

Fortgeschrittenenpraktikum

X-Ray Diffraction from a Semiconductor Heterostructure with the X'Pert PRO PANalytical Diffractometer

Location: Semiconductor-Physics Building - Room 907

Time: Usually Friday, 1.00 p.m. - 4.00 p.m.

Need: 1 USB-stick



The X'Pert PRO PANalytical Diffractometer

Contents

1	Introduction	1
1.1	About This Manual	1
1.2	Motivation	1
2	Scattering Theory Basics	5
2.1	Light Scattering by a Point Charge	5
2.2	Light Scattering by an Ensemble of Charges	6
2.3	Scattering by Crystals	7
2.3.1	Bragg’s Law, Connection With Lattice Parameter	8
2.4	Scattering from Crystals of Finite Size	10
2.4.1	Example: Block-Shaped Crystals	12
2.4.2	Example: Superlattice	12
3	Experimental Setup and Methods	15
3.1	Reciprocal Space Coordinates	15
3.1.1	Connection Between Angles and Reciprocal Space Coordinates . . .	15
3.1.2	Scan Directions, Asymmetrical Reflections	18
3.1.3	Determination of a Layer’s Strain Status	19
3.1.4	Reciprocal Space Mapping	22
3.2	Setup of a High Resolution X-Ray Diffractometer	24
3.2.1	General	24
3.2.2	Sample Stage	25
3.2.3	Sample Alignment	27

3.3	Operating the X'Pert PRO PANalytical Diffractometer	28
3.3.1	Diffractometer Elements	29
3.3.2	Mounting a Sample	31
3.3.3	Aligning a Sample	33
3.3.4	The Measurement	35
4	Experiment	41
	Appendix	42
	Structure Factor of a Crystal	43
	Shape Functions of Finite Crystals	44
	Useful Numbers	46
	Lattice Parameters of $\text{Si}_{1-x}\text{Ge}_x$ and $\text{Si}_{1-y}\text{C}_y$	47
	Data Extraction	48
	Bibliography	48

Chapter 1

Introduction

1.1 About This Manual

The following scriptum is intended as a manual to perform an x-ray diffraction experiment in the course of the "Fortgeschrittenenpraktikum". It tries to give first some motivation on the investigated samples. Then some theory is included, describing the scattering of x-rays by matter.

Thereafter the experimental setup is explained in detail. The measurements can most easily be interpreted by plotting them in the coordinate system of *reciprocal space*, therefore a large part of this section is concerned with reciprocal space coordinates. In order to give sort of a "complete" picture of the method, also some aspects that are not absolutely required in order to perform this course have been included.

In the next section, the alignment of a sample is explained, first more generally, and then step-by-step for the specific x-ray diffractometer which will be used. Again, although for this course not all steps of alignment are really required (it is, however, instructive to do a full alignment), the complete alignment procedure for a sample in the so-called *coplanar scattering geometry* is explained.

Finally, the actual problem of this course is presented in Sec. 4. The appendix contains some further details concerning the scattering theory, as well as some tables of numbers useful for the interpretation of the results, and some graphs required for the same purpose.

1.2 Motivation

In recent years the fabrication of fast electronic devices by means of heterostructures grown by several techniques like molecular beam epitaxy (MBE), chemical vapor deposition (CVD) or liquid phase epitaxy (LPE) has been developed to a considerable high

standard, in particular with the GaAs/AlGaAs system [1]. Great efforts have been made to transfer this technique to silicon based devices. There are two possible group-IV materials for the formation of heterostructures with silicon: Germanium, which has a bandgap smaller than silicon, and carbon (diamond), which exhibits a band gap approximately five times that of silicon [2]. For the lattice parameters see the following table [2]:

Element	Atomic No.	Lattice const. ($\text{\AA}$)
C	6	3.567
Si	14	5.431
Ge	32	5.657

Germanium was the first group-IV material introduced on purpose to the silicon system to fabricate heterostructures. It is miscible with silicon in any concentration, and therefore SiGe layers are rather easy to fabricate with conventional techniques. If a SiGe epitaxial film with a certain Ge concentration is grown on top of a Si substrate, it will imitate the lateral structure of the substrate up to a certain "critical thickness" of the layer [3], known as pseudomorphic growth. As a consequence of the lattice mismatch between Si and Ge (dependent on the Ge concentration of the layer) the layer will be tetragonally distorted, i.e. the lateral lattice constant will be smaller than the lattice constant in growth direction, the film is under biaxial compressive strain. For this strain state the degeneracy of the six conduction band valleys in $\mathbf{k}$ -space along Γ -X direction [4] vanishes, and the resulting band alignment is indicated in Fig.1.1a [5], [6].

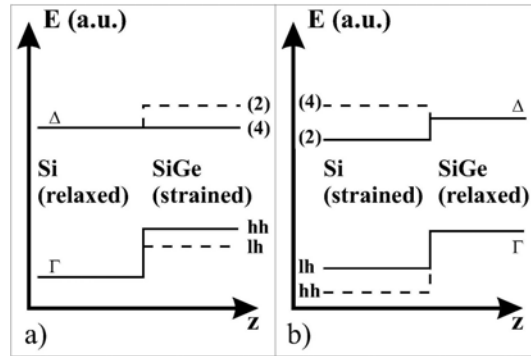


Figure 1.1: Illustration of the band offsets in a Si/SiGe heterostructure for different strain states.

If the structure is again overgrown with silicon, the SiGe layer forms a quantum well for holes, but no confinement for electrons in the conduction band is achieved. Such a layer system can be exploited technically e.g. for the construction of fast devices (as the hetero-bipolar transistor [HBT]) [7], [8], [9]. To obtain the proper band offset within the conduction band for a quantum well for electrons, as would be needed if something similar to CMOS devices are aimed, a biaxially tensile strained layer has to be grown rather than

a biaxially compressive strained one [5]. Possible ways to obtain such a layer is the growth of a SiGe layer on top of a Ge substrate or of a Si layer on top of a relaxed SiGe buffer layer, as indicated in Fig.1.1b (note that there exists still a valence band offset, making a simultaneous confinement of holes in the SiGe layer and of electrons in the Si layer possible).

Another way to fabricate quantum wells for electrons was developed recently (e.g. in [10], [11], [12]). The incorporation of carbon, which has a lattice constant approximately 50% smaller than that of silicon, in the Si lattice yields a similar strain status than for Si on SiGe, i.e. C containing layers on Si substrates exhibit a biaxial tensile strain. However, the incorporation of C in Si cannot be obtained by growth methods operating in the vicinity of thermodynamical equilibrium, as the bulk solubility limit for C in Si is as low as $3.5 \cdot 10^{17} \text{cm}^{-3}$ (for oxygen-free silicon at the melting point; for oxygen-saturated Czochralski-grown Si values of up to $2 \cdot 10^{18} \text{cm}^{-3}$ have been obtained, for details see [13]). Nevertheless carbon can be incorporated to a significantly higher amount into the silicon lattice by means of techniques like MBE and at rather low growth temperatures of about 500°C . Although as a consequence of the high lattice mismatch between C and Si, the local neighborhood of a carbon atom will be considerably distorted, which may have significant influence on the electronic properties of SiC-based devices, some devices are already available [14].

Chapter 2

Scattering Theory Basics

This chapter contains a short overview over the scattering theory relevant for the interpretation of the x-ray measurements. The descriptions follow a lecture "Theoretische Physik II, Elektrodynamik" by U.M. Titulaer [15], and a book of M.A. Krivoglaz [16].

2.1 Light Scattering by a Point Charge

In order to understand the scattering of x-rays by a crystal, let's start considering first a single charged particle, and afterwards extend the consideration to an ensemble of particles and finally to a crystal. First of all, x-rays are electromagnetic waves, so we may apply the description found in any textbook on electrodynamics. A monochromatic, plane electromagnetic wave acting on a point charge will lead to a vibration of this charge around its normal position, leading to a dipole moment $\mathbf{p}_\omega = \alpha(\omega)\mathbf{E}_\omega^0$, with $\mathbf{E}_\omega^0$ the electric field of the incoming wave and $\alpha(\omega)$ the polarizability of the particle, which depends on the frequency of the electromagnetic wave ω . This vibrating dipole, as any accelerated electrical charge, emits an electromagnetic wave. We want to know the strength of this wave in a certain direction from the vibrating charge. If we are far away from the charge, the emitted wave is a spherical wave. Let us consider the particle in a position $\mathbf{r} = 0$, then an observer in point $\mathbf{r}$ with $r = |\mathbf{r}|$ large compared to the wavelength λ of the incoming wave will "see" an electric field of [15], [17]

$$|\mathbf{E}_\omega| = -\alpha(\omega) |\mathbf{E}_\omega^0| \frac{e^{ikr}}{r}, \quad (2.1)$$

$k = |\mathbf{k}_i| = |\mathbf{k}_f|$ is the length of the wave vectors of the incoming and scattered radiation $\mathbf{k}_i$ and $\mathbf{k}_f$, respectively. I.e., we consider only elastic scattering. The scattering geometry is depicted in Fig. 2.1. Here, $\mathbf{k}_i$ is along the x axis, while $\mathbf{k}_f$ points towards the observer. The following relations hold between the wavelength and the wave vector and frequency (c denotes the speed of light):

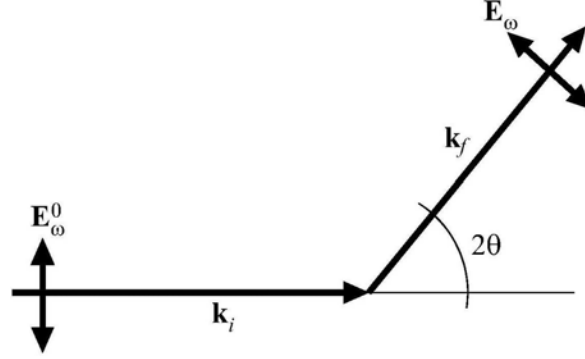


Figure 2.1: Scattering geometry for a single particle.

$$\begin{aligned}
 k &= |\mathbf{k}_i| = |\mathbf{k}_f| = \frac{2\pi}{\lambda} \\
 \omega &= 2\pi \frac{c}{\lambda}.
 \end{aligned}
 \tag{2.2}$$

2.2 Light Scattering by an Ensemble of Charges

If we consider an ensemble of identical light scatterers located at positions $\{\mathbf{r}_j\}$, and neglect the possibility of multiple scattering as well as the weakening of the incoming wave and the effect of the scattered wave on the scattering charges, we get

$$\mathbf{E}_{\omega,j} = -e^{i\mathbf{k}_i \cdot \mathbf{r}_j} \alpha(\omega) |\mathbf{E}_\omega^0| \frac{e^{ik|\mathbf{r}-\mathbf{r}_j|}}{|\mathbf{r}-\mathbf{r}_j|}$$

for the j^{th} scatterer. I.e., we have to consider the phase shift of the incoming wave "illuminating" the j^{th} scatterer $e^{i\mathbf{k}_i \cdot \mathbf{r}_j}$, and we have to take the position of the j^{th} scatterer with respect to the observer into account in $|\mathbf{r}-\mathbf{r}_j|$. With the above assumptions we are considering the so-called kinematical scattering theory.

If $kr_j \ll 1$ and $kr \gg 1$, $k|\mathbf{r}-\mathbf{r}_j|$ can be replaced by

$$\begin{aligned}
 k|\mathbf{r}-\mathbf{r}_j| &= k\sqrt{(\mathbf{r}-\mathbf{r}_j) \cdot (\mathbf{r}-\mathbf{r}_j)} = k\sqrt{r^2 - 2\mathbf{r}\mathbf{r}_j + r_j^2} \approx kr\sqrt{1 - \frac{2\mathbf{r}\mathbf{r}_j}{r^2}} \approx \\
 &\approx kr \left(1 - \frac{\mathbf{r}\mathbf{r}_j}{r^2}\right) = kr - \mathbf{k}_f \cdot \mathbf{r}_j
 \end{aligned}
 \tag{2.3}$$

as for a large distance to the scattering ensemble $\mathbf{k}_f \parallel \mathbf{r}$. Therefore the electric field has the form

$$\begin{aligned}
\mathbf{E}_{\omega,j} &= -e^{-i(\mathbf{k}_f - \mathbf{k}_i) \cdot \mathbf{r}_j} \alpha(\omega) |\mathbf{E}_{\omega}^0| \frac{e^{ikr}}{r} \\
&= \mathbf{E}_{\omega,\text{sgle}} \cdot e^{-i(\mathbf{k}_f - \mathbf{k}_i) \cdot \mathbf{r}_j}
\end{aligned} \tag{2.4}$$

with $\mathbf{E}_{\omega,\text{sgle}}$ the field of a single scatterer as in Eq. (2.1). For the whole ensemble of charges the total scattered electric field is therefore

$$\mathbf{E}_{\omega} = \mathbf{E}_{\omega,\text{sgle}} \sum_j e^{-i(\mathbf{k}_f - \mathbf{k}_i) \cdot \mathbf{r}_j} \tag{2.5}$$

For a charge density $\rho(\mathbf{r})$ this yields

$$\mathbf{E}_{\omega} = \mathbf{E}_{\omega,\text{sgle}} \int d^3\mathbf{r}' \rho(\mathbf{r}') e^{-i(\mathbf{k}_f - \mathbf{k}_i) \cdot \mathbf{r}'}. \tag{2.6}$$

We see that actually $\mathbf{E}_{\omega}$ depends only on the difference between the wave vectors of scattered and incoming radiation, i.e., on the so-called momentum transfer $\mathbf{Q} = \mathbf{k}_f - \mathbf{k}_i$. $\mathbf{Q}$ is also called the scattering vector, and in most cases the scattered electrical field or the scattered intensity is written as a function of the scattering vector

$$\begin{aligned}
E(\mathbf{Q}) &= E_{\omega,\text{sgle}} \int d^3\mathbf{r}' \rho(\mathbf{r}') e^{-i\mathbf{Q} \cdot \mathbf{r}'} \\
I(\mathbf{Q}) &= I_{\omega,\text{sgle}} \left| \int d^3\mathbf{r}' \rho(\mathbf{r}') e^{-i\mathbf{Q} \cdot \mathbf{r}'} \right|^2
\end{aligned} \tag{2.7}$$

We note that in this approximation the scattering amplitude is proportional to the *Fourier transform* of the charge density. IN fact, only the electrons have a considerable polarizability, hence the scattering amplitude is proportional to the Fourier transform of the electron density of a sample.

