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Wedge Polishing as Sample Preparation Method for Transmission Electron Microscopy: A Systematic Study

MASTER THESIS

For obtaining the academic degree
Diplom-Ingenieurin

Master Programme of
Technical Physics

Graz University of Technology

Supervisor:

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Institute for Electron Microscopy and Fine Structure Research (FELMI)

Graz, November 2011

Deutsche Fassung:
Beschluss der Curricula-Kommission für Bachelor-, Master- und Diplomstudien vom 10.11.2008
Genehmigung des Senates am 1.12.2008

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Abstract

Wedge Polishing is a mechanical sample preparation method for Transmission Electron Microscopy samples. The final shape of the sample is a small wedge with an electron transparent edge and a thicker side which provides good handling properties. In this thesis, wedge polishing was performed semiautomatically with the *Multiprep*^{TM} *System* from *Allied High Tech Products, Inc.*

The preparation method is performed in two steps: At first side polishing, the initial sample in shape of a small rectangular prism gets thinned plan and parallel to the abrasive platen. A smooth surface is obtained by successively applying finer abrasives until the final polishing act is performed chemical-mechanically with colloidal silicon dispersed in an alkaline liquid. After the last polishing step, the sample gets detached from the pyrex of the sample holder and reattached upside down, so that the other surface gets in contact with the abrasive platen. At second side polishing, the wedge angle of about 1° to 5° gets introduced by tilting the sample. Again the succession of the applied abrasives is from coarse to finer abrasives until the colloidal silicon gets applied.

Amorphous layers lower the TEM image contrast and are therefore highly unwanted. With wedge polishing, amorphisation of the surface occurs due to mechanical stress. The amorphised layers can be removed to a great extend by polishing them away with the abrasive of the next grain size in succession and with the last chemical-mechanical polishing step.

In this thesis, suitable wedge polishing procedures for samples of silicon, sapphire and tungsten carbide (in cobalt) were found out, and preparation induced artifacts investigated. The quality of the samples was checked in the TEM by images, relative thickness maps and high resolution investigations.

Silicon is highly suitable for wedge polishing. Preparation procedures which lead to high quality high resolution TEM images were developed, whereby only a negligibly thin amorphisation layer was induced.

Sapphire is difficult to polish because of its hardness and brittleness. Electron transparent areas of sufficient size could be obtained with a special polishing procedure which also kept the crystallinity of the sample.

Samples which consist of tungsten carbide grains in a cobalt binder could be thinned to electron transparency, but crystal defects were induced during sample preparation.

In conclusion, semiautomatic wedge polishing is a powerful preparation method which can provide high quality TEM samples of some materials if optimised preparation procedures are applied.

Kurzfassung

Das sogenannte *Wedge Polishing* ist eine mechanische Probenpräparationsmethode in der Transmissionselektronenmikroskopie. Die fertig präparierte Probe ist keilförmig, mit einer elektronentransparenten Kante und einer dickeren Seite, die für eine gute Handhabung sorgt. In dieser Masterarbeit wurde das Wedge Polishing halbautomatisch mit dem *Multiprep*^{TM} *System* von *Allied High Tech Products, Inc* durchgeführt. Diese Präparationsmethode wird in zwei Schritten ausgeführt: Beim Polieren der ersten Seite dünnt man eine Probe, die anfänglich die Form eines Quaders aufweist, plan und parallel in Bezug auf die Schleifscheibe. Eine glatte Oberfläche wird erzielt, indem man sukzessiv immer feinere Körnungen anwendet. Zum Schluss erfolgt das Polieren chemisch-mechanisch mit kolloidalem Silizium, das in einer alkalischen Flüssigkeit dispergiert ist. Danach wird die Probe vom Pyrex der Probenhalterung abgelöst und umgedreht, so dass die gegenüberliegende Oberfläche mit der abrasiven Scheibe in Berührung kommt. Beim Polieren der zweiten Seite wird der Keilwinkel von ungefähr 1° bis 5° durch Kippen der Probe eingestellt. Die Abfolge der Abrasive erfolgt wieder von groben zu feineren Körnungen, bis zum Schluss das kolloidale Silizium verwendet wird. Amorphe Schichten verringern den TEM-Bildkontrast und sind darum höchst unerwünscht. Beim Wedge Polishing tritt auf Grund von mechanischen Spannungen eine Amorphisierung der Oberfläche auf. Diese amorphen Schichten können zum Großteil entfernt werden, indem man sie mit der nächst feineren Körnung wegschleift und als letzten chemisch-mechanischen Polierschritt das kolloidale Silizium anwendet. In dieser Masterarbeit wurden geeignete Wedge Polishing-Verfahren für Proben, deren Hauptbestandteil Silizium, Saphir und Wolframcarbid in Cobalt ist, ermittelt und präparationsinduzierte Artefakte untersucht. Die Qualität der Proben wurde im TEM durch Bilder, relative Dicken und hochauflösende Untersuchungen überprüft. Wedge Polishing eignet sich sehr gut als Präparationsmethode für Silizium. Es wurden Präparationsverfahren entwickelt, die zu hochauflösenden TEM-Bildern von hoher Qualität führten, wobei nur eine vernachlässigbar dünne amorphisierte Schicht induziert wurde. Saphir ist ein Material, das sehr schwierig zu polieren ist, da es sehr hart und spröde ist. Ausreichend große elektronentransparente Bereiche konnten mit einem speziell angepassten Wedge Polishing-Verfahren erreicht werden, bei dem auch die Kristallinität der Probe erhalten wurde. Proben, die aus Wolframcarbid-Körnern in Cobalt bestehen, konnten bis zur Elektronentransparenz gedünnt werden. Es wurden jedoch Kristalldefekte durch das Wedge Polishing eingebracht. Abschließend kann man sagen, dass halbautomatisches Wedge Polishing eine leistungsfähige Präparationsmethode ist, mit der man aus einigen Materialien, bei Anwendung optimierter Verfahren, hoch qualitative TEM-Proben herstellen kann.

Acknowledgement

Upon the completion of my master thesis and my studies I would like to express my sincere gratitude to the persons who made this possible.

First of all I want to thank a lot my supervisors Werner Grogger and Evelin Fisslthaler for guiding me through my thesis. Thanks for all the time you spend on professional and personal support which was indispensable for my work.
A great “thank you” is also dedicated to Martina for the introduction into TEM sample preparation and the whole laboratory team for their help.
Moreover I want to thank Arno who shared his knowledge about wedge polishing of sapphire with me, and showed me many important preparation tricks.
I also would like to extend my thanks to Franz and Nadejda for the interesting discussions about so many topics.
The whole FELMI team provided an inspiring environment in which I felt very comfortable due to the extraordinary helpfulness and friendliness I experienced at this institute. To mention just a few of them, I want to thank Christian, Gerald, Thomas, Wernfried, Steffi, Sebastian, Claudia, Harald, Thomas G. and Roland.

Special thanks also to Jörg for accompanying me through the end of my studies and always encouraging me.

Most importantly, my eternal gratitude goes to my family, especially to my parents for providing me the possibility to study. I also want to thank them, as well as my sisters Susanne and Bernadette and my brother Benjamin, for their everlasting support and encouragement in every situation of my life.

Last, but not least, I want to thank all my fellow physics students who made my years of study so unforgettable awesome.

Contents

1 Motivation

Within the last decades, the rising research field of nanotechnology and -materials has gained more and more importance, not only in science, but also in modern industry. Medical and electronic devices, coatings, sensors and energy generation are only some examples for the wide ranged field of application, and so this area of expertise has also become economically relevant. However, a crucial point in successful nanoengineering is the ability to investigate small features. This is why Transmission Electron Microscopy (TEM) is an indispensable tool for the analysis of nanoscale structures.

The capability of obtaining satisfying results from TEM is directly associated with the quality of sample preparation. The more sophisticated the microscope, the higher the requirements to the sample. Due to the methodology of TEM, the sample has to be very thin ($<$50 nm) so that the electrons can transmit. Especially for high resolution TEM images of high quality, the sample thickness should not exceed the mean free path in this material, to avoid multiple scattering of the beam electrons. The original structure of the sample must not be modified during the preparation process. Particularly surface amorphisation is highly unwanted because it lowers the image contrast.

The *Institute for Electron Microscopy and Fine Structure Research (FELMI)* is not only constantly refining proven sample preparation methods, but also researching novel ones and testing them. Therefore the intention of this thesis is to introduce a new preparation technique to the laboratory at FELMI and to systematically establish wedge polishing procedures for different materials.

Wedge polishing is a mechanical sample preparation method that can provide TEM samples within a few hours. The final shape of the sample is a small wedge which includes both, an electron transparent edge and a thicker side with good handling properties. This technique can be applied to a great variety of materials, such as metals, semiconductors, ceramics, minerals, polymers and composite materials. They can be bulk, organised in mono- or multilayers or consist of fine particles which have to be embedded. The samples can be of medium to high hardness. One of the greatest advantages of wedge polishing compared with other preparation methods is, that the final sample features only a negligibly thin preparation induced amorphised layer. In this thesis suitable semiautomatic wedge polishing procedures with the *Multiprep*^{TM} *System* from *Allied High Tech Products, Inc* were ascertained for samples like silicon, sapphire and tungsten carbide. The assignment also comprised alignment of the instrument and optimising system parameters of the polishing processes in dependence of the material. The quality of all samples was checked in the TEM by acquiring images and relative thickness maps. In some cases also high resolution investigations were performed.

2 Fundamental and Theoretical Issues

2.1 The Principle of Transmission Electron Microscopy

Transmission Electron Microscopy (TEM) enables scientific investigation of materials on the atomic scale [1]. A beam of accelerated electrons transmits a sample of low thickness from tens to a few hundreds of nanometers. The interactions between the electrons and the material give information about the structure and chemical composition of the transmitted area. Therefore, the resulting picture is a two dimensional projection of the three dimensional sample, averaged through its thickness.
To keep the electron beam coherent and prevent it from scattering outside of the sample, transmission electron microscopes (see figure 2.1) operate in vacuum.

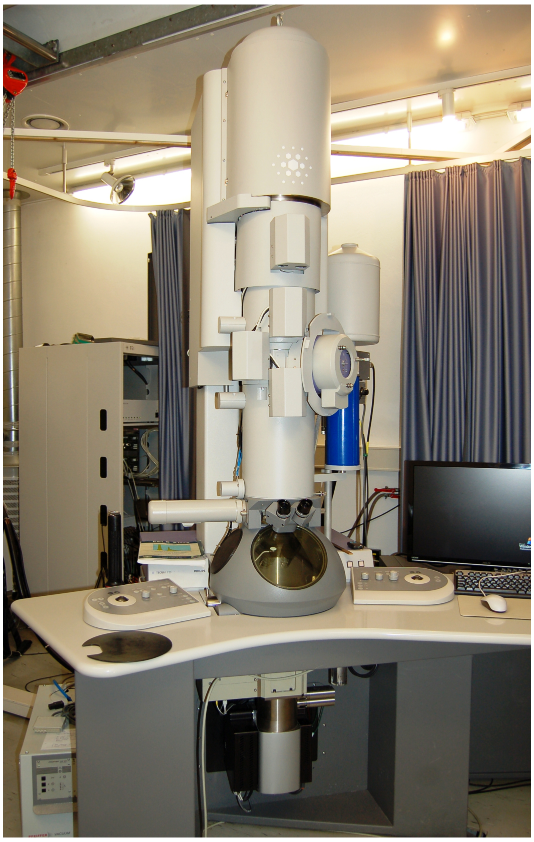


Figure 2.1: Transmission Electron Microscope FEI Tecnai G2 F20

Figure 2.2 shows which interactions take place when an electron beam transmits matter. In Transmission Electron Microscopy following signals are frequently detected and analysed:

- direct beam
- elastically scattered electrons: no loss of energy
- inelastically scattered electrons: loss of energy
- x-rays

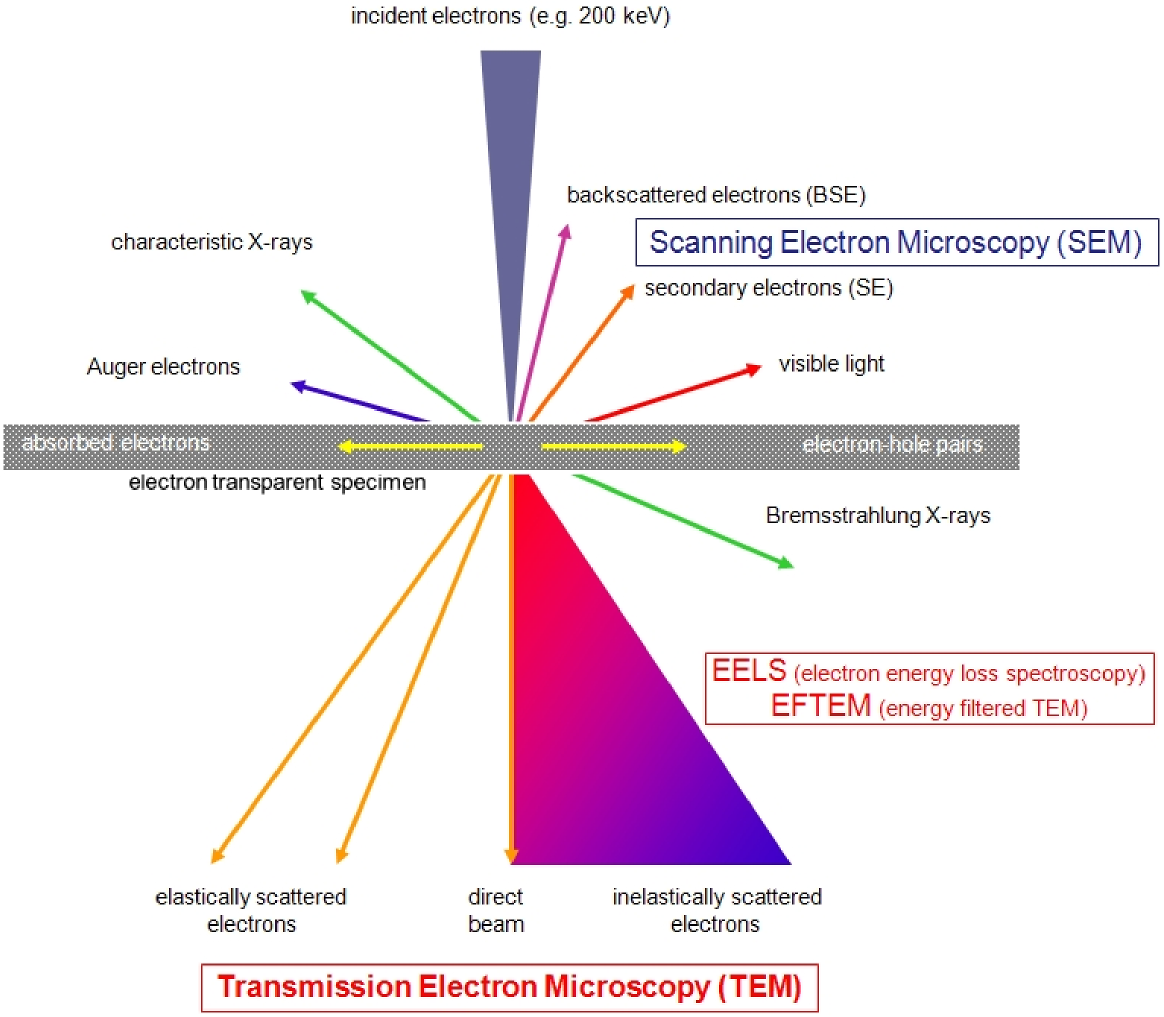


Figure 2.2: Interactions of electrons with matter and the respective electron microscope technique (modified from [2])

TEM measurements are based on the analysis of scattering events inside the sample. The probability that a particular electron interacts with an atom of a certain material is defined by the interaction cross section σ_{atom} ($[\sigma_{atom}]$=m^2). It is a function of the effective radius r ([r]=m) of a single, isolated atom - the scattering center (see equation 2.1).

$$\sigma_{atom} = \pi\, r^2 \tag{2.1}$$

If the sample consists of N atoms per unit volume ([N]=$\frac{1}{m^3}$), the total cross section σ_{total} ($[\sigma_{total}]$=$\frac{1}{m}$) can be expressed as equation 2.2. The subsequent transformation, where N_0 is Avogadro's number ([N$_0$]=$\frac{1}{mol}$), A the atomic weight ([A]=$\frac{kg}{mol}$) and ρ the density ($[\rho$=$\frac{kg}{m^3}]$), shows that σ_{total} is the number of scattering events per unit distance that the electron experiences when passing through the sample.

$$\sigma_{total} = N\, \sigma_{atom} = \frac{N_0\, \sigma_{atom}\, \rho}{A} \tag{2.2}$$

The probability of scattering in a sample of thickness t therefore is (equation 2.3):

$$\sigma_{total}\, t = \frac{N_0\, \sigma_{atom}\, \rho\, t}{A} \tag{2.3}$$

The mean free path of an electron λ ([λ]=m) is the inverse total interaction cross section (see equation 2.4). It defines the average distance between two scattering events.

$$\lambda = \frac{1}{\sigma_{total}} = \frac{A}{N_0\, \sigma_{atom}\, \rho} \tag{2.4}$$

Typical values for λ in TEM are in the order of tens of nanometers. If the sample thickness is about the same size, plural scattering will not influence the measured signals and the TEM images are more easily interpretable.

