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Highly crystallized as-grown superconducting MgB2 thin films prepared by molecular beam epitaxy

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Abstract

This report illustrates the growth of superconducting thin films of magnesium diboride, a material that has been known since 1950, but only recently discovered to be superconductive at a remarkable high critical temperature of 40 K. The films are grown \textit{in situ} by molecular beam epitaxy on Si(111) covered with a 50 Å seed layer of magnesium oxide. The growth temperature was varied between 200 and 350 °C and superconducting films were obtained in the range of 200-325 °C. Other parameters like the Mg:B flux ratio, deposition rate and background pressure were also varied in order to determine which growth parameters provide the highest quality films. It was found that highly crystallized films can already form at 250 °C, but that there is a narrow window of growth parameters in which high quality films form. For each growth temperature one has to choose carefully the Mg:B flux ratio and deposition rate, and the background pressure should be lower then \( \sim 3 \times 10^{-8} \) Torr. The highest critical temperature of 35.2 K with a sharp transition width of 0.3 K was observed for a film grown at 300 °C. Using a capping layer of 30 Å of MgO proved to be highly beneficial for the preservation and the smoothness of the films. Together with the fact that MgO proved to be a good seed layer for thin films of MgB\(_2\) makes it an ideal candidate for serving as a barrier in MgB\(_2\) Josephson junctions.
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Chapter 1. Introduction

MgB₂ is a material that has been known since the early 1950's, but only recently it is discovered that it is a superconductor with a remarkably high critical temperature ($T_c$) of about 40 K [1]. This discovery revived the interest in the field of superconductivity, especially non-oxides, and initiated a search for superconductivity in related boron compounds. It was hoped that this material was the first in a series of diborides with a much higher $T_c$, but up to date MgB₂ holds the record among diborides. Since the discovery the physics community has shown a large interest in MgB₂ [2]. One might ask why, after all its critical temperature is only 40 K, more than three times lower than the 134 K attained by the mercury-based high-$T_c$ superconducting (HTSC) cuprates. Besides, there are already wires made of high-$T_c$ copper oxides which operate above liquid nitrogen temperature (77 K). An important reason is the cost, HTSC wires are for 70% composed of silver and therefore expensive. Other reasons are that the coherence length of MgB₂ is longer than those in HTSC cuprates, and its grain boundaries have a far less detrimental effect on superconducting current transport [3]. These properties hold tremendous promise for electronic applications. MgB₂ promises a higher operating temperature and higher device speed than the present electronics based on Nb. Moreover, high critical current densities ($J_c$) can be achieved in magnetic fields by oxygen alloying [4], and irradiation shows an increase of $J_c$ values [5].

The fabrication of superconducting MgB₂ thin films is very important for many electronic applications. There are several reports on MgB₂ thin film preparation techniques [4,7-12], however most of these methods require a post-deposition annealing process to produce superconducting MgB₂ thin films. These methods provide fair quality MgB₂ thin films, but as-grown thin films are preferable for fabricating junctions and multilayers. This report describes the synthesis of highly crystallized as-grown superconducting thin films of MgB₂ using molecular beam epitaxy (MBE). In chapter 2 it is reviewed what is known in literature about the properties of MgB₂ and its structure, how according to the thermodynamics of the Mg-B system thin MgB₂ films can be grown and what is already accomplished up to date. Chapter 3 describes how MBE works and the instruments that can be used for analyzing a sample. The experimental setup is described in chapter 4 together with the cleaning of the different substrates and how the samples are grown and analyzed. Chapter 5 shows the results obtained from Auger electron spectroscopy, resistance versus temperature measurements, X-ray diffraction, and atomic force microscopy. These results are discussed in chapter 6, some conclusions are drawn from the comparison with the results of other groups and it is described how the MgB₂ thin films can be improved and used for junction fabrication.
Chapter 2. MgB$_2$ in literature

2.1 Introduction

Akimitsu's group reported the superconductivity of MgB$_2$ on January 10$^{th}$ 2001 at a conference in Sendai, Japan [13]. Since then a lot of studies have appeared about this superconductor with an average of 1.3 papers/day until the end of July 2001. The topics of these studies cover a wide area of subjects, such as preparation in the form of bulk, thin films, wires, tapes; the effect of substitution with various elements on $T_c$, isotope and Hall effect measurements, thermodynamic studies, critical currents and fields dependencies, to microwave and tunneling properties. Buzea and Yamashita wrote a review article on the main normal and superconducting state properties of MgB$_2$ based on these studies [2]. This chapter describes some of the properties of MgB$_2$ and reviews the most important results for the growth of thin films by MBE.

2.2 MgB$_2$ structure and properties

MgB$_2$ possesses the simple hexagonal AlB$_2$-type structure (space group P6/mmm) which is common among borides (figure 2.1).

It contains graphite-type boron layers which are separated by hexagonal close-packed layers of magnesium. The magnesium atoms are located at the center of hexagons formed by boron atoms and donate their electrons to the boron planes. MgB$_2$ appears to be superconductive by the Bardeen-Cooper-Schrieffer (BCS) mechanism [14] and seems to be a low-$T_c$ superconductor with a remarkably high critical
temperature, its properties resembling that of conventional superconductors rather than of HTSC cuprates. This includes an isotope effect [15,16], a linear \( T \)-dependence of the upper critical field with a positive curvature near \( T_c \) [17], and a shift to lower temperatures of both \( T_{c^{onset}} \) and \( T_{c^{zero}} \) at increasing magnetic fields as observed in resistivity measurements [18,19].