2.3 Scattering by Crystals

For crystals $\rho(\mathbf{r})$ can be identified with the total electron density as long as $\omega \gg \omega_B$, the Bohr frequency, i.e., the electrons can be treated as at rest on a timescale relevant for the scattering process. Now the electrons are distributed around the atoms at sites

$\mathbf{R}_n$. Because the charge density is invariant under a translation by a lattice vector $\mathbf{R}$, $\rho(\mathbf{r}) = \rho(\mathbf{r} + \mathbf{R})$, it may be written as a Fourier series

$$\rho(\mathbf{r}) = \sum_{\mathbf{G}} \rho_{\mathbf{G}} e^{i\mathbf{G}\mathbf{r}}. \quad (2.8)$$

Considering this in the condition $\rho(\mathbf{r}) = \rho(\mathbf{r} + \mathbf{R})$, we see

$$\rho(\mathbf{r} + \mathbf{R}) = \sum_{\mathbf{G}} \rho_{\mathbf{G}} e^{i\mathbf{G}\mathbf{r}} e^{i\mathbf{G}\mathbf{R}} = \sum_{\mathbf{G}} \rho_{\mathbf{G}} e^{i\mathbf{G}\mathbf{r}} = \rho(\mathbf{r}). \quad (2.9)$$

Which is obviously only satisfied for arbitrary $\mathbf{r}$ if for all vectors $\mathbf{G}$ $e^{i\mathbf{G}\mathbf{R}} = 1$ holds. This is a definition of $\mathbf{G}$, and all vectors $\mathbf{G}$ satisfying this relation form the so-called *reciprocal lattice* of a crystal lattice. The definition of the reciprocal lattice vectors may also be written in the more common form

$$\mathbf{G} \cdot \mathbf{R} = 2\pi m \quad (2.10)$$

with m an integer. Inserting Eq.(2.8) into Eq.(2.7), and recalling the definition of the Dirac- δ function $\int_{-\infty}^{\infty} d^3\mathbf{r} e^{i\mathbf{k}\mathbf{r}} = \delta(\mathbf{k})$, we get

$$\begin{aligned} I(\mathbf{Q}) &\sim \left| \sum_{\mathbf{G}} \rho_{\mathbf{G}} \int d^3\mathbf{r} e^{i(\mathbf{Q}-\mathbf{G})\mathbf{r}} \right|^2 \\ &= \left| \sum_{\mathbf{G}} \rho_{\mathbf{G}} \delta(\mathbf{Q} - \mathbf{G}) \right|^2, \end{aligned} \quad (2.11)$$

i.e., scattering occurs only in some exceptional directions, namely when the momentum transfer $\mathbf{Q}$ equals a reciprocal lattice vector $\mathbf{G}$. This is the Laue-condition for scattering from a perfect crystal:

$$\mathbf{Q} = \mathbf{G} \quad (2.12)$$

2.3.1 Bragg's Law, Connection With Lattice Parameter

The scattering geometry for a crystal can now be depicted as in Fig. 2.2, the so-called Ewald construction. Instead of orienting the coordinate axes along the incident wavevector $\mathbf{k}_i$, we rather draw the scattering geometry in the coordinate system of the reciprocal lattice

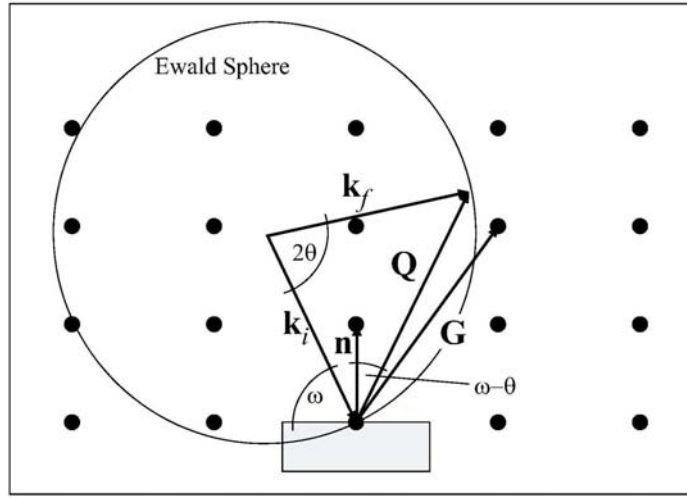


Figure 2.2: Scattering geometry depicted in reciprocal space.

of our sample. The reciprocal lattice points $\mathbf{G}$ are indicated as black dots. The incident beam $\mathbf{k}_i$ has a certain orientation with respect to the sample, given by the incidence angle with respect to the surface ω (sorry, we used the same symbol for the wavelength before, but from now on, ω always denotes the incidence angle). As the scattering is elastic, all possible wave vectors of the scattered beam describe the so-called Ewald circle (or Ewald sphere, but here we restrict ourselves to a twodimensional case, where $\mathbf{k}_i$, $\mathbf{k}_f$ and the sample's surface normal $\mathbf{n}$ lie in one plane; this geometry is therefore also called *coplanar* scattering geometry). Scattering occurs only for a momentum transfer $\mathbf{Q}$, if the Ewald circle intersects a reciprocal lattice point, i.e., for $\mathbf{Q} = \mathbf{G}$. The scattering angle between $\mathbf{k}_f$ and $\mathbf{k}_i$ is called 2θ (the factor 2 is a historical convention). From the figure it is obvious that the length of the scattering vector is

$$\frac{|\mathbf{Q}|}{2} = k \sin \theta = \frac{2\pi}{\lambda} \sin \theta,$$

The reciprocal lattice vector in the case of a cubic lattice (the only type of crystal lattice which we will consider in this course) can be written as

$$\begin{aligned} \mathbf{G} &= \frac{2\pi}{a}(h, k, l) \\ |\mathbf{G}| &= \frac{2\pi}{a}\sqrt{h^2 + k^2 + l^2}. \end{aligned}$$

For $\mathbf{Q} = \mathbf{G}$, and hence also $|\mathbf{Q}| = |\mathbf{G}|$, we obtain the Bragg condition for scattering from a perfect crystal:

$$2 a \sin \theta = \sqrt{h^2 + k^2 + l^2} \lambda, \quad (2.13)$$

or, with the spacing between the $\{hkl\}$ planes being $d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}}$ and an integer m

$$2 d_{hkl} \sin \theta = m \lambda. \quad (2.14)$$

2.4 Scattering from Crystals of Finite Size

In order to describe scattering from a finite crystal, we may start from Eq. (2.11), but the integration is restricted to a finite region of space. Consequently, the Dirac- δ will not appear, however, if the crystal is much larger than the spacing of the atoms, it is still "in some sense" infinite, i.e., the condition for scattering $\mathbf{Q} = \mathbf{G}$ is still valid, but the reflections are no longer infinitely sharp, but somewhat extended in reciprocal space. In order to see this more quantitatively, we may use a small "trick": the shape function Ω of the crystal, allowing us to expand the integration in Eq. (2.11) to the whole direct space. Let us consider the function Y^∞ describing the structure of the ideal crystal (of infinite size)

$$Y^\infty(\mathbf{r}) = \sum_{\text{lattice sites } s} \delta(\mathbf{r} - \mathbf{R}_s)$$

and the function Ω describing the shape (and size) of the crystal

$$\Omega(\mathbf{r}) = \begin{cases} 1 & \text{inside} \\ 0 & \text{outside} \end{cases} \text{ the crystal}$$

the amplitude of the scattered radiation is

$$\begin{aligned} E(\mathbf{Q}) &= f(\mathbf{Q}) \int d^3\mathbf{r} Y^\infty(\mathbf{r}) \Omega(\mathbf{r}) e^{i\mathbf{Q}\mathbf{r}}. \\ f(\mathbf{Q}) &= \sum_{\gamma=1}^{\nu} \int d^3\mathbf{r}' \rho_\gamma(\mathbf{r}') e^{i\mathbf{Q}\mathbf{r}'} \end{aligned} \quad (2.15)$$

is the so-called structure factor of the crystal, containing the details of the electron distributions ρ_γ around the ν atoms in the unit cell, i.e., information on the basis of the crystal. It modifies the intensities of the individual Bragg reflections $\mathbf{G}$. But as $f(\mathbf{Q})$ is the Fourier transform of the structure of one unit cell, which is small in real space, it is a function varying slow in reciprocal space compared to the Fourier transform of the shape

function of the whole crystal. Therefore we omit it in the following consideration, but we must not forget that when comparing, e.g., intensities of *different* Bragg reflections $\mathbf{G}_j$ and $\mathbf{G}_k$, these intensities are modified by $f(\mathbf{Q})$.

Equation (2.15) contains the product of two functions in real space. This is transformed to the convolution of the Fourier-transformed functions in reciprocal space. We introduce the Fourier transforms of Y^∞ and Ω (v is the unit cell volume)

$$Y^\infty(\mathbf{r}) = \frac{1}{8\pi^3} \int d^3\mathbf{k} \tilde{Y}^\infty(\mathbf{k}) e^{-i\mathbf{k}\mathbf{r}} = \sum_{\mathbf{G}} Y_{\mathbf{G}}^\infty e^{-i\mathbf{G}\mathbf{r}} \quad (2.16)$$

$$\tilde{Y}^\infty(\mathbf{k}) = \int d^3\mathbf{r} Y^\infty(\mathbf{r}) e^{i\mathbf{k}\mathbf{r}} \quad (2.17)$$

$$Y_{\mathbf{G}}^\infty = \frac{1}{v} \int d^3\mathbf{r} Y^\infty(\mathbf{r}) e^{i\mathbf{G}\mathbf{r}} = \frac{1}{v}$$

$$\Omega(\mathbf{r}) = \frac{1}{8\pi^3} \int d^3\mathbf{k} \tilde{\Omega}(\mathbf{k}) e^{-i\mathbf{k}\mathbf{r}} \quad (2.18)$$

$$\tilde{\Omega}(\mathbf{k}) = \int d^3\mathbf{r} \Omega(\mathbf{r}) e^{i\mathbf{k}\mathbf{r}}.$$

For Y^∞ we have used two forms, the conventional Fourier transformation as well as a Fourier series: because Y^∞ is periodic with the lattice vectors of the crystal lattice, it can be written as a discrete Fourier series, where the vectors $\mathbf{G}$ are again the vectors of the reciprocal lattice of the crystal. Using these expressions, we get for the amplitude of the scattered wave

$$E(\mathbf{Q}) \sim \frac{f(\mathbf{Q})}{8\pi^3} \int d^3\mathbf{k} \tilde{Y}^\infty(\mathbf{k}) \tilde{\Omega}(\mathbf{Q} - \mathbf{k})$$

where (inserting 2.16 into 2.17) $\tilde{Y}^\infty(\mathbf{k}) = \frac{8\pi^3}{v} \sum_{\mathbf{G}} \delta(\mathbf{k} - \mathbf{G})$, and therefore

$$E(\mathbf{Q}) = \frac{f(\mathbf{Q})}{v} \sum_{\mathbf{G}} \tilde{\Omega}(\mathbf{Q} - \mathbf{G}) \quad (2.19)$$

Hence now scattering occurs not only exactly in the Bragg points, but what we will see in the scattering experiment is the Fourier transform of the shape function $\tilde{\Omega}$ around each Bragg point, for deviations from the Bragg point $\mathbf{q} = \mathbf{Q} - \mathbf{G} \neq 0$. This is nothing else than the convolution of $\tilde{\Omega}$ and the Fourier transform of the lattice (i.e., the reciprocal lattice).

In Eq. (2.19) the sum over all reciprocal lattice vectors appears. However, when we measure the intensity distribution around one specific Bragg point, we may consider only one term

in the sum. This is justified by the fact that $\tilde{\Omega}$ decays over length scales small compared to the distance between different Bragg points, so that in the vicinity of one Bragg point contributions from another Bragg point may be neglected. In a little bit more mathematical terms: because $\tilde{\Omega}(\mathbf{q})$ differs significantly from zero only for $|\mathbf{q}| \ll |\mathbf{G}_1|$, where $\mathbf{G}_1$ is the shortest reciprocal lattice vector, for the calculation of $I(\mathbf{Q})$ only the quadratic terms have to be considered, as for small $|\mathbf{Q} - \mathbf{G}|$ the magnitude of $|\mathbf{Q} - \mathbf{G}'|$ is of the order of $|\mathbf{G}_1|$ except for $\mathbf{G} = \mathbf{G}'$:

$$I(\mathbf{Q}) = \frac{|f|^2}{v^2} \sum_{\mathbf{G}} \left| \tilde{\Omega}(\mathbf{Q} - \mathbf{G}) \right|^2,$$

i.e., we have neglected all interference terms. In the vicinity of a specific $\mathbf{G}_j$, only the term $\tilde{\Omega}(\mathbf{Q} - \mathbf{G}_j)$ will be significantly different from zero.

Let us now consider two typical shape functions (the derivation of $\tilde{\Omega}(\mathbf{q})$ is given in more detail in the appendix):

2.4.1 Example: Block-Shaped Crystals

For a rectangular block with dimensions $\{L_i\}$ for the Fourier transformed of the shape function

$$\left| \tilde{\Omega}(\mathbf{q}) \right|^2 = \prod_{i=1}^3 \frac{\sin^2\left(\frac{q_i L_i}{2}\right)}{\left(\frac{q_i}{2}\right)^2}$$

holds. This function decreases with q_i on the average as q_i^{-2} and has an effective peak width δq_i defined via $|\tilde{\Omega}(q_i = 0)| \cdot \delta q_i = \int_{-\infty}^{\infty} dq_i |\tilde{\Omega}(q_i)|$ of $\delta q_i = \frac{2\pi}{L_i}$. It also exhibits oscillations with a frequency in reciprocal space equal to δq_i .

