Institutionen för fysik, kemi och biologi

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Polytype formation in sublimation epitaxy of SiC on low off-axis substrates

Björn Lundqvist
Examensarbetet utfört vid IFM
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Contents

1.0 Introduction

Silicon carbide (SiC) is a wide-bandgap semiconducting material with many promising qualities. Its properties make it suitable for applications such as high-power and high-frequency electronics. The thermal and chemical stability is high and it has a high tolerance to radiation damage. For hetero-epitaxial growth SiC would be an excellent substrate for other materials to be grown on. [1]

SiC crystallizes in many different ways, i.e. it has many polytypes (more than 200). Each polytype is slightly different from the others and the material properties vary between them. Controlling the crystal growth so that only the wanted polytype is formed is difficult and a major reason why SiC has not already replaced other semiconductor materials, such as silicon, in products available today, despite its superior performance. There are also several types of defects that arise in the manufacturing methods that have not yet been mastered to a sufficient degree for large scale production to be viable. [2]

The most commonly used growth technique today is Chemical Vapor Deposition (CVD). Epitaxial layers produced with this method are of good quality with respect to defects and polytype stability, but the growth rate is low (typically 5-10 µm /hr) which means that only thin layers are commercially viable. This limits the use in some applications, such as high power devices. [3]
Another growth method is sublimation epitaxy. With this method it is possible to grow SiC films with growth rates up to 1 mm/h. The layer thicknesses of the produced films can be made with good enough quality to be used in components that require higher voltage levels to work properly, while the background doping is still a challenge. [4] Yet another promising application is as white LED material for general lighting, where a thick layer of doped SiC is used to convert UV-light into the visible spectrum. This is an emerging research area at LiU in cooperation with other research groups in Germany, Denmark and Japan. [5]

One way to influence polytype formation is the use of specific substrates. Depending on what material you grow your SiC on different polytypes will form more easily and could also form unintentionally. The surface of the substrate and its orientation influences the growth, together with growth conditions for initial nucleation on a particular substrate. [2] In this thesis SiC has been grown on 6H-SiC substrates using a sublimation method working at 1700-1900^{o}C. The two dominant polytypes on this kind of substrates are the hexagonal 6H- and the cubic 3C-SiC. Previous research has shown that growth on on-axis substrates favors 3C-formation, so that there is heteroepitaxial polytype growth, while growth on substrates with a miscut larger than 3.5 degrees in the <1, 1,-2, 0> -direction is better for 6H-formation so that the polytype from the substrate is maintained. [3]

In this work investigations of growth on low off-axis substrates (0.8, 1.0, 1.2 degrees) were performed in order to see when and where the transition occurs. Growth was performed at different temperatures and gas pressures, in order to gain further knowledge about what kind of growth parameters are affecting the growth in different cases. The samples were characterized by optical microscopy and atomic force microscopy (AFM).

2.0 SiC

SiC only rarely occurs naturally and then in the form of moissanite mineral. It is very hard (9-9.5 on the Mohs scale) and transparent, i.e. quite similar to diamond. It has been mass produced as SiC powder (used as an abrasive) since 1893 and the electronic properties have been investigated since the beginning of the twentieth century (first SiC LED 1907!). Material qualities that make SiC so appealing are wide bandgap, high electron mobility, high thermal conductivity and high electric field breakdown strength. [6]

2.1 Structure

The fundamental unit of the SiC crystal consists of one silicon atom bonded to one carbon atom. A structural description of a SiC crystal starts with one two-dimensional (flat) layer of SiC molecules, the layers on top of it are described in relation to the first one whose orientation is called A. As the next layer forms on top of the first, each SiC unit can attach itself in one of two ways, called B and C. The sequence of different layers determines the polytype of the crystal. Cubic 3C-SiC for instance is made up of layers stacked like ABCABC, while the hexagonal 6H-SiC has the stacking sequence ABCACB. [3]

2.2 Properties

In order for SiC to be used in electronic applications there are several criteria that must be met.

- Defect freedom: The presence of defects (described below) in the material rapidly degrades its performance. High purity SiC is necessary to avoid significant losses of component efficiency.
- Stress freedom: Stresses in the material make it more fragile and likely to break down at a much earlier point in time. Structures with high stress levels are very unstable.
- Surface smoothness: A common step in the production of electronic components is the addition of a layer of one material on top of another. If the added upon surface is too rough it will cause problems, such as growth disturbances, stresses and defects which reduce the quality of the component. [3]

2.3 Techniques

Different techniques come with different challenges to be met, and are used for different kinds of crystal production. The two major techniques are CVD and PVT.

2.3.1 Chemical Vapor Deposition (CVD)

There are *many* different types and variations of CVD. The general principle, however, is that SiC is deposited on the substrate through the use of chemical reactions. The CVD process produces very smooth crystals of very high purity. The drawback is that it is very slow, which makes it expensive for growth of thicker layers. [4]

2.3.2 Physical Vapor Transport (PVT)

With this method SiC vapor is transported to and deposited on the seed. In sublimation growth a temperature gradient is used to sublime[1] a SiC source and bring the vapor to a seed where it nucleates. It is a lot faster than CVD, up to 1 mm/h, but also more difficult to control to a degree that allows production of material suitable for electronic devices. The longer distance (5-30 mm) between source and seed causes disturbances on the vapor species when they are transported. Most commonly the PVT method is used for growth of boules from which substrates are produced for subsequent epitaxial growth. [4]

2.3.3 The Fast Sublimation Growth Process (FSGP)

The type of sublimation epitaxy that was used in this thesis was the FSGP, which has been developed at LiU. The FSGP sublimes a solid polycrystalline SiC source (unordered polytype) , and the vapor species need only to be transported a short distance onto a substrate. It operates at a lower typical temperature than other sublimation methods but has a high growth rate.

Because the source to substrate distance is short, the interactions with the graphite walls are minimized, which is good for vapor species control.

The lower temperature of the method gives higher material quality. The method also enables rapid growth of thick 3C-SiC, something that is impossible with other methods since 3C is not stable at higher temperatures. [4]

[1] SiC does not melt. Instead it sublimes, i.e. it goes directly from solid to vapor.

2.4 Growth mechanisms

The way that the SiC crystal grows is of great importance for the quality and polytype of the crystal. Each mechanism comes with built in problems that have to be understood in order to produce a good result. There are several factors that influence the growth, but a main principle is that the incoming atoms nucleate at the most energetically favored position. An energetically favored position is usually a step, an island or a defect related protrusion of some sort, rather than the middle of a flat surface. [2]

2.4.1 2D-Nucleation

Two dimensional nucleation is also known as island growth. The 2D nucleation growth occurs when there are large flat areas. The first atom of a growing layer adheres to a random spot on a flat surface on the substrate, as for some reason there are no spots of lower energy within its reach. This becomes the nucleation site for the following atoms. They attach to positions next to the first one and the layer grows outwards like an expanding island. The next layer will start to form on top of the first one when it has reached a size that forces an incoming atom to nucleate on the top of the island rather than diffusing to the island edge.

One common problem with this type of growth is that many islands will start to form on different sites of a flat area, usually not with the same orientation, which causes problems when the islands eventually coalesce and create defective domain boundaries. The domains can also have different orientation by different stacking and orientations, like ACB and ABC, which causes inversion (twin) domains. [4]

2.4.2 Step flow growth

Step flow growth is used to increase the crystalline similarity between the grown layer and the substrate, in the case of SiC. It is based on that an atom that nucleates at a step site must assume the orientation of the substrate, while nucleation on a flat surface offers two possible orientations. By cutting the substrate at an angle from a (flat) low index surface, a surface with evenly distributed straight steps is produced. When the SiC atoms are adsorbed they may nucleate at the step sites and all of them assume the same orientation. Step flow growth makes it possible to take a thin crystal of high quality (often slowly and expensively produced) and use it as a seed for fast growth of the same or even better high quality. This growth mode suffers from problems with step-bunching. [4]

2.5 Defects

There are several different types of defects that can arise during the formation of the crystal, and which degrade the performance of the finished material. These are the most important ones.

2.5.1 Inclusions

During growth something unwanted is included in the material. An inclusion can consist of many things, from equipment pollution to an area of the wrong kind of SiC polytype. Simply put it is anything that does not belong in the ideal single crystal SiC you are striving for and it degrades performance and disturbs growth. [3]

2.5.2 Dislocations

Dislocation is a common defect that arises when a part of the growing layer is dislocated relative to the rest. It can happen for many reasons (stress, growth disturbance etc.) and is characterized by a displacement vector. [4]

2.5.3 Double positioning boundaries (DPB)

Specific for 3C-SiC, a DPB has its origins in the stacking sequence of the growing layer. The cubic polytype can grow in two ways: ABCABC… or ACBACB. A DPB is formed when two neighboring regions with different stacking order coalesce. [3]

2.5.4 Step bunching

In 6H-SiC there are three types of terraces of the steps (A, B and C type of terrace). Material grown with step flow growth displays a phenomenon known as step-bunching. This arises due to the fact that steps grow with different lateral speeds, causing the faster ones to catch up to the growth fronts of slower steps in front of it to form larger steps. The mechanism behind this is not yet fully understood in SiC but the most widely spread model is that the surface free energy of the A,B and C terraces is different, which affects the nucleation rate of adatoms and thereby the lateral speed of these steps. In the characterization of step-bunching one considers microsteps, made up of several SiC bilayers, and macrosteps, made up of many bilayers. Step-bunching reduces the epilayer quality and it may also leave traces of altered impurity/dopant concentration in the grown material. [3]

2.6 Parameters affecting growth

There are many factors that influence the growth in different ways. These are the major ones.

2.6.1 Temperature

The temperature is a very important parameter; it affects the growth in many ways.

- The ramp up rate influences the initial nucleation, which lays the foundation for the growth.
- Ramp up smoothness (controlled and steady temperature increase) is also important. Sudden changes cause shifts in the phase of the growing material and give rise to defects.
- It influences the sublimation rate of the source which influences the Si/C ratio and the supersaturation. It also affects the vapor composition.
- The diffusion length of adsorbed atoms is highly dependent on temperature. Higher temperature makes it possible for the adatoms to move farther on the substrate to find a better nucleation site.
- A rising temperature causes an expansion of the materials, which may in turn cause stresses if the thermal expansion rates are different for substrate and SiC crystal.
- The polytype appearance is to a large extent influenced by the temperature, and the substrate surface is one way to try to control polytype formation. [3]

2.6.2 Gas ambient and pressure

The background conditions, under which the vapor transport takes place, will obviously affect the growth. There are two main considerations:

- The pressure will influence sublimation and vapor transport rate. Higher pressure makes it harder for the SiC vapor species to travel from the source to the substrate.
- Different gases will or will not participate in the growth, i.e. dope the crystal [3]
  The way that a gas dopes the layer may also influence the polytype. A relevant example of this is that on-axis growth in 0.5 mbar (RT) nitrogen ambient stabilizes the formation of 6H. This is possible because nitrogen substitutes for carbon in the growing layer which in turn shifts the Si/C-ratio to a region that favors 6H. [7]

2.6.3 Si/C ratio

The foundation of sublimation growth is transport of Si and C-containing gas species. Therefore it is vital to know the vapor composition. Primary constituents of sublimed SiC are Si, Si_2C and SiC_2. The most commonly used measure of the composition is the Si/C-ratio, which gives the relative distribution between Si and C. The ratio has a strong influence on the polytype stability and keeping it stable at a suitable value is important for the crystal quality.

Since graphite is the most common material for heating components (e.g. crucibles, spacers) used in sublimation growth there will often be excess carbon present in the reactor. When the ratio shifts towards carbon in this way, the source and eventually also the substrate will become graphitized. Graphitization means that carbon is deposited on the surface and will decrease the growth rate significantly.

To avoid this, one can either add extra Si or remove excess C. In our setup a Ta-foil is placed under the source in the crucible. The foil will absorb C through formation of TaC on its surface. [3]

2.6.4 Substrate

The substrate is the starting point and foundation of the growth. It will affect the growth in many ways. The two main considerations are these:

- Material: The type of material, its structure, orientation and behavior during growth (e.g. thermal expansion) will affect the initial growth and to a large extent determine the result of the finished layer.
- Surface quality: The smoothness, cleanness and defect freedom of the surface are of great importance, as defects in many cases will be reproduced or give rise to problems during growth. [2]

3.0 Experiment construction

For this experiment series a sublimation epitaxy technique was used in order to be able to produce thick enough samples at relevant conditions in a reasonable time frame. In this section the equipment setup will be described and an outline of the sample series presented.

3.1. Equipment setup

A SiC source and a substrate, separated by a graphite spacer, are placed inside a graphite crucible. The crucible is then placed in insulating graphite foam inside a quartz tube. After sealing the tube and setting up the atmosphere inside it in the desired way (e.g. vacuum or Ar) the crucible is then inductively heated by an RF coil positioned around the tube. The position of the coil relative to the crucible determines the temperature gradient. Inside the crucible there is also a Ta foil which acts as a carbon getter, i.e. it absorbs excess carbon, in order to prevent the Si/C ratio to shift more than necessary during growth.

3.2 Growth process

The crucible containing source, spacer, substrate and Ta foil was placed in the insulating foam and loaded into the tube. Then the vacuum pump was turned on and left to pump over night, ensuring a vacuum pressure below $5x10^{-4}$mbar. If a gas ambient was wanted, the tube was then filled with gas to atmospheric pressure and then pumped down to the starting point. The crucible was then heated to growth temperature using a ramp up of 20 K/min. The temperature was measured on the top of the crucible through a hole in the insulation. When the growth temperature was reached the temperature was kept steady for thirty minutes and then power was switched of and the sample cooled.

