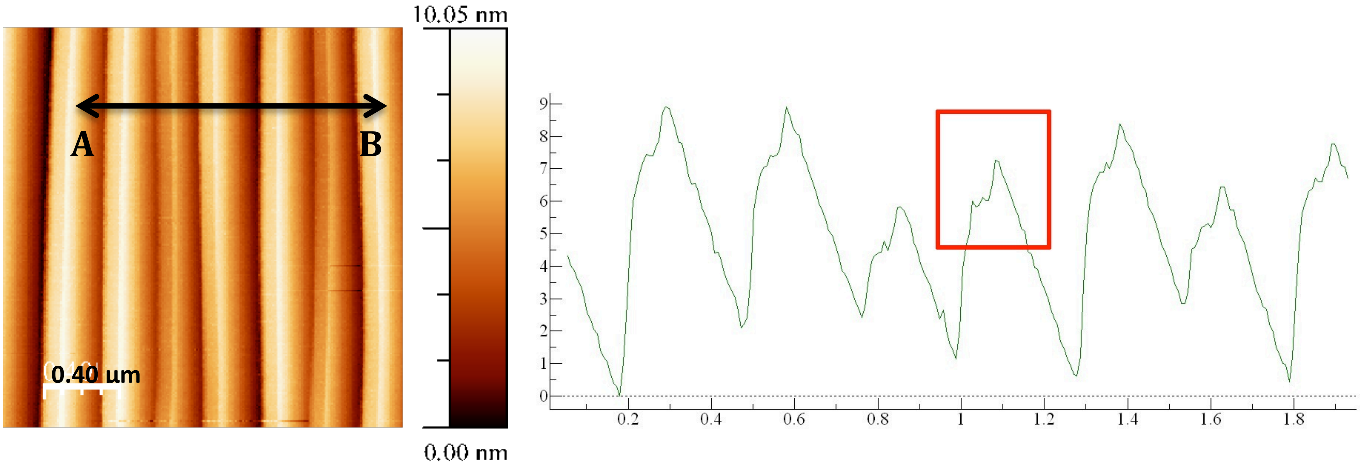


Fig. 24. The step profiles from the chosen point A to B of the 2 degrees off-oriented 4H-SiC substrate sample grown at 1750 °C. The image was taken in AFM at the magnification 2x2 μm. The scale at left corner is 0.4 μm.

Furthermore, Fig. 25 shows the relation between the step height and the step width taken from AFM images at 5x5 μm magnification for different off-axis cutting angle of the substrate. As a reference, in Fig. 25(a) the step height and the step width of 4H-SiC epilayers grown on 8 degrees off-axis angle shows a very clear trend [25]. When the temperature gets higher, the step widths also get higher values because of the step-bunching effect. In Fig. 25(b), the step height and the step width of the 4H-SiC grown on substrates with lower off-axis cutting angles, namely 2 degrees and 4 degrees, are shown. The trend is not as clear as the one on the 8 degrees substrate. Therefore, generally speaking, in these step height vs. step width figures, the trend of increasing step width and step height when increasing temperature are not pronounced in the low off-axis angle, such as 2 degrees and 4 degrees, most probably due to an emerging competition between two dimensional growth and step flow growth.