On the other hand, the quadratic \( T \)-dependence of the penetration depth \( \lambda(T) \) [20-22] as well as the sign reversal of the Hall coefficient near \( T_c \) [23] indicates unconventional superconductivity similar to cuprates. The key of the higher \( T_c \) may be the layered structure of MgB\(_2\) [2].

### 2.3 Thermodynamics of the Mg-B system

Due to the high volatility of Mg, difficulties in fabricating MgB\(_2\) thin films by in situ depositions are anticipated. On the other hand, such volatility can greatly simplify composition control by enabling adsorption-controlled film growth [24]. An understanding of the thermodynamics of the system can help to identify the appropriate growth region for materials containing a volatile constituent [25-29].

Zi-Kui Liu et al. presented a thermodynamic analysis of the Mg-B system with the thermodynamic calculation of phase diagrams (CALPHAD) modeling technique using a computerized optimization procedure [30]. They found that the MgB\(_2\) phase is thermodynamically stable only under high Mg partial pressures. Figure 2.2 shows the pressure-temperature phase diagram for the Mg:B atomic ratio \( x_{Mg}/x_B \geq 1/2 \) that they obtained extended to \( 10^{10} \) Torr.
The region of Gas+MgB$_2$ represents the thermodynamic stability window for the deposition of MgB$_2$ thin films. There are three intermediate compounds, MgB$_2$, MgB$_4$, and MgB$_7$, in addition to the gas, liquid, and solid (hcp) magnesium phases and the $\beta$-rhombohedral boron solid phase [31]. From a thermodynamic perspective, deposition of a single-phase MgB$_2$ film becomes easy when the growth conditions (substrate temperature and Mg overpressure) fall within the window where the thermodynamically-stable phases are the desired MgB$_2$ phase and gas phases. Within this growth window MgB$_2$ does not decompose and excess Mg does not condense on the MgB$_2$ surface, thus the formation of single-phase MgB$_2$ is adsorption controlled and automatic. As long as the Mg:B ratio is above the stoichiometric $1:2$, any amount of extra Mg above the stoichiometry will be vaporized and the desired MgB$_2$ phase will result. The more critical requirement for controlling the stoichiometry is to avoid insufficient Mg supply, which will lead to MgB$_4$, MgB$_7$, or solid B phases.

The thermodynamic stability window for MgB$_2$ film deposition is best illustrated by the pressure-temperature phase diagram shown in figure 2.2. If the Mg overpressure is too low, it is thermodynamically favorable for MgB$_2$ to decompose into MgB$_4$ (+Gas). If it is too high, it is favorable for Mg to condense on to the MgB$_2$ surface. For a given deposition temperature, one can find the Mg partial pressure range to keep the MgB$_2$ phase thermodynamically stable.

The kinetics of crystal growth require that an in situ film deposition process takes place at sufficiently high temperatures. The optimum temperature for epitaxial growth is typically about one half of the melting temperature which means a temperature for deposition of epitaxial MgB$_2$ film is around $\sim$1080 °C [30]. For MgB$_2$ to be stable at that temperature, a
Mg partial pressure of at least 11 Torr is required, which is impossible for many thin film deposition techniques. The minimum temperature though for epitaxial growth can be much lower [32]. For example, the minimum epitaxial growth temperature for metals is about 1/8 of the melting temperature [33].

2.4 As-grown MgB_{2} thin films

The main problem in forming MgB_{2} films is the high volatility of Mg. Kenji Ueda et al. obtained as-grown superconducting films in the limited growth temperature range of 150-320 °C [34]. Their films were poorly crystallized but showed a sharp (ΔT≤1 K) superconducting transition of as high as 36 K (\(T_{c,\text{ onset}}\)). The films were grown on SrTiO_{3} (001), sapphire r-plane and c-plane substrates, and Si (111).

Figure 2.3 shows the molar concentration of Mg (compared with that of B_{2}) in the film as a function of the growth temperature on the basis of inductively-coupled plasmaspectrometer (ICP) analysis. The films formed above 350 °C show a significantly deficit in Mg even with a 10 times higher Mg rate. They suggested that this is the growth temperature limit in terms of forming as-grown superconducting MgB_{2} films using molecular beam epitaxy. The films formed below 300 °C contained excess Mg which may effect the resistivity of the film, but should not effect the critical temperature (\(T_{c}\)).

![Figure 2.3. Molar concentration of Mg in the film versus growth temperature.](image-url)
They studied the crystal structure with 2θ X-ray diffraction (XRD). Only tiny peaks were observed for films formed on Si(111) and sapphire C which were attributed to MgB₂ (00l) peaks (figure 2.4).

The films formed in the growth range of 150-320 °C showed superconductivity in spite of their poor crystallinity.
Figure 2.5 shows the resistivity versus temperature curves of MgB$_2$ thin films on Si(111) as a function of the growth temperature.