After each growth run SiC had been deposited on the inner side of the crucible lid and on the spacer. To remove this, a resublimation procedure was performed in which the lid was used as bottom, with a dummy spacer inside with the spacer on, and a dummy bottom as lid. This setup was then heated to high temperature, letting the unwanted SiC deposits sublime and nucleate on the dummy parts. After this procedure carbon traces were removed and the crucible part could be used for the next growth experiment.

3.3 Sample series description

The experiment series was performed on substrates with off-axis 1.2, 1.0 or 0.8 degrees, except for the last sample which was grown on a 2.0 degrees substrate. In this section the overall plan for the series will be presented. It consists of four main stages.

3.3.1 Stage one: Dynamic vacuum and 0.5 mbar N_2

The main part of the series, which was also the only part planned beforehand, can be divided further into two parts: Growth in dynamic vacuum (continuous pumping throughout the experiment) and growth with a 0.5 mbar N_2 ambient. Each of these parts consists of nine samples, three different substrates (1.2, 1.0, 0.8) each with growth performed at three different temperatures. See Table 3.1.

Table 3.1	Dynamic Vacuum			N_2		
	1750°	1800°	1850°	1750°	1800°	1850°
1.2	X	X	X	X	X	X
1.0	X	X	X	X	X	X
0.8	X	X	X	X	X	X

3.3.2 Stage 2: N_2 at 1900°C

The results of the N_2 growth made further investigations interesting, wherefore we decided to grow three additional samples (1.2, 1.0, 0.8) in N_2 at one more temperature: 1900°C since the formation of 3C-SiC was enhanced at higher temperature in the first stage. See Table 3.2.

Table 3.2	N_2
	1900°
1.2	X
1.0	X
0.8	X

3.3.3 Stage 3: Growth in Argon

In order to determine whether the domain widening effect was caused by the presence of N_2 specifically or by the pressure during growth, we decided to make two runs in an ambient of 0.5 mbar Ar. The choice fell on argon because it is an inert gas that unlike nitrogen does not participate in the growth other than by limiting the vapor transport from source to substrate. Nitrogen is actually incorporated in the grown crystal and thus affects the material in additional ways.

See Table 3.3 for overview.

Table 3.3	Ar	
	1800°	1850°
1.2		
1.0	X	
0.8		X

3.3.4 Stage 4: 2.0 degrees off-axis in 0.5mbar N_2 at 1900°C.

To get further indications as to the influence of off-axis on high temperature growth in N_2, one last sample was grown on a 2.0 degree substrate.

The entire series is summarized in Table 3.4.

Table 3.4	Dynamic Vacuum			N_2			N_2	Ar	
	1750°C	1800°C	1850°C	1750°C	1800°C	1850°C	1900°C	1800°C	1850°C
2.0							X		
1.2	X	X	X	X	X	X	X		
1.0	X	X	X	X	X	X	X	X	
0.8	X	X	X	X	X	X	X		X

4.0 Characterization

The SiC can be characterized by many methods: TEM, SEM, AFM, XRD, OM etc. and before characterization the samples can be treated in many ways, e.g. etching, polishing or cutting. For the purposes of this thesis we only needed to use OM and AFM which will be described further in the following sections. Overview pictures were taken using high resolution optical microscopy scanning and the polytype coverage was calculated using imaging software.

4.1 Optical microscopy (OM)

An optical microscope was used for the most of the surface studies. Magnification was available at 50X, 200X, 400X and 1000X. Reflection as well as transmission light was used for studies of different features.

A Nomarski differential interference contrast prism was used in order for us to be able to distinguish small details in high magnification. The function of the prism is splitting the incoming beam into an ordinary and an extraordinary polarized component. After reflection on the sample the beams return through the prism. A difference in the optical path gives a shift between the two components, which enables distinction between objects with different heights that would have been very hard to see without the prism. [4]

The hexagonal 6H and cubic 3C polytypes of SiC are easily distinguished in an optical microscope: 3C is yellow and 6H is colorless or greenish when doped with nitrogen.

4.2 Atomic force microscopy (AFM)

The principle of AFM is that the movement of a cantilever is detected by a laser. On the tip of the cantilever there is a very small filament attached with a tip that is only a few atoms thick. This tip interacts with the forces from the surface you scan and moves the cantilever. The interaction with the surface can be performed in various modes with different results for the scan. The obvious advantage of AFM is that it allows a detailed scan on an atomic level. The disadvantage is that scanning is a slow process, which means that the scanned area usually only covers a small percentage of the total area (a typical image size is 2x2 µm, the sample is ~10x10 mm.).

In this thesis we used AFM to image the steps on the 6H-SiC samples grown at lower temperature since the 6H-SiC clearly dominated the surface. The purpose was to measure step height and width to get a general view of the quality of the step-flow growth and understanding of step-bunching on low off-oriented substrates. [8]

4.3 High resolution X-ray diffraction (HRXRD)

The HRXRD is a non-destructive technique that delivers information on the structure and composition of grown epilayers and bulk material. It is based on the scattering of X-ray radiation at the lattice planes of crystals. By varying the angles of incidence and detection in certain ways, details about things like strain, polytype uniformity, crystal bending and lattice misorientation can be studied.

Often the HRXRD is used for obtaining a measure of the quality of the material, but the samples produced in this study were in general too rough for measurements like these to make sense. Instead it was utilized in order to investigate the orientation of substrates. [4]

5.0 Results and discussion

The samples that were grown in this work show clear correlations between the 6H/3C coverage ratio and off-axis degree and temperature. There were also indications of a domain enlarging effect for 3C grown in nitrogen atmosphere.

In this section the results will be presented according to which stage of the series they belong to. Initially, growth rate and markings of substrate orientation will be commented upon. Then I present the vacuum grown samples, followed by samples grown in N_2 ambient and a comparison between the two. Thereafter the 1900°C N_2 samples, the argon samples on 1.0 and 0.8 degrees substrates and lastly the 2.0 degrees substrate sample grown in N_2 are displayed. The final section will address the AFM pictures of low temperature N_2 grown 6H to discuss the step heights and widths.

5.1 General Comments, growth rates

The overall surface characteristics are similar for samples of the same category. Due to this fact all magnifications of all samples in optical microscopy will not be presented here, but rather a representative selection will be shown.

A few comments are given below about the growth rates:

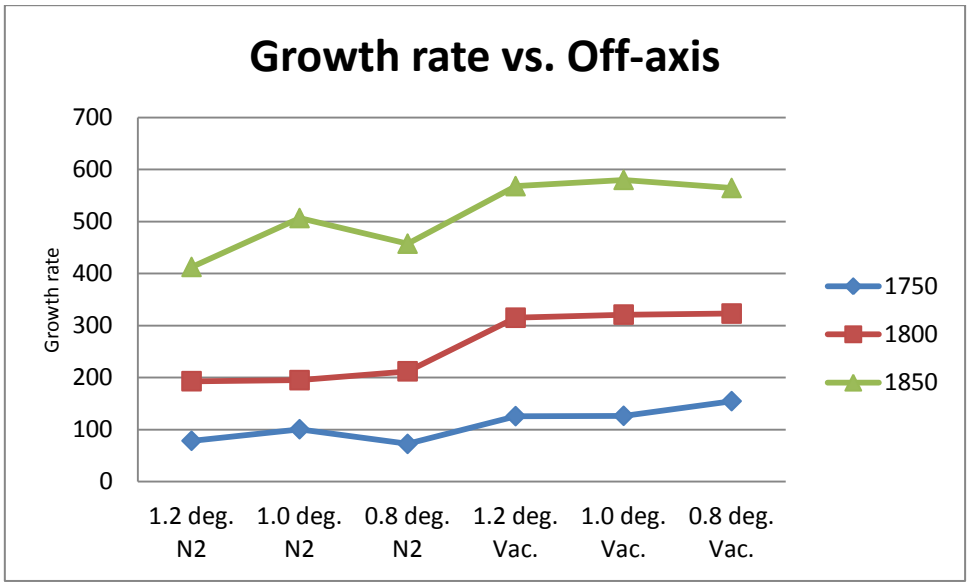


Fig. 5.1 Growth rate is plotted against off-axis degree and ambient.

From this chart a few facts are revealed:

- The deciding factor for growth rate is the temperature.
- Growth rate is lowered in the presence of a gas ambient.
- The rule of thumb that the growth rate doubles when the temperature in large off-axis substrate is increased by 50 degrees [9] applies to growth on low off-axis substrates as well.

5.2 Substrate orientation

The substrates are generally cut with an angle towards the <1, 1,-2, 0>-direction. This direction is then marked by the addition of a primary and a secondary flat. Fig.5.2 displays the principle.

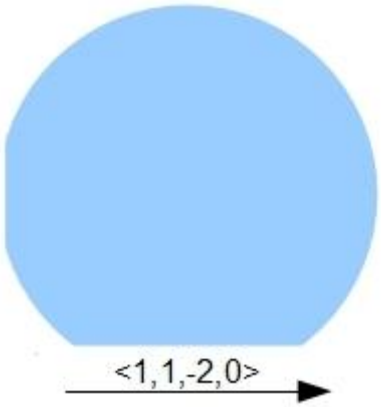


Fig. 5.2 A schematical view of a substrate. On the bottom we see the wide primary flat and on the left hand side the narrower secondary flat.

Epitaxial SiC growth (independent of polytype) is expected to occur in this direction by the step flow growth mechanism. [3] This was the case for the 1.2 degrees substrates, but after producing the first samples of 1.0 and 0.8 degree off-axis the growth direction appeared to have been rotated by approximately 60°. Either this was a new phenomenon that appeared when growing on low off-axis substrates, or a mistake had been made by the substrate manufacturer. Since the rotation was the same for 1.0 and 0.8 substrates as well as independent of the growth conditions, the second explanation seemed more likely. This was then confirmed by HRXRD of the substrates.

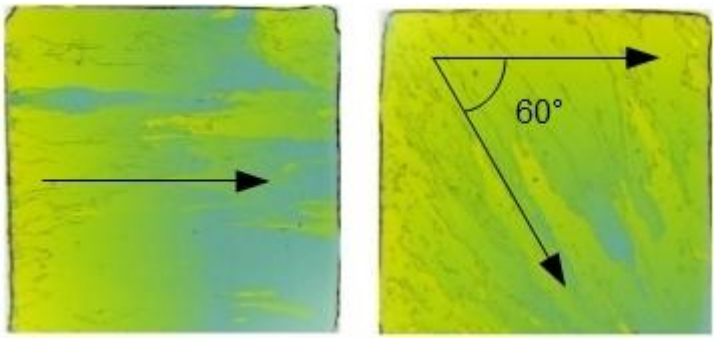


Fig. 5.3 An example of the 60 degree rotation of growth direction between 1.2° (left) and 1.0°/0.8° (right).

The reason for what appears to be differences in sample growth between 1.2° and 1.0°/0.8° is thus a production mistake. Other than altering the growth direction of the layers, this has no further implication for the growths in this thesis since the heating is symmetric and thus growth is determined by off-axis and growth parameters.

5.3 Samples grown in vacuum

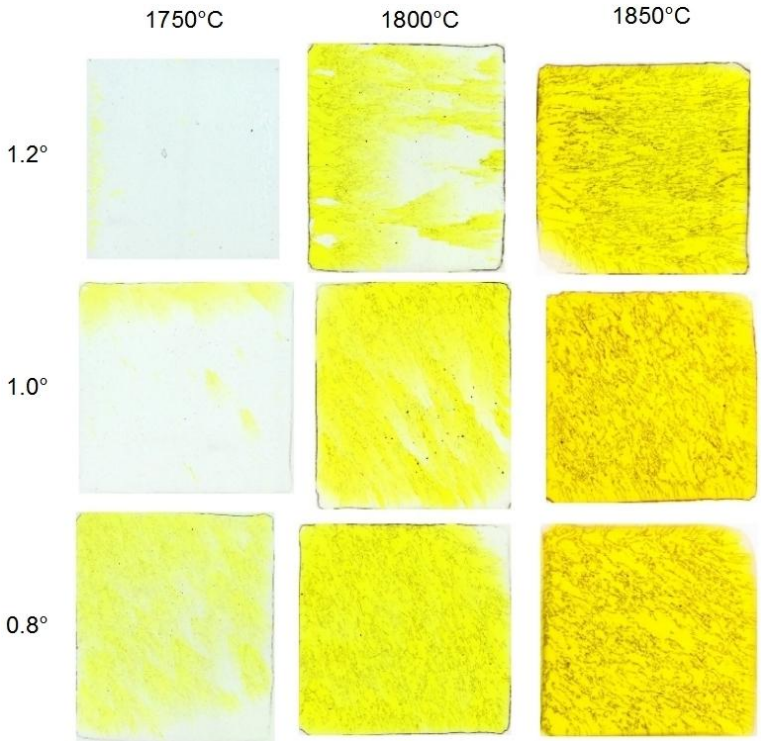


Fig. 5.4 An overview of samples that were grown under vacuum conditions.

The samples grown in vacuum have clear tendencies: 3C coverage is affected even by the small off-axis angles. This is seen in the images by shades of grey, where light grey is 6H and darker grey is 3C. In the reality, the polytypes are easy to distinguish since 3C is yellow and 6H almost transparent, or green when doped with nitrogen.

It is clear that when the off-angle of substrates is lower, i.e. closer to on-axis, the percentage of 3C covering the surfaces is higher.

The images reveal that the amount of 3C also varies with the temperature: Higher temperature means more 3C. This is to be expected since higher temperature shifts the concentration of vapor species to a level that is more favorable for the formation of 3C. [3]

It is worthwhile to note that there is almost complete coverage of 6H-SiC in the sample grown on 1.2 degree substrate at the lowest temperature (1750°C).