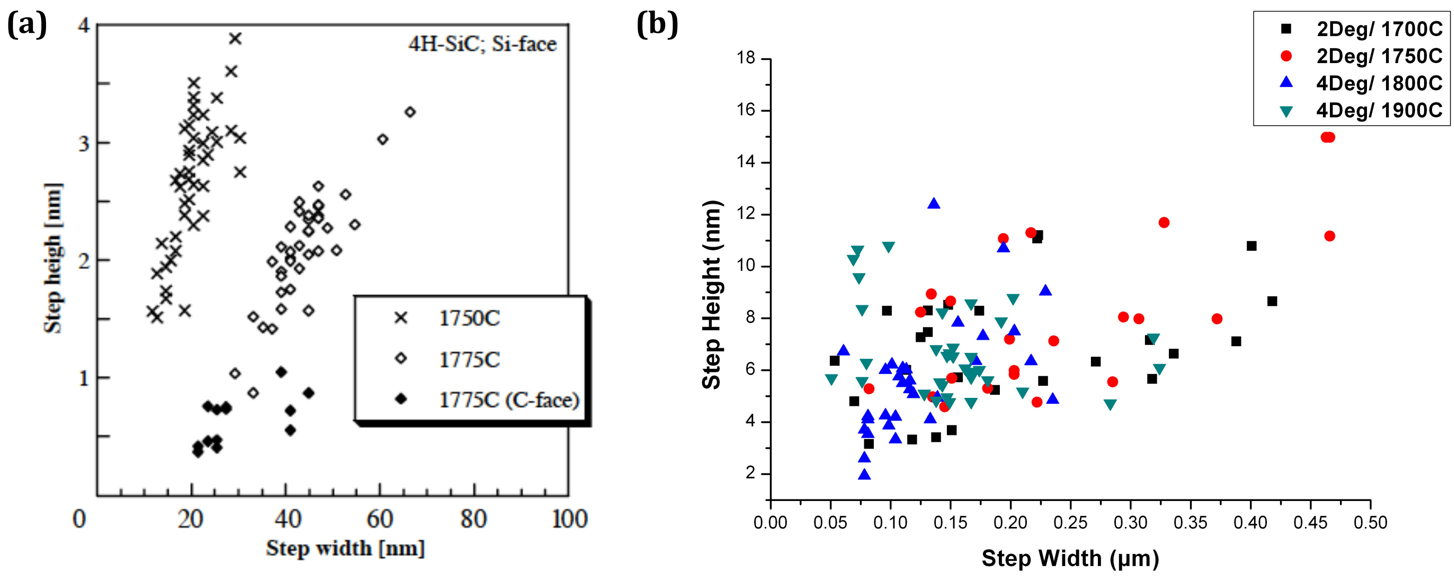


Fig. 25. Step height vs. step width from AFM measurements for (a) 4H-SiC epilayers, which have 8 degrees off-axis cutting angle grown on the Si-face at 1750°C and 1775°C and on the C-face at 1775°C [25]. (b) 4H-SiC epilayers, which have 2 degrees and 4 degrees off-axis cutting angles at 1700°C , 1750°C and 1800°C , 1900°C respectively by sublimation epitaxy.

The Root Mean Square (RMS)

The root mean square (RMS) shows the roughness and the average height of the epilayer. The higher the RMS value, the rougher the surface is. In each chosen sample, three different magnifications images were taken, 10 X10 μm^2, 5 X5 μm^2 and 2 X2 μm^2. The RMS values in each image in the sample were measured and shown in Fig. 26.