The transition temperatures ($T_c^{\text{onset}}-T_c^{\text{zero}}$) were 12.2-5.2, 26.3-25.2, and 33.0-32.5 K for 150, 183, and 283 °C, respectively. The $T_c$ increased as the growth temperature increased. Films grown above 320 and below 83 °C were semiconducting or insulating and did not show superconductivity.
Chapter 3. Molecular Beam Epitaxy

3.1 Introduction

Molecular beam epitaxy was developed in early 1970 as a means of growing high purity epitaxial layers of compound semiconductors. Since that time it has evolved into a popular technique for growing III-V compound semiconductors as well as elemental semiconductors. MBE may also be used to grow insulating layers and single-crystal metal films [35]. A newer branch is the growth of multilayers of metallic films for superconducting applications.

Molecular beam epitaxy is an epitaxial growth process involving the reaction of one or more thermal beams of atoms or molecules with a crystalline surface under ultrahigh vacuum (UHV) conditions. It can produce clean surfaces in many crystallographic orientations and well-defined compositions. Since the growth is conducted in UHV, many surface diagnostic instruments may be incorporated into the system. Thus the analysis of a surface can be performed immediately after concluding the growth of a fresh surface without exposure to atmospheric contamination. MBE can produce high quality layers with very abrupt interfaces and good control of thickness, doping, and composition. Because of the high degree of control it is a valuable tool in the development of sophisticated electronic and optoelectronic devices.

3.2 System configuration

Figure 3.1 displays a schematic of a MBE chamber. In MBE, molecular beams generated from thermal Knudsen crucibles or e-gun sources interact on a cooled/heated crystalline substrate to form thin epitaxial layers. Each source contains one of the constituent elements or compounds required in the grown film. The temperature of each source is chosen so that films of the desired composition may be obtained. The sources are arranged around the substrate in such a way as to ensure optimum film uniformity both of composition and thickness. Additional control over the growth process can be achieved by inserting mechanical shutters between each individual source and the substrate. The flow of components from the source to the substrate is in the molecular and not hydrodynamic flow region [35]. Thus the beams can be considered, for all practical purposes, as unidirectional with negligible interaction within them. The insertion of a mechanical shutter between source and substrate will then effectively stop the beam from reaching the substrate, and so allow different crystal compositions to be superimposed on each other.
Crystal quartz monitors are used for monitoring the beam fluxes. These use the piezoelectric properties of crystalline quartz to determine the deposition rate [36]. To obtain high purity layers, it is critical that the material sources are extremely pure and that the entire process is done in UHV. The sources and the growth environment are therefore usually surrounded by liquid nitrogen cooled cryopanels to minimize unintentional impurity incorporation in the deposited layers from the residual background, and the whole is confined within an ultrahigh vacuum environment where base pressures are $\sim 5 \times 10^{-11}$ Torr.

### 3.3 Surface analysis

Because MBE is a UHV technique the whole range of surface analysis techniques can be used to monitor the growth process before, during, and after deposition. Reflection High Energy Electron Diffraction (RHEED) is often used because it gives information about substrate cleanliness and growth conditions. Mass spectrometry is useful for residual gas analysis and leak detection.
Auger Electron Spectroscopy (AES), which is described in detail elsewhere [37], is used for analyzing the topmost 10-30 Å of a sample with a detection limit of 0.1-1% of a monolayer. AES is used principally for measuring elemental atomic concentrations but it is also sensitive to the chemical environment. Shifts in Auger peaks can be used to provide information on compounds. When used together with an Ar⁺ ion sputter gun, compositional depth profiles can be built up, although the technique is not sensitive enough to follow dopant concentrations. It can also be used to laterally map the distribution of atomic species across a surface with a spatial resolution <0.2 μm. AES is primarily used in MBE for the study of surface stoichiometry.

X-Ray Photoelectron Spectroscopy (XPS) is a near surface technique similar to AES [38]. However, the spatial resolution is low (>200 μm). It differs from AES in that large chemical shifts in peak position dependent on chemical composition can be observed. This makes it for example a very useful technique for the study of the type of compounds formed on substrate surfaces after chemical etching.
Chapter 4. Experimental

4.1 Experimental setup

A schematic of the MBE setup that is used for the experiments is displayed in figure 4.1. The setup consists of a high vacuum chamber (load lock), an ultra high vacuum chamber (MBE chamber) and a sample manipulator (probe). The MBE chamber is surrounded by a liquid nitrogen cooled cryoshroud which acts as an effective pump for many of the residual gasses in the chamber. The chambers are separated by a gate valve (GV).

![Figure 4.1. Schematic of the MBE setup.](image)

The load lock is used to bring samples into and out of the vacuum environment while maintaining the vacuum integrity of the MBE chamber. It also has a glow discharge facility. After the insertion of a sample it is pre-pumped by a roughing pump (Leybold Trivac) to achieve a medium vacuum state and then a turbo pump (Leybold Turbovac) is used to create a high vacuum state.

The MBE chamber is pumped by a cryo pump (CTI Cryogenics Cryo Torr 8) and an ion pump (Varian Vacion 200 1/s). They create a base pressure of $10^{-10}$ Torr after the pre-pumping by the pumps in the load lock and maintain a pressure of $10^{-9}$ Torr during evaporation. The MBE chamber houses two e-guns which can contain five different sources each and it contains four Knudsen cells (K-cells) so a total of fourteen different sources can be placed simultaneously in the chamber. Each K-cell is controlled by a deposition controller (Leybold Infinicon XTC/2) and two of the controllers are also used to monitor the deposition rate when the e-guns are used.
Samples can be analyzed by Auger Electron Spectroscopy (AES) (Physical Electronics Industries model 15-255GAR) and X-ray Photoelectron Spectroscopy (XPS) (Physical Electronics Industries model 04-500) or sputtered by an ion gun (Physical Electronics Industries model 04-191). There is also a mask manipulator present which contains seven different masks and makes in situ junction fabrication possible. The Residual Gas Analyzer (RGA) (MKS Instruments PPT 100EM) can be used for the detection of the various constituents in vacuum.