2.4.2 Example: Superlattice

A superlattice can be seen as a one-dimensional series of block-shaped crystals, i.e., the shape function $\Omega(x)$ is the sum of individual shape functions:

$$\Omega(x) = \sum_{j=1}^N \Omega_{\text{block}}(x - jd)$$

with the individual shape function

$$\Omega_{\text{block}}(x) = \Theta(x)\Theta(b - x),$$

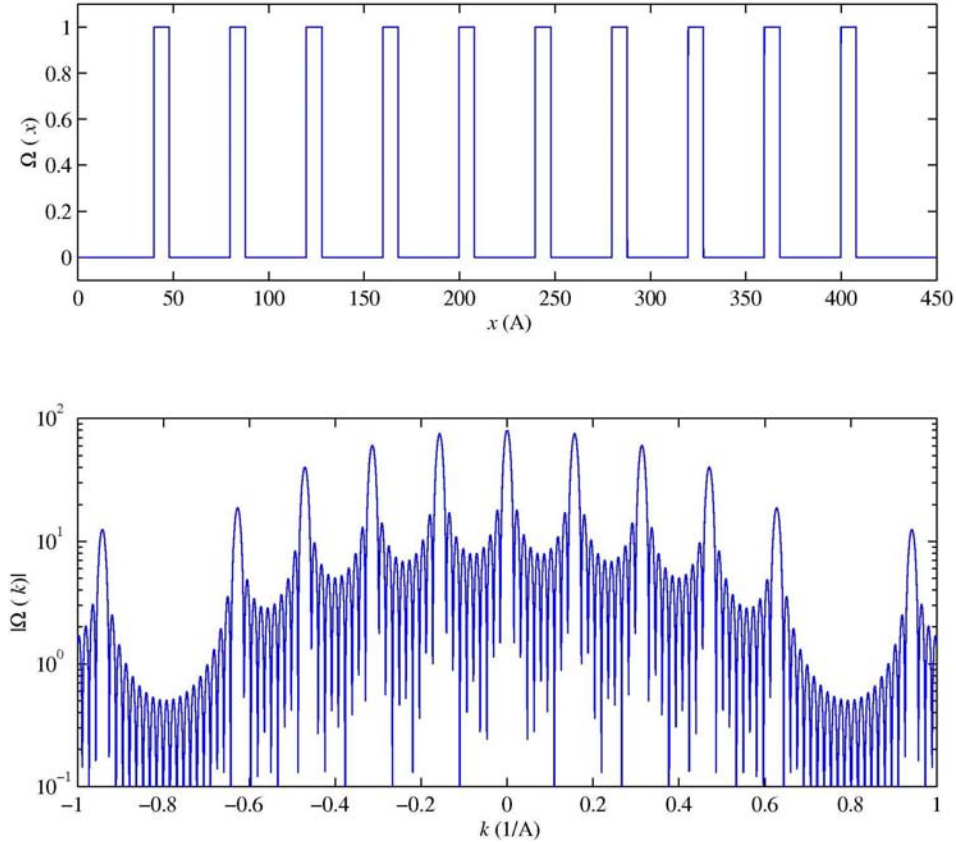


Figure 2.3: Shape function $\Omega(x)$ of a superlattice with a "block width" of $8 \text{ \AA}$ and a superlattice period of $40 \text{ \AA}$, together with the Fourier transform of the shape function $\tilde{\Omega}(k)$

where d is the superlattice period, and $b < d$ is the width of one "block". $\Theta(x)$ is the so-called Heaviside step function, $\Theta(x) = 1$ for $x \geq 0$ and $\Theta(x) = 0$ for $x < 0$. For the square of the absolute value of $\tilde{\Omega}$ we obtain:

$$|\tilde{\Omega}(k)|^2 = \frac{4}{k^2} \sin^2 \frac{kb}{2} \frac{\sin^2 \frac{Nkd}{2}}{\sin^2 \frac{kd}{2}} \quad (2.20)$$

An example of the shape function and its Fourier transform is shown in Fig. 2.3 for $d = 40 \text{ \AA}$, $b = 8 \text{ \AA}$. The remarkable features of the Fourier transform of the shape function are obvious:

- a rapid oscillation with a period of $\frac{2\pi}{Nd}$ indicating the total thickness.
- so called superlattice peaks with a distance of $\frac{2\pi}{d}$
- an envelope of the satellites, where every d/b^{th} peak is suppressed

Chapter 3

Experimental Setup and Methods

3.1 Reciprocal Space Coordinates

3.1.1 Connection Between Angles and Reciprocal Space Coordinates

The connection between the goniometer angles ω and 2θ and the reciprocal space coordinates used in Chap.2 can easily be seen from Fig.3.1. The diffraction vector $\mathbf{Q} = \mathbf{k}_i - \mathbf{k}_s$ is directly connected to the scattering angle θ via

$$\begin{aligned}\frac{|\mathbf{Q}|}{2} &= |\mathbf{k}_{i,s}| \sin \theta \\ Q &= 2k \sin \theta = \frac{4\pi}{\lambda} \sin \theta\end{aligned}\tag{3.1}$$

We consider coplanar scattering, i.e., incident and scattered wavevector $\mathbf{k}_{i,f}$ and the surface normal $\mathbf{n}$ lie in a common plane. For this geometry we denote the direction of $\mathbf{n}$ as the z -direction, and the direction parallel to the sample surface as x -direction. Then we get for the components of the scattering vector $\mathbf{Q} = (Q_x, 0, Q_z)$

$$\begin{aligned}Q_x &= \frac{4\pi}{\lambda} \sin \theta \sin (\omega - \theta) \\ Q_z &= \frac{4\pi}{\lambda} \sin \theta \cos (\omega - \theta)\end{aligned}\tag{3.2}$$

Inverting these expressions, we can calculate the goniometer angles from the reciprocal space coordinates via

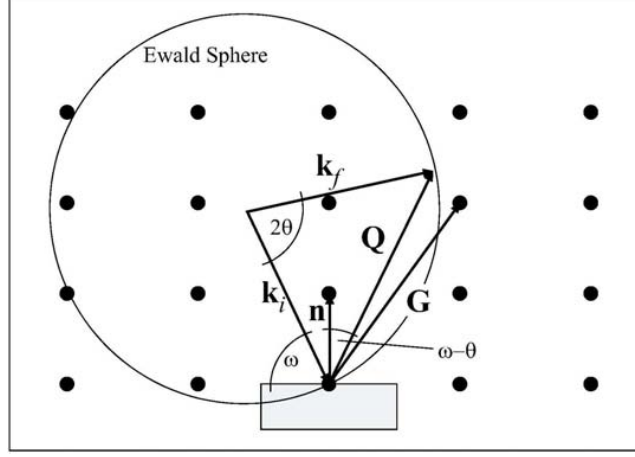


Figure 3.1: Picture of reciprocal space for the sample surface perpendicular to $\langle 001 \rangle$ and the in-plane vector (parallel to the sample surface in the drawing) $\langle 110 \rangle$.

$$\begin{aligned}\theta &= \arcsin\left(\frac{\lambda}{4\pi}Q\right) \\ \omega &= \theta + \arccos\frac{Q_z}{Q}\end{aligned}\tag{3.3}$$

For a cubic lattice the reciprocal lattice vectors have a simple form: $\mathbf{G} = \frac{2\pi}{a}(h, k, l)$, with a the lattice constant of the sample. The z component of $\mathbf{G}$ is $G_z = \mathbf{n} \cdot \mathbf{G}$, the x component can be written as $G_x = \mathbf{n} \times \mathbf{n} \times \mathbf{G}$ ($\mathbf{n}$ has length 1). In our case, $\mathbf{n} = (0, 0, 1)$ holds. For the most common Bragg reflections, the sample azimuth (x -direction) will be either along the crystallographic $[110]$ or $[100]$ directions. For the two cases, the connection between $\mathbf{Q} = \mathbf{G}$ and the lattice parameter are the following:

$$\begin{aligned}a &= \frac{2\pi}{Q_z}l \\ &= \frac{2\sqrt{2}\pi}{Q_{x,[110]}}h \\ &= \frac{2\pi}{Q_{x,[100]}}h\end{aligned}\tag{3.4}$$

For tetragonally distorted samples, as for the SiGe- and SiC-layers investigated here, the relation have to be modified. With $\mathbf{g} \equiv (h, k, l)$ one can write $\mathbf{G}$ as

$$\mathbf{G} = \frac{2\pi}{a_{\perp}} \mathbf{n} (\mathbf{n} \mathbf{g}) - \frac{2\pi}{a_{\parallel}} \mathbf{n} \times (\mathbf{n} \times \mathbf{g},)$$

i.e., we have to consider the lattice parameters in growth direction $a_{\perp}$ (along $\mathbf{n}$) and parallel to the surface $a_{\parallel}$ separately. Consequently, for tetragonal distortion, where the lattice parameters in x - and y -directions are equal, Eq.(3.5) has to be slightly modified:

$$\begin{aligned} a_{\perp} &= \frac{2\pi}{Q_z} l \\ a_{\parallel} &= \frac{2\sqrt{2}\pi}{Q_{x,[110]}} h \\ a_{\parallel} &= \frac{2\pi}{Q_{x,[100]}} h \end{aligned} \tag{3.5}$$

Some other probably useful relations are:

$$\begin{aligned} l &= \frac{2a_{\perp}}{\lambda} \sin \theta \cos (\omega - \theta) \\ h = k &= \frac{\sqrt{2}a_{\parallel}}{\lambda} \sin \theta \sin (\omega - \theta) \\ \theta &= \arcsin \left(\frac{\lambda}{2} \sqrt{\frac{h^2 + k^2}{a_{\parallel}^2} + \frac{l^2}{a_{\perp}^2}} \right) \\ \omega &= \theta + \arccos \frac{\frac{l}{a_{\perp}}}{\sqrt{\frac{h^2 + k^2}{a_{\parallel}^2} + \frac{l^2}{a_{\perp}^2}}} \end{aligned} \tag{3.6}$$

Note that here we are always considering the lattice parameters of a primitive cubic cell. Silicon, germanium and carbon (diamond) and their compounds crystallize in the diamond lattice structure. It is composed of two fcc-lattices which are shifted by a quarter of the space diagonal along the space diagonal with respect to each other. Hence the primitive cubic cell contains 8 atoms, which leads to the following rule for the Bragg reflections:

Only reflections are allowed with either

- h , k , and l even *and* the sum $h + k + l$ a multiple of 4, or
- h , k , and l odd.

3.1.2 Scan Directions, Asymmetrical Reflections

For the recording of x-ray data three major scan modes are used, which can be seen from Fig. 3.2. An ω -scan is performed by rotating the sample around the ω -axis. In the coordinate system of the sample this corresponds to a rotation of $\mathbf{k}_i$, $\mathbf{k}_s$, and $\mathbf{Q}$ around the origin of reciprocal space, as indicated in Fig. 3.2a. Figure 3.2b illustrates an ω - 2θ -scan, where the ω - and 2θ -drives of the goniometer are moved simultaneously, the 2θ -drive at twice the angular speed than the ω -drive. The effect is a scan along the $\mathbf{Q}$ -direction in reciprocal space. As a consequence, for each point $\mathbf{Q}$ in reciprocal space the ω - and 2θ -scan directions are perpendicular with respect to each other. Another important direction is the 2θ -direction, which corresponds to a scan along the Ewald sphere in Fig. 3.2a,b.

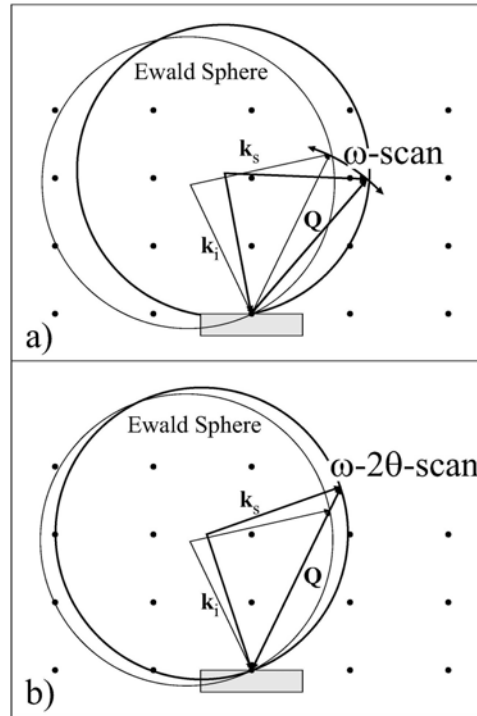


Figure 3.2: Illustration of the ω - and ω - 2θ -scan directions in reciprocal space.

For an asymmetrical reflection there are two possible scattering geometries. One is the so called "grazing exit" configuration as shown in Fig. 3.3, where the incident beam hits the sample at a relatively steep angle, whereas the scattered beam leaves the sample at a rather low angle. By exchanging the roles of incident and scattered beams, one gets to the so called "grazing incidence" configuration (see Fig. 3.7 for an example). The advantage and disadvantage of the two configurations can be seen from Fig. 3.3.

In the "grazing exit" geometry depicted in the figure, a beam with a finite width is "compressed", i.e. the scattered beam has a smaller width than the incident one. Therefore a

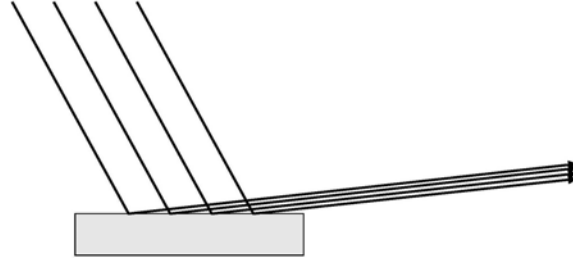


Figure 3.3: Illustration of the effect of a "grazing exit" scattering geometry: the exiting beam is considerably smaller than the incident beam, and therefore the resolution of a scan can be improved (by using a smaller detector slit) without losing intensity.

smaller detector slit can be used as in the "grazing incidence" geometry, resulting in a higher angular resolution, without any loss in intensity.

3.1.3 Determination of a Layer's Strain Status

When a layer of a certain material is grown on a substrate of a different material, the layer will adopt the lateral structure of the substrate up to a certain thickness (critical thickness [3]), known as pseudomorphic growth. Therefore, as the lattice parameter of the layer material will be different from the substrate's, the layer will be strained, i.e. its lattice parameters normal to the surface and parallel to it will be different from each other. If the film thickness exceeds the critical thickness, the layer will relax, i.e. the parallel and normal lattice constants will get more and more equal and tend towards the equilibrium lattice constant of the layer material. The mechanisms responsible for this relaxation process depend on the involved materials and growth conditions (see e.g. Ref. [18]). For a cubic material grown on top of another cubic material, as is the case for most of our samples consisting of SiGe and/or SiC layers with various Ge and C contents grown on silicon, the strained layers will have tetragonal symmetry. The situation for different strain states and equilibrium lattice constants of the layer are illustrated in Fig. 3.4.