To get a clearer overview of the polytype ratios, the 3C coverage is plotted against off-axis angle in the chart below. Each marker corresponds to one growth temperature, and lines are given between the off-orientations at each temperature as guide for the eye to high-light the changes.

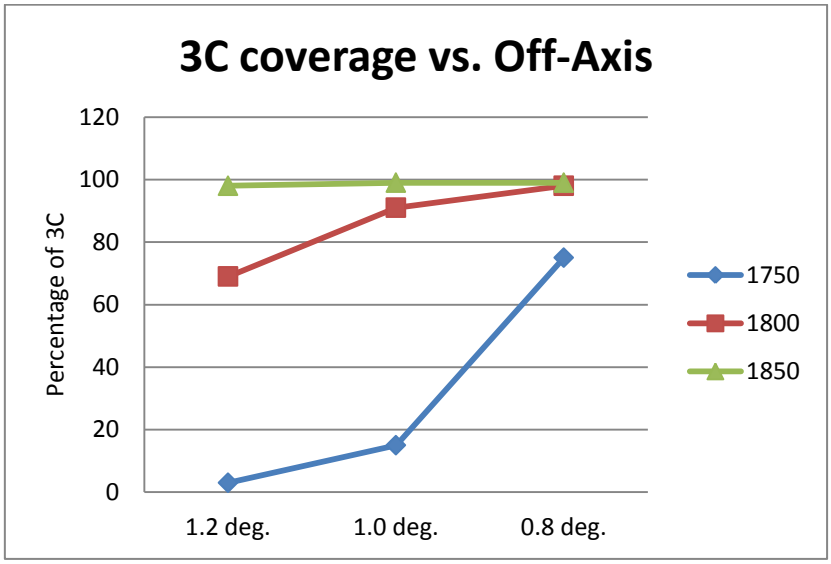


Fig. 5.5 It can be noted that 1850°C is enough to get full 3C coverage on all substrates.

Although it is clearly seen in the images, as well as in the graph, that the degree of 3C coverage decreases as the off-axis angle increases, the deciding factor for these growths is the temperature. At 1850°C the temperature is high enough that 3C growth completely dominates independently of off-axis angle. In order to utilize the off-axis influence for vacuum growth to maintain the substrate polytype, the sublimation epitaxial growth must thus be done at lower temperature.

In these transmission microscopy pictures at 50X magnification the general quality of the surface is readily available.

The defects that can be observed at this level are inclusions (probably of Si and C) and domain boundaries.

The amount of defects increases with temperature. The reasons behind this are probably related to higher growth rate.

The quality of the surfaces is determined by domains and domain boundaries. The 3C-SiC growth proceeds through the 2D-mechanism, i.e. the domains form, expand and finally coalesce. Since defects accumulate at the domain boundaries they are crucial for the quality. The images clearly show this behavior: dark uneven edges and cracks appear where two domains come together. Additionally it is interesting to see how the brighter 6H-areas do not suffer from this affliction, since they grow by step flow growth.

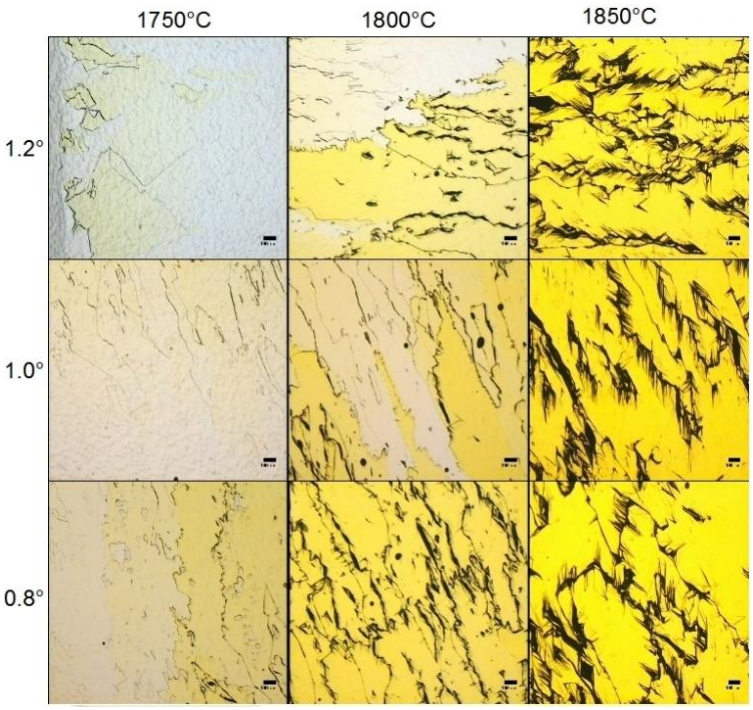


Fig. 5.6 50X magnification transmission microscopy of the vacuum grown samples. Brighter areas are 6H, darker 3C and black is boundaries and cracks.

5.4 Samples grown in N_2

We also found a strong dependence of the 3C-coverage on off-axis angle and temperature in the case of growth in an atmosphere of 0.5 mbar N_2.

Although the percentage changes of the coverage follows the same tendency as for vacuum growth it is apparent that the 3C coverage in general is markedly lower on these samples.

It is also interesting to note that an off-axis angle as small as 1.2 degrees is enough to stabilize the formation of 6H almost completely if the growth is carried out in N_2 at 1750°C. Only a minimal amount of 3C is visible at the edge of the substrate.

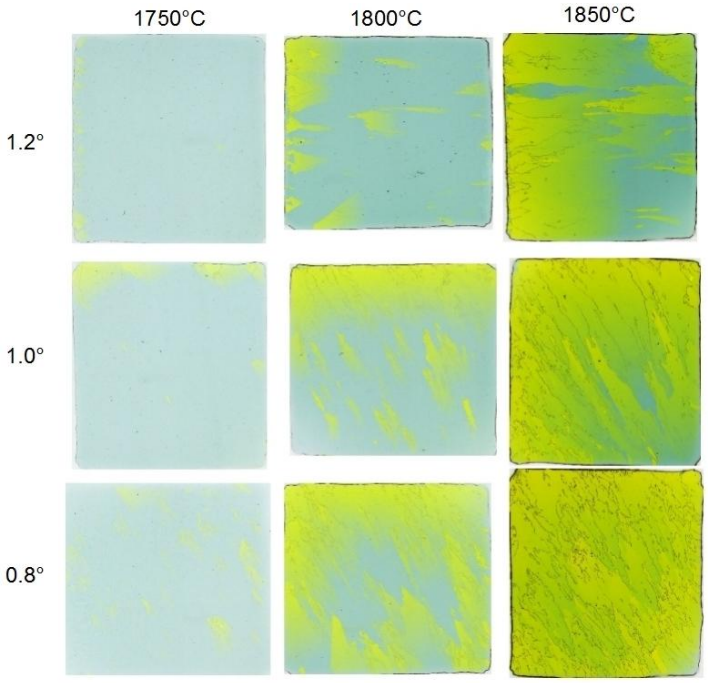


Fig. 5.7 An overview of the first samples grown in nitrogen ambient. Note the enlarged 3C domains in all samples and the high degree of 6H coverage on the samples grown at 1750°C.

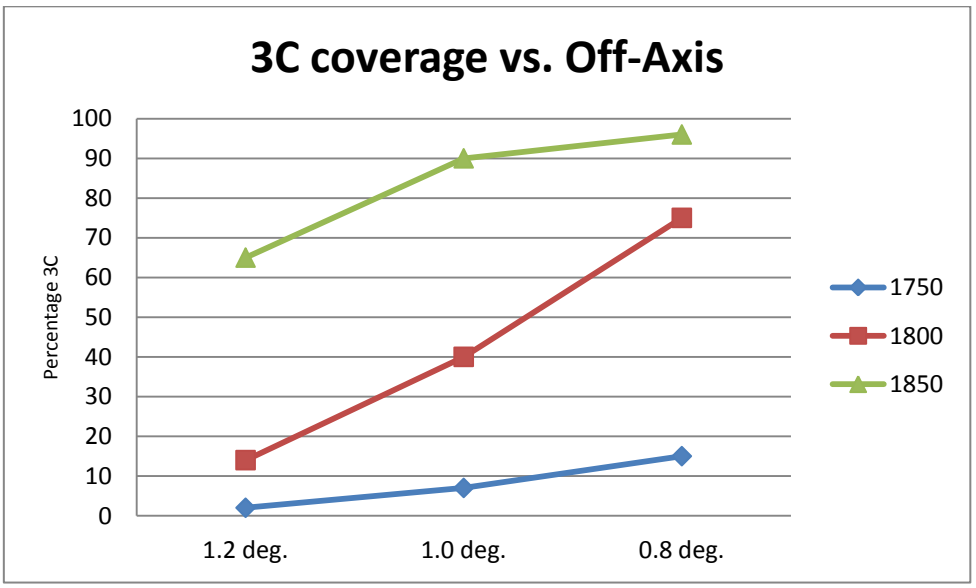


Fig. 5.8 The effect of off-axis degree on polytype formation is evident in all samples.

The coverage chart for N_2 growth shows how similar the behavior is to that in vacuum growth. The difference is that the amount of 3C has become lower in all samples, and that the highest temperature 1850°C is not enough to push the growth into the region where 3C completely dominates. For a full comparison the charts are shown in a summarizing chart below and a table with the percentages.

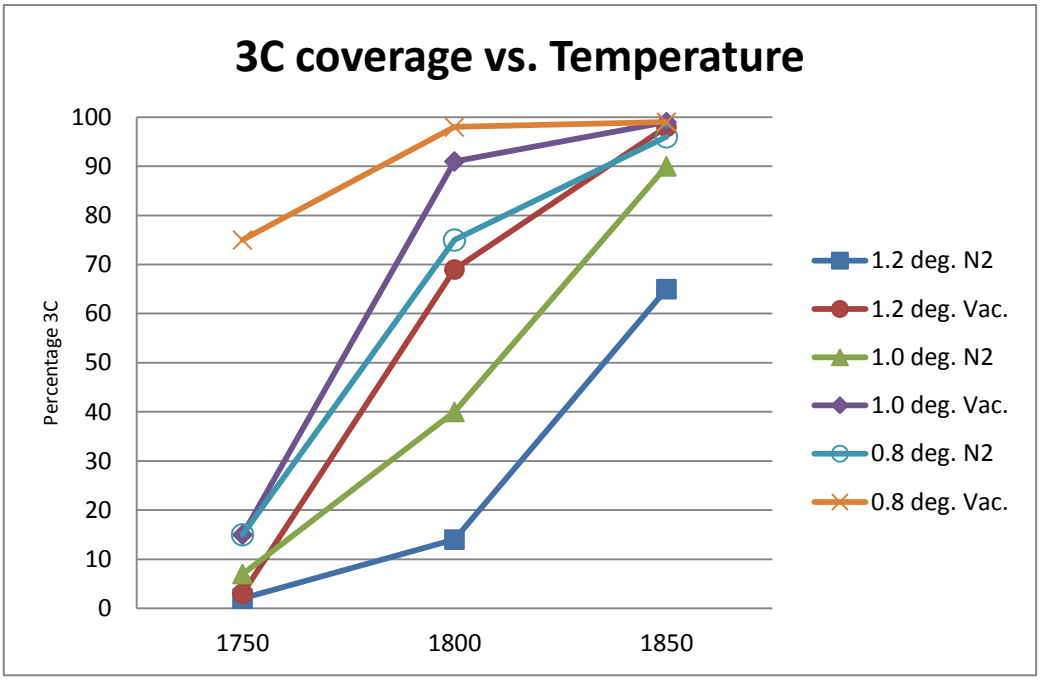


Fig. 5.9 Summary of 3C coverage for the first eighteen samples.

Table 5.1	1750	1800	1850
1.2 deg. N2	2	14	65
1.2 deg. Vac.	3	69	98
1.0 deg. N2	7	40	90
1.0 deg. Vac.	15	91	99
0.8 deg. N2	15	75	96
0.8 deg. Vac.	75	98	99

3C coverage percentages

The transmission microscopy images (50X) for the N_2 growth reveal the same type of defects as for the vacuum growth: inclusions and domain boundaries. However, a marked decrease in the number of domain boundaries is observed, as a consequence of the domains being larger.

In all samples grown in N_2 the domains are larger than in the samples grown in a vacuum ambient. This is true for all off-axis degrees.

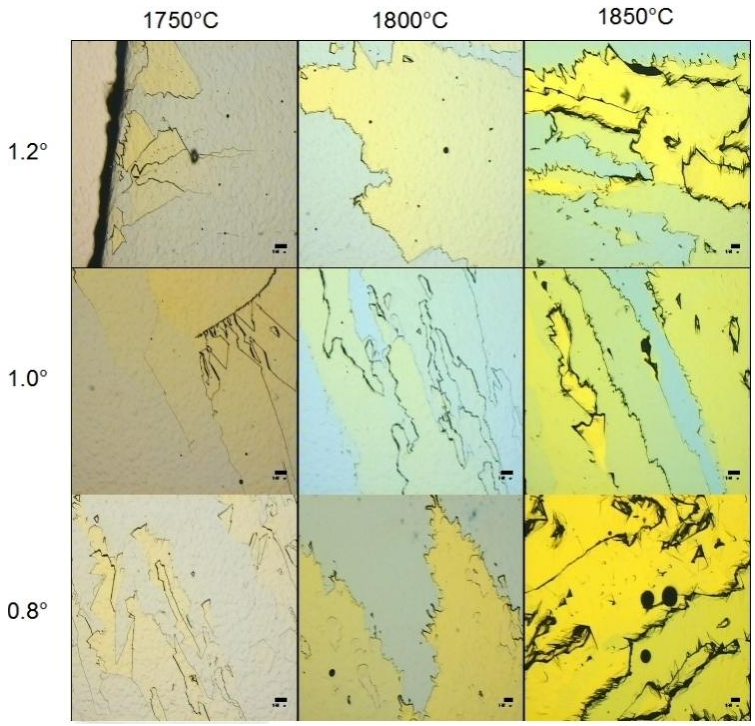


Fig. 5.10 50X transmission microscopy images of the samples grown in

An explanation for the larger domains is that less 3C -SiC nucleation centers are formed in the initial stages of growth. The nitrogen ambient causes a reduction of the supersaturation at the substrate surface. This means that at a given temperature less 3C will form than in the case of growth in vacuum. A reduced amount of 3C formed during the initial stages of growth means that there are less growth centers from which the island growth will proceed later in the process. Fewer growth centers means more distance between the islands, i.e. larger domains and a smaller number of boundaries.