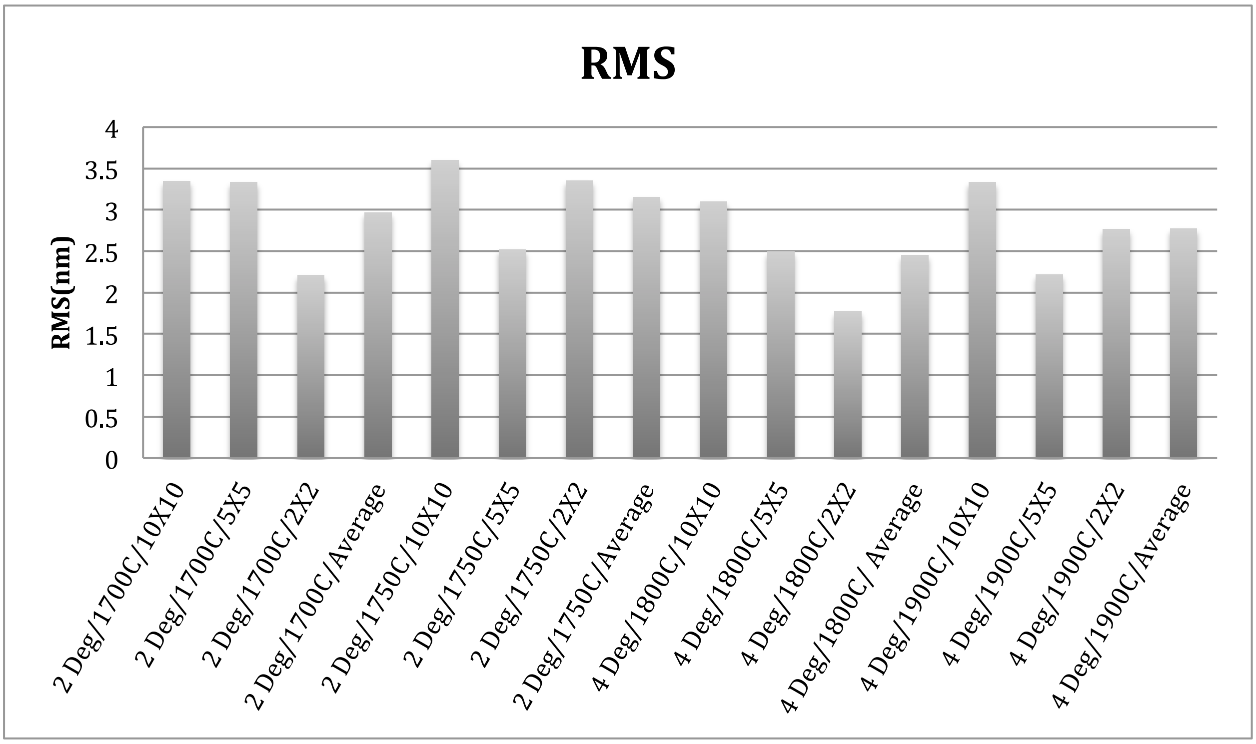


Fig. 26. The RMS values for the chosen sample in AFM, showing that different values were obtained in different magnifications images, but the variation is not so large. (At the X-axis the name of the terms are listed by the order off-axis cutting angle/ growth temperature/ the magnification of the AFM images)

The variations of RMS values between the samples are small. It means that the surface roughness in each case is similar. The samples grown on 4 degrees off-axis substrate would possibly be rougher since they have a higher density of steps, or on the other hand the steps are larger in the 2 degrees substrate growth.

8.Conclusion

The favorable criteria for growing cubic-SiC epilayer and bulk-like 3C-SiC have been studied for different parameters and discussed in respect to influencing factors in the growth method fast sublimation growth process.

The growth temperature, growth time, off-axis angle, graphite plate were changed to increase the domain size, the quality of epilayer and lower the number of defects so that we can increase the thickness of the epilayer to bulk-like 3C-SiC. This could, for example, be used as the seed to grow the homopolytypic bulk growth of 3C-SiC. It has been shown that in 4 degrees 4H-SiC substrate the 3C-SiC start to grow around 1800°C and 3C cover all the epilayer at 1950°C. Similarly, in 2 degrees 4H-SiC substrate the 3C-SiC start to grow around 1750°C and 3C cover all the epilayer at 1850°C. In addition, the growth rate follows an expected trend by increasing twice when the temperature increased by 50°C, except in some case when the growth time is long and the graphitization of the source happened. The graphite plate on the backside of the substrate lowers the growth rate, and the domain sizes are bigger. Furthermore, it was found that the lower the off-axis angle, the easier to form 3C-SiC, but the domain size is smaller so the number of defects increased. Also, higher temperatures can lead to easier formation of 3C-SiC and can cover all the epilayer, but a higher temperature results in a higher possibility of defects on the boundaries because of a larger number of nucleation sites. The growth rate, growth temperature and the supersaturation with defects forming should be balanced well to get a good morphology and good quality of 3C-SiC bulk epilayer.

In the growth temperature 1950°C, and the growth time 60 min with graphite plate on 4 degrees 4H-SiC there are large average domain sizes compared to other samples which indicates that the growth criteria are promising for full coverage by 3C and few domains.

In addition, the model of 3C-SiC lateral enlargement on 4H-SiC off-axis substrate has been set. Therefore, if the thickness of the epilayer when the basal plane is formed is known, the lateral expansion distance of the 3C-SiC on the epilayer can be estimated.

The equation goes in good agreement with the growth series study of growth temperature above the 3C transition on 4 degrees 4H-SiC substrates.

The step-bunching effect during the growth was studied regarding step height and the terrace width using atomic force microscopy. Wider terraces were measured in the case where 3C-SiC start to form in the epilayer. As the temperature gets higher, the step bunching becomes more serious, so the terraces get wider. At lower off-axis angle, fewer numbers of steps are observed and the terraces become wider.

9. Future Work

To find an optimal growth condition and get more information for growing 3C-SiC bulk material, in the future, more different off-axis angles could be used for the growth. From the experiments, the off-axis angles can be regarded as one of the most important factors to influence the domain size, number of defects and the morphology of the epilayer. More series should be done to know how to related finding optimal criteria in on-axis growth case, and possibly know if we can conquer the terrace width of the off-axis angle to allow growth of the 3C-SiC on the 4H-SiC substrate.

Not only the 4H-SiC substrate may be used as substrate, different polytypes give different growing mechanism for 3C-SiC epilayer. The 6H-SiC substrates, though it is also hexagonal structure, can be an interesting comparison to know how the stacking faults affect the growth. Furthermore, the difference in chemical bond strength on these two polytypes can also make the growth conditions different.

All the series in the thesis were done in the vacuum ambient. During the growth, Nitrogen and Argon can be introduced to the chamber, and even if resulting in a lower growth rate, there could be doping and interplay in vapor phase between Nitrogen, Argon atoms and Silicon, Carbon atoms. More electronic applications could be obtained using doped materials.

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