If a TEM is equipped with an energy filter, zero loss filtered bright field images can be acquired and relative thickness maps calculated from them. Bright field images are formed by the direct beam that has transmitted the sample. Unfiltered images depict the whole electron energy loss spectrum (EELS), while in zero loss images only electrons with negligible energy loss can pass the filter (see figure 2.4).

A sample's TEM thickness map gives an estimate for the sample thickness. These maps show the thickness in units of the mean free path ($\frac{t}{\lambda}$), which corresponds to equation 2.3, in dependence of the spatial coordinates (see figure 2.3(c)). If the mean free path λ is known, the absolute thickness can be concluded.

The relative thickness map is obtained by the ansatz:

$$I(t) = I_0 \exp^{-\sigma_{total} \cdot t} \tag{2.5}$$

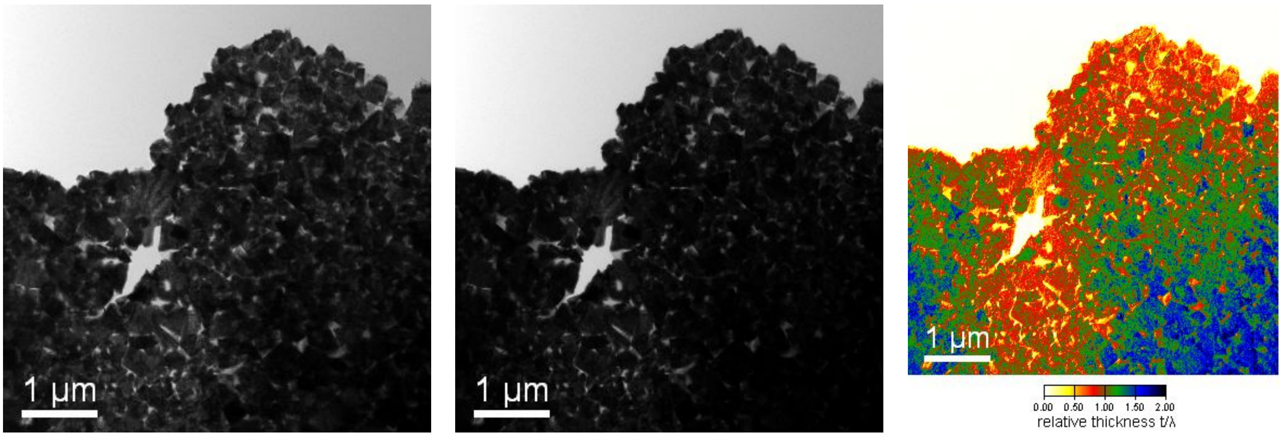
Poisson statistics can be applied because the collisions are independent events [3]. *I(t)* is the total intensity in the low loss EEL spectrum in dependence of the sample thickness. Intensities at higher energy losses are negligible small [1]. I(t) corresponds to the intensity of the unfiltered bright field

image I$_{unfiltered}$. I_0 is the intensity without losses and therefore the intensity of the zero loss filtered image I$_{zero-loss\,filtered}$.
This leads to equation 2.6 which shows that the relative thickness map can be calculated from the intensities of an unfiltered bright field ($I_{unfiltered}$) (see figure 2.3(a)) and an zero loss filtered bright field image ($I_{zero-loss\,filtered}$) (see figure 2.3(b)) at the same position of the sample [2].

$$\frac{t}{\lambda} = \ln \frac{I_{unfiltered}}{I_{zero-loss\,filtered}} \tag{2.6}$$

Resumed, according to the methodology of Transmission Electron Microscopy, samples have to satisfy the following conditions:

- *Vacuum stability:* Transmission Electron Microscopy is performed in vacuum and therefore the sample has to stand it.
- *Cleanliness:* Contamination of the sample causes artifacts. Amorphous carbon layers can grow onto the material and lower the contrast of the TEM images (see section 2.4).
- *Thinness:* The sample must be at least electron transparent. As the resulting picture is an thickness-averaged projection, the material should be thin to obtain significant results (desirable thickness <50 nm).
- *No modifications:* The sample should not be modified in structure or chemical composition. Often it cannot be determined if a special feature is property of the material, or if it is an artifact that has been induced e.g. during preparation. A prevalent artifact caused by preparation is amorphisation. Amorphised layers also contribute to the measured signal and cause a decrease in image contrast (see section 2.4).



(a) unfiltered (b) zero loss filtered (c) resulting thickness map

Figure 2.3: Thickness measurement via different TEM images of a tungsten carbide sample

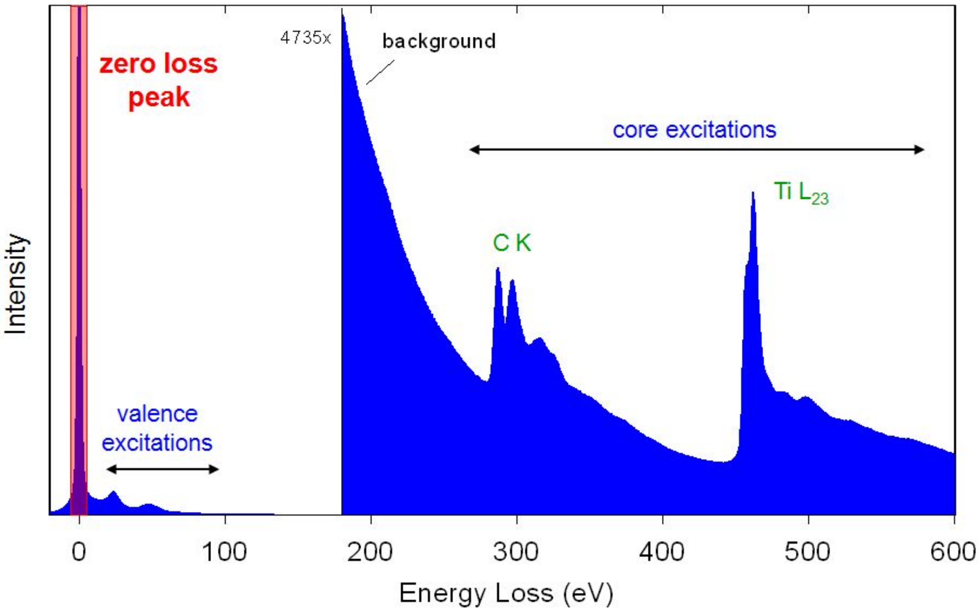


Figure 2.4: Electron Energy Loss Spectrum of a 20 nm thin titanium carbide sample (modified from [2]). Unfiltered images are formed by signals of electrons of all energies while zero loss filtered images are built up only by the intensity of the zero loss peak that is indicated in red.

2.2 Wedge Polishing

2.2.1 General Information

The purpose of wedge polishing is to form a wedge-shaped sample by mechanical abrasion. The advantage of this shape is that it combines electron transparency at the edge with good handling properties at the thicker part (see figure 2.5).

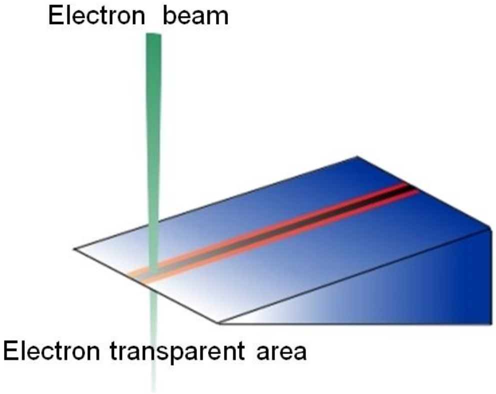


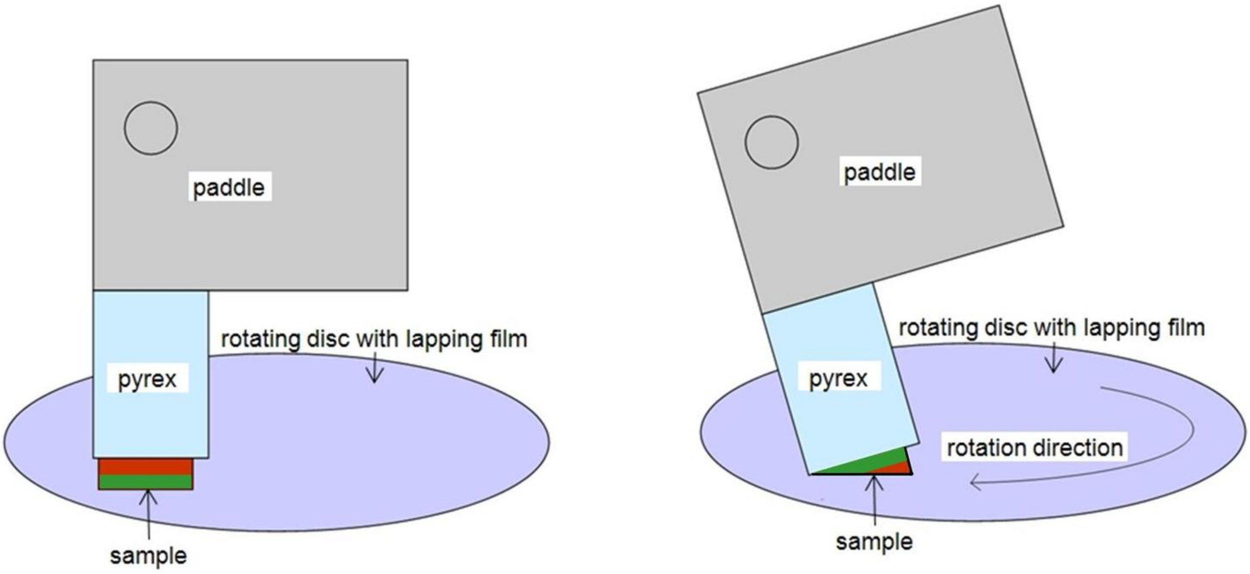
Figure 2.5: Schematic drawing of a wedge polished sample sandwich

At first the sample has to get into a proper shape for polishing. The adequate form is a rectangular prism with a base area that does not extend the geometrical dimensions of the disc-shaped TEM-grid (see figure 3.11). Otherwise it may not fit into the sample holder of the TEM. The right shape is achieved by preceding work, such as cutting, sawing, breaking, embedding and gluing. Sometimes sandwiches, consisting of stripes that are glued together face to face, have to be prepared to prevent the layer of interest from damage [4].
Before starting the polishing procedure, the prism gets mounted onto a pyrex stub of either the manual wedge polishing tool (see subsection 2.2.2) or the thinning paddle of the semiautomatic system (see subsection 2.2.3). It is important to use adhesives that can be dissolved in acetone, as it should be possible to unmount the sample without much effort.

The abrasion process is realised by a rotating platen, equipped with so called *lapping films*. They are polymer films which contain resin bonded particles of diamond, aluminum oxide, silicon carbide or silicon oxide. The grain size refers to the size of these particles. In subsection 3.3 optical microscope photos of lapping films of different grain sizes and the corresponding polished surface are depicted. The preparation proceeds by successively applying different lapping films, starting with the coarsest, ending with the finest one. Water or special lubricants serve as cooling and lubrication during the polishing process. The final polishing step is done with a felt cloth soaked with colloidal silica. This liquid is a suspensions of abrasive nanosized particles, dispersed in a liquid that exhibit an alkaline pH value.

Therefore final polishing is not only a mechanical procedure, but also features a chemical component.

The wedge polishing technique is performed in two steps: first side and second side polishing (see figure 2.6(a) and figure 2.6(b)).
At first, one side of the sample is polished parallel with respect to the platen and the pyrex. As described before, a perfectly smooth surface is obtained by decreasing the grain size of the abrasive film successively. A sufficient thin wedge edge of good TEM investigation properties can only be obtained if both, first and second side, are of perfect smoothness. After the last polishing step, the sample is unmounted from the pyrex. It is turned around and fixed again with the polished surface glued onto the pyrex.
At second side polishing, initially the sample is thinned parallel with a coarse lapping film until the sample is thin enough to proceed with finer abrasives. As abrasion of a large amount of material with small grain sizes is very time-consuming, this practice accelerates the preparation progress. In the following step the wedge angle gets introduced. For protecting the edge of the sample, it is important that polishing is always performed from behind so that the abrasive grains hit the thicker side of the wedge first (see figure 2.6(b)). The succession of the abrasives is the same as for first side polishing.



(a) First side polishing: plane polishing

(b) Second side polishing: wedge-shaped sample caused by tilting the paddle with the pyrex

Figure 2.6: Schematic drawing of first and second side polishing: The sample is fixed onto the pyrex stub of the paddle which can be tilted for second side polishing (proportions do not correspond to reality).

It is absolutely essential that the surface of the pyrex is even and parallel to the grinding platen. Otherwise all geometrical adjustments and requirements cannot be fulfilled properly. If the pyrex does not satisfy these conditions it must be polished plane before use.

The advantage of wedge polishing is that it can be applied to a variety of materials in any section and of any orientation [4]. It is often used for hard, brittle and multiphase samples. This technique enables high quality surfaces at a preparation time of several hours [5]. Depending on the wedge angle, the observable area can be very large. When performed without ion milling as subsequent treatment, the preparation induced damage layer is very thin (see section 2.4).
There are also some disadvantages of this preparation method, like the differing abrasion rate in multiphase samples which consist of materials of different hardness. Depending on the material, the wedges also can be very sensitive to handling after polishing, like mounting on a TEM-grid and transport. The wedge edges of very brittle materials can easily tear off before electron transparency is reached. In this case ion thinning as a subsequent treatment has to be performed (see subsection 2.2.4).

2.2.2 Manual Wedge Polishing

Conventional manual polishing is performed with a special tool, that was developed at IBM in 1988 [6]. It is called *tripod polisher* (see figure 2.7) and typically consists of following parts [4, 5]:

- a circular main body
- three feet which can be adjusted in length by micrometer screws
- three locking screws for the micrometer screws
- a L-shaped bracket equipped either with a glass or pyrex cylinder or rectangular prism which serve as sample holder
- a spirit level

During the abrasion process, the tripod is situated on the abrasion disc and held by hand. The index finger is put onto the L-bracket to feel the specific behavior of the sample and to apply the optimal pressure. If performed with regular sample movements perpendicular to the platen rotation direction, the resulting spiral abrasion track avoids damage due to polishing residues. The debris has to be removed from the lapping film with lint-free paper and water before using the same area again.

Before starting with first side polishing, the micrometer screws have to be adjusted so that the feet of the tripod and the sample are in the same plane which is parallel with respect to the rotating abrasive platen. This can be achieved by use of a leveling table and a spirit level. The depth of abrasion is limited by retracting the 3 micrometer screws. When the adjusted amount of material is removed,

Figure 2.7: Upside down view of a Tripod Polisher® from South Bay Technology with Si-Sample glued on pyrex stub

the feet and the sample are in one plane again. For the next polishing step the desired retraction has to be set up again.

To introduce the wedge angle at second side polishing, the two rear feet have to be lowered via the micrometer screws. Depending on the desired wedge angle, the recommended change of these screws from the level position is between 300 μm and 500 μm. This gives a wedge angle between 0.3° and 0.7° [4, 5]. Again the retraction of the feet determines abrasion depth.

It is obvious that for achieving high quality samples by conventional tripod polishing, the operator needs great skill and a lot of experience.

The Tripod Polisher® is a registered trademark by South Bay Technology. There exist similar tools which are nearly equal in construction and handling (e.g. T-Tool from T&T Group or TEM Wedge Polisher from Allied).