4.2 Substrate preparation

Films were grown on various substrates (Si(111), sapphire, SiC, and GaN) and most substrates required a different cleaning method. A clean surface is an important prerequisite for epitaxial growth, since contaminants from the atmosphere or other sources can easily contaminate a clean substrate and cause crystal defects or degrade the electrical characteristics of the epitaxial layer.

Si(111) substrates were first rinsed with deionised water to get rid of dust and then cleaned ultrasonically with isopropyl alcohol. After that they were treated with a sulfuric acid (H$_2$SO$_4$ 10%) and subsequently etched with a 10% hydrogen fluoride (HF) solution.

Sapphire was first cleaned ultrasonically in a deionised water-detergent mixture and then treated with alcohol in a condensor. SiC was cleaned the same way as Si(111). GaN was first treated with TCE, Acetone, and methanol and subsequently etched with a 20% solution of hydrochloric acid (HCl). After the chemical cleaning ex situ, the samples were cleaned in situ by heating them 25 °C above the growth temperature ($T_s$).

4.3 Film growth

Si(111) substrates were first covered with a 50 Å thick seed layer of MgO which was deposited on the substrate at the same temperature as the Mg-B growth temperature which ranged from 200-350 °C. The flux ratio of Mg to B varied from 2 to 6 times the stoichiometric ratio of 1:2 to compensate for the volatility of Mg. The deposition rate varied from 1.5 to 3 Å/s and the film thickness was typically 1000 Å. This is the thickness calibrated from what is detected by the crystal quartz monitors. The actual thickness is smaller (~ 400 Å) mostly because of the volatility of Mg. After the Mg-B depositions a cover layer of 30 Å of MgO was deposited for protection of the film. Films without this protective layer started to deteriorate even after a couple of hours.

Films on sapphire, GaN, and SiC were grown without a seed layer and at a substrate temperature of 300 °C. The flux ratio of Mg to B was approximately 5-6 times the nominal ratio for the films grown on those substrates because of difficulties with the evaporation of boron. When cooling down B one has to be very careful because it explodes upon rapid cooling.
4.3 Film analysis

The samples were analyzed by Auger Electron Spectroscopy (AES), Atomic Force Microscopy (AFM), X-Ray Diffraction (XRD), and by determining the resistance versus temperature.

AES measurements were performed *in situ* before the protective MgO cover layer was deposited. The beam voltage was 3 kV and the step size 300 ms. The resolution was 0.5 eV and the scan had a range from 10-1300 eV when Si(111) was used as a substrate, 10-1500 eV for GaN, and 10-1700 eV for SiC.

The resistance versus temperature characteristics were determined by connecting the sample to a sample holder and subsequently lowering the sample holder in a liquid helium filled dewar. The voltage of the sample was determined by a four point measurement with a current of 10 mA for the first few measurements. Later a current of 10 μA was used to minimize the influence of the current on the $T_c$. Contacts were made by either depositing 1500 Å indium on a seed layer of 30 Å chromium in thin stripes across the sample, or by pressing fresh cut indium directly on the sample. Copper wires and silverpaint were then used to connect the sample to the sample holder. Using only silverpaint for the contacts gave large fluctuations during measurement because it reacted with the film.

The AFM scans were made with a burleigh METRIS 2000 microscope. First a topographic image was made on a scale of 50,000 x 50,000 nm and then the magnification was increased until the resolution was too poor to observe any structures, usually at 100 x 100 nm.

XRD spectra were obtained with a Rigaku RTP 500 RC diffractometer. It used a wavelength of 1.54 Å and the 2θ-θ scans were made in the range from 10 to 70°.
Chapter 5. Results

5.1 Auger Electron Spectroscopy

Figure 5.1 shows an Auger spectrum of a Si(111) substrate after the cleaning procedure described in the previous chapter. The spectrum was made with a beam energy of $V_b = 3$ keV and a resolution of 0.5 eV.

![Auger spectrum of a clean Si(111) substrate.](image)

The spectrum contains no peaks from contaminations which indicates that the substrate has a clean surface. Figure 5.2 shows two spectra of a superconductive MgB$_2$ film grown at 300 °C with a Mg:B flux ratio of 2.0:1.0 measured after different time intervals.

![Auger spectra of a superconductive MgB$_2$ film measured after different time intervals.](image)
The numbers in the figure indicate the atomic concentration. From the scans it can be seen that the film has an off-stoichiometric composition and that it is contaminated with oxygen. Furthermore, it shows that the oxygen concentration almost doubled after half an hour, even while the film remained in a UHV chamber with a background pressure of \(6.0 \times 10^{-10}\) Torr.

Figure 5.3 shows two scans obtained from different MgB\(_2\) films, one grown at a temperature of 300 °C and a Mg:B flux ratio of 2.0:1.0 (a), and the other with \(T_s = 310\) °C and a Mg:B flux ratio of 1.8:1.0 (b).