The upper three drawings of Fig. 3.4 (a to c) show from left to right the case of a fully strained (pseudomorphic), a partially relaxed and a fully relaxed layer (grey) with bigger equilibrium lattice constant than the substrate (white), the case of a layer with equilibrium constant smaller than the substrate's is illustrated in Fig. 3.4d to f.

Now, if we consider a Si substrate and a $\text{Si}_{1-x}\text{Ge}_x$ layer on top of it, the reciprocal space of this system looks as depicted in Fig. 3.5(a,b) if the $\text{Si}_{1-x}\text{Ge}_x$ is fully relaxed, if it is fully pseudomorphic, the arrangements are like in Fig. 3.5(c,d). In the first case, the symmetry of both lattices is the same, and consequently the reflections with the same (hkl) lie in the same directions from the origin. On the other hand, if the epilayer is pseudomorphic, the

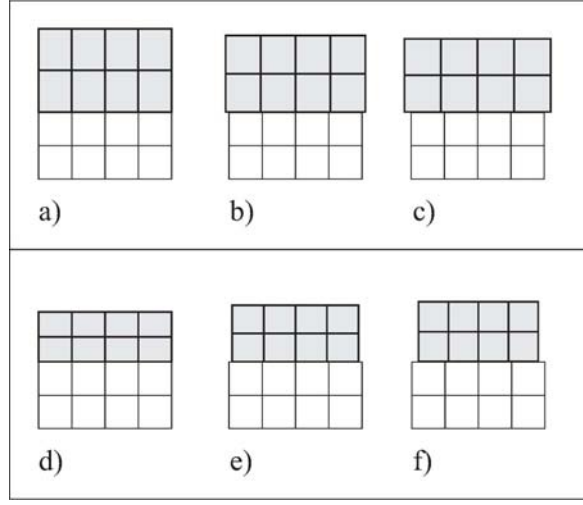


Figure 3.4: Different strain states for a layer with bigger lattice constant (a to c) and a layer with smaller lattice constant (d to f) than the substrate. The strain states are fully strained (pseudomorphic; left side), partially relaxed (middle), and fully relaxed (rightmost figures).

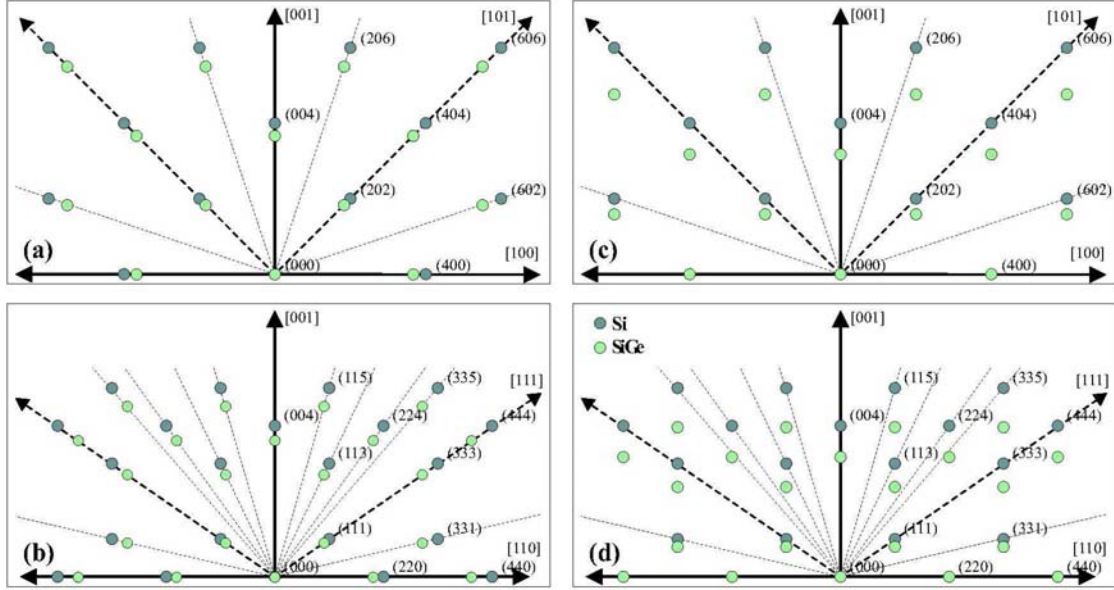


Figure 3.5: Reciprocal space for a $\text{Si}_{1-x}\text{Ge}_x$ layer on a Si substrate for the case of complete relaxation of the epilayer (a,b) and for completely pseudomorphic growth (c,d). Panels (a,c) are with $Q_x \parallel [100]$, panels (b,d) for $Q_x \parallel [110]$, Q_z is always parallel to $[001]$.

in-plane lattice parameter $a_{\parallel}$ is the same, and consequently the reflections for the same (hkl) are aligned vertically over each other. However, $a_{\parallel}$ and $a_{\perp}$ are not independent of each other, but are determined by the elastic properties of the material. Roughly speaking, if the lattice parameter $a_{\parallel}$ is compressed to a smaller value than the bulk lattice parameter

a_{bulk} of the $\text{Si}_{1-x}\text{Ge}_x$ alloy, the layer will respond by an increase of $a_{\perp}$. For tetragonal distortion of cubic materials with the perpendicular direction parallel to $[001]$ direction the relation

$$a_{\perp} = a_{\text{bulk}} + \frac{2C_{12}}{C_{11}} (a_{\text{bulk}} - a_{\parallel}) \quad (3.7)$$

holds, where C_{11} and C_{12} are the elastic constants of the epilayer. In the case of $\text{Si}_{1-x}\text{Ge}_x$ or $\text{Si}_{1-y}\text{C}_y$ layers, these elastic constants as well as a_{bulk} depend on the composition x or y (see the appendix for details). Often, instead of the lattice parameters, the strain of an epilayer *with respect to its bulk lattice parameter*

$$\begin{aligned} \varepsilon_{\parallel} &= \frac{a_{\parallel} - a_{\text{bulk}}}{a_{\text{bulk}}} \\ \varepsilon_{\perp} &= \frac{a_{\perp} - a_{\text{bulk}}}{a_{\text{bulk}}} \end{aligned} \quad (3.8)$$

is given, or alternatively the strain of an epilayer *with respect to the substrate lattice parameter* a_{sub}

$$\begin{aligned} \epsilon_{\parallel} &= \frac{a_{\parallel} - a_{\text{sub}}}{a_{\text{sub}}} \\ \epsilon_{\perp} &= \frac{a_{\perp} - a_{\text{sub}}}{a_{\text{sub}}}. \end{aligned} \quad (3.9)$$

Obviously, $\epsilon_{\parallel} = 0$ for a fully pseudomorphic layer, while it has some finite value depending on $a_{\text{bulk}}(x, y)$ for a relaxed layer. Vice versa, $\varepsilon_{\parallel} = 0$ for a fully relaxed layer, while it has some finite value for a pseudomorphic layer. Therefore, in order to quantify the relaxation, sometimes also the *degree of relaxation* is given:

$$\mathcal{R} = \frac{a_{\parallel} - a_{\text{sub}}}{a_{\text{bulk}} - a_{\text{sub}}} \quad (3.10)$$

For fully relaxed layers $a_{\parallel} = a_{\text{bulk}}$ and hence $\mathcal{R} = 1$, while for pseudomorphic layers $a_{\parallel} = a_{\text{sub}}$ and hence $\mathcal{R} = 0$ hold.

If we enlarge, as an example, the region around the (224) Bragg point in Fig. 3.5(b,d), we obtain the so-called *relaxation triangle*, shown in Fig. 3.6.

The reciprocal lattice point of a fully strained layer lies directly beneath or above the corresponding reciprocal lattice point of the substrate in an asymmetrical reflection, as

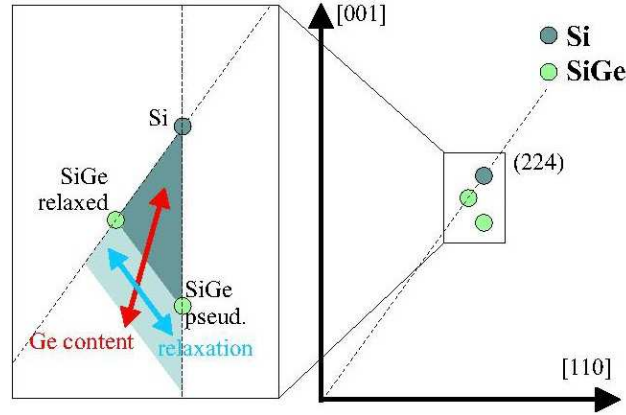


Figure 3.6: Illustration of the effect of strain on the position of the reciprocal lattice points, the so-called *relaxation triangle*. A fully strained layer exhibits the same lattice constant parallel to the growth plane than the substrate and therefore the corresponding reciprocal lattice point lies exactly beneath or above the substrate's in an asymmetrical reflection [here the (224)], depending on whether the layer's equilibrium lattice constant is bigger or smaller than that of the substrate. If the layer is fully relaxed, the position of the corresponding reciprocal lattice point lies in the same direction in reciprocal space than that for the substrate, provided that the layer material has the same symmetry than the substrate. The reciprocal lattice point for a partially relaxed layer lies between those two positions. Hence changing the strain status of a layer will result in a shift of the peak in reciprocal space as indicated by the blue arrow, while changing the Ge content shifts the peak as indicated by the red arrow.

the lattice constants parallel to the growth plane are equal. If the layer is fully relaxed and the material has the same symmetry than the substrate (as is the case for our samples), then the reciprocal lattice point of the layer will lie in the same direction in reciprocal space than the substrate's. If the layer is partially strained, the reciprocal lattice point lies in between those two positions.

3.1.4 Reciprocal Space Mapping

To obtain precise data on the lattice parameters of a sample (which may consist of several layers on a substrate), the intensity distribution around a reciprocal lattice point is recorded by "mapping" an entire region of reciprocal space. This is done by performing a series of ω - 2θ -scans (see below) at different ω -settings (or vice versa). An overview over the region of reciprocal space accessible within our scattering geometry is given in Fig. 3.7.

The solid dots denote the position of the accessible reciprocal lattice points. The maximum length of the scattering vector is twice the length of the $\mathbf{k}$ -vectors (backscattering of the incident beam), and is limited by the wavelength of the x-ray source. This length is denoted by the solid outer half circle in Fig. 3.7. Of course, in an experiment the source and detector cannot be at exactly the same position, and therefore the biggest scattering angle 2θ will

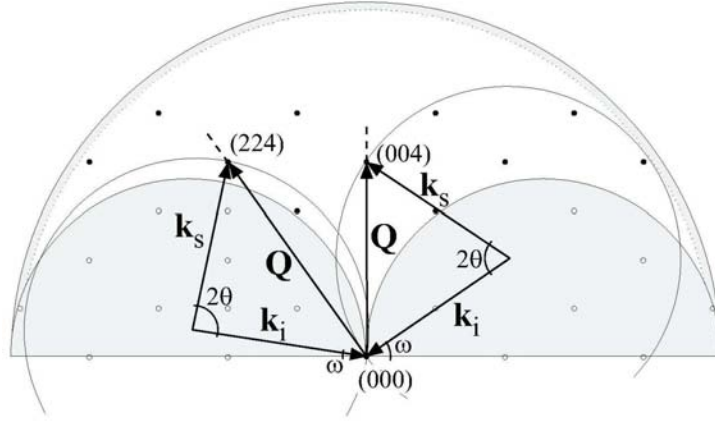


Figure 3.7: Picture of the region of reciprocal space accessible within the scattering geometry used with the MRD for a sample grown on a $\langle 001 \rangle$ -substrate and in plane vector $\langle 110 \rangle$. For a detailed discussion see the text.

in practise be somewhat smaller than 180° , which is denoted by the dotted line and the shaded region inside the outer half circle. The two shaded half circles in Fig. 3.7 denote the so-called Laue zones. To "reach" a Bragg reflection within one of this zones, either the incident or the scattered beam have to pass through the sample, which is not possible for our samples (the absorption length is in the order of $100\mu\text{m}$, the extinction length even smaller). The lower half-plane (not shown in Fig. 3.7) corresponds to a scattering geometry where the incident or the scattered beam or both have to pass through the sample. Also indicated in Fig. 3.7 are the most frequently used Bragg reflections for the recording of our data, namely the (symmetrical) (004)- and the (asymmetrical) (224)-reflection.

From a reciprocal space map around a Bragg reflection, i.e., of a region as depicted in Fig. 3.6, the position of a peak due to an epilayer is obvious, allowing to see immediately whether a layer is relaxed or pseudomorphic. The strain parallel and normal to the growth plane can be calculated quantitatively from the lattice constants of the layer (which are obtained from the coordinates of the reciprocal lattice points).

It is clear that from a map around a symmetrical reflection, only the lattice constant normal to the growth plane can be determined, whereas from an asymmetrical reflection both, the normal and parallel lattice constants can be obtained. We use the reciprocal lattice of the substrate (which lattice constant is known very accurately) as a reference for the determination of positions of reciprocal lattice points of the investigated layers, and the lattice parameters obtained thereof.

3.2 Setup of a High Resolution X-Ray Diffractometer

3.2.1 General

The principal setup of the diffractometer is shown in Fig. 3.8a. The incident angle ω is the angle between the incident beam and the sample surface, the scattering angle 2θ is the angle between incident and scattered beam. The x-ray source is a conventional x-ray tube with a copper anode and a maximum electric power of 2kW. Our data will be recorded using a electric power of 1.6kW (acceleration voltage 40 kV, electron current to the anode 40 mA). Included in the primary beam optics is a four-crystal Ge (220) Bartels-type monochromator. Its setup is shown schematically in Fig. 3.8b.