2.2.3 Semiautomatic Wedge Polishing

Semiautomatic wedge polishing is a further development of conventional manual tripod polishing. As the *Multiprep*^{TM} *System* from *Allied High Tech Products, Inc* (see figure 2.8) was used in this thesis, this subsection gives a closer description of it.

The high precision polishing tool consists of [7]:

- corpus
- 8″ aluminum rotation platen
- control panel
- vertical adjustment knob
- suspension arm
- spindle with spindle riser
- cam lock adapter
- water supply and drain
- TEM wedge thinning pyrex paddle and cross-sectioning paddle
- front and rear digital indicator
- two micrometer screws
- load adjustment screw

As figure 2.9 shows, the free-floating spindle is supported by the suspension arm which can be adjusted in height via the vertical adjustment knob. The rear digital indicator shows the position of the suspension arm.

At the bottom of the spindle, the cam lock adapter can be attached. The paddle (see figure 2.10), which serves as sample holder, is fixed at this adapter. With the spindle riser the spindle, and therefore also the sample, can be lifted and lowered without changing the vertical position of the suspension arm. The digital indicators, with a resolution of one micrometer, can be zeroed and used as reference points. Again first side and second side polishing differ only in the wedge angle that has to be introduced at the second side. These angular adjustments can be done via the micrometer screws situated at the lower part of the spindle. The left screw is for front-to-back adaptations (pitch), the right one is for left-right adjustments (roll). The pitch is for introducing the wedge angle. The roll can be important for angular corrections, for example when the sample is not mounted completely plane (see subsection 3.1.1).

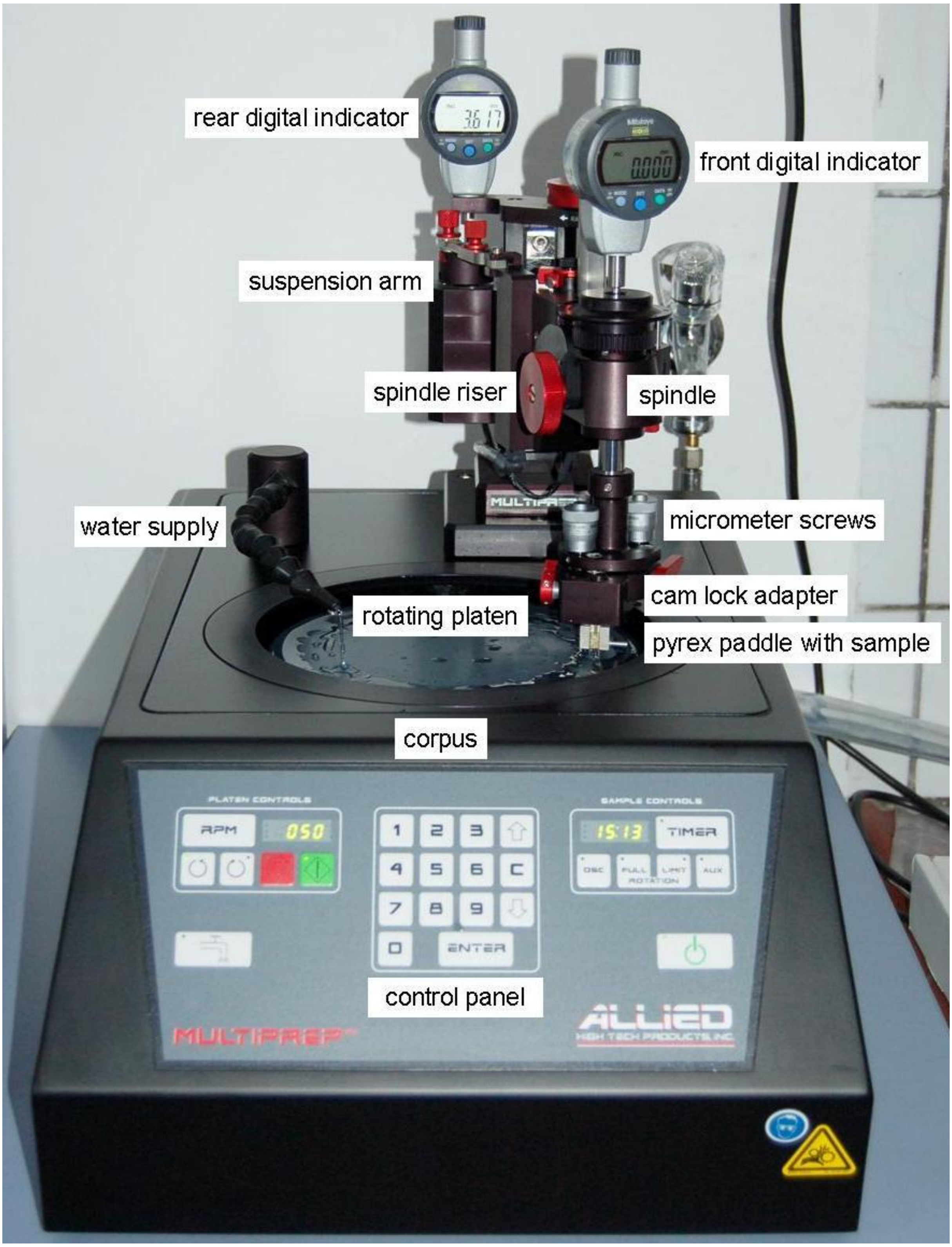


Figure 2.8: The Multiprep^{TM} System from Allied High Tech Products, Inc with labeled components in front view

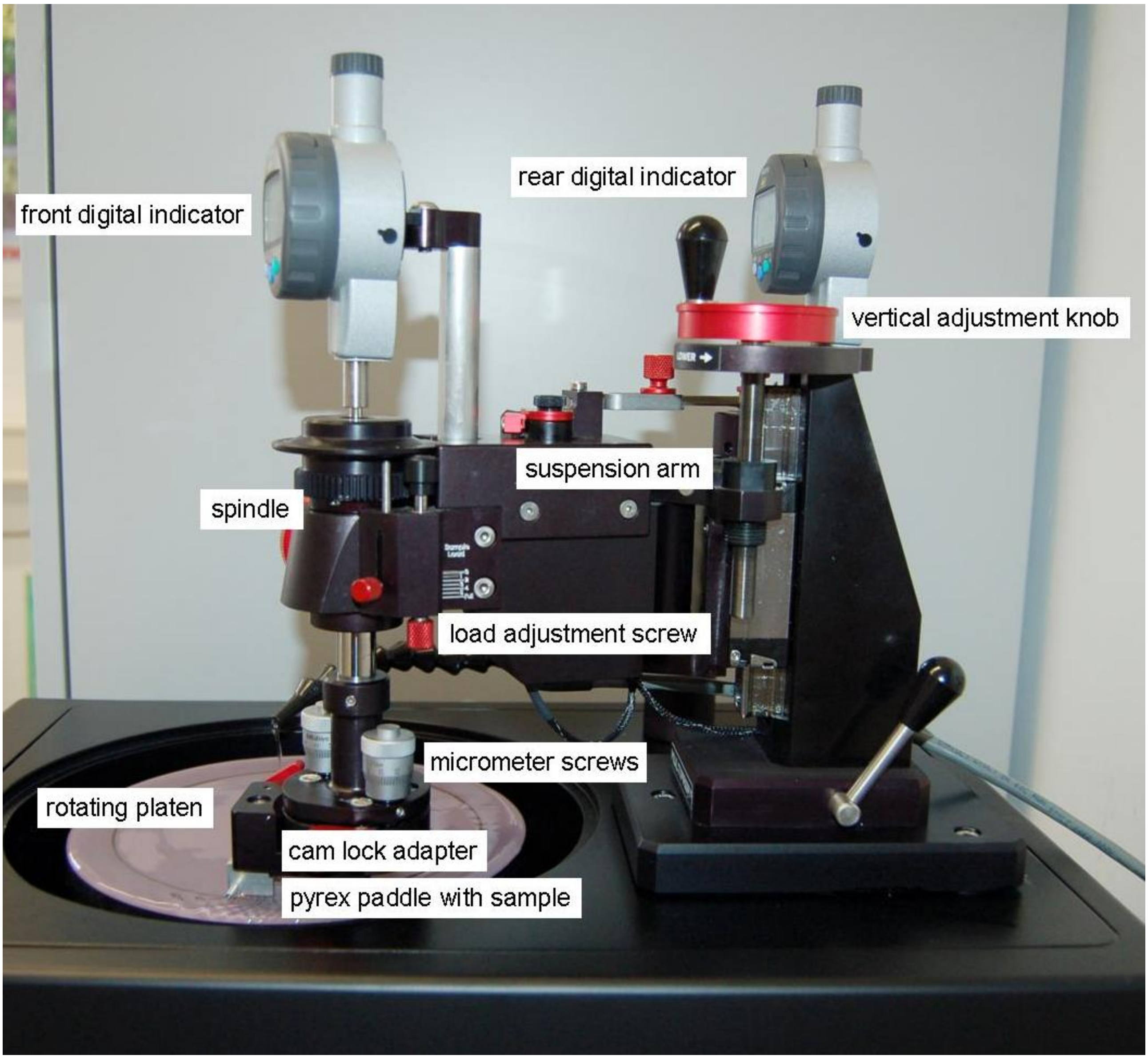
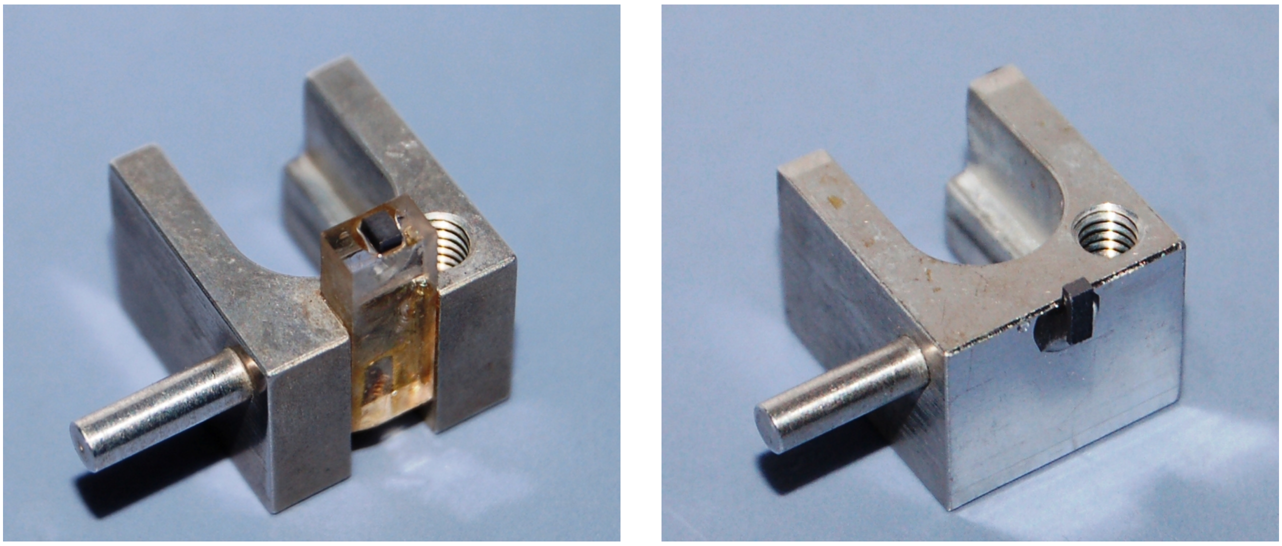


Figure 2.9: The Multiprep$^{\text{TM}}$ System from Allied High Tech Products, Inc with labeled components in side view

Each bar of the micrometric screws corresponds to 0.02°. Therefore, one full rotation, which equals 50 bars, creates a 1° angle on the sample. The range of angular adjustments goes from -2.5° to 10°. There are two kinds of paddles (see figure 2.10). With the pyrex paddle, first and second side polishing of thin samples can be performed. The cross-sectioning paddle is for first side polishing of thicker ones, especially when both base areas are inappropriate for using the pyrex paddle.


(a) Pyrex thinning paddle with a silicon sample fixed with mounting wax

(b) Cross-sectioning paddle with a silicon sample fixed with mounting wax

Figure 2.10: Two types of paddles for thinning the sample with the Multiprep System

The process of semiautomatic wedge polishing with the Multiprep$^{\text{TM}}$ System is described in section 3.1.

2.2.4 Subsequent Treatments

Chemical Etching

If the polished material is silicon, a nearly pure crystal can be obtained by a short acid dip right before the TEM investigation [8]. The thin amorphous layer, caused by the last polishing step, oxides in air to SiO_2. By putting the sample into a 1% HF (hydrofluoric acid) for one minute, the oxide layer is etched away. It is not recommended to repeat the procedure many times, as the surface of the wedge will become rougher and rougher. As the acid has some very dangerous properties, awareness of safe handling is absolutely necessary.

Ion Thinning

Sometimes a sufficient thinness cannot be achieved only by wedge polishing. In this case subsequent ion milling and/or polishing has to be applied for further sample thinning (see subsection 2.3.1). The problem is that ion thinning causes additional, non-removable artifacts. Samples only prepared by properly performed wedge polishing do not show amorphised layers to that extend (see section 2.4).

2.3 Other TEM Sample Preparation Methods

The following subsections give a short overview of other important preparation methods for TEM samples (based on [4]). In subsection 2.3.9 these preparation techniques are compared among themselves and wedge polishing.

2.3.1 Conventional Method

Precedent Thinning

From a bulk material thin slices are cut with a diamond saw. Then discs of 3 mm in diameter are stamped out and get mechanical polished from two sides until the thickness does not exceed 100 μm.

Dimpling

Dimpling thins the center of the rotating disc, through concave abrasion. Due to a rotating vertical wheel, in combination with a paste of abrasive grains and a lubricating cooling fluid, the center of the disc gets successively ground down to about 20 μm.

After the dimpling process the sample is still unfeasible thick for TEM investigations, and therefore it is necessary to apply a further thinning procedure, commonly ion thinning.

Ion Thinning

Material can be removed successively by impacts of ions of high enough kinetic energy. Typically argon (Ar^+) ions get accelerated by a electric field and are shot at that part of the sample that should be taken off. This process has to take place in a vacuum. The two ion thinning instruments that were applied on several samples described in the experimental part of this thesis are:

- *PIPS:* The *Precision Ion Polishing System* (PIPS) from Gatan thins the sample with Ar^+ ions which have energies of 500 eV to 6 keV. Two ionic beams sputter either concurrent or alternating in adjustable angles from 0° to 10°. The purpose of the PIPS is final ion milling of pre-thinned samples that were prepared through wedge polishing or dimpling. Figure 2.11 shows a schematic image of the ion milling process.
- *NEID:* NEID stands for the German expression *Niedrigenergetische Ionendünnung (Low Energy Ion Thinning)*. A beam of Ar^+ ions impacts the pre-thinned sample in adjustable angles from typically 5° to 10°. The energy of these ions can be very low from 0 eV to 2 keV.
  The NEID can be used for final ion polishing of dimpled or wedge polished samples and also for cleaning them. Often the amorphous layer, that was induced by former preparation steps like wedge polishing or ion milling, are removed in the NEID. Due to the low energies, the abrasion of the material takes place more gently but also more slowly.

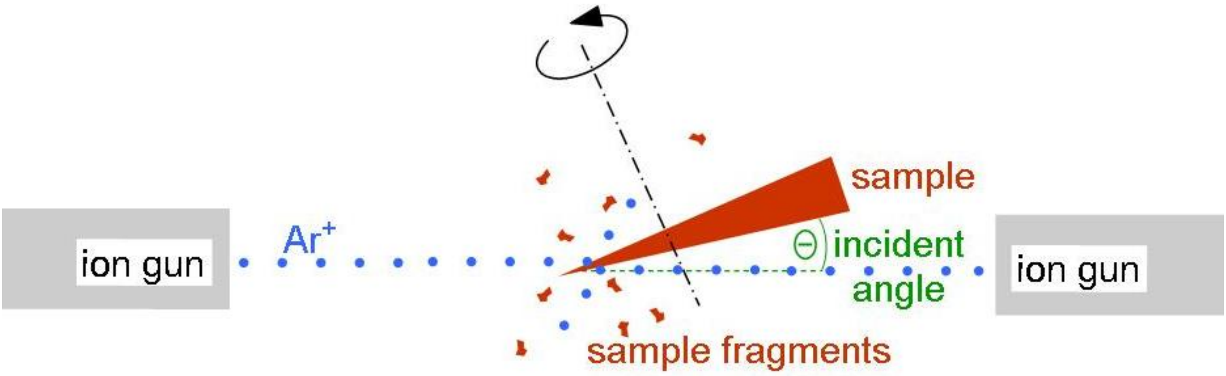


Figure 2.11: Schematic image of the ion milling process (proportions do not correspond to reality)

2.3.2 Methods Specific to Fine Particles

Crushing

If the sample does not consist of micro- or nanometrical sized particles, they can be achieved by crushing. The material is cracked mechanically by a crusher, mortar or mill until a fine powder is obtained.