The oxygen contamination in both films is almost the same, however the magnesium and boron concentrations are different. The film with the Mg:B ratio closest to the stoichiometric ratio of 1:2 has the highest critical temperature as can be seen from the figure.

### 5.2 Resistance measurements

Figure 5.4 shows the resistance versus temperature for three MgB\(_2\) films grown on a Si(111) substrate covered with a 50 Å thick MgO seed layer. The transition temperatures \((T_c^{\text{onset}}-T_c^{\text{zero}})\) are 21.3-19.2, 29.9-29.4, and 35.2-34.9 K, respectively for films grown with a Mg:B flux ratio and critical temperature of 1.0:1.0 and 200 °C, 3.0:1.0 and 250 °C, 1.5:1.0 and 300 °C.
Figure 5.4. Resistance versus temperature for three different MgB$_2$ films.

The used Mg:B flux ratio and the temperature of the substrate during growth ($T_s$) are also indicated in the figure. The critical temperature increases with growth temperature, but the resistance of the film varies independently.

A total of 46 films were grown under various conditions and on different substrates. A table with the characteristics of all the grown samples can be found in appendix A. One can see that only 12 of the 46 samples grown showed superconductive behavior. This depended on $T_s$, the Mg:B flux ratio, the pressure during deposition ($p_{dep}$), the deposition rate, and the substrate or seed layer. Films showed superconductivity at a growth temperature range from 200 to 325°C, however only when the background pressure was lower than $\sim 3.0 \times 10^{-8}$ Torr. All films grown with $p_{dep} > \sim 3.0 \times 10^{-8}$ Torr showed insulating behavior. Some samples grown at lower background pressures still showed insulating behavior because the chosen Mg:B flux ratio or the deposition rate was not suitable compared with the chosen growth temperature to get superconductive films.

5.3 X-Ray Diffraction

Figure 5.5 and 5.6 display the XRD-spectra of different MgB$_2$ films. Scans were made with $\lambda = 1.54$ Å and $V_b = 60$ kV. The $T_c$ (left), Mg:B flux ratio (middle) and $T_s$ (right) are also indicated for each film.
Figure 5.5. XRD-spectra of different MgB$_2$ films.

Figure 5.6. XRD-spectra of highly crystallized MgB$_2$ films.
The spectra in figure 5.5 contain some other peaks besides the ones that correspond to MgB\(_2\). The peak that appears around 15° in the \(T_s = 200\) and 325 °C spectra corresponds to a direction in B\(_2\)O\(_3\), but might originate from another compound. The same spectra contain a second substrate peak at ~59° that is caused by a false reflection which can also be observed in a XRD pattern of a bare substrate. The peak around 35° in the \(T_s = 200\) and 300 °C spectra originates from solid Mg in the corresponding films.

From the figures it can be seen that the degree of crystallization is not only a function of growth temperature, but also a function of the Mg:B flux ratio that is used. Figure 5.5 shows that even very poorly crystallized films are superconductive, however with a reduced \(T_c\). A higher degree of crystallization corresponds with a higher critical temperature. Figure 5.6 shows that the as-grown MgB\(_2\) films can already be highly crystallized at a growth temperature of “only” 250 °C. The \(c\) lattice constant is 3.512 in agreement with the bulk value [16]. The full width at half maximum (FWHM) for the film grown at 300 °C is 0.25° for the (001) peak and 0.39° for the (002) peak which corresponds to a grainsize of \(3.3 \times 10^{-8}\) m and \(2.3 \times 10^{-8}\) m respectively.

5.4 Atomic Force Microscopy

The samples were also analyzed with an atomic force microscope to determine the grainsize and the roughness of the films. Figure 5.7 shows the picture obtained from a film grown at 300 °C that had no MgO capping layer.

This film had a \(T_c\) of 35 K and was grown with a Mg:B flux ratio of 1.5:1.0. The average size of the features in the picture is 320 nm which is 10 times larger than the grainsize obtained from XRD measurements of the same film. The RMS roughness is 255 Å.
Figures 5.8-5.10 show the pictures of three films that had a MgO capping layer and were grown at different temperatures.

The films were grown at $T_s = 200$, 250, and 300 °C and have a Root Mean Square (RMS) roughness of 246, 4.2, and 120 Å, respectively. Figure 5.9 shows the AFM picture at a scale 10 times smaller than the other two pictures.

The critical temperatures were 22, 30, and 16 K for the films in figures 5.8, 5.9, and 5.10, respectively. The film with the highest $T_c$ has the smoothest surface. The films in the figures 5.7 and 5.10 were both grown at 300 °C but the first one did not have a MgO capping layer and has twice the RMS roughness compared with the second one. The average size of the features in both figures however is approximately the same.
5.5 XRD compared with AFM

Figure 5.11 displays the grainsize versus $T_s$ according to XRD results and figure 5.12 shows the size of the features in the AFM images versus $T_s$. The numbers in the figures represent the Mg:B flux ratios for each particular film.

![Figure 5.11. Grain size versus growth temperature according to XRD.](image1)

![Figure 5.12. Size of features in AFM image versus growth temperature.](image2)

When comparing the grainsize obtained from XRD measurements with the size of the features in the AFM pictures one can see that at $T_s = 200$ °C the grains are 10 times smaller than the features, at $T_s = 250$ °C the sizes are comparable, and at $T_s = 300$ °C the grains are 5 times smaller.