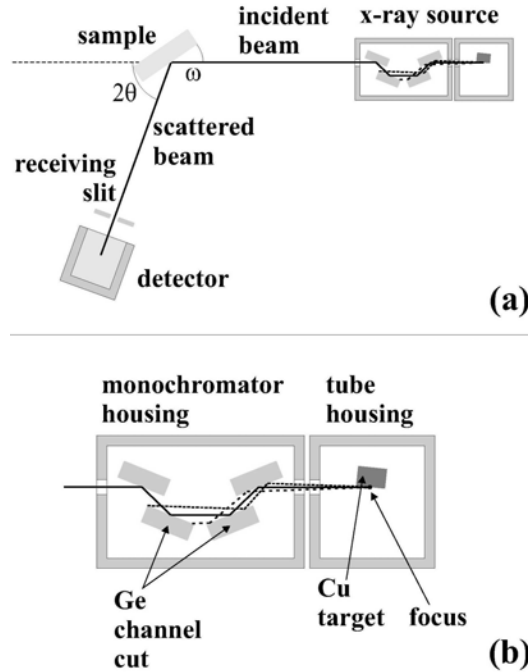


Figure 3.8: Scheme of the scattering geometry used with a high-resolution diffractometer (top view): a) shows the beam geometry, b) illustrates the functionality of the four-crystal Ge (220) Bartels-type monochromator (for details see the text).

The functionality of the monochromator can be seen from Fig. 3.8b and Fig. 3.9. The first crystal "selects" a different angle for each wavelength of the polychromatic x-ray source, indicated by the different line styles in Fig. 3.8b, the solid line in Fig. 3.8b indicates the part of the beam with the desired wavelength (and Bragg-angle). The second crystal is aligned exactly parallel to the first one, because it is a part of the same single crystal (the two "reflecting" surfaces are fabricated by cutting a groove or channel into a single crystal of Ge, therefore this type of two-reflection crystal is called a "channel cut" crystal. Hence

the incidence angle of any radiation reflected by the first crystal is exactly equal for both crystals. In other words, the relation between scattering angle and wavelength (Bragg-law) of first and second crystals is equal (Fig. 3.9a) and no further "monochromating" effect by the second reflection is obtained. The third crystal is mirrored as compared to the second one. (Third and fourth crystal are in reality again a channel cut crystal.) Therefore the part of the beam reflected by the second crystal at a somewhat higher angle than the intended one incides the third crystal at a somewhat too low angle and is therefore blocked (Fig. 3.9b), and the same is true for the part of the beam reflected by the second crystal at a somewhat too low angle. Only the part of the incident beam with almost exactly the intended wavelength therefore passes the third crystal (the finite width of the Bragg reflections of the monochromator crystals is indicated by the "finite" linewidth in Fig. 3.9). The fourth crystal — similarly to the second one — has only a small effect on the monochromacy of the primary beam and is used to reflect the beam in the same direction it had in front of the monochromator, so that the monochromator can be removed without the need of changing something else in the beam geometry. The wavelength of the primary beam in our case is set to the copper $K_{\alpha 1}$ -line at $\lambda = 1.5406 \text{ \AA}$, and the line width obtained is 12 arcsec. The detection resolution is determined by the width of a slit in front of the detector in double crystal mode.

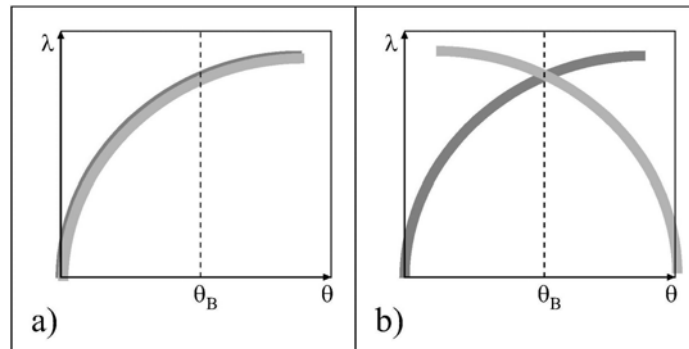


Figure 3.9: Schematic illustration of the monochromacy obtained with two subsequent Bragg reflections (at crystals of the same type; "duMond"-diagrams). The wavelength λ for which the crystal reflects is shown as a function of the scattering angle θ , i.e., the diagram is a graphic representation of Bragg's law. The curves have a finite width indicating a finite acceptance angle of the crystals. For two crystals, the region where both curves overlap denote determine the resulting resolution in wavelength and scattering angle: a) case of parallel crystals, the resolution is almost the same as with only one reflection; b) case of crystals tilted by twice the Bragg angle against each other, considerable increase in resolution as compared to a single reflection (for further details see the text).

3.2.2 Sample Stage

The sample is fixed to the sample holder using a small pump, with adhesive tape or with a glue. When putting the sample on the sample holder, it will be tilted around three axes,

as indicated in Fig. 3.10:

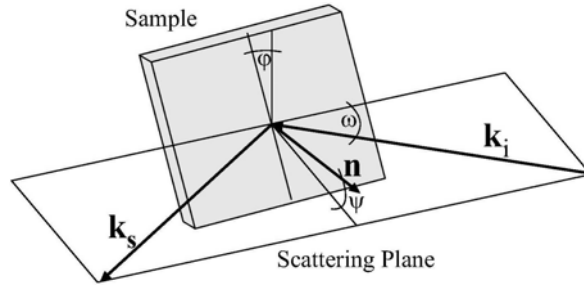


Figure 3.10: Definition of the goniometer angles: ω is the angle between incident beam and sample surface in the scattering plane, ψ is the tilt of the surface normal of the sample with respect to the scattering plane, and φ describes the rotation of the sample around its surface normal.

- ω is defined as the angle between the sample surface and the incident beam, but the angle measured by the instrument is rather the angle between the "sample holder surface normal" and the incident beam. Therefore we have to expect a certain angle offset in ω when a sample is put on the sample holder, e.g. because of some particles between the sample and the holder or scratches in the holder's surface, or because of a certain "miscut" of the samples: usually, the surface of a sample is oriented only with a certain precision of several tenths of a degree with respect to a certain crystallographic orientation, in our case the (001) direction.
- The ψ -angle, i.e., the angle between the sample's surface normal and the scattering plane defined by the incident and scattered beam, will exhibit a similar offset for the same reasons.
- As the sample is put on the holder, the φ -position, i.e. the tilt of some direction parallel to the sample surface (e.g. the $\langle 110 \rangle$ -direction for the measurements to be carried out) with respect to the scattering plane (i.e. φ describes a rotation of the sample around its surface normal) is determined by the eye only, which causes a misalignment of φ of up to several degrees (and again the measured angle is connected to the holder, not the sample).

Beside the angle offsets discussed above, there is an additional mechanism introducing a misalignment to the goniometer angles: The goniometer angles are controlled by the stepping motors, and therefore additional offsets exist caused by the finite accuracy of the

gears connecting the motors with the goniometer axes. Therefore, also the angle 2θ should be calibrated.

In summary, when putting a sample onto the instrument, it has to be aligned before any measurement can be carried out. To align all goniometer angles, a series of scans has to be performed, as the influences of the particular goniometer axes positions on the alignment are not independent from each other, which can be seen from the following.

3.2.3 Sample Alignment

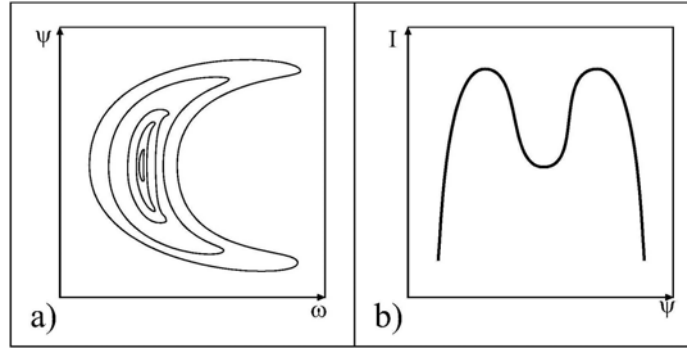


Figure 3.11: Scheme of the dependence of the intensity in the vicinity of a Bragg peak on the goniometer alignment. a) intensity distribution for different ω and ψ position, and b) a cross section through a) at a constant ω -angle at the right side of the maximum.

After mounting the sample, the nominal Bragg angle 2θ and incident angle ω are calculated, and the goniometer is set to this position. Now, the substrate reflection of the sample is aligned. All samples used in this course are heterostructures deposited on Si (001) wafers. Hence we will align the sample using the Bragg reflections from Si. As a first step of alignment, one will perform an ω - and a 2θ -scan with low resolution (e.g. by using a broad detector slit or the open detector) around this theoretical values to find a starting position for further optimization steps. Usually one will for this purpose use a "symmetrical reflection", i.e., a reflection at lattice planes parallel to the sample surface, as for this reflections a misalignment in φ is not important. If we imagine the sample is ideally aligned on a symmetrical reflection (i.e., $\omega = \frac{2\theta}{2}$) and then it is tilted around the ψ -axis in Fig. 3.10, ω is effectively decreasing. To retain an "almost aligned" position, ω as a consequence has to be increased, so that the incident angle with respect to the lattice planes again fulfills the Bragg condition. As the lattice planes are no longer perpendicular to the scattering plane, and for a beam divergent normal to the scattering plane, as is the case for our setup, the Bragg condition is fulfilled for a part of the beam only, leading to a decrease in the scattered intensity. The resulting intensity distribution in the vicinity of a Bragg reflection is indicated in the contour map of Fig. 3.11a.

There are several ways to obtain the proper value for ψ . One possibility is to perform a

series of ω -scans at different ψ settings to find the intensity maximum, which is found for the scan which has at the same time the lowest ω -value for the peak and will in most cases show the smallest peak width. Another possibility is to perform an ψ -scan at a constant ω value, which yields a double-peak as indicated in Fig. 3.11b, as the starting ω -position will usually be on one of the "wings" in Fig. 3.11a. If ψ is set to the center between the peaks before a subsequent ω -scan is performed, the resulting ω and ψ values will lie close to the optimum positions. "Close", because the shape of the iso-intensity lines of Fig. 3.11a may be somewhat distorted if, e.g., φ is misaligned. Therefore here we use the first and "safer" method.

After ψ has been aligned, we use an asymmetrical reflection (lattice planes tilted with respect to the sample surface) to align φ . If we now imagine a rotation of the sample around the surface normal, the angle between the incident beam and the scattering planes (which are not perpendicular to the surface normal) will again decrease, and to retain a "almost aligned" position, this angle has to be increased. As the latter angle differs from ω by a certain constant value (namely exactly the tilt of the scattering planes with respect to the surface), the resulting intensity distribution is very similar to Fig. 3.11a, but with φ replacing ψ . Therefore the aligning procedure will also be the same as described before. As was noted above, the angles φ and ψ are not independent from each other, i.e. rotating the sample around the "sample holder's surface normal" will also change its tilt with respect to the scattering plane, unless the sample is precisely aligned and the "sample holder's surface normal" coincides with the samples surface normal. As a consequence, the alignment procedures have to be repeated several times (at least once), until the remaining offsets are negligible.

3.3 Operating the X'Pert PRO PANalytical Diffractometer

In the following a description of the X'Pert PRO PANalytical diffractometer is given, intended as a short reference for this course. The instrument is designed as a full-protection unit concerning radiation safety, therefore there is no danger of getting exposed to x-rays. However, as the diffractometer is a high precision mechanical instrument, and the anode is operated at high electrical power, everything should be handled carefully. If anything differs from the description here, **please ask** the tutor or somebody experienced, **do not just try things!**

Please read this instruction completely before starting the experiment. Then, it is best to make all settings in the control software (setting up data directories etc.) before mounting and aligning the sample, and finally performing the experiment.

3.3.1 Diffractometer Elements

The main parts of the diffractometer are:



Figure 3.12: The x-ray tube and the monochromator housing



Figure 3.13: The sample stage



Figure 3.14: The x-ray detector arm and the vertical divergence slit

The x-ray tube, the monochromator, the sample stage, the vertical divergence slit and the detector are shielded behind a metal-housing with glass-doors, so that there is no danger of getting exposed to x-rays.



Figure 3.15: The display-unit and the buttons for turning on/off the shutter and the light



Figure 3.16: The cooling-unit for the x-ray tube



Figure 3.17: The PC for processing the diffractometer

The sample stage offers 6 degrees of freedom for the sample movement, listed in the order of stacking, i.e., all elements below a certain element in the following list are moved together with this element:

- incidence angle Ω (motorized)
- sample translation Z (motorized, translation along "surface normal")
- tilt angle Ψ (motorized)
- azimuth angle Φ (motorized)
- sample translation X (motorized)
- sample translation Y (motorized)

In addition, the detector can be moved around a vertical axis through the common center of rotation of all axes

- detector angle 2Θ (motorized)

3.3.2 Mounting a Sample

As told before, there are a few possibilities to mount a sample on the samplestage:

- Use a doublesided adhesive tape
- Use a glue (removeable)
- Use a vacuum pump

In our case we use the glue. Therefore we have to move the samplestage in a horizontal position. To do so, do the following procedure:

1. Close the doors of the diffractometer
2. Start the program 'X'Pert Data Collector' at the PC
3. Enter username and password
4. Connect the PC with the diffractometer
5. Go into the 'Instrument Settings'-menu and move all values to '0' (see Fig. 3.18)
6. Move the value for ' ψ ' to '90°'

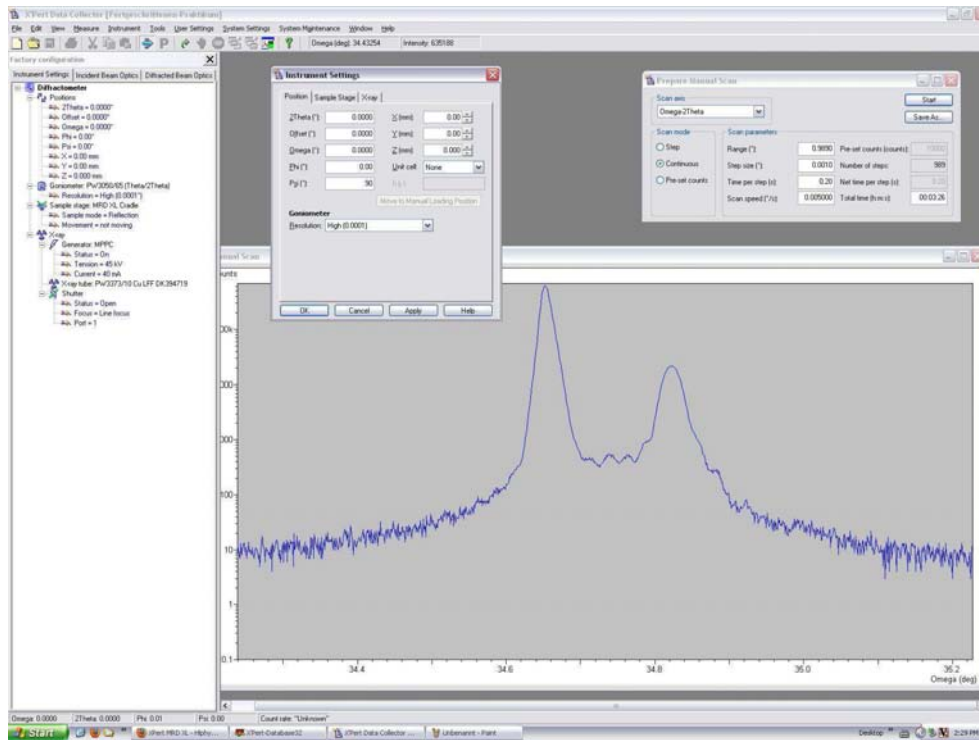


Figure 3.18: The 'Instrument Settings'-menu



Figure 3.19: The sample glued to the samplestage

7. Push the 'Close Shutter'-button and open the doors
8. Glue the sample at the corners carefully on the cleaned center of the samplestage (see Fig. 3.19)
9. Wait for about 3 minutes to let the glue time to harden

Now you have successfully mounted the sample.