5.5 Vacuum vs. N_2

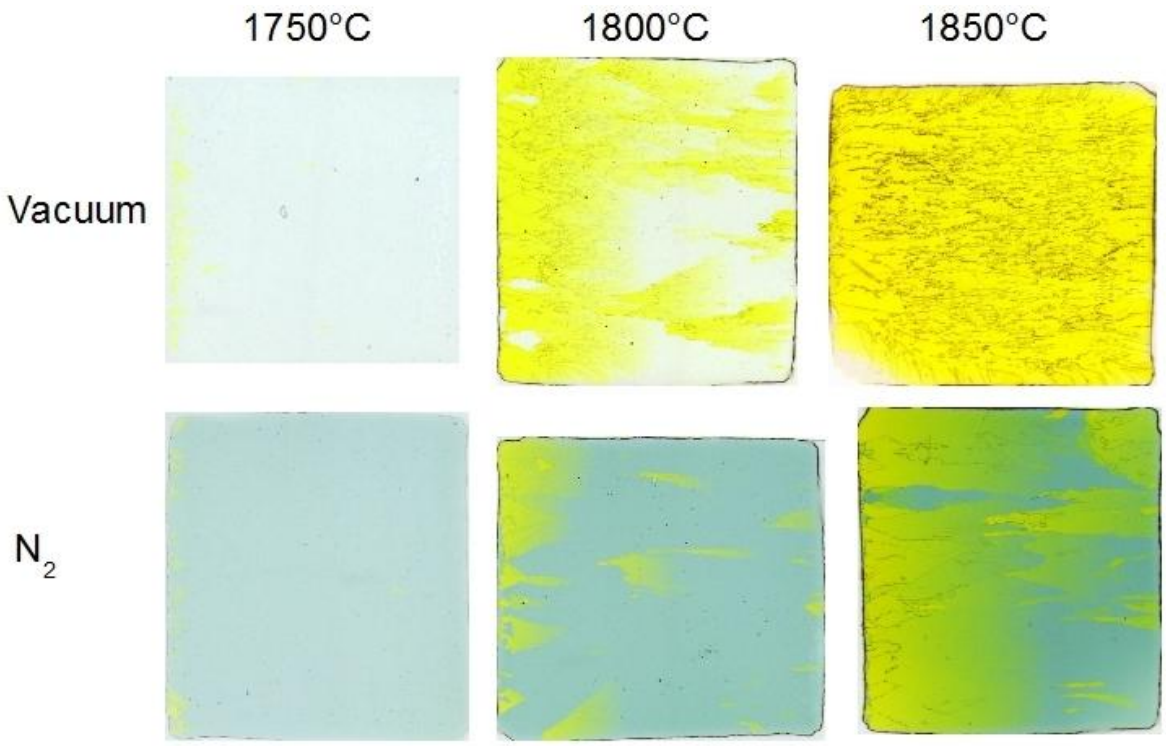


Fig. 5.11 Comparison image of the 1.2 degree substrates grown in vacuum and nitrogen respectively.

The window parameter of growth temperature effect is most clear in samples grown on 1.2 degree substrates. At low temperature the 6H homoepitaxial growth is dominating, while at high temperature growth the 3C formation is dominating. This image of the 1.2 degree off-axis samples, grown in vacuum and N_2 respectively, clearly demonstrates the lower 3C coverage of samples grown in the N_2 atmosphere. A part of the explanation is that the growth rate is lower, like discussed in section 5.1, probably due to limiting effects on the vapor transport from the presence of N_2. High grow rate is a known factor that promotes the growth of 3C. Another part of the explanation is that N_2 has a stabilizing influence on formation of 6H using on-axis substrates, an effect that previous work of the research group has shown, but not fully explained yet. A difference is that on on-axis substrates the growth mode is two-dimensional and the 6H-SiC grows by spiral growth. In off-axis substrates the 6H-SiC grows by step-flow growth. A possible reason for any stabilizing effect by nitrogen could be the difference in the 6H-SiC growth when comparing polytype growth stability of on-axis and off-axis in nitrogen ambient. [7] A notable difference is the low number of domain boundaries and large 3C domain size that can be seen in the N_2 samples, relative to the vacuum grown ones.

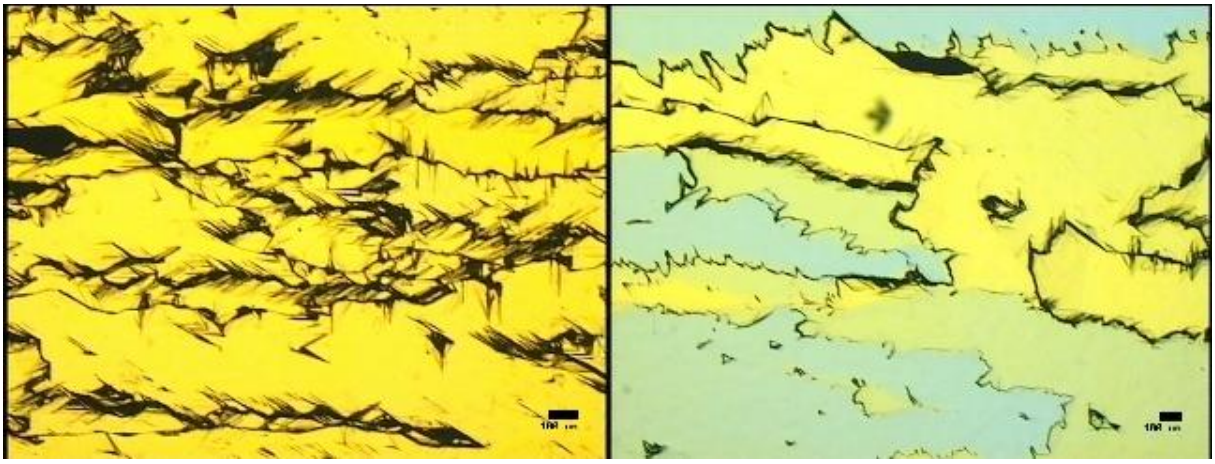


Fig. 5.12 50X transmission microscopy images. Substrate grown in vacuum to the left and in nitrogen to the right.

The details seen in the above 50X transmission images of the samples grown at 1850°C make the difference even more obvious. The large number and rougher appearance of the boundaries on the vacuum grown sample on the left image can in part be attributed to higher growth rate, but the definite reduction of the number of domains needs a different explanation.

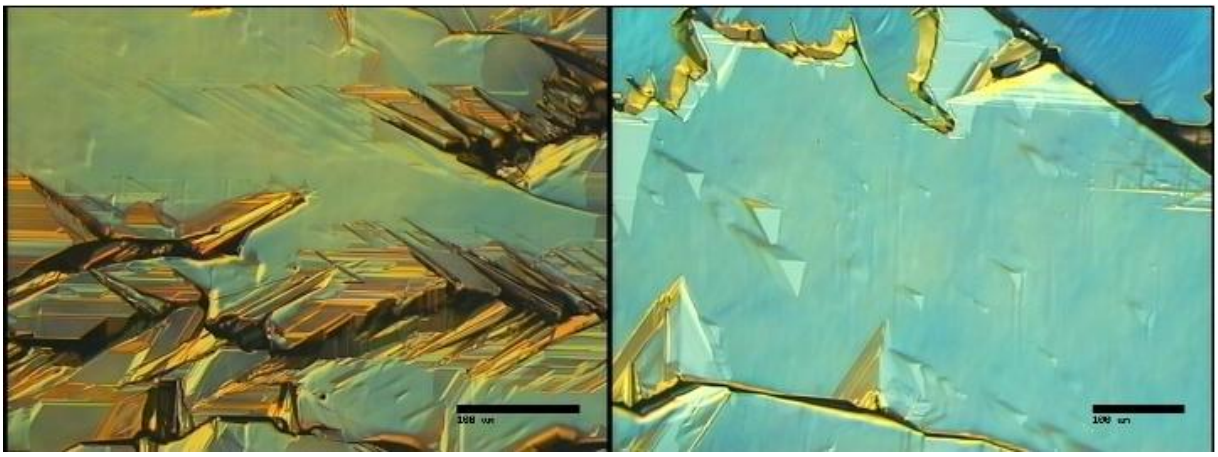


Fig. 5.13 200X reflection microscopy images. Substrate grown in vacuum to the left and in nitrogen to the right.

Further magnification shows more of the same characteristics. Above are 200X reflection images. In the right image triangular defects typical of 3C are visible. It is quite evident that the domain boundaries are substantially more defective in vacuum grown samples.

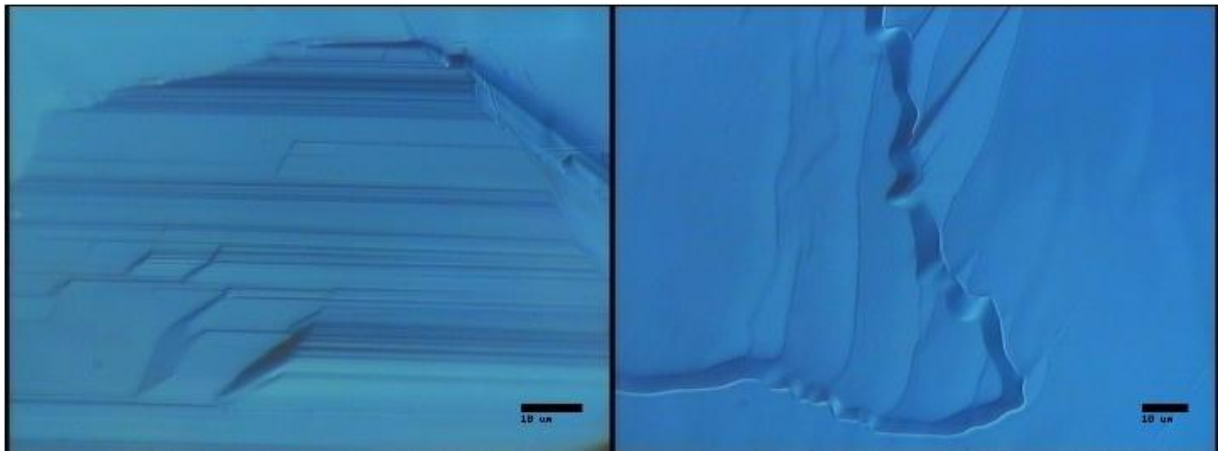


Fig. 5.14 1000X reflection microscopy images. Substrate grown in vacuum to the left and in nitrogen to the right.

At 1000X the difference is still clear. Macrosteps are visible in the right picture, but the general appearance of the surface is much smoother than in the left picture.

In summary we can clearly say that there are more domain boundaries in the samples grown in vacuum and that these boundaries are more defective than their nitrogen grown counterparts. This suggests that the domains do not adapt to each other as well in vacuum as in nitrogen when they coalesce.

5.6 0.5 mbar N_2 at 1900°C

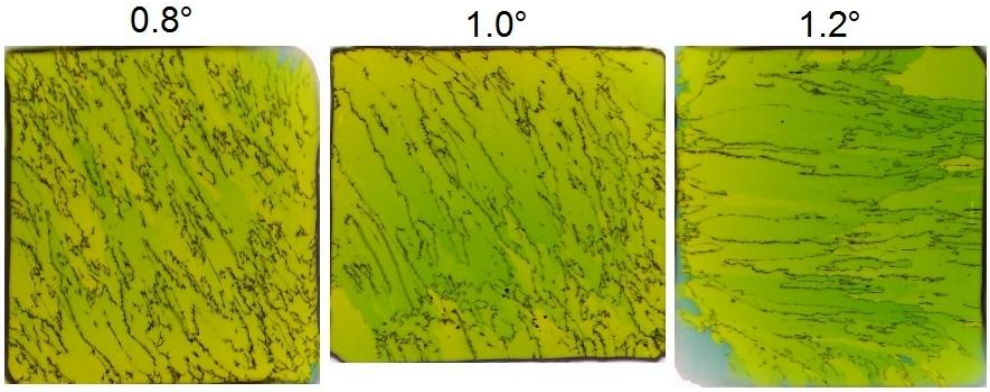


Fig. 5.15 The samples grown in nitrogen at 1900°C.

At the higher temperature the substrates were completely covered by 3C-SiC, though the thickness is not uniform, as can be seen by the shifts in color when using transmission light microscopy. It is also clearer in these samples that the increase in domain size also gets more pronounced with higher off-axis degree. The rotation of the substrate off-axis from [11-20] due to the production mistake is seen, the domains extend with an angle from the edge in 0.8 and 1.0 degree substrates, while in the 1.2 degree substrate the extension is parallel to an edge. The quarters are cut parallel or 90 degrees to the primary and secondary flat.

The growth rate for these samples was very high, ~900 µm/h, and as a consequence of the rapid growth the surfaces are quite rough. There is also an increased presence of triangular defects. The images below are reflection mode images at 50X magnification, off-axis degree increasing from left to right.