Dispersion of Fine Particles

The electron transparent fine particles get ultrasonically dispersed in a proper solvent. The suspension is dropped onto the support film of a TEM grid. The liquid evaporates and the particles can be investigated.

2.3.3 Chemical Polishing

The sample is cut and polished until either a thin slice of 100 μm is obtained (full bath chemical thinning) or cut, stamped and polished until a disc of 3 mm and 50 to 200 μm thickness is achieved (twin jet chemical thinning). Foils or thin discs of single phased samples can be thinned by immersing them in a chemical bath. One side of the sample is protected by a varnish that avoids the surface to get in contact with the chemical solution.

2.3.4 Electropolishing

Depending on the specific technique, the process starts with preparing either a thin slice of 100 μm thickness by cutting and polishing (full bath electrolytic thinning) or a disc of 3 mm and 50 to 200 μm thickness by cutting, stamping and polishing (twin jet electrolytic thinning).

Electroconductive single or, under certain conditions, multi phased materials can be thinned electrochemically in a bath of proper electrolyte. The sample, which serves as the anode, gets thinned due to an oxidation process when electrical voltage is applied.

2.3.5 Focused Ion Beam (FIB)

This technique is used to obtain an electron transparent lamella from a certain position in a sample. A beam of Ga^+ ions of the FIB is focused by electric lenses and sputters away the surroundings of the selected area so that only a thin slice is left. The lift-out process of the lamella is done by means of a micromanipulator and a welding process. The FIB can be applied to various materials. Due to amorphisation of surface regions Sometimes ion polishing as subsequent treatment is necessary.

2.3.6 Replica Technique

This method can be applied when a sample cannot be inserted into the TEM due to geometrical or material reasons. The surface topography gets captured by direct or indirect replica techniques with a carbon film deposit serving as mold.

2.3.7 Ultramicrotomy

Ultramicrotomy is a procedure for cutting off ultrathin slices of 30-100 nm thickness from a small sample. This is realized with a tool called *Ultramicrotome* that is equipped with a glass or diamond knife, a sample holder and a goniometer. Sometimes the sample has to be embedded to enhance the suitability for this preparation method.

If the material is too soft for proper cutting, cyro-ultramicrotomy can be applied. This method is similar to ultramicrotomy but it is performed at a temperature below room temperature.

2.3.8 Wedge Cleavage

Plates of monocrystalline substrates can be cleaved along atomic planes that give perfect edges. The resulting fragments are in shape of rectangular prisms with base areas of about 0.6 mm^2. The fragment, which should be investigated, is raised and tilted, so that the electron beam goes through two lateral surfaces at one edge of the initial prism. Therefore the beam transmits a wedge, which is determined by two surfaces that originate from cleaving. The edge of this wedge is electron transparent.

2.3.9 Comparison of Different Preparation Methods

This subsection refers to [9].

Table 2.1 shows which preparation method can be applied to which materials. Often preceding preparation steps like embedding, cutting or polishing are required.

	metal	semiconductor	ceramic	mineral	polymer	biological material	composite material
chemical polishing	×	×	×	×			×
electropolishing	×	×					
ion thinning	×	×	×	×	×		×
focused ion beam	×	×	×	×	×		×
ultramicrotomy	×	×	×	×	×	×	×
replica technique	×	×	×	×	×	×	×
wedge cleavage		×	×				
wedge polishing	×	×	×	×	×		×
crushing			×	×			×
fine particle dispersion	×		×	×	×	×	×

Table 2.1: Suitability of different preparation methods for particular material groups

Table 2.2 lists the suitability of each preparation method in dependence of the hardness of the sample.

	very hard	hard	medium	soft	very soft
chemical polishing	×	×	×	×	×
electropolishing	×	×	×	×	×
ion thinning	×	×	×	×	
focused ion beam	×	×	×	×	
ultramicrotomy		×	×	×	×
replica technique	×	×	×	×	
wedge cleavage	×	×			
wedge polishing	×	×	×		
crushing	×	×	×		
fine particle dispersion	×	×	×	×	×

Table 2.2: Suitability of different preparation methods for materials of different hardness

Table 2.3 shows which preparation methods can be applied at samples of different fragility.

	resistant	fragile	fairly fragile	ductile
chemical polishing	×	×	×	×
electropolishing	×	×	×	×
ion thinning	×	×	×	×
focused ion beam	×	×	×	×
ultramicrotomy		×	×	×
replica technique	×	×	×	×
wedge cleavage		×	×	
wedge polishing	×	×	×	
crushing		×	×	
fine particle dispersion	×	×	×	×

Table 2.3: Suitability of different preparation methods for materials of different fragility

An overview of suitable preparation methods for different initial organisation of the material is shown in table 2.4. Some preparation methods require preceding work like embedding, cutting or polishing.

	bulk	monolayer	multilayer	fine particles
chemical polishing	×	×		
electropolishing	×	×		
ion thinning	×	×	×	×
focused ion beam	×	×	×	×
ultramicrotomy	×	×	×	×
replica technique	×			×
wedge cleavage	×		×	
wedge polishing	×	×	×	×
crushing	×	×	×	×
fine particle dispersion				×

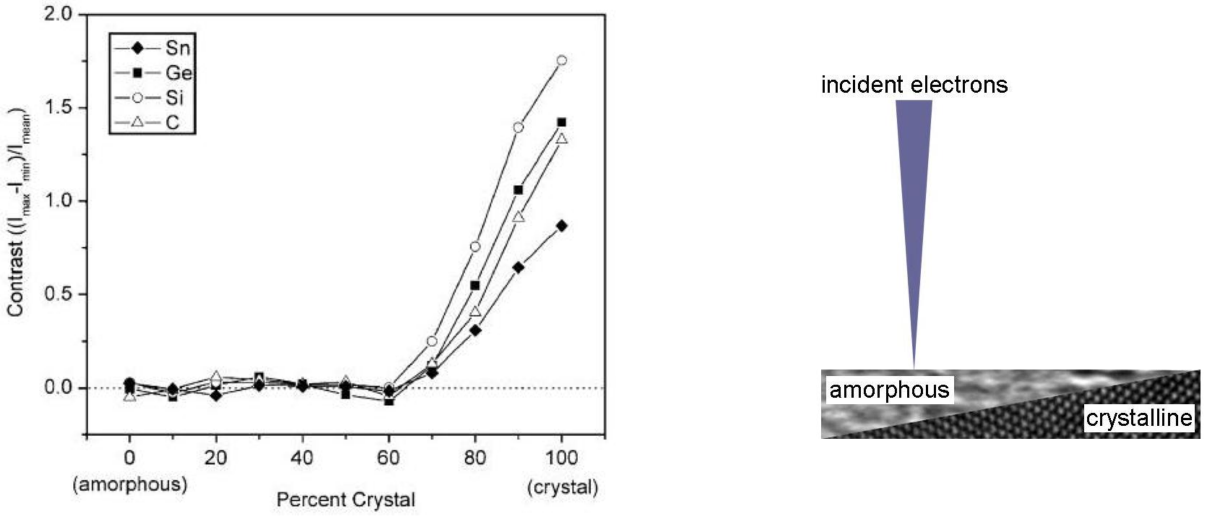
Table 2.4: Suitability of different preparation methods for materials of different organisation

In conclusion, wedge polishing is a preparation method that can be used for a great variety of materials, like metals, semiconductors, ceramics, minerals, polymers and composite materials. It is suitable for materials from very hard to medium hardness. Soft or ductile samples can not be wedge polished very well. This preparation method can be applied to bulk, mono- and multilayer samples, as well as fine particles which have to be embedded previously.

2.4 Prevalent Artifacts Induced by Sample Preparation

During sample preparation, the material often suffers physical and/or chemical modifications [10]. These artifacts affect the TEM investigation, lead to distorted results and limit resolution. Especially the quality of images, that were acquired with high resolution C_S-corrected transmission electron microscopes, is restricted this way [11].
Therefore the damage due to chemical, mechanical, ionic or thermal influences has to be reduced as much as possible.

A widespread artifact is amorphisation of the surface layer which causes a decrease in spatial resolution [12]. The electron beam loses some of its initial energy which converts into a Gaussian-like background. Additionally the remaining intensity suffers a change of defocus that depends on the thickness of the amorphous layer. Figure 2.12(a) shows the calculated contrast of 300 $Å$ thin tin, silicon, germanium and carbon samples, according to the proportion of the thickness of crystalline and amorphous layer. Due to this simulation, which assumes that the beam passes the amorphous layer first (see figure 2.12(b)), the atomic columns are only visible if the amorphous layer is less than 30% in thickness compared to the whole sample, independent of the material.



(a) Calculated contrast of 300 $Å$ thick sample of several materials in dependence of their composition [12]

(b) Schematic image of the relation between amorphous and crystalline layer

Figure 2.12: Influence of amorphisation layer on TEM investigation

The following subsections introduce the two preparation methods that were applied in this thesis, as well as their typical artifacts and how to minimize them.

2.4.1 Wedge Polishing - Artifacts and their Elimination

The overwhelming damage caused by wedge polishing is amorphisation of the surface layer. Due to mechanical stress [10], formerly crystalline material undergoes a phase transition and ends up in an amorphous state. Beside this structural change there are also other unwanted effects like:

- matter displacement
- tears, cracks and fractures
- linear (1D) and plane (2D) structural defects
- inclusion of abrasive grains
- material selective abrasion

Based on the *trinity of damage* [8], formula 2.7 shows a simple rule to estimate the thickness of the amorphisation layer ($t_{Amorphisation\,Layer}$) according to the grain size (s_{Grain}) of the applied lapping film.

$$t_{Amorphisation\,Layer} = 3 \cdot s_{Grain} \tag{2.7}$$

That means that a polishing film causes an amorphous layer about 3 times deeper than its grain size. The concept of minimizing artifacts intends to polish away the damage with the subsequent film. When starting with a grain size of 30 μm, a layer of at least 90 μm has to be removed by the 15 μm-film. Then the 6 μm-film has to polish away at least 45 μm and so on.

This way the layer of damaged material gets thinner and thinner. Final chemical-mechanical polishing with colloidal silicon also lowers the thickness of the preparation induced amorphous layer until it is negligible slim. The remaining amorphous layer can be removed completely by a chemical treatment (see subsection 2.2.4).

2.4.2 Ion Thinning - Artifacts and their Elimination

Ion thinning induces (micro)structural changes, which lead to amorphisation of the surface due to ionic effects [10].

Other artifacts caused by this preparation technique are:

- implantation of ions
- material selective abrasion
- redeposition of the sample material
- vacancies, cavities and dislocations

The thickness of the amorphous layer ($t_{Amorphisation\ Layer}$) depends on the ion energy (E_{Ion}), which is adjustable via the acceleration voltage, and the angle of incidence (Θ) [13, 14] with respect to the direction parallel to the surface. Formula 2.8 shows the relation between these parameters.

$$t_{Amorphisation\ Layer} \propto E_{Ion} \sin\Theta \tag{2.8}$$

In figure 2.13 the amorphous layer thickness is imaged as a function of the acceleration voltage and the angle of incidence in a three dimensional diagram.

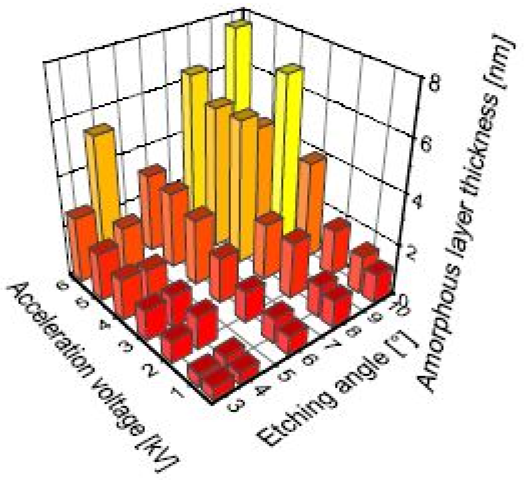


Figure 2.13: 3D diagram of the amorphous layer thickness of a Silicon sample as a function of the acceleration voltage of the Ar^+ ions and the angle of incidence [15]

Figure 2.14 depicts TEM images of a crystalline silicon sample with an ion milling induced amorphisation layer. It shows the dependence of the thickness of the amorphous layer on the acceleration voltage. The higher the voltage, the more amorphisation.

It is not possible to obtain a totally damage-free sample which was thinned by ion milling. For example if the material is perovskite, there is always an amorphous layer, even if ion milling was performed under gentle conditions like low incident angle, low energy and low temperature [11]. With the wedge polishing technique, in contrary, the majority of the damage layer can be eliminated very well. In some cases it can be removed completely.

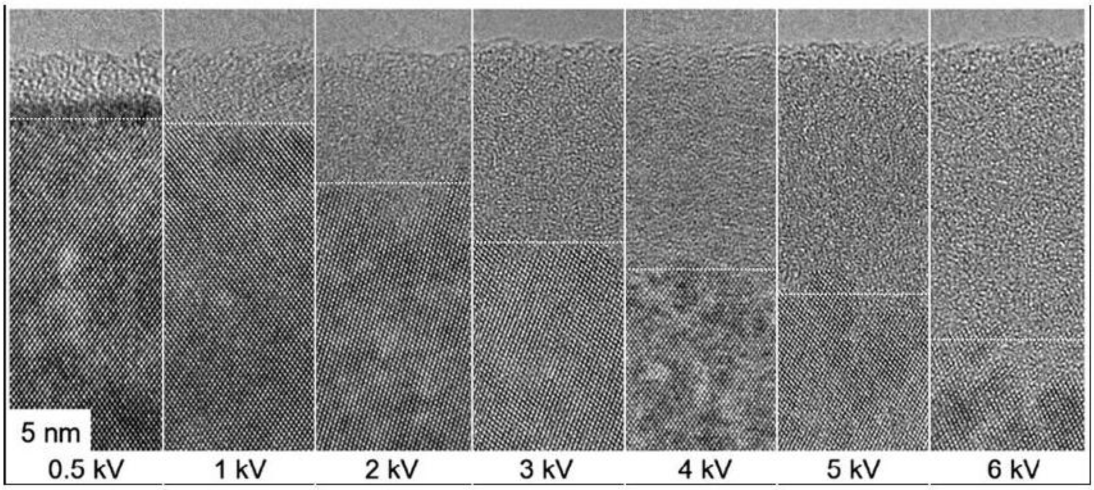


Figure 2.14: TEM images of a silicon sample that was ion milled at 3° at different acceleration voltages. The thickness of the amorphised layer increases with the voltage (modified from [15])

2.5 Materials with High Wedge Polishing Suitability

Table 2.5 shows a list of materials which have been successfully prepared via the wedge polishing technique. As the properties of the main or substrate material are crucial for the preparation, it is sorted by them, even if the focus of the scientific investigations is on smaller regions or layers of other materials. In the following columns the treatment after wedge polishing, the polishing method, some additional information and the reference are listed.