Hint: You can also mount two samples simultaneously at the positions $y = + 20$ mm and $y = - 20$ mm instead of mounting one sample in the center at $y = 0$ mm.

3.3.3 Aligning a Sample

Correct angles can only be measured, if the surface of the sample is in the middle of the x-ray beam. It is also to consider, that the atomic layers of the sample are usually slightly tilted to the surface. To get the right results, do the following procedure:

1. Set back the value for ' ψ ' to '0°'
2. Enter the correct value for 'y'

In our case this is one of the values $y = 0$ mm or $y = + 20$ mm or $y = - 20$ mm

3. Also the sample is only about 1 mm thick, enter the value of '16 mm' for 'z'

Now it is time to make the first scan. You move the sample in z-direction and look at the intensity. As the detector has no angle to the beam, the sample is in the beam-center, when half of the intensity is absorbt.

4. Do this 'z-scan' (see Fig. 3.20)

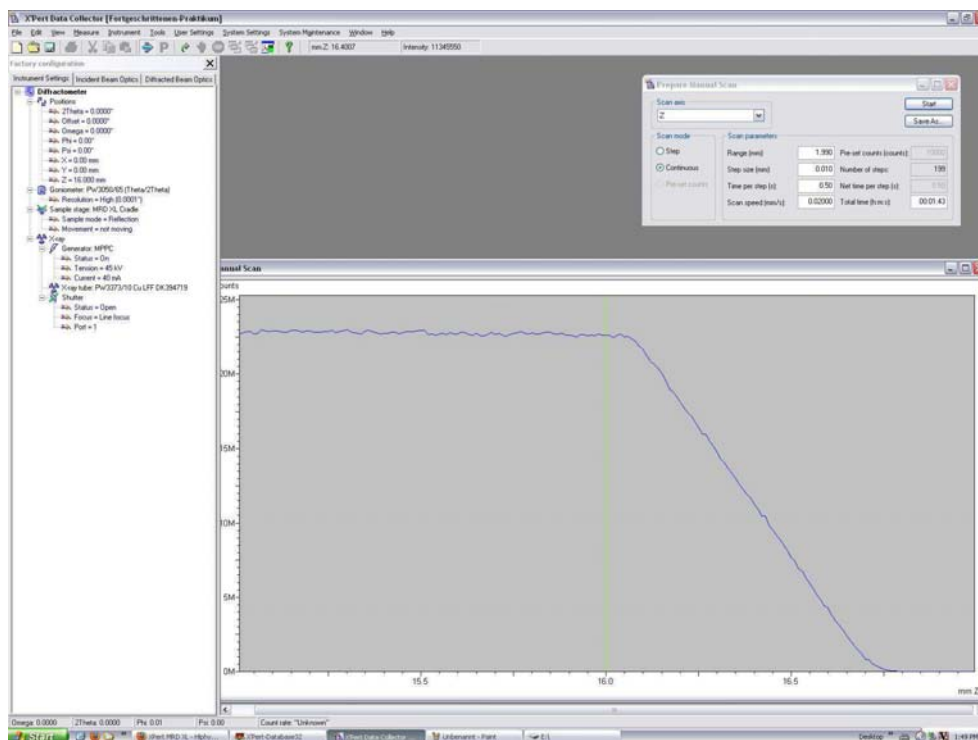


Figure 3.20: The 'z-scan'

The scan should look like as in the picture. Now the position in which the value of 'z' is dropped to one half can be found.

5. Enter this 1/2-value for 'z'

We are looking for the 0-0-4-peak of Si. This peak has the nominal values:

$$\omega = 34,56^\circ$$

$$2\theta = 69,12^\circ$$

6. Run an ω -scan and find this peak (see Fig. 3.21)

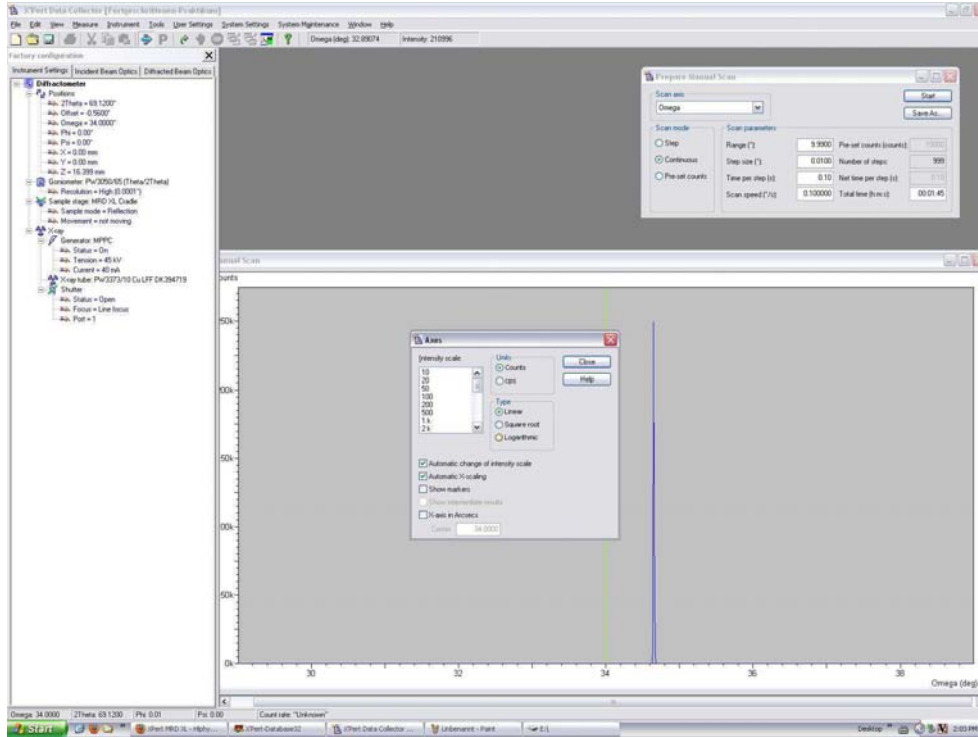
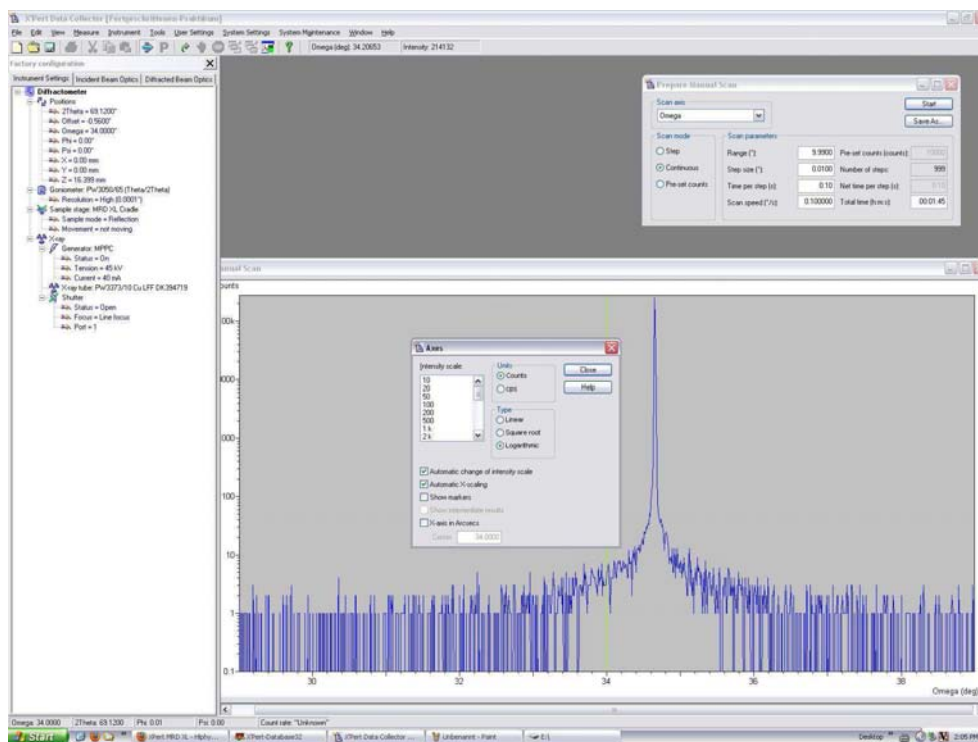


Figure 3.21: The ' ω -scan - linear scale'

7. Change in the 'axes'-menu the scaling from *linear* to *logarithmic* to find even less intense peaks (see Fig. 3.22)
8. Move to this peak in the 'Peak Parameters'-menu (see Fig. 3.23)
9. Run a ψ -scan (see Fig. 3.24)
10. Move to this peak in the 'Peak Parameters'-menu
11. (Run a ϕ -scan)

As our peak is a symmetric peak, this scan has practically no influence to the results. So you can skip this step.

Important: This procedure suggerates that all axes can be controlled without any influence to the other axes, but this is not true! You can only move and rotate the whole sample and therefore if one value is changed, all other values are also changed!

Figure 3.22: The ' ω -scan - logarithmic scale'

12. Repeat the steps 6 - 11 until all values are (practically) constant
13. Run an ω -2 θ -scan (you should find more than one peak!) (see Fig. 3.25)
14. Move to the position in the middle of the two (or more) peaks

You have now correctly aligned the sample. (Don't be troubled by the fact that in many cases aligning the sample takes longer than the measurement itself.)

3.3.4 The Measurement

There are a few possibilities to get the results in which you are interested. In practical use the most limiting factor is the *measuring-time*. So you have to consider:

1. How many data-points do I need?
2. How long will I measure one point?
3. How often will I repeat my measurement?

In our case 1 measurement with approximately 1000 data-points and a measuring-time of about 15 - 20 minutes will be a good choice. So:

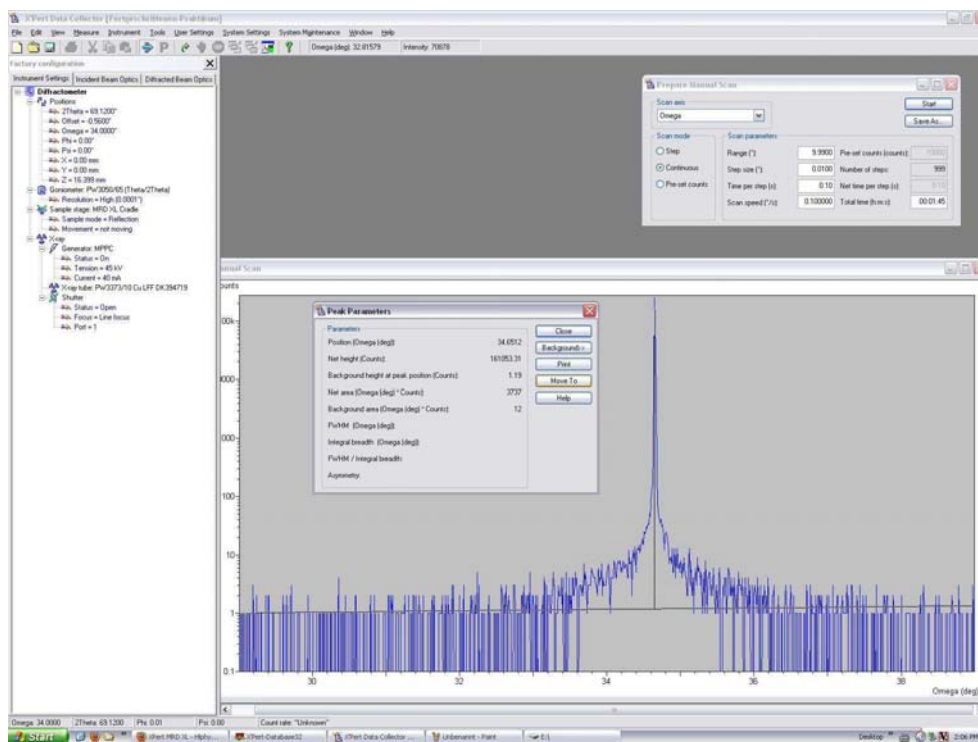


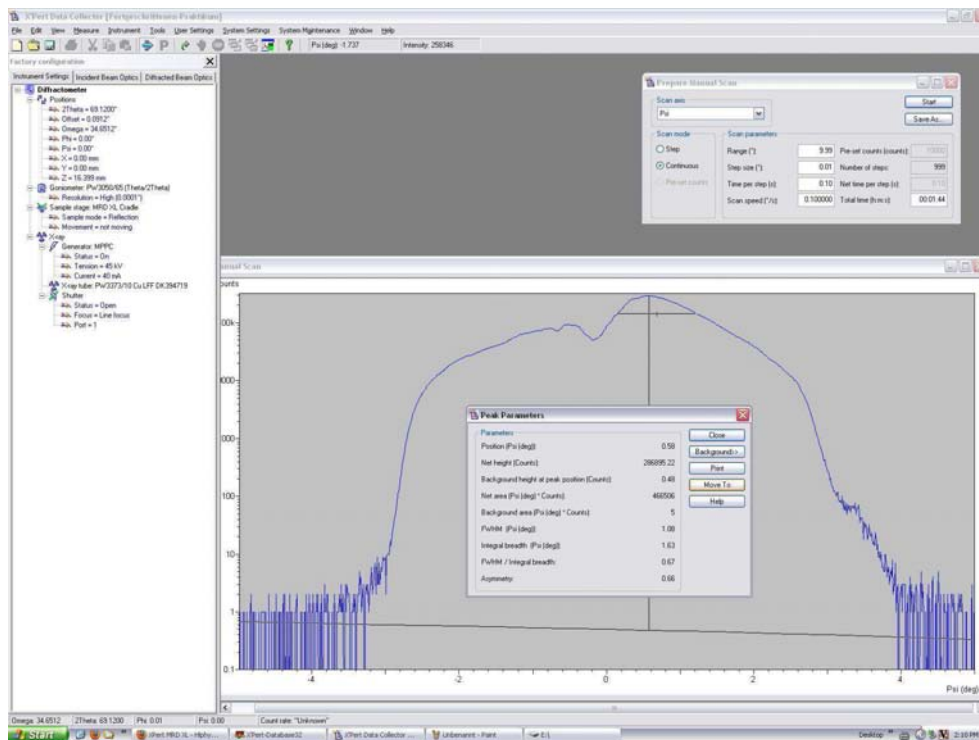
Figure 3.23: The 'Peak Parameters'-menu

1. Enter these values in the 'Prepare Manual Scan'-menu
2. Click the 'start'-button
3. Wait until the measurement is completed.
4. Click 'Save As...' in the 'File'-menu and choose a proper directory and filename (see Fig. 3.26)
5. Open this directory, click at the filename and choose 'Convert' (see Fig. 3.27)