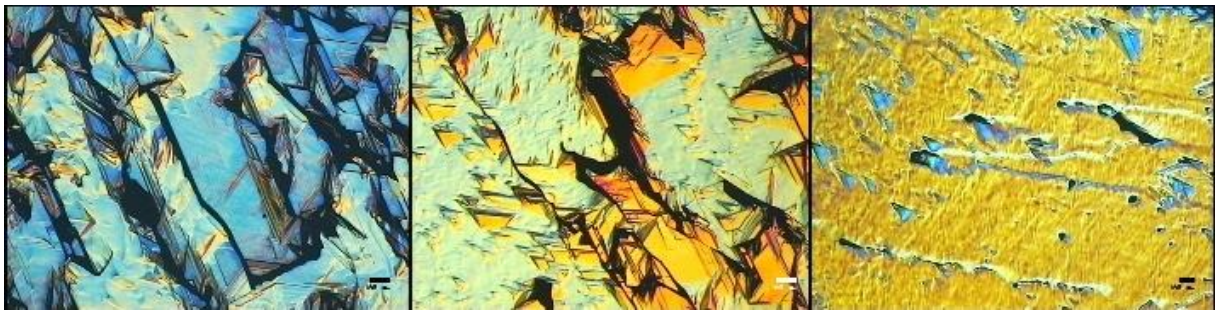


Fig. 5.16 50X reflection microscopy images. 0.8-1.2 degrees off-axis from left to right.

The dark areas in the images are wide cracks with jagged edges between the domains. The darkness here shows that the cracks are deep, and further it can be noted that the cracks run in a very irregular pattern. At 200X magnification (next page) the surfaces still look rough. It is especially clear how severe the interfaces between the domains are.

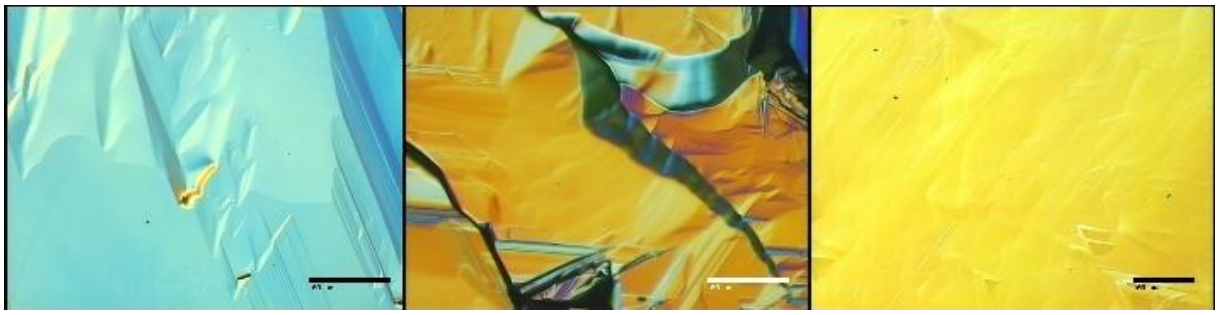


Fig. 5.17 200X reflection microscopy images.

Despite this it seems that growth within the domains can be more stable, which indicates that a more precise growth situation with a generally better quality is possible. The issue of 3C-SiC growth is to achieve large domains at stable parameters.

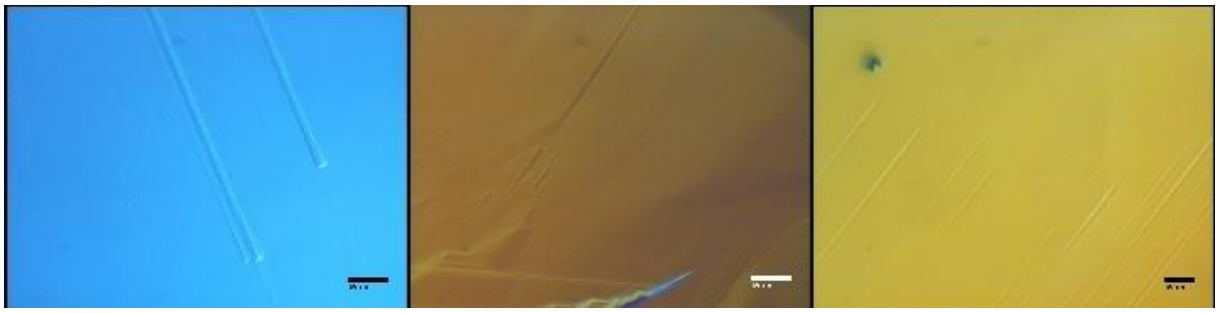


Fig. 5.18 1000X reflection microscopy images.

The local smoothness is confirmed in the 1000X pictures.

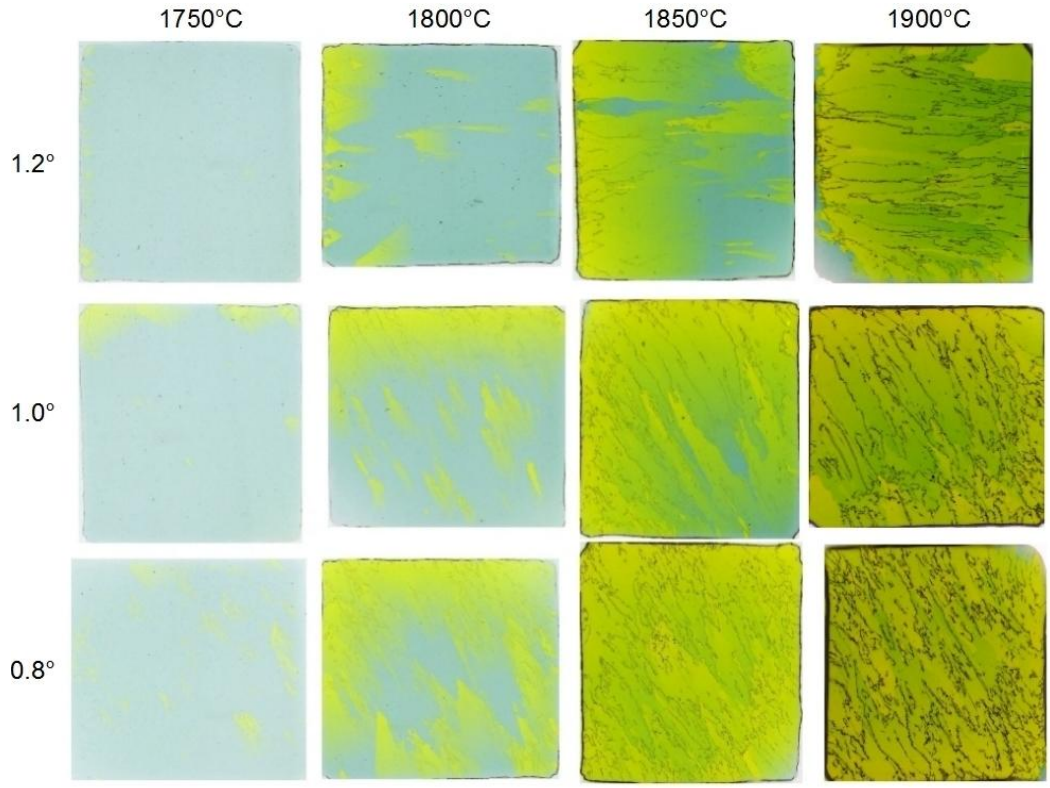


Fig. 5.19 An overview of all samples that were grown in nitrogen on 0.8, 1.0 or 1.2 degree off-axis substrates.

In the overview of all nitrogen grown samples, the tendencies mentioned above are visible. The high temperature samples fit the pattern well and no unexpected phenomena appear. The noticeably darker color of the high temperature samples is a consequence of their thickness.

5.7 Samples grown in Ar

In order to get indications about the reason for the domain widening effect depending on growth ambient, two samples were grown in argon at the same initial pressure as the nitrogen and compared with vacuum growth. The comparison is to be made between argon and nitrogen to explore the effect of gas pressure and gas type. Images of the corresponding vacuum samples are added for reference.

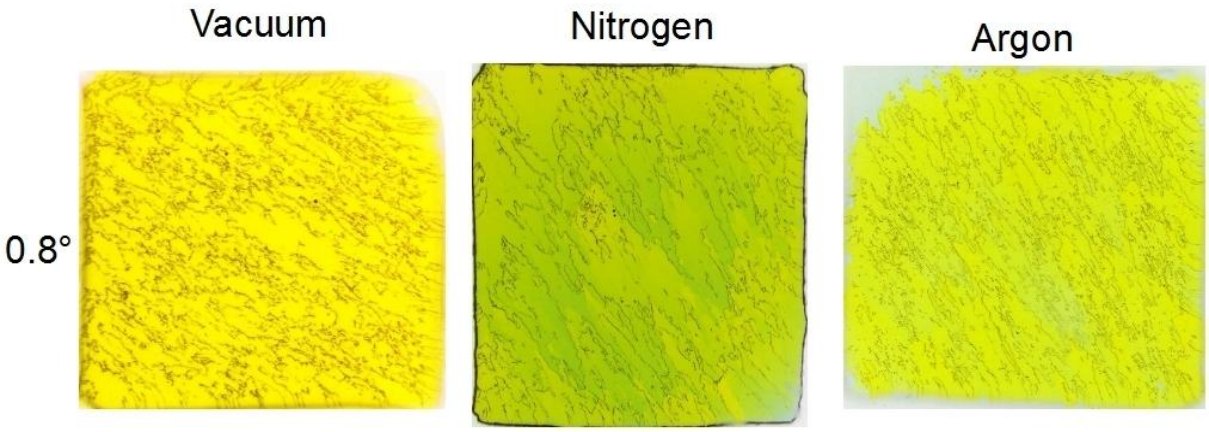


Fig. 5.20 Samples grown on 0.8 degree substrates at 1850°C in different ambient.

The conditions of using 0.8 degree off-oriented substrate and growth temperature at 1850°C were chosen due to the complete 3C coverage. The main idea is to understand the initial nucleation of the 3C-SiC, and the extension of domains.

The growth rates were similar, 409 µm/h in argon and 457 µm/h in nitrogen (568 µm/h in vacuum). The domains in the Argon sample appear to be somewhat smaller than on the nitrogen sample, but these two are still quite similar to each other, while neither of them is as similar to the vacuum grown sample. Apart from similar domain size, the argon and nitrogen samples also have similar 3C-coverage and we observed that the domain boundaries are more defective in the sample grown in vacuum than in the other two.

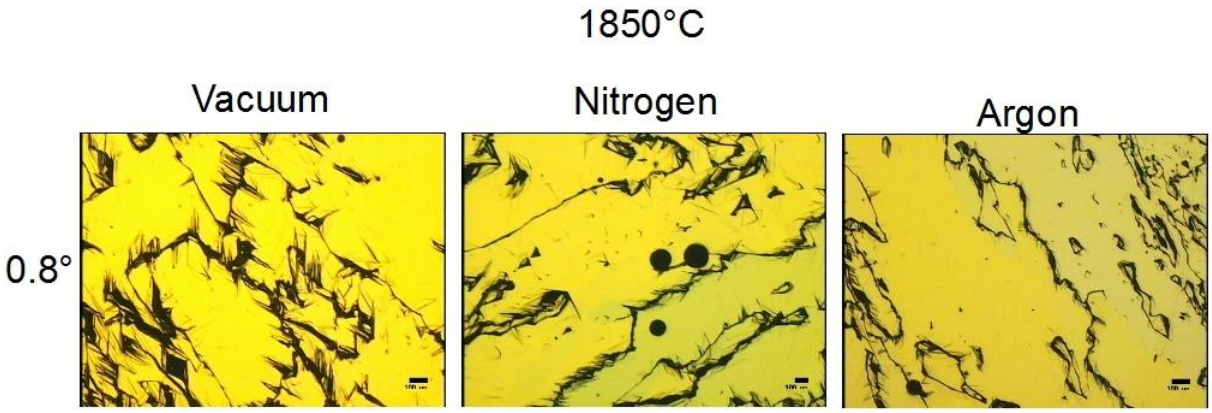


Fig. 5.21 50X transmission microscopy images.

In the above 50X transmission images it is clearer that the argon and nitrogen samples again resemble each other more than the one done in vacuum. The most pronounced difference is that there are rougher boundary edges in the vacuum grown sample.

As second sample set to compare the formation of 3C-SiC and homoepitaxial growth of 6H-SiC at different ambient conditions, the condition of 1.0 degree off-oriented substrate at growth temperature 1800°C was selected. The reason for choosing this comparative growth parameter point is that the nitrogen sample appears to be in a region right in between where 3C or 6H growth is dominating.

The growth rates were similar for these samples as well, 181 µm/h in argon and 195 µm/h in nitrogen (321 µm/h in vacuum). From the sample overview pictures below, we can also see that the growth characteristics in nitrogen and argon ambient are quite similar, though the amount of 3C is slightly lower on the argon sample.