substrate material	**subsequent treatment**	**polishing method**	**additional information**	**reference**
sapphire samples:				
sapphire (Al_2O_3) with GaN	none	semiautomatic	- gallium nitride grown on sapphire - glued together face to face	[16] 4.3
sapphire (Al_2O_3) with metallized GaN	ion thinning	manual	- metallized gallium nitride grown on sapphire	[17]
sapphire (Al_2O_3) with InGaN/GaN quantum wells	ion thinning	manual	- indium gallium nitride/ gallium nitride grown on sapphire	[18]
silicon carbide (SiC)	none	semiautomatic	- SiC/SiC thin foils - crystalline SiC fibres in submicron SiC-matrix - embedded in epoxy resin	[19]
tungsten carbide (WC) with Co	none and ion thinning	semiautomatic	- tungsten carbide in matrix of cobalt	4.4
indium phosphide (InP)	none	manual	- doped with iron - GaInAs/InP heterostructures grown on substrate	[20]
magnesium oxide (MgO)	ion thinning	manual	with $YBa_2Cu_3O_7$ thin film	[21]

substrate material	**subsequent treatment**	**polishing method**	**additional information**	**reference**
strontium titanate samples:				
strontium titanate ($SrTiO_3$) with pervoskite thin films	none or ion thinning	semiautomatic	- $PbTiO_3$, $SrRuO_3$ and $LaFeO_3$ films of 10-50 nm were grown on $SrTiO_3$-substrate - if electron transparency was not achieved by wedge polishing, ion thinning was performed	[11]
strontium titanate ($SrTiO_3$)	none or ion thinning	manual	with $YBa_2Cu_3O_7$ thin film	[22] [21]
strontium titanate ($SrTiO_3$) with $BaTiO_3$	ion thinning	semiautomatic	- atomic resolution EELS maps	[23]
ceramic fibres:				
silicon carbide (SiC), oxide (O) and alumina (Al) ceramic fibers in epoxy	ion thinning	manual	- impregnated with epoxy - coated with carbon, zirconia, alumina, mullite, calcium hexaluminate, lanthanum phosphate, and combinations thereof	[24]
ceramic fibers in epoxy	ion thinning	manual	- impregnated with epoxy - ceramic coated	[25]
yttria-stabilized zirconia (YSZ)	ion thinning	manual	with $YBa_2Cu_3O_7$ thin film	[21]
yttrium barium copper oxide ($YBa_2Cu_3O_7$)	none	manual	$YBa_2Cu_3O_7$ as bulk ceramics	[22]

substrate material	**subsequent treatment**	**polishing method**	**additional information**	**reference**
silicon samples:				
silicon (Si)	none	semiautomatic	- sandwich of 2 Si-platen	4.2
silicon wafer	none	semiautomatic	- sandwich of 2 parts of a Si-wafer	4.2
silicon with SnO_2-layer	none	semiautomatic	- tin dioxide on Si-substrate	4.2
silicon doped with Sb	chemical etching	semiautomatic	- hydrofluoric acid (HF) removes oxide layer	[8]
silicon-based nanostructures	ion thinning	manual	- metal coated - polycristalline	[26]
silicon semiconductor	chemical etching	manual	- $TiSi_2$, WSi, $Si0_2$ and SiN_x on Si - chemical etching with potassium hydroxide for selective decomposition of silicon over silicide	[27]
silicon with $Nd_2Fe_{14}B$	ion thinning	manual	- channeled Si-wafer with $Nd_2Fe_{14}B$-powder mixed with Gatan G1 epoxy	[28]
aluminum indium nitride (AlInN)	none or ion thinning	not specified	- multilayered AlInN/AlN/GaN heterostructures - ion thinning only as clean-up when necessary	[29]
stainless steel with YSZ	ion thinning	manual	- plasma sprayed yttria-stabilized zirconia on smooth surfaces of different types of stainless steel	[30]

Table 2.5: Materials with high wedge polishing suitability sorted by the main component

3 Experimental

3.1 The Semiautomatic Polishing Process

The steps, which have to be done to perform semiautomatic sample polishing with the Multiprep$^{\text{TM}}$ System are listed below. Some of these steps are described in more detail after the list.

1. Mount the sample onto the paddle.
2. Place the platen onto the platen base.
3. Wet the platen with distilled water, put the lapping film thereupon and sweep it with a rubber squeegee. This causes the water beneath the film to be removed and to affix the lapping film.
4. Attach the paddle with the sample to the cam lock adapter at the bottom of the spindle.
5. Rise the spindle with the vertical adjustment knob so that the sample will not touch the lapping film when the spindle riser is lowered.
6. Lower the spindle riser.
7. Set the front digital indicator to zero.
8. Lower the sample into the abrasive via the vertical adjustment knob until 100 μm more than the desired abrasion are displayed at the front indicator.
9. Lift the spindle riser.
10. Start the rotation of the platen with 10 rpm.
11. Apply the lubricant.
12. Lower the spindle riser carefully.
13. Immediately set the front digital indicator to zero.
14. Set desired rotation speed between 5 rpm and 350 rpm.

Additionally it is possible to choose the following adjustments [31]:

- clockwise or counterclockwise platen rotation
- horizontal position of the sample
- automatic sample oscillation with 6 sweep speeds
- automatic full or limited sample rotation with 8 speeds
- digital timing
- sample load from 0 to full
- spindle lock for orientation in a certain direction

The front digital indicator, that is set to zero at step 7, will indicate “0” until the sample is in contact with the lapping film. Then the indication is displayed with a negative sign. Step 8 of the polishing procedure causes that the spindle pulley does not have contact to the arm. Therefore it goes downwards due to gravity as abrasion proceeds. The amount of load with which the sample is pressed onto the lapping film, can be adjusted by a knurled screw (see figure 2.9). At full load, the whole weight of the spindle, cam lock adapter and paddle, which is about 500 g, is applied. The pressure at the sample can be reduced in 6 steps until zero, whereby the load scale is relative to the total weight.

After polishing away a sufficient amount of material, the spindle pulley makes contact with the arm again and the vertical traveling is stopped. Therefore, the value shown at the front digital indicator at point 8, is the maximal abrasion. During polishing it indicates how much of the sample thickness already has been abraded.

Step 9 to 12 are performed for starting the abrasion gently.

3.1.1 Polishing Hints

Geometrical Alignment

Before starting the preparation of a new sample it is highly recommendable to align the Multiprep™ System through mechanical adjustments which are described in the operation manual [7]. The alignment procedure is important for obtaining perpendicular and parallel reference surfaces that are necessary for high precision working.

After aligning, the pyrex of the paddle has to be polished plan and parallel to the grinding platen, to fit to the adapted reference surfaces. This is performed with a 3 μm lapping film at full load, oscillation at level 3 and a rotation speed of 80 rpm.

Abrasion Debris

During polishing, the abraded particles have to be caught to prevent them from hitting again the polished surface and scratching it. Lint free paper or small sponges are held on the polishing surface to pick up the debris. It is important not to use the same sponge or paper for different grain sizes. Some particles may escape and if they are larger than the current grain size and scratch over the sample surface, they can cause serious damage.

Angular Correction

The final shape of the sample is influenced by its parallelism to the pyrex edge and the evenness and flatness of the adhesive line [32]. Right after starting the wedge, the sample shows a top view angle (see figure 3.1), if not mounted properly. To prevent a sample, that is orthogonal trapezoidal shaped in horizontal plane, angle correction has to be performed. The correction angle ($\alpha_{correction}$) has to be adjusted at the right rear micrometer screw and depends on the wedge angle (α_{wedge}) and the top view angle ($\alpha_{topview}$):

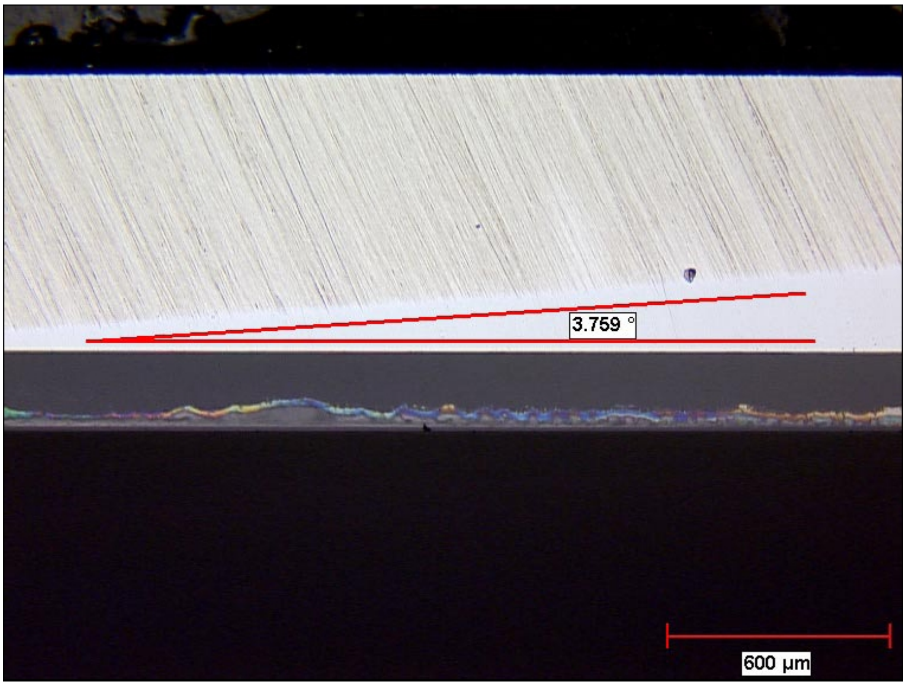


Figure 3.1: Top view angle after introducing the wedge angle at TEM cross-section preparation of an Integrated Circuit [32]

$$\alpha_{correction} = 0.01\, \alpha_{wedge}\, \alpha_{topview} \tag{3.1}$$

After correction, the top view angle is zero.

Colloidal Silica

As pointed out in subsection 2.2.1, final polishing with colloidal silica is a chemical-mechanical process. To remove the colloidal silica, the sample gets rinsed with water and immersed into a basic solution consisting of organic soap and warm water. Nevertheless in many cases some of the silica-particles remain on the sample surface and agglomerate (see figure 4.9).

Cleanliness

Cleanliness is an absolute necessity when working with the Multiprep$^{\text{TM}}$ System. It is important to clean the machine each time after use and to protect it with a dust cover.
It is also advisable to apply only distilled water during polishing to avoid chalk stains.

After having contact with acetone, the sample should be washed with isopropanol before it has dried. This prevents schlieren.

Wedge Geometry

The goal is to polish a wedge with an edge as fine as possible. Therefore the geometrical figure of the wedge shaped sample in lateral view is a right-angled triangle to which trigonometric functions can be applied (see figure 3.2). The height of the final wedge h_{wedge} corresponds to the opposite side, the wedge length l_{wedge} to the adjacent side, and the angle is the introduced wedge angle α_{wedge}. The wedge length can be easily measured with an visible light microscope, and the wedge angle also is known. Therefore equation 3.2 shows:

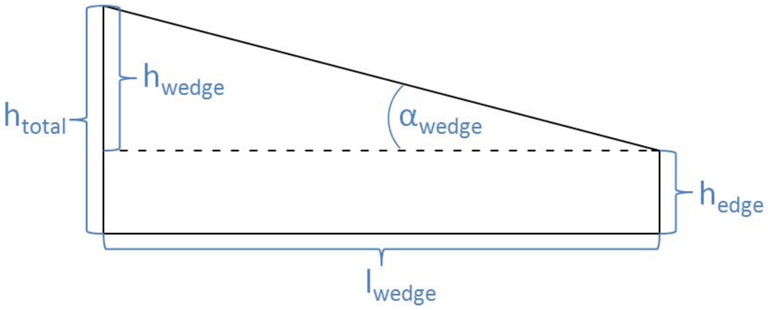


Figure 3.2: Schematic drawing of the sample shape in lateral view during the polishing process

$$h_{wedge} = l_{wedge} \tan \alpha_{wedge} \tag{3.2}$$

The height of the thicker side of the sample h_{total} must not exceed h_{wedge} to achieve a theoretical infinite fine wedge edge. In other words, the thickness of the edge h_{edge} is the current height of the thicker side h_{total}, minus h_{wedge}.

3.2 General Preparation Hints

3.2.1 Gluing and Adhesives

The choice of adhesive influences the result of the preparation significantly. There are three application fields for the glues, which demand different properties:

- *Preparing sample sandwiches:* If the sample is not a bulk material but layers on a substrate that should be investigated, it is a common practice to cut off two equally sized pieces and glue them together face to face. This way, the layers are protected by the substrate during polishing. Preparing sandwiches with the sequence: glass cover slip - sample - glass cover slip, is also an often used method.
  The splice has to be very reliable to withstand high forces during polishing, and the glue must be insoluble in acetone. The thinner the glue line, the better the performance. To obtain a narrow splice, the freshly cured sandwich is clamped into a special tool (see figure 3.3). Via a screw and springs, pressure is applied at the top and bottom of the sample during the curing process that normally takes place at higher temperatures. Teflon lining prevents the sample from sticking to the tool due to oozing adhesive.

- *Mounting sample onto pyrex:* Ideally, the sample is mounted parallel to, and close beneath, the front edge of the pyrex. If it is fixed over the pyrex edge, the sample bends and/or breaks during polishing. If it is attached with a large distance to the front edge, the pyrex also gets abraded significantly at second side polishing.
  If the sample separates from the pyrex during polishing it can get lost or completely destroyed. In particular at second side polishing, when the wedge angle has already been introduced and the sample is very thin, it is nearly impossible to reattach the sample in a way, that the preparation work can be continued properly. Therefore the adhesive has to sustain pressure and forces that occur during polishing. Solubility in acetone is required, as it is necessary to unmount the sample after polishing.

- *Mounting sample onto TEM grid:* For the mounting process (see subsection 3.4.2), the adhesive should be sticky even when it is not fully cured. It does not have to stand high mechanical stress, but vacuum stability is required.

In this thesis following types of adhesives were used:

- *EpoxyBond 110^{TM} [33, 34]:* This is a hard and fast-curing two component epoxy adhesive that was used for sample sandwiches due to its insolubility in acetone. The glue lines get thin because of the low viscosity. The mixing ratio is 10:1 and the curing time is from 5 min to 30 min at 150 °C to 100 °C.

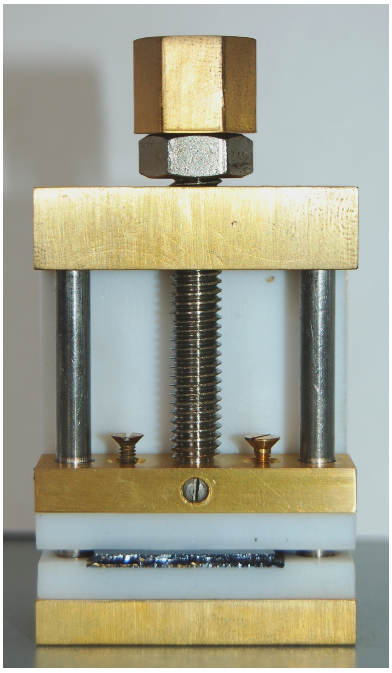


Figure 3.3: Gluing tool for obtaining thin glue lines. The sample is inserted and pressure is applied via the big top screw and springs at the two smaller screws.

- *Gatan G1:* The G1 is a two component epoxy adhesive which was used for sample sandwiches. It is insoluble in acetone and shows a mixing ratio of 10:1. Its low viscosity provides thin glue lines. The curing time is about 2 h at 150 °C.
- *M-Bond 610 [35]:* It is a two component adhesive which is insoluble in acetone. It was used for gluing sample-sandwiches and fixing the sample onto the TEM-grid. The viscosity is low, and therefore it provides thin glue lines. The optimal curing temperature and the curing time are shown in a diagram at the instruction bulletin. Optimal curing proceeds at 160 °C (during 2 h) to 190 °C (during 1 h). It is important not to apply a high amount of the M-Bond 610 because its boiling point is far beneath the curing temperature and bubbles will develop that cannot escape.
- *Loctite® 460™ [36]:* It is a one component alkoxyethyl cyanoacrylate glue that is well soluble in acetone. A relatively thin glue line can be achieved due to its medium viscosity. It was used for fixing the sample to the pyrex and for mounting it onto the TEM-grid. Loctite® 460™ is not recommendable for use as an adhesive between sample and pyrex, because when in contact with water during longer lasting polishing, it detaches.
  The best rate of cure is achieved at a humidity of 40% to 60% in the working environment at 22 °C. Depending on the material, the cure time can vary from 2 s to 300 s until functional strength is obtained, while the full chemical and solvent resistance is developed after 24 h.

- *Loctite® 435™ [37]:* This adhesive is a one component ethyl cyanoacrylate glue. Because of the low viscosity it yields a very thin glue line. The Loctite® 435™ was used for mounting the sample onto the pyrex and onto the TEM-grid. It is very reliable and therefore highly recommendable to use it as an adhesive between sample and pyrex in longer lasting polishing procedures or at critical processes. The disadvantage is, that due to its miscible solubility in acetone, it takes longer to unmount the sample.
  Normal atmospheric moisture at room temperature starts the curing process. The rate of cure depends on the material and is between less than 5 s to 105 s until functional strength is achieved. Nevertheless, curing continues for at least 24 h until full chemical and solvent resistance is developed.