5.6 Other substrates

Films were also grown on sapphire, SiC and GaN. The cleanliness of the SiC and GaN substrates was checked with AES and those spectra can be found in appendix B. They show that the substrates were contaminated with oxygen. Each substrate was only tried once and the films were grown under less beneficial conditions like lower Mg and B fluxes and higher base pressures. They all showed insulating behavior.
Chapter 6. Discussion and conclusions

As-grown thin films of MgB$_2$ were prepared by molecular beam epitaxy on Si(111) covered with a 50 Å seed layer of MgO. As was shown in the results, MgO and MgB$_2$ were deposited on a clean Si(111) surface without any contaminations. The growth temperature varied from 200-350 °C and superconducting films were obtained in the range from 200-325 °C. This is consistent with the results of Ueda et al [34] who obtained superconducting films in the range of 180-320 °C. Their best film on Si(111) had a $T_c$ of 32.8 K ($\Delta T = 0.5$ K) and was grown at 283 °C. This is lower than the highest critical temperature of 35.2 K ($\Delta T = 0.3$ K) that we obtained for a film grown at 300 °C on Si(111) with a 50 Å MgO seed layer. Their films showed no crystallinity irrespective of the growth temperature. Our films showed already a high degree of crystallization at a growth temperature of 250 °C. This indicates that higher quality MgB$_2$ films on Si(111) are possible when grown on a seed layer of MgO. Magnesium oxide was also used as a capping layer and this proved to be highly beneficial for preserving the films. This makes MgO an ideal candidate for serving as a barrier in MgB$_2$ tunnel junctions.

The fact that highly crystallized films already form at such a low temperature is quite remarkable. Typically epitaxial growth takes place at about one half of the melting temperature which means at ~1080 °C for MgB$_2$. For metals the minimum temperature for epitaxial growth is about 1/8 of the melting temperature [35] which corresponds with 270 °C for MgB$_2$. This is close to the temperature at which we observed the growth of highly crystallized films indicating a metallic behavior of MgB$_2$.

Liu et al [32] predicted a thermodynamic stability window for the growth of MgB$_2$ films. Deposition of a single-phase MgB$_2$ film would become easy when the growth conditions fall within the window where the thermodynamically-stable phases are the desired MgB$_2$ phase and gas phases. Within this growth window MgB$_2$ should not decompose and excess Mg should not condense on the MgB$_2$ surface, thus the formation of single-phase MgB$_2$ would be adsorption controlled and automatic. We did not find this convenient growth window, reflecting perhaps the kinetics of the deposition at a rate of ~2 Å/s. Based on AES measurements, we found that it is difficult to obtain exactly the MgB$_2$ compound. The films were off-stoichiometric: too much Mg flux resulted in MgB$_2$ + Mg solid, and too little resulted in Mg$_x$B$_{2-x}$ with perhaps Mg$_4$, or Mg$_2$ with Mg vacancies. This caused a reduced $T_c$ or insulating samples, however, it was shown that when the Mg:B concentration approached the right stoichiometry, the $T_c$ increased.

The quality of the films was also highly dependent on the background pressure which was shown by the fact that all the films grown with $P_{\text{depo}} > 3.0 \times 10^{-8}$ Torr were insulating. A higher background pressure means a higher partial pressure of oxygen which is the main reason for the degradation of the films. Oxygen is highly reactive with magnesium as was shown by AES and contaminates the film. A higher background pressure also means a lower partial Mg pressure, while high partial Mg pressures are needed to compensate for its volatility, particularly at higher temperatures.
The off-stoichiometry and contamination with oxygen are possible explanations for the films having a lower critical temperature or being insulating. Another reason might be the deposition rate during growth. It was found that growing samples with a deposition rate lower than 1 Å/s resulted in films with insulating characteristics. This is probably due to the low partial Mg pressure when depositing at low rates. Furthermore, at a low deposition rate there is more time for the oxidation of magnesium which increases the oxygen concentration in the film.

XRD measurements showed that even very poorly crystallized films still show superconductivity. This is in agreement with the results of Ueda et al, however in our case a higher $T_c$ corresponded with a higher degree of crystallinity and their films remained poorly crystallized even when having a $T_c$ of 32.8 K.

The XRD patterns of the films grown at different temperatures and with different Mg:B flux ratios showed again that MgB$_2$ growth is not adsorption controlled or automatic. With every chosen growth temperature one has to choose carefully the Mg:B flux ratio and deposition rate. However when the right parameters are used, high quality MgB$_2$ films form already at a temperature of 250 °C.

AFM measurements showed that the roughness of the films is a function of growth temperature. When increasing $T_s$ from 200 to 250 °C the films become smoother, but at 300 °C the roughness increased again. This is probably because the quality of the film is also important. The film grown at 300 °C had a $T_c$ of 16 K and was poorly crystallized. However it still showed that using a capping layer of MgO significantly increased the smoothness of the films. The high quality film grown at 300 °C without the cover layer was twice as rough as the poor quality film with the MgO capping layer. The fact that high quality films with a cover layer of magnesium oxide are very smooth makes them ideal for the preparation of MgB$_2$ tunnel junctions.