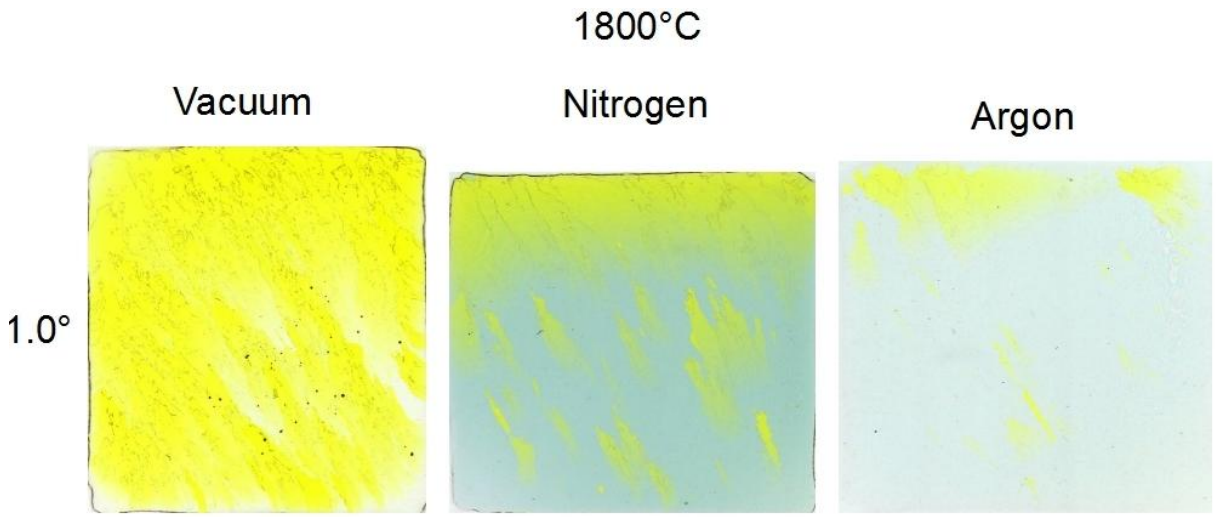


Fig. 5.22 An overview of the samples grown on 1.0 degree substrates at 1800°C with different gas backgrounds.

The 50X transmission images look the same as for the previous comparison, with the same relative properties.

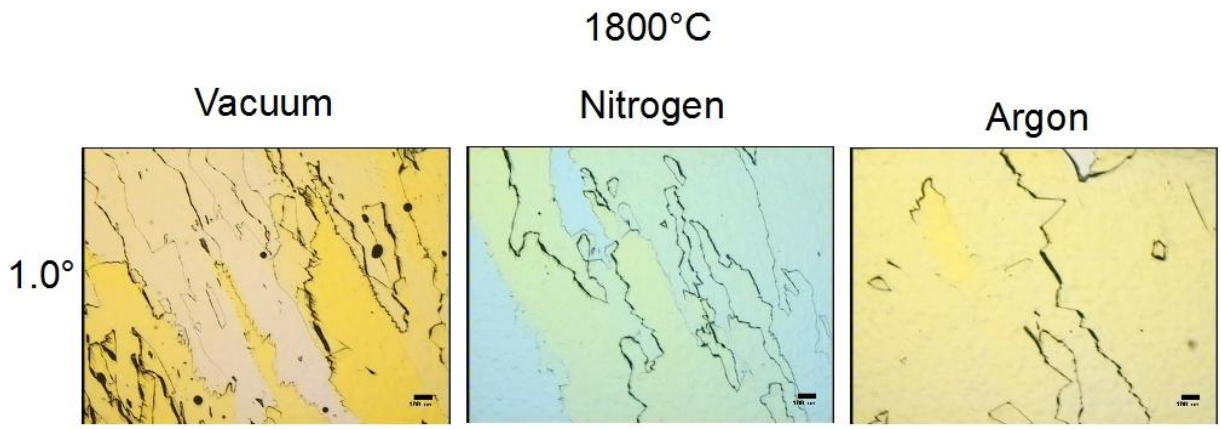


Fig. 5.23 50X transmission microscopy images.

The growth rates of these samples are given in overview in the diagram below.

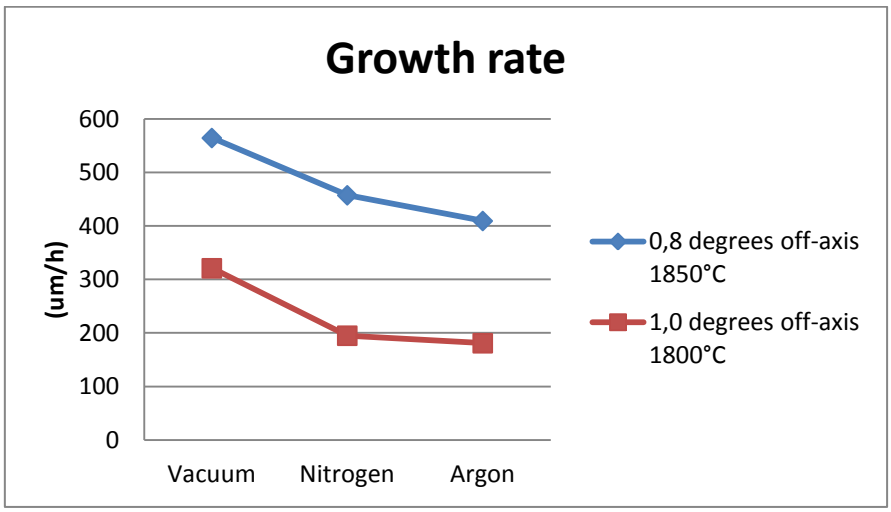


Fig. 5.24 Growth rates at different temperatures and ambients. The rate gets lower with the presence of gas in the chamber and is also slightly lower in argon than in nitrogen.

Although the above results indicate that it is the pressure rather than the nitrogen that causes the domain enlargement, there are some issues that could be high-lighted:

- Nitrogen and argon do not behave in the same way during the temperature ramp up. This means that although the initial pressures are the same, the actual growth will take place at different pressure levels.
- The fact that nitrogen is a doping gas means that there will be a larger shift in pressure during the nitrogen growth.

These differences make the comparison not completely applicable, although the tendencies of influence by growth rate, growth temperature and off-axis substrate orientation are clearly seen.

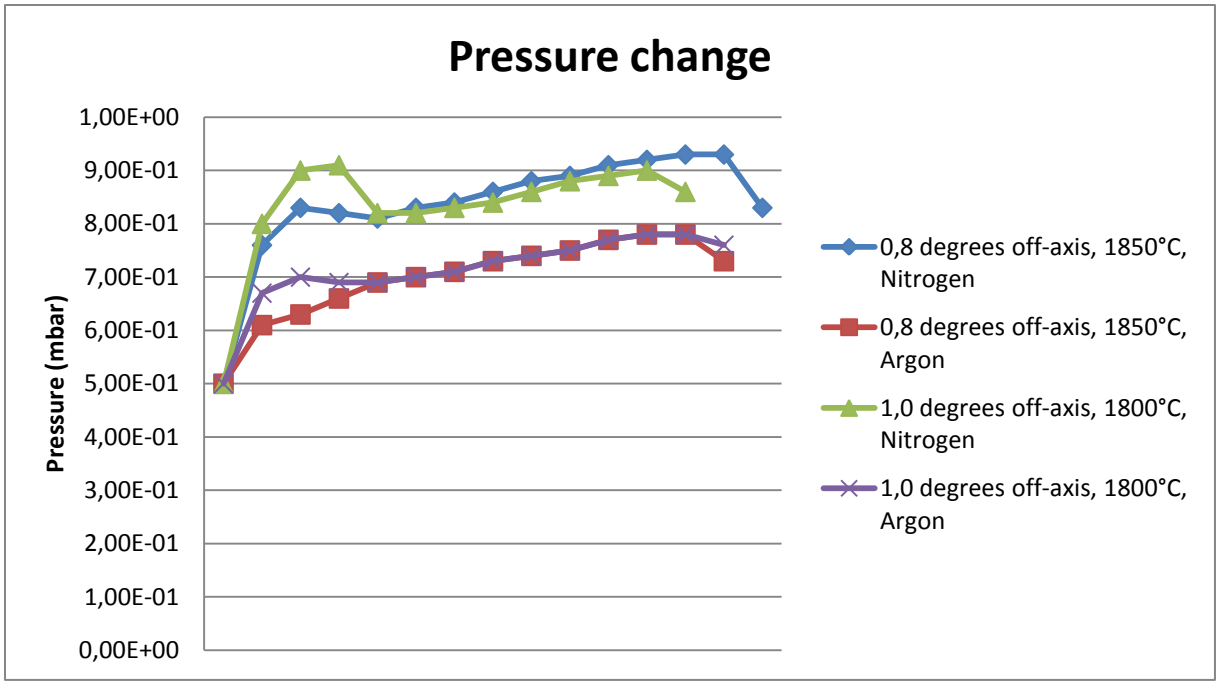


Fig. 5.25 The pressure during entire growth runs, from start to finish. The last bend of the curve corresponds to the drop in pressure during the growth phase.

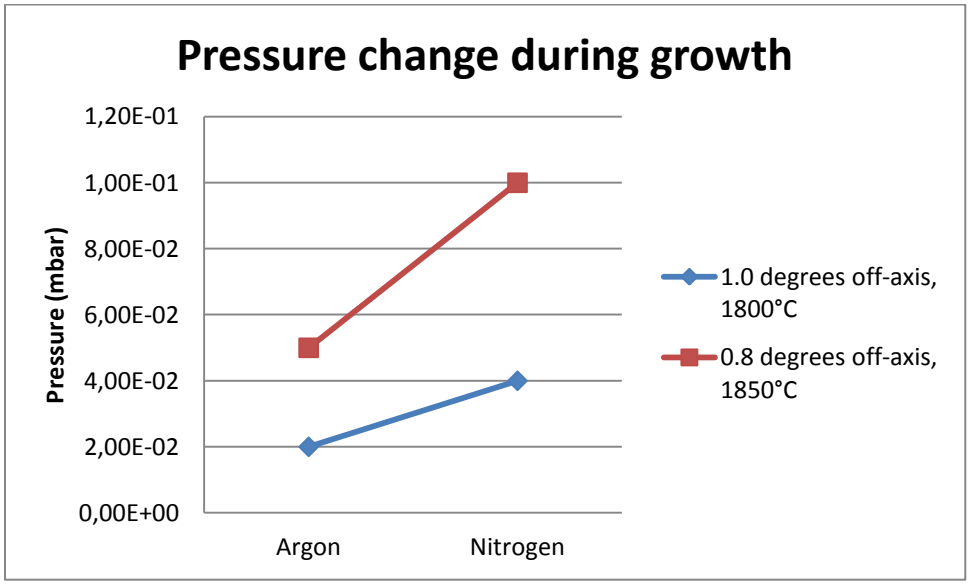


Fig. 5.26 For clarity purposes, this additional graph shows the total drop in pressure from growth start to growth end.

The above plots of the pressure during heating and growth clearly show the differences between nitrogen and argon ambient. Although the initial pressure at room temperature is the same, the pressure at growth will be higher with nitrogen in the chamber. We also see a difference in how much the pressure drops during growth. The drop is larger when using nitrogen, possibly since it participates in the growth, which argon does not.

5.8 2.0 degrees off-axis substrate in N_2 at 1900°C

To further explore the window parameter of growth temperature and substrate off-axis orientation, growth on 2 degree off-axis substrates was studied to see how the trend of the high temperature nitrogen growth would develop at still higher off-axis.

The growth rate of this sample was 929 µm/h, which is very high. High growth rate means that a lot of material from the source has been used and because of this the source has been graphitized. The following images illustrate the difference between the levels of source graphitization in growth at very high and lower temperature respectively. On the left hand side a source used for growth at 1900°C and on the right a source from a 1750°C growth.

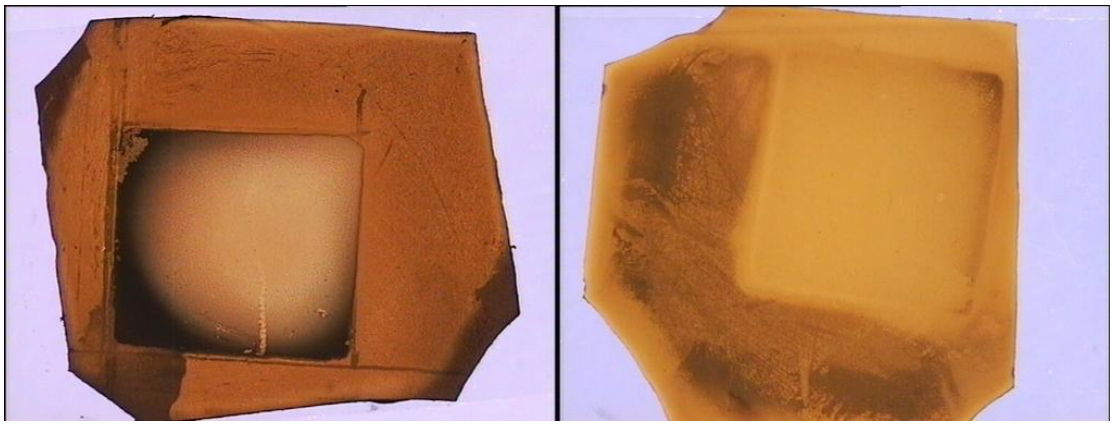


Fig. 5.27 Two SiC sources that have been used for growth at different temperatures. It is the square area in the middle that is of important for the growth. Graphitized areas are black.

The overview image of the sample is shown beside. From this we can observe that enlarged domains are still present but the higher off-axis degree has started to affect the 3C stability. The high temperature is no longer sufficient to reach full 3C coverage like in use of lower off-axis.

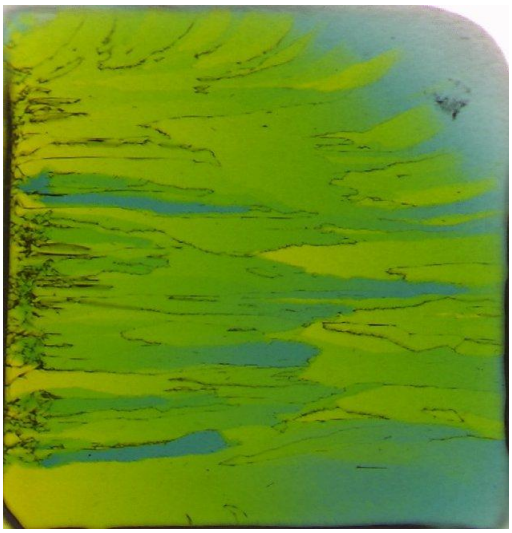


Fig. 5.28 2.0 degree sample grown in nitrogen at 1900°C.