This will generate two new files. If you open the file with the extension '.xy' you will see two rows with numbers (see Fig. 3.28). The first row is the ω -angle and the second the measured intensity at this angle. This data can be imported with programs like Microsoft EXCEL, Origin, Open Office or something equal.

Now you have successfully completed your measurement.

6. You can now adjust and measure the 2nd mounted sample
7. Finish measuring: Put in the 'Instrumental Settings'-menu all values to '0' and the value for ' ψ ' to '90°'
8. Remove your sample(s) from the samplestage

Figure 3.24: The ' ψ -scan'-menu

9. Copy the datafiles to the USB-stick

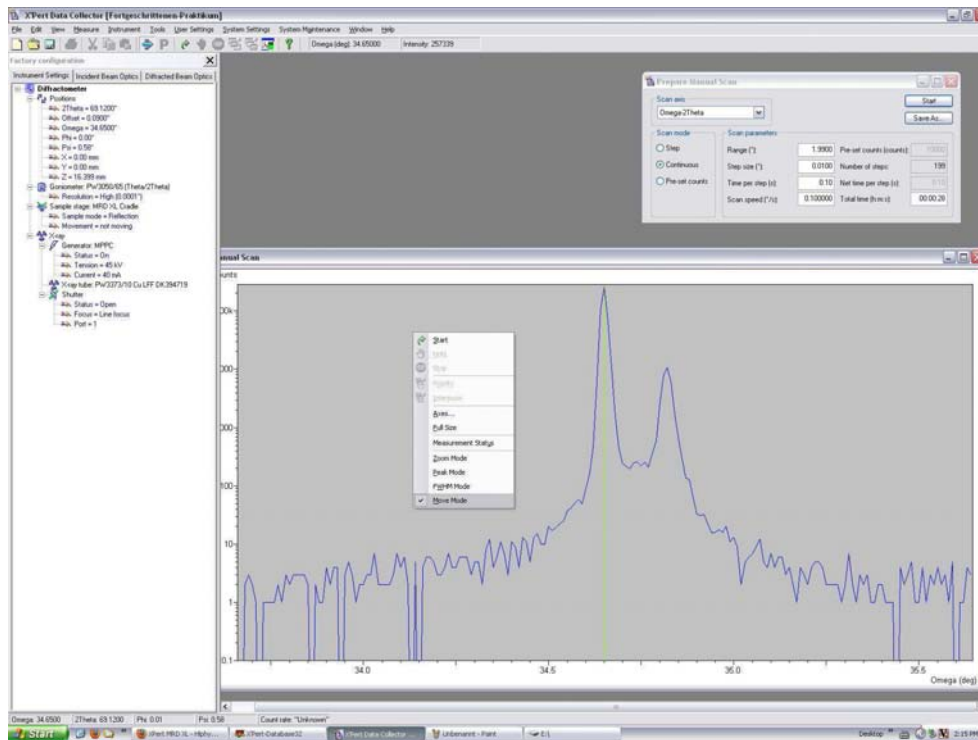
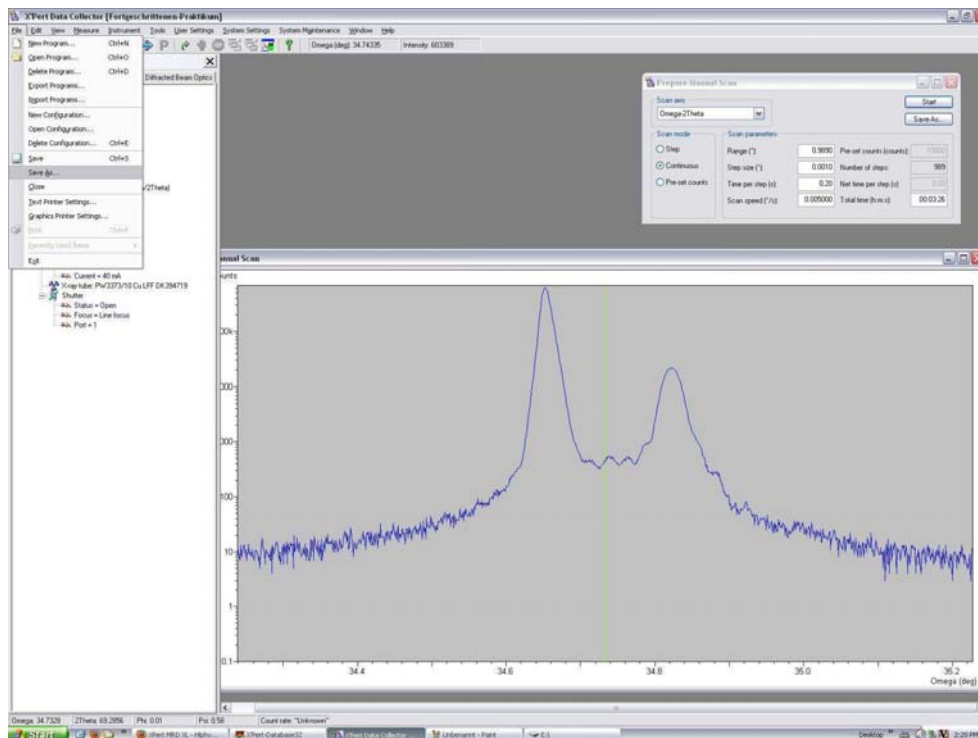
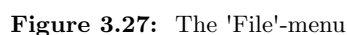
Figure 3.25: The ' ω -2 θ -scan'-menu

Figure 3.26: The 'File-convert'-menu



Chapter 4

Investigation of a Si/SiGe(C) Heterostructure

The Problem

You have got two out of three samples for this course. The three structures are depicted in Fig. 4.1: a single $\text{Si}_{1-x}\text{Ge}_x$ layer on top of a Si substrate, or a single $\text{Si}_{1-y}\text{C}_y$ layer on a Si substrate, or a 10 period Si/ $\text{Si}_{1-x}\text{Ge}_x$ superlattice. In any case the substrate is a Si wafer with (001) orientation. Therefore You should align for the Si (004) reflection. For these type of samples, a short alignment procedure may be used: You need to align only Psi at the symmetric (004) reflection. No Phi alignment at an asymmetrical reflection is required, hence also no iterative refinement of Phi and Psi.

After the sample is aligned, You should measure an ω - 2θ -scan around the Si (004) peak. Use a typical scan range of 5° . You should see the typical peaks of the Si substrate and the SiGe, SiC, or Si/SiGe-superlattice layers already in a fast scan. Choose a position in the middle of the peaks for a fine scan, and select a useful scan range. Then, perform a fine scan with a step widths of 0.001° to 0.002° and 1 to 2 seconds counting time per point, depending on available time (the scan should last approximately 15 - 20 minutes). After the scan has finished, save the measurement (select a proper directory and file name) and convert this file.

From the scan, You should extract the following information:

- In the case of the $\text{Si}_{1-x}\text{Ge}_x$ or $\text{Si}_{1-y}\text{C}_y$ layer:
 - determine the lattice parameter in growth direction (i.e., along the surface normal direction).
 - assuming that the layer is grown pseudomorphically onto the Si substrate, determine the Ge or C content using the dependence between lattice parameter and composition given in the appendix.

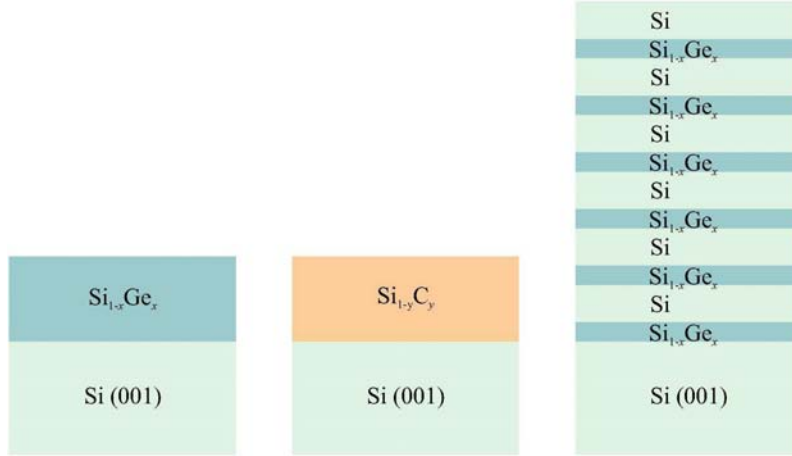


Figure 4.1: Schema of the three different samples available for this course.

- from the width of the peak corresponding to $\text{Si}_{1-x}\text{Ge}_x$ or $\text{Si}_{1-y}\text{C}_y$, or from the spacing of the thickness oscillations, determine the layer thickness.
- In the case of the $\text{Si}/\text{Si}_{1-x}\text{Ge}_x$ superlattice:
 - determine the *average* lattice parameter in growth direction (i.e., along the surface normal direction) from the position of the zero-order superlattice reflection (the highest peak of the series of satellites indicating the superlattice).
 - assuming that the layer is grown pseudomorphically onto the Si substrate, determine the *average* Ge content using the dependence between lattice parameter and composition given in the appendix.
 - assuming a thickness ratio of 1:4 of the $\text{Si}_{1-x}\text{Ge}_x$ and Si layers, determine the Ge content of the $\text{Si}_{1-x}\text{Ge}_x$ layers.
 - from the spacing of the superlattice peaks, determine the superlattice period.

Note: In order to calculate layer thicknesses or periods, it is convenient to convert the data set into reciprocal space coordinates first. To do so, and also to calculate lattice parameters, the angle coordinates should always be corrected, so that the substrate peak lies at the nominal values.

Appendix

The Structure Factor of a Crystal

For crystals $\rho(\mathbf{r})$ can be identified with the total electron density as long as $\omega \gg \omega_B$, the Bohr frequency, i.e., the electrons can be treated as at rest on a timescale relevant for the scattering process. Now the electrons are distributed around the atoms at sites $\mathbf{R}_n$. Therefore the electron density can be written as

$$\rho(\mathbf{r}) = \sum_{n=1}^{N_0} \rho_n(\mathbf{r} - \mathbf{R}_n) = \sum_{s=1}^N \sum_{\gamma=1}^{\nu} \rho_{s\gamma}(\mathbf{r} - \mathbf{R}_s - \mathbf{R}_\gamma) \quad (4.1)$$

where we have divided the sum over the atom sites n into a sum over the lattice sites s and the sum over the sites in the basis γ . $N_0 = N \cdot \nu$ is the total number of atoms in the crystal, N the number of unit cells and ν the number of atoms per unit cell. By inserting 4.1 into 2.7, we obtain

$$\begin{aligned} I(\mathbf{Q}) &\sim \left| \sum_{n=1}^{N_0} f_n e^{i\mathbf{Q}\mathbf{R}_n} \right|^2 = \left| \sum_{s=1}^N \sum_{\gamma=1}^{\nu} f_{s\gamma} e^{i\mathbf{Q}(\mathbf{R}_s + \mathbf{R}_\gamma)} \right|^2 \\ f_n(\mathbf{Q}) &= \int d^3\mathbf{r}' \rho_n(\mathbf{r}') e^{i\mathbf{Q}\mathbf{r}'} \\ f_{s\gamma}(\mathbf{Q}) &= \int d^3\mathbf{r}' \rho_{s\gamma}(\mathbf{r}') e^{i\mathbf{Q}\mathbf{r}'} \end{aligned} \quad (4.2)$$

$f_n(\mathbf{Q})$ is the so-called structure factor of the crystal. For perfect crystals, f_n depends only on the arrangement and types of atoms in the unit cell:

$$\begin{aligned}
I(\mathbf{Q}) &= \left| \sum_{s=1}^N f_s e^{i\mathbf{Q}\mathbf{R}_s} \right|^2 \\
f_s(\mathbf{Q}) &= \sum_{\gamma=1}^{\nu} \int d^3\mathbf{r}' \rho_{\gamma}(\mathbf{r}') e^{i\mathbf{Q}\mathbf{r}'}, \tag{4.3}
\end{aligned}$$

and as f_s is the same for all unit cells, the index s is usually omitted and simply f is used as the structure factor of a crystal.