- *Hot Mounting Wax [38, 34]:* This mounting wax mainly consists of phthalic anhydride and melts at a temperature of 120 °C. It is good soluble in acetone and was used for fixing the sample onto the pyrex. The glue lines are thicker than with the Loctite® glues. It is important to apply a sufficient amount of wax, otherwise the sample detaches. This can also happen when polishing is performed for a longer time and the wax gets softened by the cooling water. The curing process is performed at room temperature.

Most of the glues were applied with a wooden toothpick that was copped with a scalpel if necessary. For application of a very small amount at a tiny area, also a hair of the eyebrow can be used as a brush [5].
The surfaces that are glued together have to be accurately cleaned before.

If curing has to be performed in an oven, do not preheat it. If the adhesive is heated too fast, bubbles may occur and cannot escape due to the cured adhesive in their surroundings. This lowers the performance of the splice significantly.

3.2.2 Thickness Control

At second side polishing, it is necessary to know the actual sample thickness for a proper, well timed succession of the lapping films. To apply the appropriate grain size in dependence of the thickness of the wedge is crucial for a good preparation result. Starting too early with a fine lapping film highly increases the preparation time, which is also a challenge for the material and adhesive. If polished too long with a coarse grain size, the strategy of successively polishing away the amorphous layer that is induced by the previous lapping film (see subsection 2.4.1), cannot be realised because there is not enough material left.

In the following paragraphs, methods for thickness control are described.

Visible Light Microscope

In some cases, the height of the thicker side of the wedge can be measured with a visible light microscope. For this, the paddle with the pyrex has to be placed in a way that the wedge gets observed from the back. The thickness at the edge can be concluded as described before (subsection 3.1.1). This method works only as long as the sample is not too thin. If it is mounted too far away from the back edge of the pyrex, focusing problems can occur.

If the visible light microscope is able to display the z-value for the focus, the sample height is obtained by focusing on the sample surface and then on the pyrex. The difference between these z-values gives the thickness at the measured position. The thickness of the glue line has to be estimated and subtracted for getting the real sample thickness. In this thesis, the *Infinite Focus Microscope* from *Alicona* was used for this thickness control method.

Thickness Due to the Front Digital Indicator

As pointed out in subsection 2.2.3, the front digital indicator shows the reduction of the sample thickness. Theoretically the sample thickness can be measured once and then be monitored via subtraction of the displayed value. In reality, a far too few abrasion is indicated when a sample load of zero is applied. As at the last polishing steps the load is always set to zero the indication only can be used as an estimation.

Optical Interference Fringes

If the sample is thin enough (approximately 2 μm for silicon), optical interference fringes appear at the edge [39] (see figure 4.8, 4.14, 4.18 and 4.23). The effect is due to destructive interference of rays that were reflected and transmitted inside the sample and can be seen only via the visible light microscope. The condition that fringes appear is that the sample is not too opaque. In this thesis, fringes were observed in sapphire and silicon samples but not in tungsten carbide.

Color

The thickness of silicon samples can be monitored easily through the color of the edge. At a thickness of about 4 μm, the color of silicon turns from brown to red when observed in transmission. At about 2 μm, it appears yellow and the thinner it gets, the brighter it looks. By comparison with a color standard (see figure 3.4), the thickness of these samples can be determined.

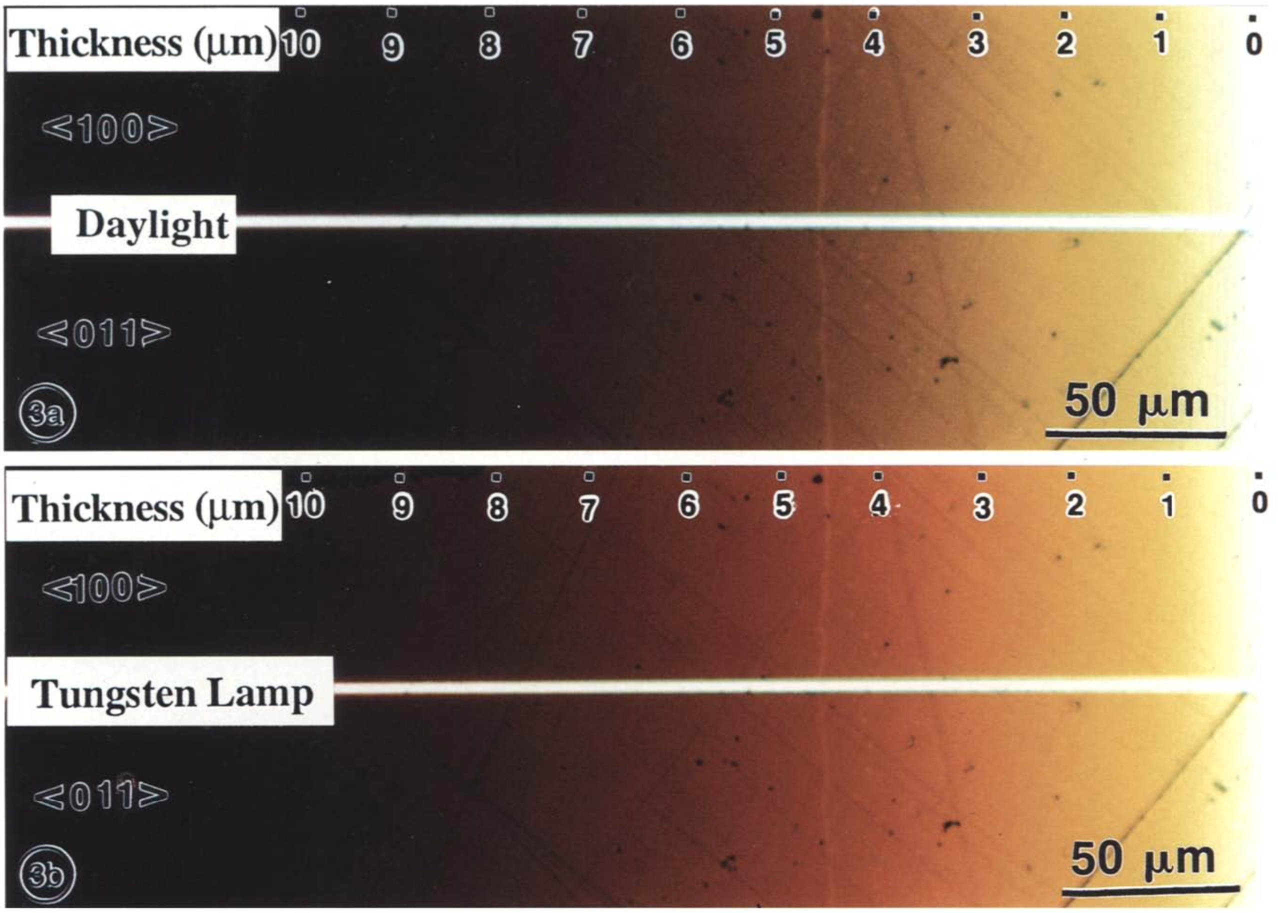
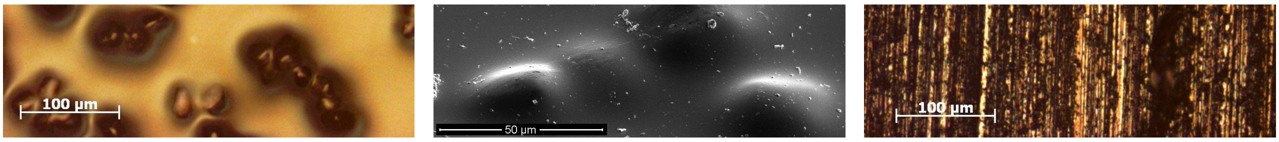


Figure 3.4: Color gradient in dependence of the sample thickness of silicon single crystal in two orientation directions <100> and <011> [39]

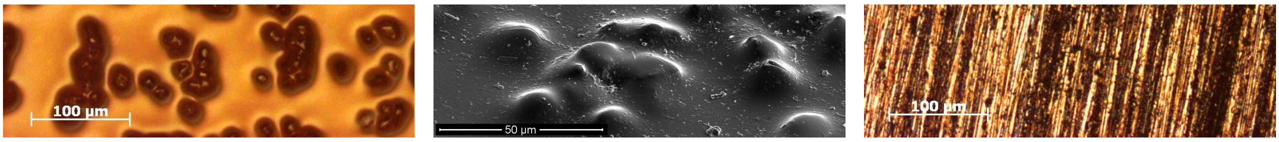
3.3 Lapping Films and Polishing Results

Figures 3.5, 3.6, 3.7, 3.8, 3.9 and 3.10. show visible light microscope and Scanning Electron Microscope (SEM) images of different lapping films and the surface of a highly doped silicon sample, polished with the corresponding grain size. The SEM images were taken at different magnifications from 1200× to 5000×. Further settings of the polishing process are pointed out at subsection 4.2.4 at first side polishing.



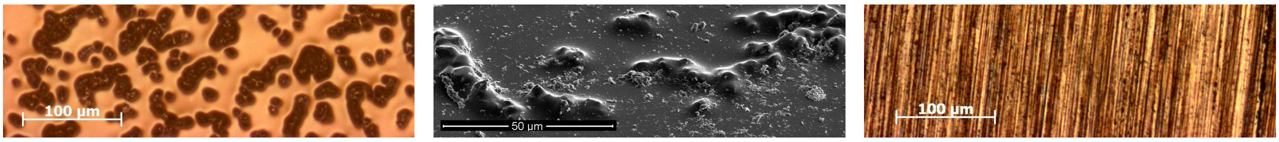
(a) (b) (c)

Figure 3.5: Visible light microscope (a) and SEM images (b) of a 30 μm diamond lapping film and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 1200×.



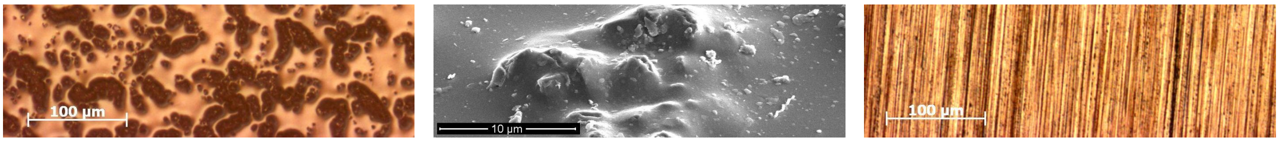
(a) (b) (c)

Figure 3.6: Visible light microscope (a) and SEM images (b) of a 15 μm diamond lapping film (a) and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 1200×.



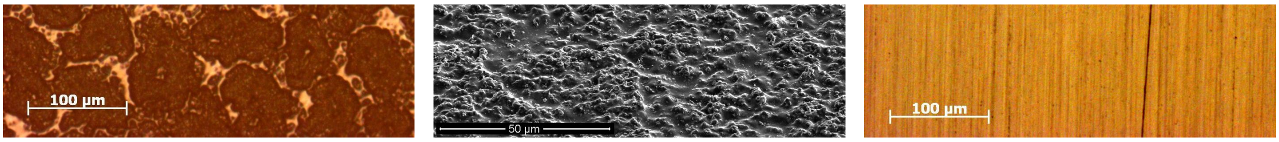
(a) (b) (c)

Figure 3.7: Visible light microscope (a) and SEM images (b) of a 6 μm diamond lapping film (a) and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 1250×.



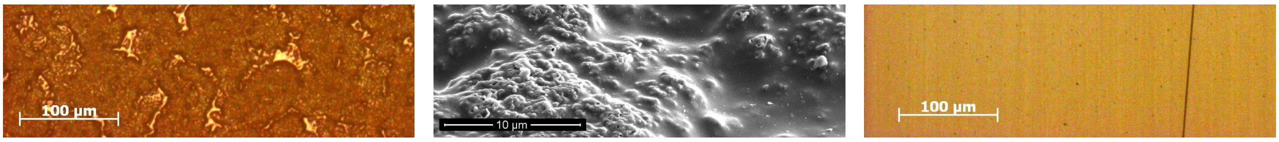
(a) (b) (c)

Figure 3.8: Visible light microscope (a) and SEM images (b) of a 3 μm diamond lapping film (a) and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 5000×.



(a) (b) (c)

Figure 3.9: Visible light microscope (a) and SEM images (b) of a 1 μm diamond lapping film (a) and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 1250×.



(a) (b) (c)

Figure 3.10: Visible light microscope (a) and SEM images (b) of a 0.1 μm diamond lapping film (a) and the corresponding polished surface of a silicon sample (c). The SEM image was taken at a magnification of 5000×.

3.4 Treatment after Polishing

3.4.1 TEM Grids

For investigation in TEM, the prepared samples have to be mounted onto special support grids (TEM grids). They are flat discs of about 13 μm in thickness and 3.05 mm in diameter, which exactly fit into the standardised TEM sample holders. There is a variety of different grid types which have to be selected depending on the sample. Some of them are depicted in figure 3.11. Also the grid material (e.g. gold, copper, molybdenum, nickel, palladium, diamond) can be chosen. Some types of grids also are covered with a support film, which is a thin, electron transparent carbon or polymer film.

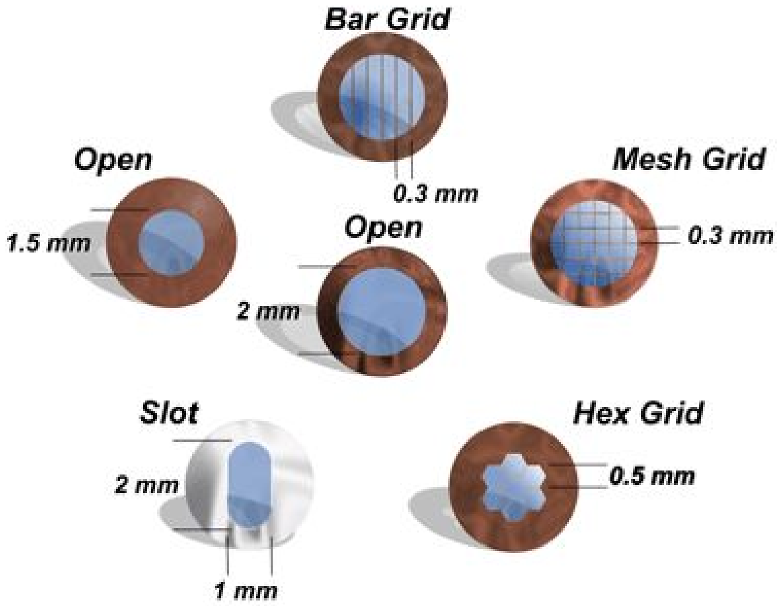


Figure 3.11: Different types of LuxFilm$^{\text{TM}}$ TEM support grids [40]

The sample is investigated only at regions that are situated above holes of the grid, so that the electron beam does not hit the grid.

3.4.2 Mounting on TEM Grid

To perform Transmission Electron Microscopy, it is necessary to fix the sample to a TEM grid (see subsection 3.4.1). In this thesis, slot grids (see figure 3.11) were used, where the electron transparent wedge edge has to be situated in the slot, while the thick side must not exceed the grid.
As the thin edge is very sensitive to any kind of mechanical influences, detaching the sample and mounting it onto the TEM-grid is a delicate process.

A good working method for gluing the sample onto the grid is to put a small amount of glue onto the grid and let it dry a little bit, so that it becomes sticky. Then the grid is taken with tweezers and the part with the glue is put onto the sample, which has been unmounted from the pyrex before and is lying on a piece of aluminum foil or a dry filter paper. When the sample sticks to the grid, the grid has to be turned around and further curing has to be performed. In some cases the sample starts to “swim” on the glue. If this happens, it must be brought into the right position gently with a toothpick.

In this thesis, two ways of unmounting the sample from the pyrex were performed:

Upright Pyrex in Acetone

The pyrex with the sample that is still attached, is put into a small glass equipped with a filter paper at the bottom. Acetone is added until the sample is totally covered. After a sufficient waiting time that depends on the type of the used glue, the next step can be performed. With a small pipet some acetone is soaked up and released again so that a gentle acetone flow is created (see figure 3.12). The flow direction is from the edge to the thicker side, but the edge should not be hit by the flow directly. The sample glides over the pyrex and gets caught by a piece of aluminum foil that is hold to the back edge of the pyrex. Now the aluminum foil with the sample is lifted out of the acetone carefully. The sample gets cleaned with isopropanol before the acetone is evaporated.