The reason for the reduced 3C coverage is that the step-flow growth mechanism, which produces 6H in this case, is starting to be more favorable than island growth, at this higher off-axis degree. The higher degree makes it more likely that adatoms will attach at step sites rather than on the flat terrace surfaces where they create the starting points for the 3C-generating island growth.

Additionally the growth no longer proceeds mainly in the <1, 1,-2, 0> direction. Especially at the upper edge of the substrate an outward bending of the growth is noticeable. Initially we believed this to have happened due to a changed temperature profile at high growth temperature, but this was shown not to be the case by numerical simulations performed by Philip Hens, Erlangen University. A more likely explanation is a situation where step flow growth in the middle of the sample competes with on-axis nucleation at the edges. Similar behavior has been observed as a consequence of non-uniform supersaturation, the Berg effect, see [10].

The 50X reflection images (left hand side image below) show a coarse surface with deep gashes between domains. The transmission picture on the right hand side reveals how different the thicknesses of the 3C- and 6H-layers are: yellow 3C on the right, bluish 6H to the left and green overlapping layers covering the rest. Further magnification reveals nothing beyond more roughness and indications of rapid and uneven growth.

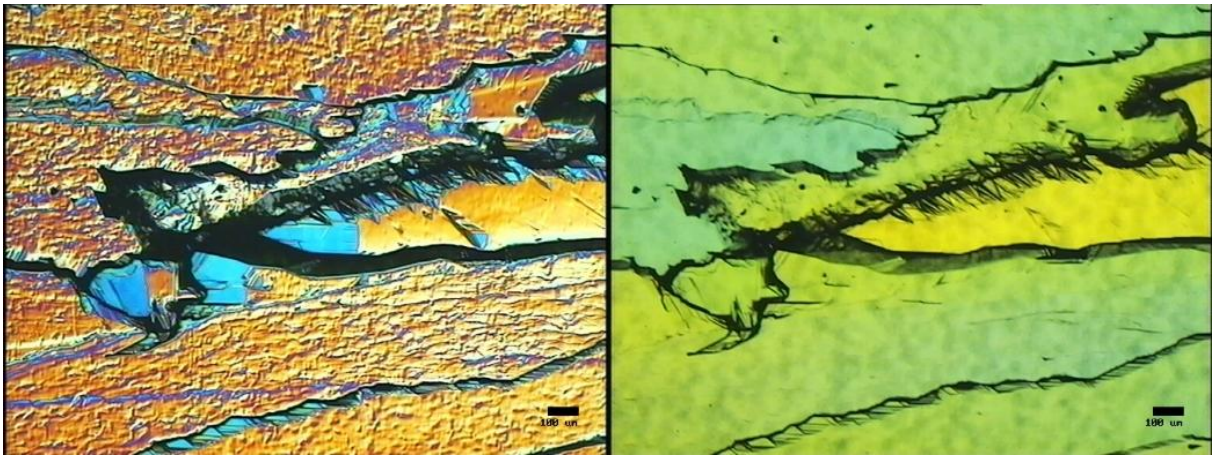


Fig. 5.29 50X images. Reflection mode on the left and transmission mode on the right.

However, the 400X (left) and 1000X (right) images below are fine examples of how something disturbs the step growth and causes a line defect that continues their extension in the <1,1,-2,0> direction.

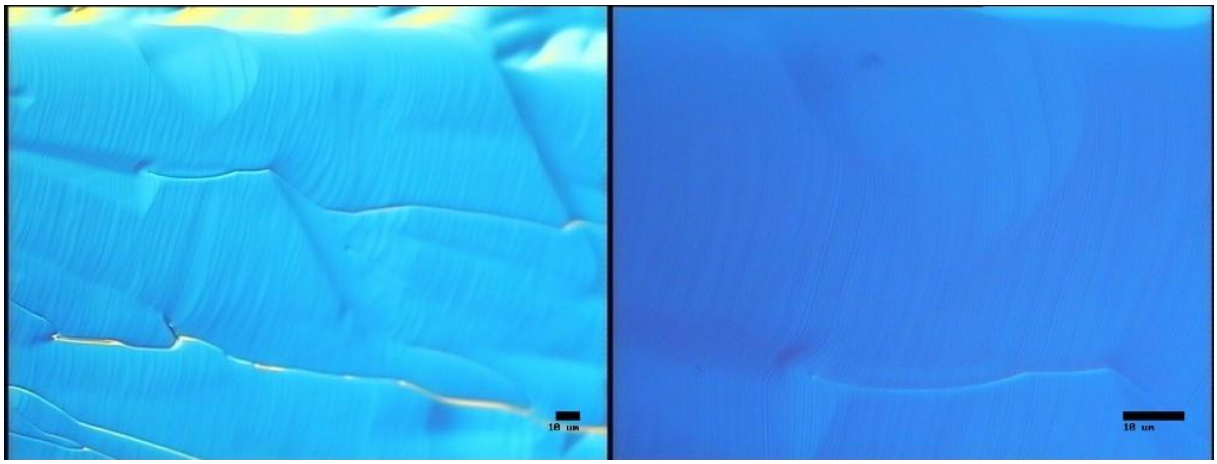


Fig. 5.30 400X reflection microscopy image on the left and 1000X reflection microscopy on the right. A disturbance initiates a line defect that grows with the step flow.

5.9 AFM characterization of steps in layers grown at 1750°C in N_2.

The growth on off-axis substrates proceeds by step-flow growth. Typically the earlier experience in vacuum growth on 3.5 degree substrates shows a step-bunching with steps which are 1.5-3.5 nm high and having terraces with 20-70 nm wide [11].

The samples grown in vacuum and nitrogen ambient at 1750°C on 1.2 degree substrates displayed 6H surfaces that were uniform and smooth enough that we decided to do a more detailed study with AFM. Two 2x2 µm images were taken of each sample. The height and width of the steps were measured and the roughness calculated. The same was done with the 0.8 and 1.0 degrees off-axis samples that were grown at 1750°C in nitrogen ambient, since they too displayed surfaces that to a large extent were covered by 6H-SiC of reasonable quality.

The images showed large similarity. During the step flow growth quite straight steps had formed and proceeded to flow in an ordered manner across the surface in the <1, 1,-2, 0> direction. Their widths and heights did not vary very much. As they are quite similar not all the images will be included. One is seen below.

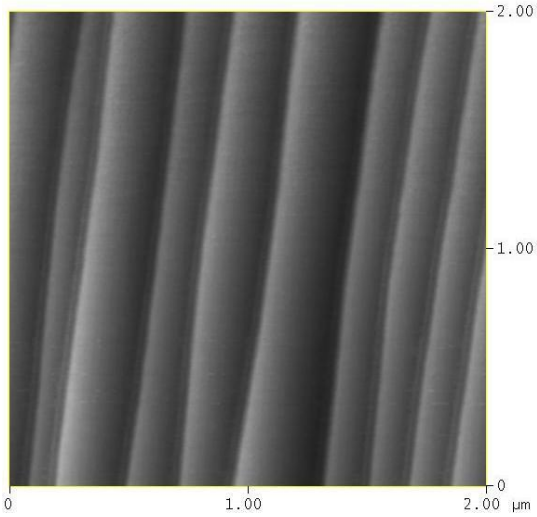


Fig. 5.31 Image no.2 of ELS201, 1.2 degree off-axis grown with a background of nitrogen at 1750°C.

Table 5.2 shows an overview of the samples selected for AFM characterization.

Experiment number	Off-axis degree	Ambient	Growth rate
ELS198	1.2	Vacuum	125.8
ELS201	1.2	Nitrogen	78.4
ELS213	1.0	Nitrogen	100.6
ELS207	0.8	Nitrogen	72.4

Table 5.2

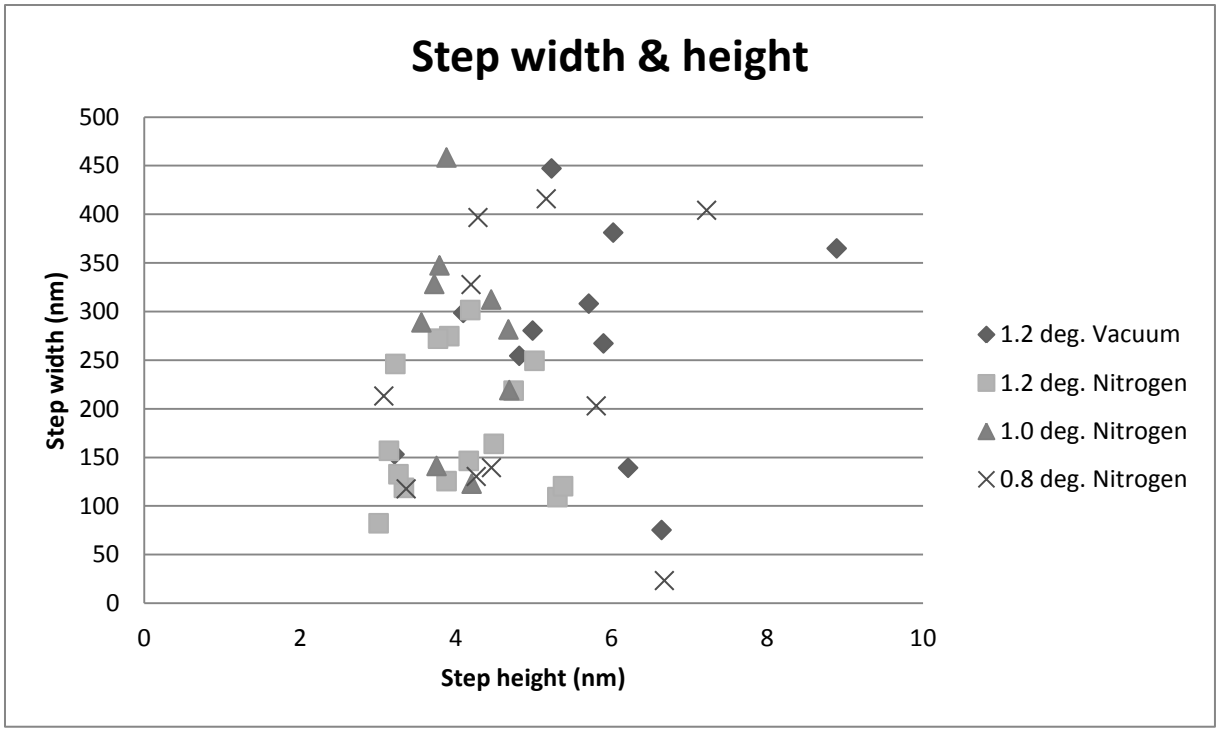


Fig. 5.32 Step heights and widths of all measured samples.

The above graph displays the height and width of all measured steps. It can be seen that most of the steps are grouped in a similar region and that the sample grown in vacuum stands out with a much larger dispersion than the nitrogen grown ones.

5.9.1 Average step height

Between eleven and fifteen step heights were measured for each sample. An average value for each sample was calculated, as well as the standard deviation of the values as an indication of the height dispersion.

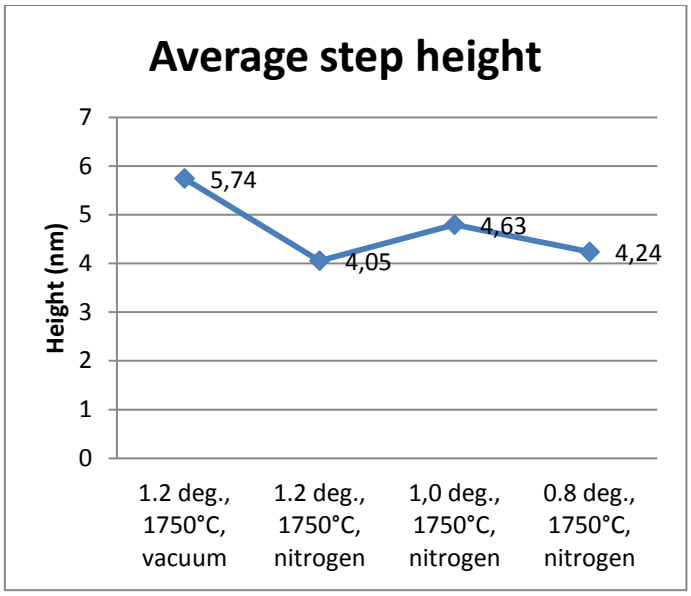


Fig. 5.33 Average step height of the different samples.

A comparison between the 1.2 degree off-axis samples (grown in vacuum and nitrogen) shows distinctive differences between the two ambients. The steps on the vacuum sample are generally larger and have wider height dispersion than on the sample grown in nitrogen. On the vacuum sample the growth fronts of the steps are of a more wavy nature than on the nitrogen samples where they are quite straight. Additionally, out of all the characterized sites, one on the vacuum grown sample shows the only occurrence of step splitting (fig. 5.34). The more disordered growth on the first sample is most likely a product of the decidedly higher growth rate.