According to AFM measurements the size of the particles decreased with the increase of growth temperature. This is not consistent with XRD results. Those spectra showed an increase in grain size with a higher $T_s$ until a growth temperature of 300 °C is reached and then a decrease. The reason for this discrepancy is probably that AFM is a surface technique and XRD a probe for the bulk properties. The surface of the film reacts with water and oxygen from the air, which changes its characteristics. Films become smoother at higher growth temperatures, so smaller particles are detected, while they also become more crystallized, so larger grains are detected by XRD. The decrease in grain size above a $T_s$ of 300 °C can be explained by the high volatility of Mg. At those temperatures a much higher Mg flux is needed and the lack of magnesium causes Mg vacancies in the film instead of large MgB$_2$ grains.

The lattice constant of MgO is 4.21 Å, whereas it is 3.05 Å for MgB$_2$ implying a lattice mismatch of 28%. This means that MgB$_2$ can not grow directly on top of the MgO seed layer, but that there is an interface layer which can also cause a reduced $T_c$. This is not the case for SiC and GaN whose lattice constants are almost the same as for MgB$_2$. Unfortunately we were not able to compare the films grown on those substrates with films grown on Si(111) with a MgO seed layer in our preliminary studies because they were grown at high background pressures and with low deposition rates which caused insulating films.
In conclusion we have investigated the growth of MgB$_2$ films on various substrates and seed layers under different conditions. Mg:B flux ratio, growth temperature, deposition rate, and background pressure were varied in order to determine which growth parameters provide the highest quality films which can be used for the fabrication of tunnel junctions. The films grown on Si(111) with a seed layer of MgO proved to be highly promising to reach that goal. In the future we plan the preparation of MgB$_2$ Josephson junctions with those films and further investigate ways to improve the critical temperature by growing MgB$_2$ films on SiC and GaN.
References