Shape Functions of Finite Crystals

Block-Shaped Crystals

For a rectangular block with dimensions $\{L_i\}$ we can write the Fourier transformed of the shape function in one dimension x

$$\tilde{\Omega}(q) = \int_{-\infty}^{\infty} dx \Theta(x) \Theta(L-x) e^{iqx}$$

where $\Theta(x)$ is the so-called Heaviside step function, $\Theta(x) = 1$ for $x \geq 0$ and $\Theta(x) = 0$ for $x < 0$. This can be evaluated to

$$\begin{aligned}
\tilde{\Omega}(q) &= \int_0^L dx e^{iqx} = \\
&= \frac{1}{iq} e^{iqx} \Big|_0^L = \\
&= \frac{1}{iq} [e^{iqL} - 1] \tag{4.4}
\end{aligned}$$

The scattered intensity $I(\mathbf{q})$ is proportional to $|\tilde{\Omega}(\mathbf{q})|^2 = \tilde{\Omega}^*(\mathbf{q})\tilde{\Omega}(\mathbf{q})$:

$$\begin{aligned}
\left| \tilde{\Omega}(q) \right|^2 &= \frac{1}{q^2} [e^{iqL} - 1][e^{-iqL} - 1] = \\
&= \frac{1}{q^2} (2 - e^{iqL} - e^{-iqL}) = \\
&= \frac{1}{q^2} (2 - 2 \cos qL) = \\
&= \frac{2}{q^2} 2 \sin^2 \frac{qL}{2} = \\
&= \frac{\sin^2 \frac{qL}{2}}{\left(\frac{q}{2}\right)^2}
\end{aligned} \tag{4.5}$$

where we have used $e^{ix} = \cos x + i \sin x$ and $1 - \cos x = 2 \sin^2 \frac{x}{2}$. Performing the same calculation for all three dimensions this finally yields

$$\left| \tilde{\Omega}(\mathbf{q}) \right|^2 = \prod_{i=1}^3 \frac{\sin^2 \left(\frac{q_i L_i}{2} \right)}{\left(\frac{q_i}{2} \right)^2}. \tag{4.6}$$

Superlattice

A superlattice can be seen as a one-dimensional series of block-shaped crystals, i.e., the shape function $\Omega(x)$ is the sum of individual shape functions:

$$\Omega(x) = \sum_{j=1}^N \Omega_{\text{block}}(x - jd)$$

with the individual shape function

$$\Omega_{\text{block}}(x) = \Theta(x)\Theta(b - x),$$

where d is the superlattice period, and $b < d$ is the width of one "block". We can calculate the Fourier transform of $\Omega(x)$:

$$\begin{aligned}
\tilde{\Omega}(k) &= \int_{-\infty}^{\infty} dx \sum_{j=0}^N \Omega_{\text{block}}(x - jd) e^{ikx} \\
&= \sum_{j=1}^N \int_{jd}^{jd+b} e^{ikx} \\
&= \sum_{j=1}^N \frac{1}{ik} e^{ikx} \Big|_{jd}^{jd+b} \\
&= \frac{1}{ik} \sum_{j=1}^N \left[e^{ikjd} e^{ikb} - e^{ikjd} \right] \\
&= \frac{1}{ik} \left(e^{ikb} - 1 \right) \sum_{j=0}^N e^{ikjd} \\
&= \frac{1}{ik} \left(e^{ikb} - 1 \right) \frac{\exp(ikNd)}{e^{ikd} - 1} \tag{4.7}
\end{aligned}$$

Using again $e^{ix} = \cos x + i \sin x$ and $1 - \cos x = 2 \sin^2 \frac{x}{2}$, the square of the absolute value evaluates to

$$|\tilde{\Omega}(k)|^2 = \frac{4}{k^2} \sin^2 \frac{kb}{2} \frac{\sin^2 \frac{Nkd}{2}}{\sin^2 \frac{kd}{2}} \tag{4.8}$$

Some Useful Numbers

For the calculation of Bragg angles and Ge or C contents, the following parameters are required:

Lattice parameters ($\text{\AA}$)		
silicon	a_{Si}	5.43105
germanium	a_{Ge}	5.65785
carbon (diamond)	a_{C}	3.5668
Wavelength ($\text{\AA}$)		
Cu $\text{K}_{\alpha 1}$	λ	1.5406
Elastic Constants (10^9 Pa)	C_{11}	C_{12}
Si	1.658	0.639
Ge	1.285	0.483
C	1.076	0.125

In order to obtain properties of compounds $\text{Si}_{1-x}\text{Ge}_x$ or $\text{Si}_{1-y}\text{C}_y$, You can just linearly interpolate these quantities (although this is a simplification and not really correct).

You should obtain the following Bragg angles (for asymmetrical reflections, the grazing exit or grazing incidence geometry can be chosen, consequently two possible incidence angles ω^+ and ω^- are given):

reflection	ω^+ ($^\circ$)	ω^- ($^\circ$)	2θ ($^\circ$)
Silicon			
(004)	34.564	34.564	69.129
(224)	79.278	8.750	88.028
(113)	53.300	2.821	56.121
(115)	63.268	31.682	94.950
(404)	98.353	8.352	106.706
(206)	82.205	45.335	127.540
Germanium			
(004)	32.997	32.997	65.993
(224)	77.099	6.570	83.669
(113)	52.083	1.604	53.686
(115)	60.820	29.234	90.054
(404)	95.369	5.369	100.737
(206)	77.872	41.002	118.874
Carbon			
(004)	59.752	59.752	119.505
(113)	70.987	20.508	91.495

Lattice Parameters of $\text{Si}_{1-x}\text{Ge}_x$ and $\text{Si}_{1-y}\text{C}_y$

Figures 4.2 and 4.3 display the variation of the bulk lattice parameter a_{bulk} of $\text{Si}_{1-x}\text{Ge}_x$ and $\text{Si}_{1-y}\text{C}_y$ as a function of the compositions x and y , respectively. They also display the lattice parameter $a_{\perp}$ in growth direction (perpendicular to the surface, in our case

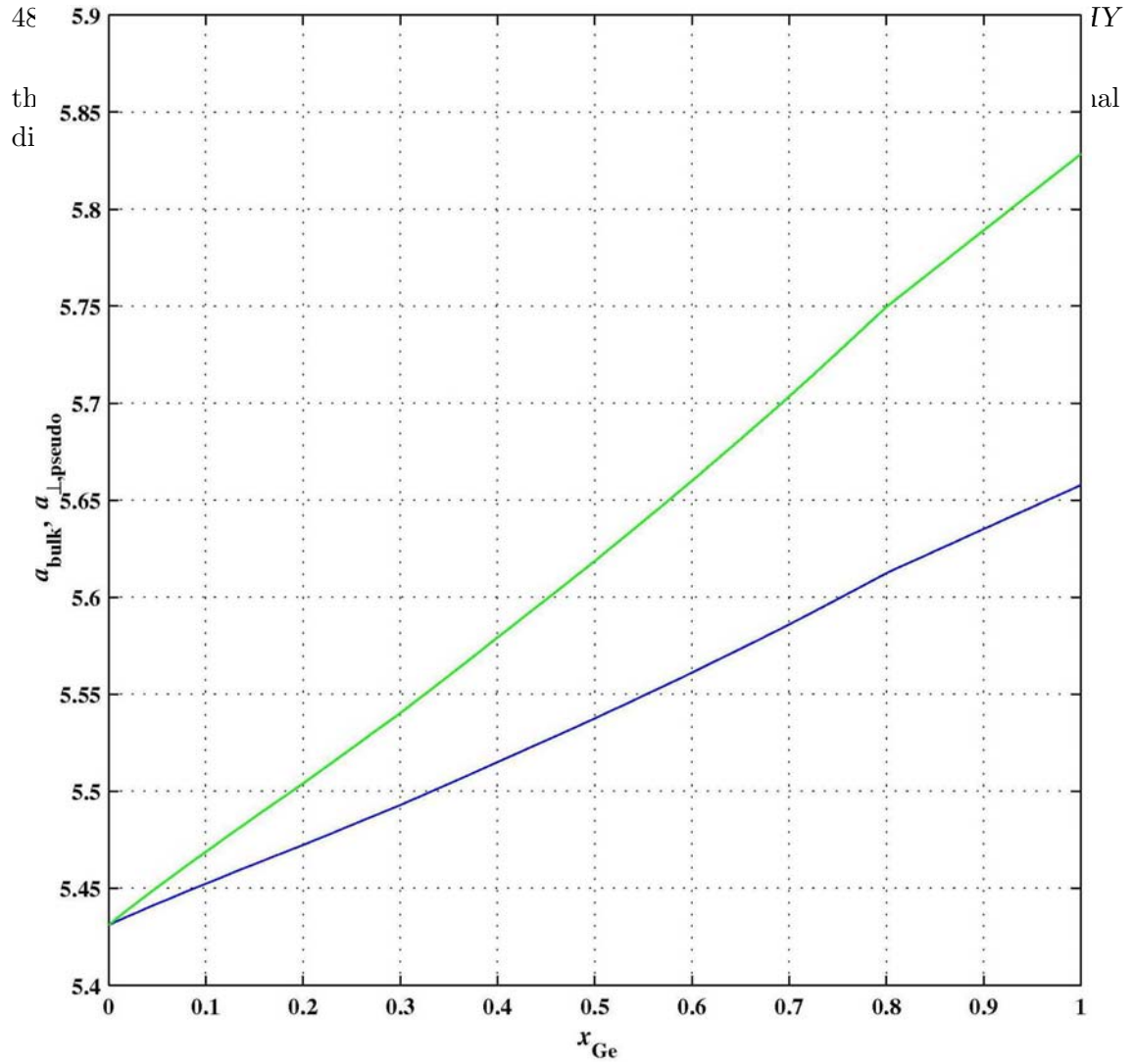


Figure 4.2: Lattice parameters $\text{Si}_{1-x}\text{Ge}_x$ as a function of x : Bulk lattice parameter a_{bulk} (blue) and perpendicular parameter $a_{\perp}$ along growth direction for fully pseudomorphic growth (green).

Data Extraction

The data are saved by the software in a binary format. You can convert into ASCII format using the PHILIPS MRD software: in the Main Menu, select Utilities, then Convert files and MRD (Dnn,Ann) to ASCII (Xnn,Ynn). Select the files to convert from the list. Copy the resulting ASCII files (with extensions Xnn) to a floppy disk.

For the ASCII data format an input routine for MATLAB is provided, XGETMRD.M. Using this script, the data can be imported into MATLAB for handling, conversion in reciprocal space coordinates etc.

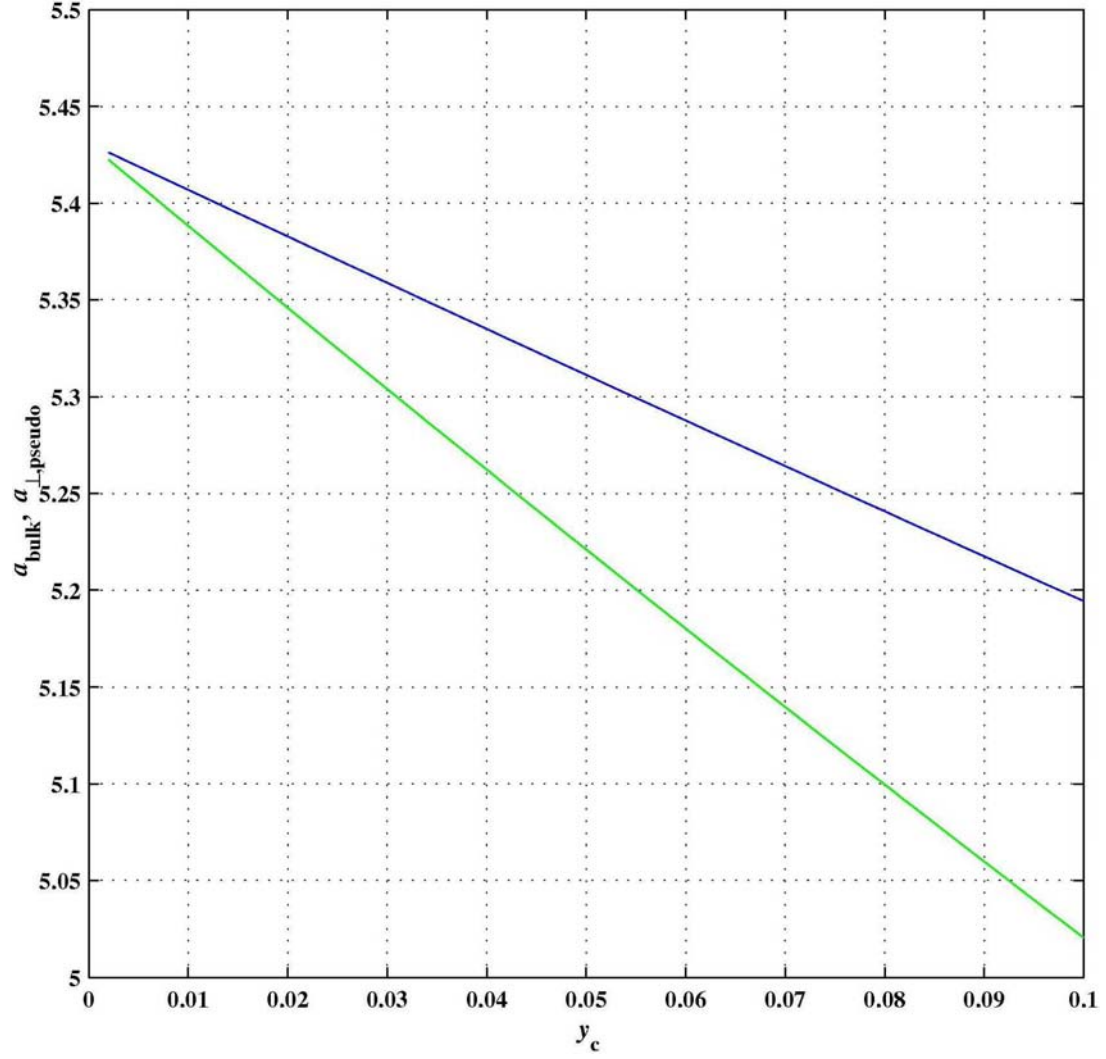


Figure 4.3: Lattice parameters $\text{Si}_{1-y}\text{C}_y$ as a function of y : Bulk lattice parameter a_{bulk} (blue) and perpendicular parameter $a_{\perp}$ along growth direction for fully pseudomorphic growth (green).

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