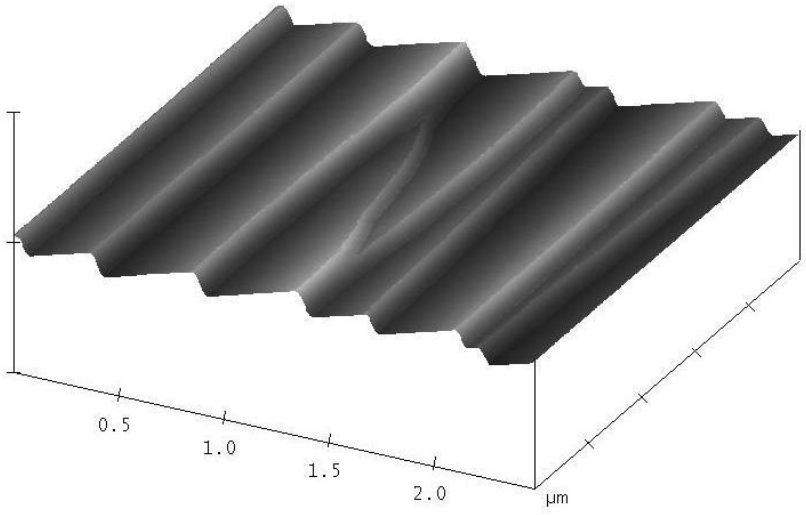


Fig. 5.34 AFM image of the sample grown in vacuum. A step splits into two smaller steps (with equal height).

When comparing the step heights on the different off-axis samples grown in nitrogen, we see that they all lie pretty closely within the same span, which is confirmed by a much lower standard deviation of the heights than in the vacuum case. There is also an increase in waviness of the growth fronts as the off-axis decrease. This comes of lowered step stabilization, due to the fact that the steps become lower and wider the closer you get to the on-axis situation, and thus lose influence.

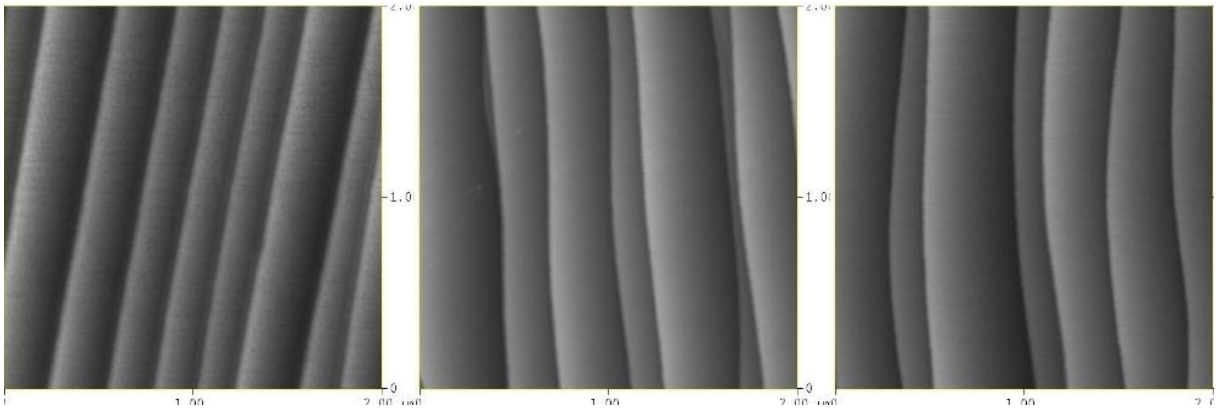
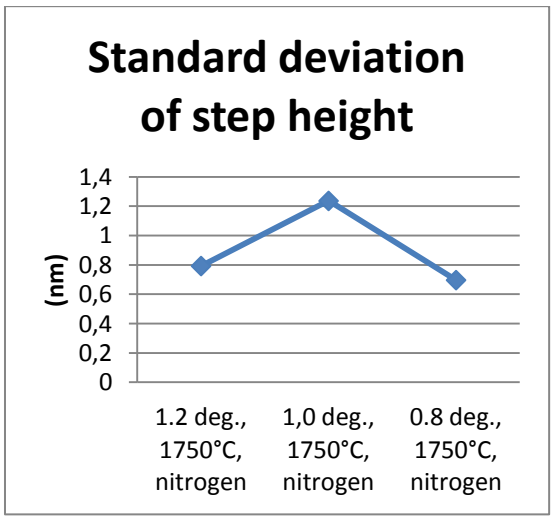


Fig. 5.35 1.2, 1.0 and 0.8 degrees off-axis samples, from left to right, with increasing waviness.

Although the average step heights for the sample grown in nitrogen ambient do not vary very much, the variation that does occur follows the variations in growth rate. This is also true for the standard deviations of step width for the three samples. In fig. 5.36 and 5.37 the striking correspondence between growth rate and the evolution of the steps is demonstrated.



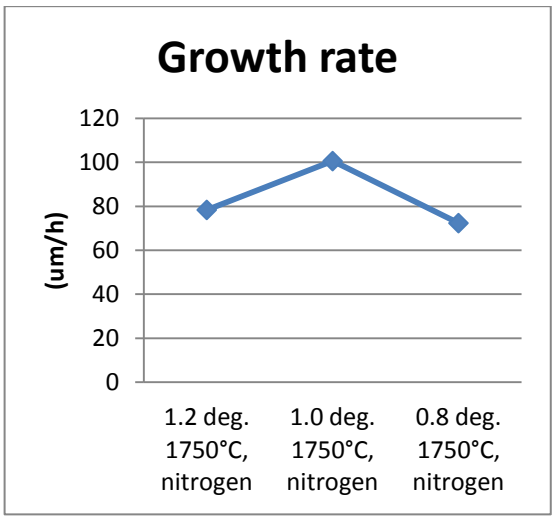


Fig. 5.36 Standard deviation of step height.

Fig. 5.37 Growth rate of the nitrogen grown 1750°C samples.

These results make it clear that growth rate is an important parameter for the step-bunching on low off-axis substrates. This information is potentially very important to have in order to achieve smooth epitaxial layers. Smoothness is necessary for further LED processing by nitride growth on doped low off-axis epilayers, i.e. making a light excitation nitride layer for a high power white LED. [5]

5.9.2 Average step width

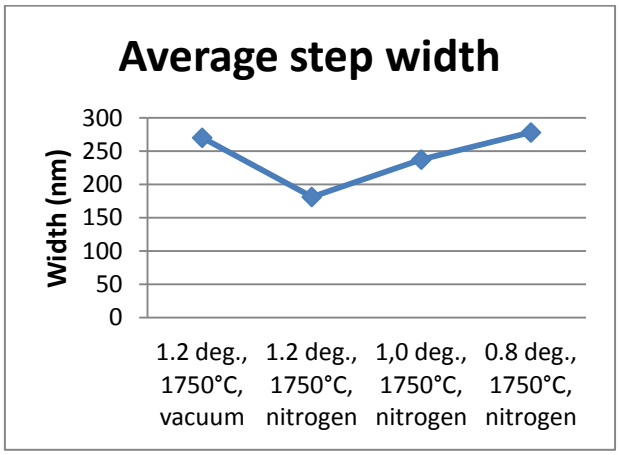


Fig. 5.38 Average step widths of the AFM-imaged samples.

The step width has a clear correlation with the off-axis degree. This is a natural consequence of the geometry of the substrates, higher cutting angle means more and thinner steps and vice versa. It seems to be a linear relationship between angle and width, but in order to make any useful predictions more measurements of more samples, cut at more angles are necessary.

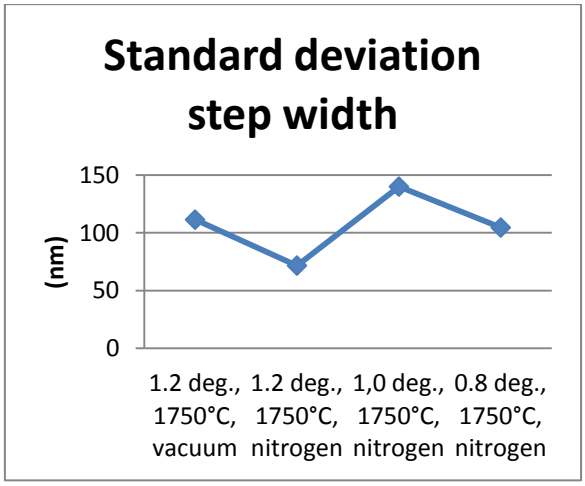


Fig. 5.39 Standard deviation of the step widths.

The dispersion of the step widths also corresponds to the growth rate, reinforcing the view that it is crucial for step bunching.

5.9.3 RMS

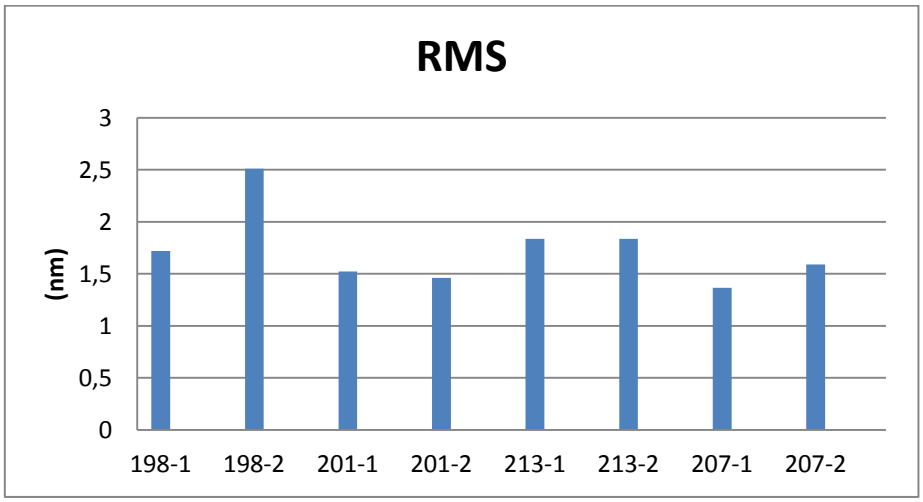


Fig. 5.40 RMS-values of all measured sites.

Fig. 5.40 shows the rms (root mean square) roughness of the eight AFM images that were made.

The surface roughness is quite similar in all samples grown in nitrogen. Once again the 1.0° off-axis sample, which had the highest growth rate, displays a slightly higher value.

The markedly higher value in vacuum samples is also a consequence of much higher growth rate.

Further device processing by nitride growth will show if as-grown surfaces with growth rates similar to those used in this thesis will be applicable, or if growth parameters (growth rate) have to be modified. The limit of surface roughness for nitride growth is in the 1-2 nm range.

Repetition of table 5.2 for references between samples and experiment numbers.

Experiment number	Off-axis degree	Ambient	Growth rate
ELS198	1.2	Vacuum	125.8
ELS201	1.2	Nitrogen	78.4
ELS213	1.0	Nitrogen	100.6
ELS207	0.8	Nitrogen	72.4

Table 5.2

6.0 Conclusions

Twenty four samples were grown by sublimation epitaxy under different conditions, in order to investigate the effects of using low off-axis substrates. They were then characterized and the following paragraphs contain the conclusions that can be drawn from the produced results.

The off-axis degree influences the growth mechanism. Even at the low degrees of substrate orientation used in this thesis, it is possible to affect which kind of polytype formation that will be the most favorable. The higher the off-axis angle, the more probable it is that 6H will be formed and the closer you get to the on-axis case, the more likely you are to get 3C.

The temperature is an important factor. The influence of growth temperature on polytype formation is very strong and may dominate and overcome the off-axis influence. Therefore it is important to be aware of the parameter window that is available for polytype steering by altering the off-axis angle at these low degrees of the substrates. This temperature window will also depend on the configuration of other growth conditions, such as gas background.

It was found that the stabilizing effect of nitrogen ambient on 6H growth, which has previously been studied in the case of growth on on-axis substrates, is also present when growing on low off-axis substrates. As long as the growth temperature is not increased beyond a certain point, the presence of nitrogen produces epilayers with almost 100% 6H coverage.

High enough growth temperature produces samples with complete 3C coverage and extremely high growth rates. However, they suffer from a lot of defects due to this rapid growth. The defects are mainly domain boundaries and problems associated with them.

The samples grown with nitrogen ambient display significantly enlarged 3C domains, at low as well as high temperatures. A pressure induced reduction of the supersaturation during the initial growth, which reduces the number of starting points for the 3C island growth, is suspected to be the reason behind this. Comparison with growth in Argon indicates that it might be the case, but some questions remain since Argon does not participate in doping while nitrogen does. At equal pressures, there will be an influence on diffusion of vapor species transport.

The critical point for growing thick 3C layers at high temperatures with full surface coverage lies between 1.2 and 2.0 degrees off-axis.

The AFM imaging of step bunching behavior on the 6H layers, grown in nitrogen at 1750°C, showed large correlation with the growth rate of the samples. Growth rate is therefore an important process parameter for the production of smooth layers on low off-axis substrates, since it greatly influences the roughness.

7.0 Further work

In this thesis some conclusions about the use of low off-axis substrates have been drawn. Before they can be really useful, however, some work still remains. This section presents suggestions for what could be done next to gain more knowledge.

Regardless of what you are trying to achieve, since this is an initial and very general study of the behavior of low off-axis growth, any specific use of the discovered characteristics first requires more detailed study. In order to find the right conditions for any particular type of growth, a fair deal of work remains. Optimization of process parameters is thus a necessary step.

The domain enlargement effect needs more investigation for certainty about the mechanism behind it. In order to investigate it properly, a series with better controlled growth pressure should be performed so that the results are more comparable. It would also be interesting to grow samples at high temperature on substrates with off-axis angles between 1.2 and 2.0 degrees, so that the critical point for 3C/6H formation can be found.

The low temperature growth of 6H in nitrogen ambient produced samples of some quality. If first more stable growth conditions are found and slightly better epilayers produced, a trial growth of an additional nitride layer on top would perhaps show if low off-axis grown layers can eventually be used in component production.

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