The filter paper at the glass bottom can help to lift the sample out of the acetone if it has missed the aluminum foil.
Aluminum foil is used because the sample does not get soaked in by fine pores, as it has occurred with not fully dried filter paper.

Tilted Pyrex in Acetone

The paddle with the pyrex and the sample is placed tilted in a bath of acetone (see figure 3.13). A piece of filter paper is placed beneath the sample. When the acetone has dissolved the glue, the sample detaches from the pyrex and falls onto the filter paper. With the filter paper, the sample can be lifted out of the acetone. The next steps are cleaning the sample with isopropanol and gluing it onto the TEM-grid.

3.4.3 Ion Thinning

As described in subsection 2.2.4, subsequent ion thinning can be performed when the sample is not thin enough or structural defects were induced through polishing.
In this thesis, wedges made of tungsten carbide and sapphire were ion thinned after wedge polishing (see chapter 4).

3.4.4 Damage due to Transport

The wedge can get damaged during transport or when inserted into the sample holder and taken out again. This happened with a silicon sample that was investigated by TEM two times. The wedge showed significant damage and a smaller thin area, in the second TEM investigation.

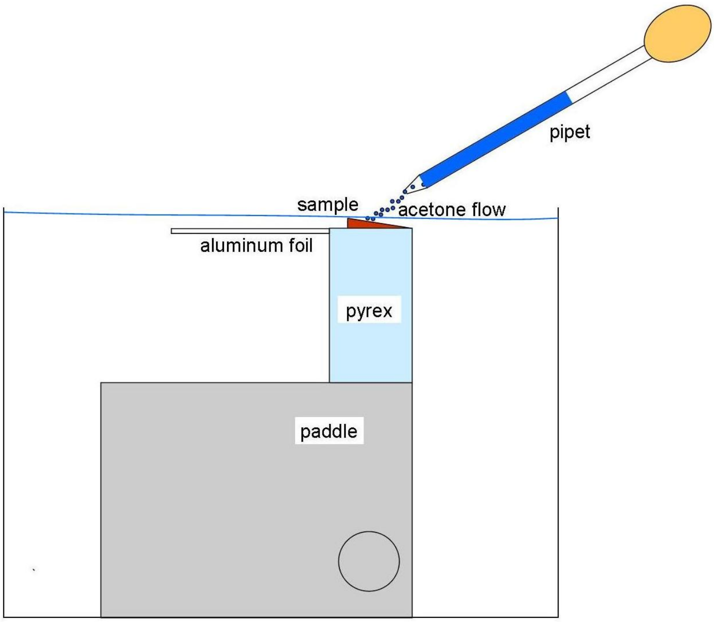


Figure 3.12: Schematic illustration of the detaching-process with upright pyrex in acetone bath (proportions do not correspond to reality).

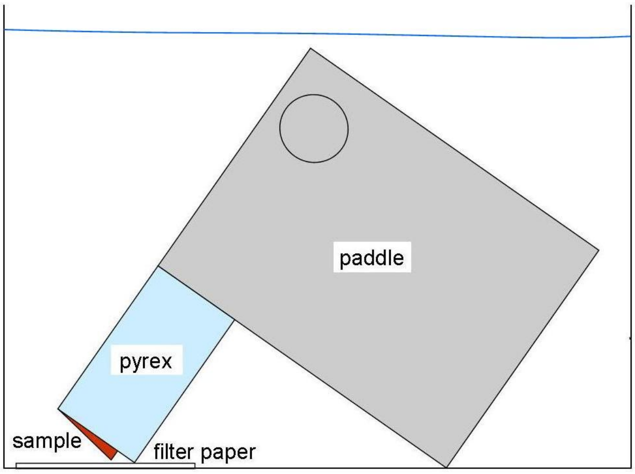


Figure 3.13: Schematic illustration of the detaching-process with tilted pyrex in acetone bath (proportions do not correspond to reality).

4 Preparation and Results

4.1 Overview

Table 4.1 shows the types of samples which were prepared via the semiautomatic wedge polishing technique.

	sample	subsequent treatment	subsection
silicon	silicon plate sandwich	none	4.2.1
	silicon wafer sandwich	none	4.2.2
	silicon with tin oxide layer	none	4.2.3
	highly doped silicon	none	4.2.4
sapphire with GaN layer	sapphire I	ion thinning	4.3.1
	sapphire II	none	4.3.2
tungsten carbide with Co	tungsten carbide I	ion thinning	4.4.1
	tungsten carbide II	none	4.4.2

Table 4.1: Overview of prepared samples

The general polishing setup for each sample is listed in tables. The succession of lapping films with the corresponding settings for sample load, rotating speed, lubricant and abrasion for first and second side polishing are also shown in the tables. Diamond lapping films were used exclusively. The abrasion in μm is the value that is displayed at the front digital indicator. However, especially at fine grain sizes and a sample load of “0”, the true amount of abraded thickness is much more. For this reason, abrasion at fine lapping films is sometimes expressed in minutes of polishing, which is also displayed by the Multiprep$^{\text{TM}}$ System, or monitored by other criteria like the change of color or the appearance of optical fringes.

As described in section 2.2, second side polishing always has to be performed with a clockwise rotating plate.
TEM slot grids were used as support (see subsection 3.4.1).

A photo of each finished sample on a TEM-grid was taken with a Zeiss Axioplan visible light microscope. Most of the TEM investigations were made with a FEI Tecnai G2 F20 (see figure 2.1). Only the sapphire I wedge was investigated with a Philips CM20 after ion thinning and the highly doped silicon with the FEI Titan ASTEM.
TEM bright field images and a colored relative thickness map are depicted for each sample. This relative thickness is the value for $\frac{t}{\lambda}$ (see section 2.1). The profiles of these thickness maps show the respective thicknesses over the wedge length. The integration width indicates the width of that part of the wedge over that is integrated. The profiles are taken from the area which is indicated by a box in the thickness map.
If satisfying preparation results were achieved, also high resolution TEM images were acquired.

4.2 Silicon

Due to its physical properties, silicon is a very suitable material for semiautomatic wedge polishing. Three kinds of samples, with silicon as main material, were prepared with different methods.

4.2.1 Silicon Plate Sandwich

The first prepared samples were sandwiches made of two silicon plates. They were prepared according to the description of the Multiprep$^{\text{TM}}$ procedure for TEM wedge preparation [32] by Allied High Tech Products, Inc. The sample was mounted onto the pyrex in a way so that the sandwich glue line was parallel to the pyrex front edge. Important preparation settings are shown in table 4.2. The rule to polish away three times more than the grain size of the former abrasive (see 2.4.1) was not applied, as it was one of the first samples. In table 4.3 the sequence of the lapping films and the corresponding sample load, rotation speed, lubricant and abrasion is listed. The wedge was started at a grain size of 0.5 μm.
Second side polishing was done as described in table 4.4. With the 0.5 μm lapping film polishing was performed until the wedge edge appeared red.

wedge angle:	1°
sample oscillation:	none
sample rotation:	limit rotation at speed 1 during polishing with colloidal Si 1^{st} and 2^{nd} side
adhesive sample-pyrex:	Loctite® 460™
adhesive sample-grid:	Loctite® 460™
unmount method sample-pyrex:	upright pyrex
thickness control:	visible light microscope color
subsequent treatment:	none

Table 4.2: Preparation details of silicon sandwich

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
15	full	75	H_20	20 μm
6	full	60	H_20	20 μm
3	3	40	H_20	10 μm
1	3	20	H_20	35 μm
0.5	3	15	H_20	50 μm
0.1	4	10	H_20	20 μm
0.04 colloidal silica	1	150		2 min

Table 4.3: Succession of the first side polishing process of a silicon-wedge

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	3	75	H_20	400 μm
15	3	75	H_20	200 μm
6	3	60	H_20	100 μm
3	3	40	H_20	200 μm
0.5	3	15	H_20	until edge red
0.04 colloidal silica	1	150		2 min

Table 4.4: Succession of the second side polishing process of the silicon-wedge

Results

Figure 4.1 shows the visible light microscope photo of the silicon wedge on a TEM-grid. The wedge edge appears totally tattered. Electron transparency was achieved at some areas at the edge. But due to lack of experience and the not optimized polishing procedure, the samples that were prepared this way, did not show good TEM investigation properties. The thin parts easily broke off during polishing and mounting onto the TEM grid, as the wedge angle is very small (see figure 4.2 and 4.3(a)).

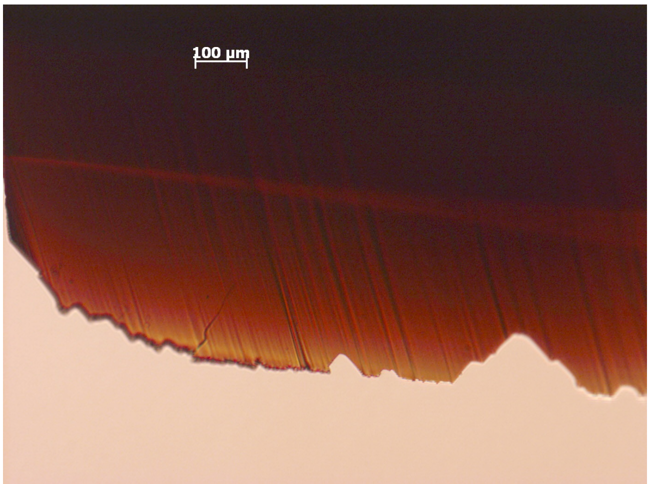


Figure 4.1: Visible light microscope photo of a silicon wedge at a magnification of 50×. The color gradient from red to yellow indicates the thickness of the edge.

Also the profile of the thickness map (figure 4.3(b)), with an integration width of 200 pixels, shows discontinuities.

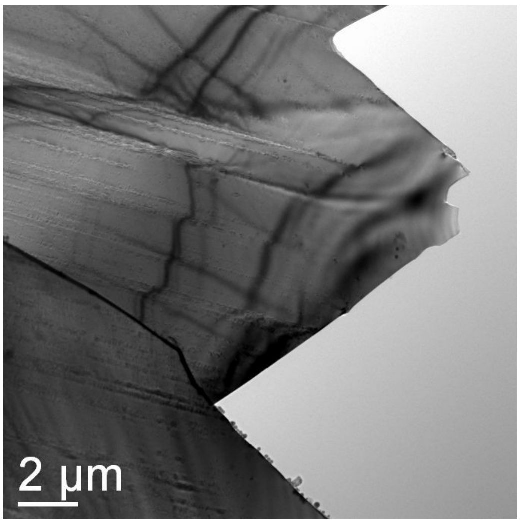
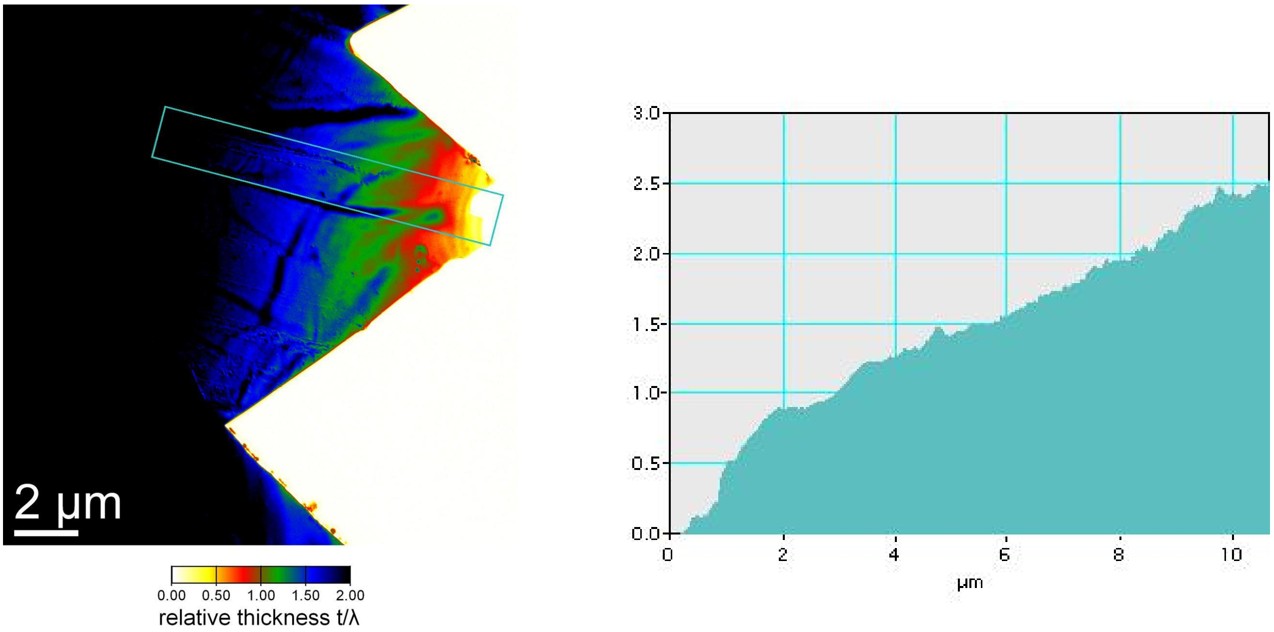


Figure 4.2: TEM bright field image of the silicon wedge



(a)

(b)

Figure 4.3: (a): Relative thickness map $\frac{t}{\lambda}$ of the silicon wedge. The box indicates the area where the profile was taken. (b): Profile of thickness map of the silicon wedge. The integration width is 200 pixels.

4.2.2 Silicon Wafer Sandwich

A sandwich, consisting of two silicon wafer plates, was prepared with the method that was described by D.A. Muller et. al. in [8]. The sample was mounted onto the pyrex in a way, that the glue line was perpendicular to the pyrex edge. The most important preparation setting are listed in table 4.5.

wedge angle:	2°
sample oscillation:	none
sample rotation:	none
adhesive sample-pyrex:	mounting wax
adhesive sample-grid:	Loctite® 460™
unmount method sample-pyrex:	upright pyrex
thickness control:	visible light microscope color
subsequent treatment:	none

Table 4.5: Preparation details of silicon wafer sandwich

Table 4.6 shows the succession of the lapping films including the setting of the Multiprep™ System at first side polishing. At second side polishing, initially the sample was polished down to a thickness of 500 μm with a grain size of 30 μm. Then, the wedge angle was introduced with the 15 μm lapping film and the process continued as shown in table 4.7.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	3	100	H_20	720 μm
15	3	100	H_20	300 μm
6	3	75	H_20	150 μm
3	3	50	H_20	50 μm
1	5	50	H_20	10 μm
0.5	5	30	Green Lube	5 μm
0.1	3	30	Green Lube	1 μm
0.04 colloidal silica	0	50		4 min

Table 4.6: Succession of the first side polishing process of the silicon-wafer-wedge with the method of D.A. Muller [8]

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	3	100	H_20	200 μm
15	3	100	H_20	250 μm
6	3	75	H_20	100 μm
3	3	50	H_20	90 μm
1	0	50	H_20	until edge red
0.5	0	30	Green Lube	4 μm
0.1	0	30	Green Lube	3 min
0.04 colloidal silica	0	50		2 min

Table 4.7: Succession of the second side polishing process of the silicon-wafer-wedge with the method of D.A. Muller [8]

Results

With the polishing recipe by D.A. Muller [8], good TEM results could be achieved, although the sample appears frayed in the picture that was made with the visible light microscope (figure 4.4).

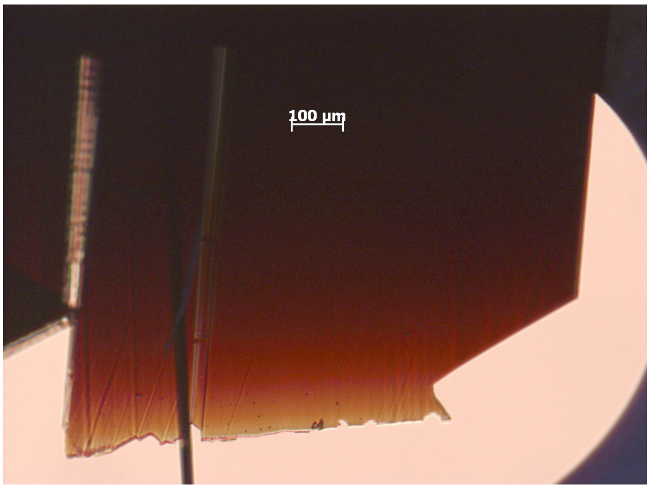


Figure 4.4: Visible light microscope photo of the silicon wafer wedge at a magnification of 50×. The color gradient from red to yellow indicates the wedge shape.