## Appendix A

Table of all the samples grown under different conditions:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Substrate/Seed-layer/Ratio</th>
<th>$T_e$ (°C)</th>
<th>$P_{dep}$ (Torr)</th>
<th>$T_e$ (K) $I$=10 mA</th>
<th>$T_c$ (K) $I$=10 µA</th>
<th>$R_{RT}$ (Ω)</th>
<th>RMS Roughness (Å)</th>
<th>DR-Mg (Å/s)</th>
<th>DR-B (Å/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>R-3X</td>
<td>Si(111)/MgO/MgB</td>
<td>300</td>
<td>2.0 ·10^{-8}</td>
<td>14.3</td>
<td>Not measured</td>
<td>6.6</td>
<td>120 (x20)</td>
<td>1.25</td>
<td>X</td>
</tr>
<tr>
<td>R-4X</td>
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<td>300</td>
<td>1.5 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>R-4Y</td>
<td>Si(111)/MgO/Mg_{1.2}B</td>
<td>300</td>
<td>7.2 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.15</td>
<td>0.25</td>
</tr>
<tr>
<td>R-5X</td>
<td>Si(111)/MgO/Mg_{2.0}B</td>
<td>300</td>
<td>1.0 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>R-5Y</td>
<td>Si(111)/MgO/Mg_{1.5}B</td>
<td>300</td>
<td>2.8 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.7</td>
<td>0.3</td>
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<tr>
<td>R-6X</td>
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<td>250</td>
<td>1.0 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>R-6Y</td>
<td>Si(111)/MgO/Mg_{1.5}B</td>
<td>250</td>
<td>1.4 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>R-7X</td>
<td>Si(111)/MgO/Mg_{2.0}B</td>
<td>300</td>
<td>~10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>0.25</td>
<td>X</td>
</tr>
<tr>
<td>R-7Y</td>
<td>Si(111)/MgO/Mg_{2.8}B</td>
<td>300</td>
<td>~10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.2</td>
<td>0.1</td>
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<td>R-8X</td>
<td>Si(111)/MgO/Mg_{3.1}B</td>
<td>300</td>
<td>1.4 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.85</td>
<td>0.2</td>
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<tr>
<td>R-8Y</td>
<td>Si(111)/MgO/Mg_{3.4}B</td>
<td>300</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>2.05</td>
<td>0.15</td>
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<td>R-9X</td>
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<td>1.1 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.3</td>
<td>0.1</td>
</tr>
<tr>
<td>R-9Y</td>
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<td>300</td>
<td>1.7 ·10^{-8}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.15</td>
<td>0.05</td>
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<td>200</td>
<td>3.0 ·10^{-8}</td>
<td>20.4</td>
<td>Not measured</td>
<td>1.9</td>
<td>246 (x20)</td>
<td>1.35</td>
<td>0.5</td>
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<tr>
<td>R-11X</td>
<td>Si(111)/MgO/Mg_{2.9}B</td>
<td>250</td>
<td>3.5 ·10^{-9}</td>
<td>28.5</td>
<td>Not measured</td>
<td>32</td>
<td>4.2 (x50)</td>
<td>1.65</td>
<td>0.25</td>
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<tr>
<td>R-11Y</td>
<td>Si(111)/MgO/Mg_{3.9}B</td>
<td>250</td>
<td>5.8 ·10^{-9}</td>
<td>31.4</td>
<td>33</td>
<td>9.1</td>
<td>3.2 (x100)</td>
<td>1.85</td>
<td>0.45</td>
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<td>283</td>
<td>4.1 ·10^{-9}</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.65</td>
<td>0.25</td>
</tr>
<tr>
<td>R-12Y</td>
<td>Si(111)/MgO/Mg_{2.0}B</td>
<td>283</td>
<td>6.0 ·10^{-9}</td>
<td>31</td>
<td>32.7</td>
<td>35</td>
<td>5.7 (x50)</td>
<td>1.5</td>
<td>0.35</td>
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<td>R-13X</td>
<td>Si(111)/MgO/Mg_{2.0}B</td>
<td>300</td>
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<td>Not measured</td>
<td>27</td>
<td>80</td>
<td>Not measured</td>
<td>2.0</td>
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<tr>
<td>R-13Y</td>
<td>Si(111)/MgO/Mg_{1.5}B</td>
<td>300</td>
<td>1.7 ·10^{-8}</td>
<td>Not measured</td>
<td>35</td>
<td>1.0</td>
<td>227 (x50)</td>
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<td>0.4</td>
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<td>Insulating</td>
<td>Not measured</td>
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<td>252</td>
<td>Not measured</td>
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<td>325</td>
<td>4.3·$10^{-9}$</td>
<td>Insulating</td>
<td>Not measured</td>
<td>30</td>
<td>301</td>
<td>Not measured</td>
<td>1.6</td>
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<td>R-15X</td>
<td>Si(111)/MgO/Mg$_{2.6}$B</td>
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<td>1.2·$10^{-7}$</td>
<td>Insulating</td>
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<td>Insulating</td>
<td>Not measured</td>
<td>1.6</td>
<td>0.2</td>
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<tr>
<td>R-15Y</td>
<td>Si(111)/MgO/Mg$_{2.3}$B</td>
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<td>2.9·$10^{-8}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.2</td>
<td>0.2</td>
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<td>R-16X</td>
<td>Si(111)/MgO/Mg$_{2.0}$B</td>
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<td>4.0·$10^{-8}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.5</td>
<td>0.35</td>
</tr>
<tr>
<td>R-16Y</td>
<td>Si(111)/Al$_2$O$<em>3$/Mg$</em>{1.8}$B</td>
<td>300</td>
<td>2.6·$10^{-8}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.1</td>
<td>0.23</td>
</tr>
<tr>
<td>R-17X</td>
<td>Si(111)/Al$_2$O$<em>3$/Mg$</em>{1.7}$B</td>
<td>300</td>
<td>2.8·$10^{-8}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>0.85</td>
<td>0.19</td>
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<tr>
<td>R-17Y</td>
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<td>Insulating</td>
<td>Insulating</td>
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<td>Not measured</td>
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<td>R-18X</td>
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<td>5.1·$10^{-8}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.2</td>
<td>0.25</td>
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<td>R-19X</td>
<td>Si(111)/MgO/Mg$_{1.9}$B</td>
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<td>Insulating</td>
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<td>31.2</td>
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<td>Insulating</td>
<td>Insulating</td>
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<td>1.45</td>
<td>0.25</td>
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<td>R-20X</td>
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<td>3.5·$10^{-8}$</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
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<td>0.15</td>
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<td>R-20Y</td>
<td>Si(111)/MgO/Mg$_{2.3}$B</td>
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<td>6.0·$10^{-8}$</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.45</td>
<td>0.2</td>
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<tr>
<td>R-21X</td>
<td>Si(111)/MgO/Mg$_{2.2}$B</td>
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<td>1.4·$10^{-8}$</td>
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<td>Insulating</td>
<td>Insulating</td>
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<td>0.2</td>
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<td>R-21Y</td>
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<td>Not measured</td>
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<td>Insulating</td>
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<td>0.25</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
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<td>0.3</td>
</tr>
<tr>
<td>R-23X</td>
<td>Si(111)/MgO/Mg$_{1.9}$B</td>
<td>330</td>
<td>10$^{-8}$·$10^{-9}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.5</td>
<td>0.25</td>
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<tr>
<td>R-23Y</td>
<td>Si(111)/MgO/Mg$_{1.9}$B</td>
<td>330</td>
<td>10$^{-8}$·$10^{-9}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.55</td>
<td>0.25</td>
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<td>Insulating</td>
<td>Not measured</td>
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<td>R-24Y</td>
<td>Si(111)/Mg$_{1.1}$B</td>
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<td>10$^{-8}$·$10^{-9}$</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
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<td>R-25X</td>
<td>Sapphire/Mg$_{1.9}$B</td>
<td>300</td>
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<td>Insulating</td>
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<td>Not measured</td>
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<td>R-26X</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.55</td>
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<td>Insulating</td>
<td>Insulating</td>
<td>Insulating</td>
<td>Not measured</td>
<td>1.45</td>
<td>0.2</td>
</tr>
</tbody>
</table>

X means unknown; First $R$-$T$ measurements were performed with a current of 10 mA, then a current of 10 μA was used; $R_{RT}$ means at room temperature; (×20) means at magnification 20 times higher then ×1 which is 50,000 × 50,000 Å$^2$; DR means deposition rate.
Appendix B

Figure B.1 shows the Auger spectrum of a SiC substrate after the cleaning procedure.

![Fig 1: Auger spectrum of cleaned SiC](image)

*Figure B.1. Auger spectrum of cleaned SiC.*

Figure B.2 shows the Auger spectrum of a GaN substrate after the cleaning procedure.

![Fig 2: Auger spectrum of cleaned GaN](image)

*Figure B.2. Auger spectrum of cleaned GaN.*

Both spectra contain a small oxygen peak which indicates that the surfaces of the substrates were still contaminated after the cleaning procedure.