Figure 4.5 depicts a TEM bright field image of the silicon wedge. The thickness map (see figure 4.6(a)) shows that the wedge is very thin over large areas. The value for the relative thickness $\frac{t}{\lambda}$ corresponds to the color according to the legend.

Due to the profile of the thickness map (see figure 4.6(b)), the sample has a proper wedge shape. It was calculated with an integration width of 200 pixels. The pictures of the crystalline silicon microstructure are of high quality (see figure 4.7) which indicates that there is hardly any preparation induced amorphisation.

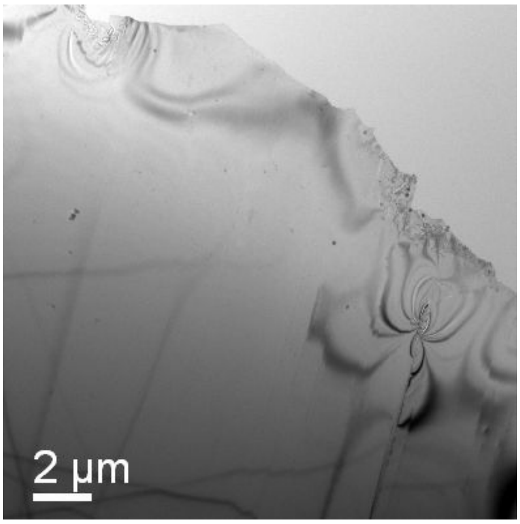
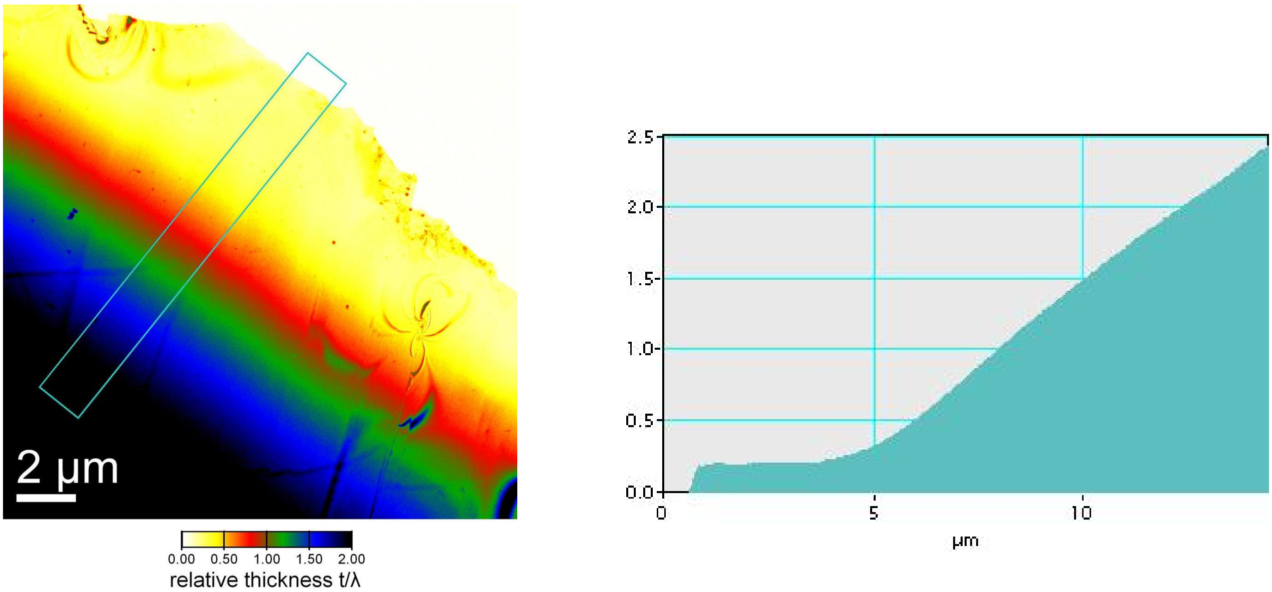


Figure 4.5: TEM bright field image of the silicon wafer wedge



(a)

(b)

Figure 4.6: (a): Relative thickness map $\frac{t}{\lambda}$ of the silicon wafer wedge. The box indicates the area where the profile was taken. (b): Profile of thickness map of the silicon wafer wedge. The integration width is 200 pixels.

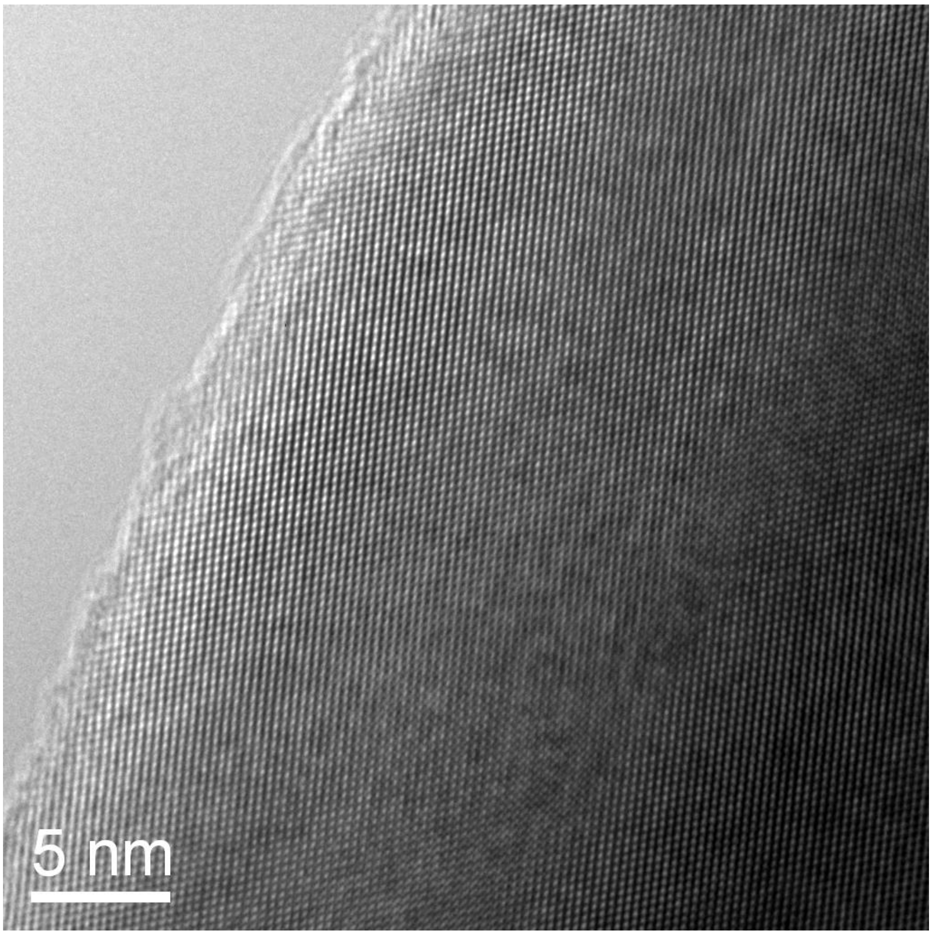


Figure 4.7: High resolution TEM image of the crystalline silicon taken along [110] zone axis

4.2.3 Silicon with Tin Oxide Layer

The sample consists of two plates of silicon substrate with a SnO_2 layer of about 60 nm, as used in gas sensors. As protection of the tin oxide, the pieces were glued together face to face, to a sandwich. The sample was glued onto the pyrex with the glue line perpendicular to the pyrex front edge. Table 4.8 shows important polishing settings.

wedge angle:	2°
sample oscillation:	at speed 3 at each grain size at 1^{st} and 2^{nd} side
sample rotation:	none
adhesive sample-pyrex:	Loctite® 435™
adhesive sample-grid:	Loctite® 435™
unmount method sample-pyrex:	tilted pyrex
thickness control:	infinite focus microscope color optical fringes
subsequent treatment:	none

Table 4.8: Preparation details of Si with SnO_2 sandwich

First side polishing was performed like listed in table 4.9. The succession of the lapping films and the corresponding sample load, rotating speed, lubricant and abrasion are shown in table 4.10. As the sample had been very slim from the beginning, no coarse grain size was applied. The wedge angle was introduced with the 3 μm lapping film.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
3	0	50	distilled H_20	200 μm
1	0	50	distilled H_20	200 μm
0.5	0	50	Green Lube	80 μm
0.1	0	50	Green Lube	60 μm
0.02 colloidal silica	0	80		2 min

Table 4.9: Succession of the first side polishing process of a silicon tin oxide wedge

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
3	0	50	distilled H_20	220 μm
1	0	50	Green Lube	40 μm
0.5	0	50	Green Lube	4 μm
0.1	0	30	Green Lube	4 min
0.02 colloidal silica	0	80		1 min

Table 4.10: Succession of the second side polishing process of a silicon tin oxide wedge

Results

The continuous sample oscillation that was applied at each polishing step provided a uniformly smooth sample surface. The photo which was taken with the visible light microscope at a magnification of 200× (see figure 4.8), shows optical fringes and a color gradient to red at the edge, which are signs of proper thinness. The silicon wedge edge is not tattered.

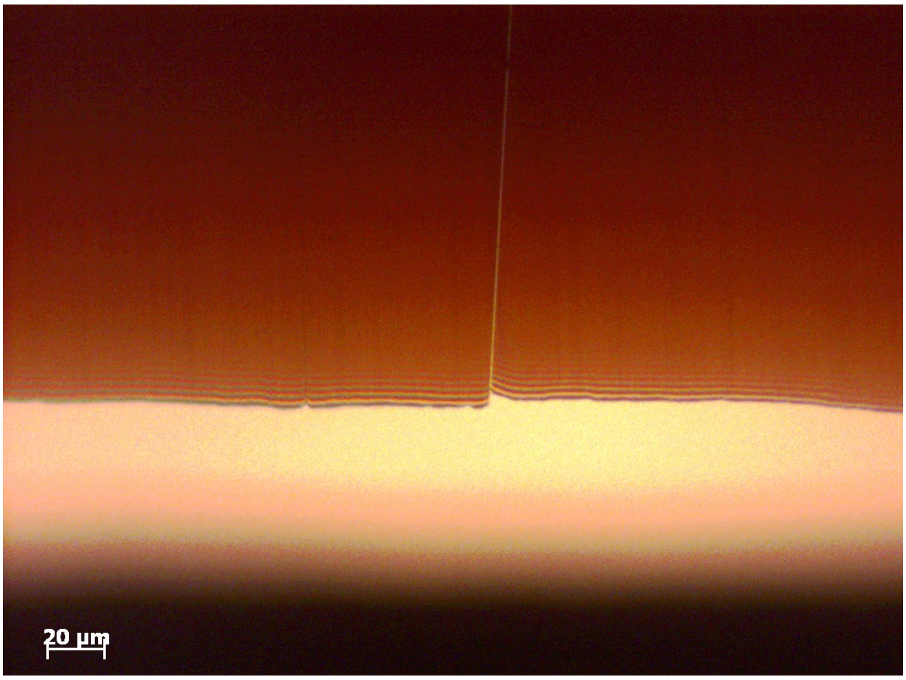


Figure 4.8: Visible light microscope photo of a silicon tin oxide wedge at a magnification of 200×. The color gradient and the optical fringes indicate the thickness of the edge.

The sample was highly suitable for TEM investigation. Figure 4.9 shows a TEM brightfield image of the central part of the sample wedge. The agglomerated spherules on the right-hand side originate from the colloidal silica suspension. The thick layer in the middle of the picture is the glue line. On the left and right side of the glue are the SnO_2 layers which are about 60 nm thick. The relative thickness map in figure 4.10(a) depicts large thin areas at the silicon substrate, and also the tin oxide layers are properly thinned over a long length. In figure 4.10(b) the profile of the thickness map is displayed. It was measured at the silicon substrate to the left of the spherules with an integration width of 200 pixels. The tin oxide layer and the crystalline silicon both could be examined accurately, and high quality high resolution images could be acquired. Figure 4.11 shows high resolution TEM pictures of the polycrystalline SnO_2 layer,whereas figure 4.12 depicts the crystalline Si in high resolution. At the upper left corner a Fast Fourier Transformation (FFT) image is added. The pattern with this kind of diffraction spots indicates crystallinity of the structure.

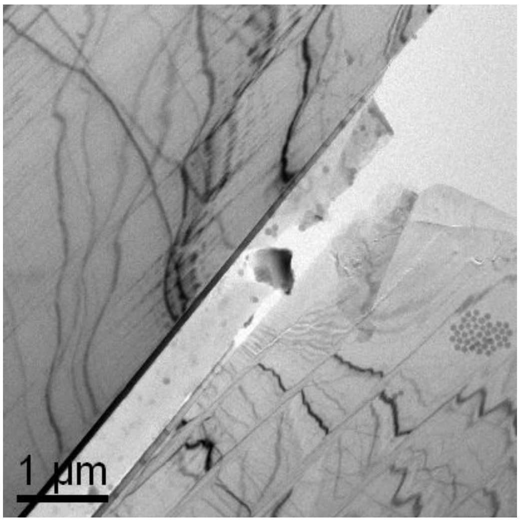
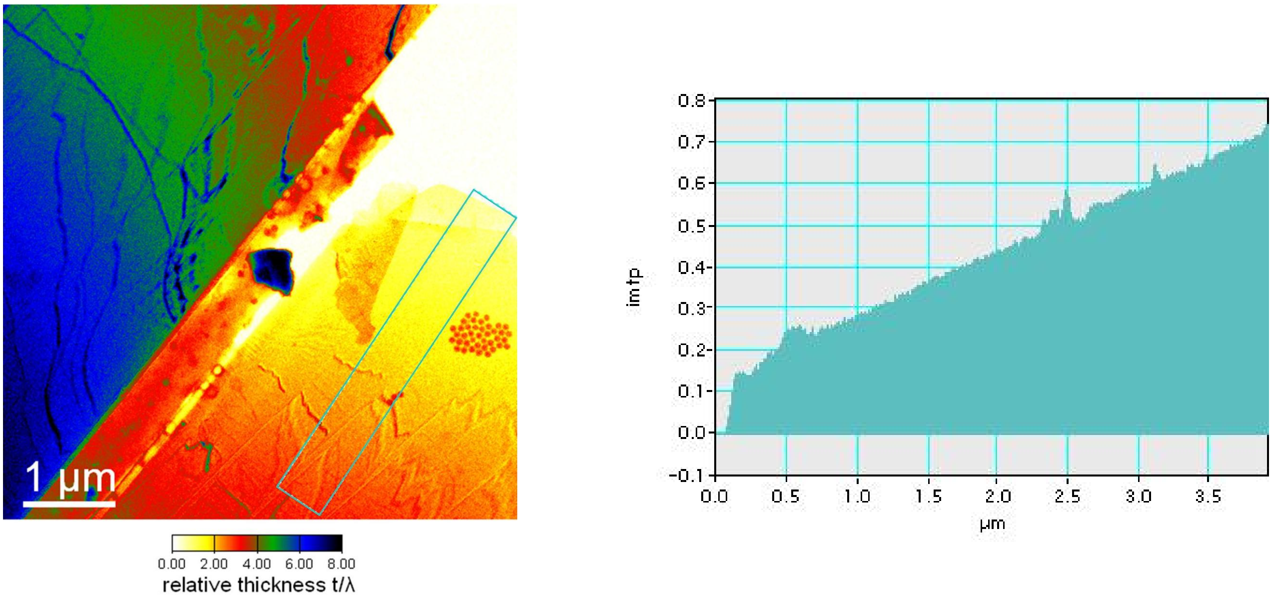


Figure 4.9: TEM bright field image of the silicon tin oxide wedge. Spherules of the colloidal silica suspension are agglomerated on the right.



(a)

(b)

Figure 4.10: (a): Relative thickness map $\frac{t}{\lambda}$ of the silicon tin oxide wedge. The box indicates the area where the profile was taken. (b): Profile of thickness map of the silicon tin oxide wedge. The integration width is 200 pixels.

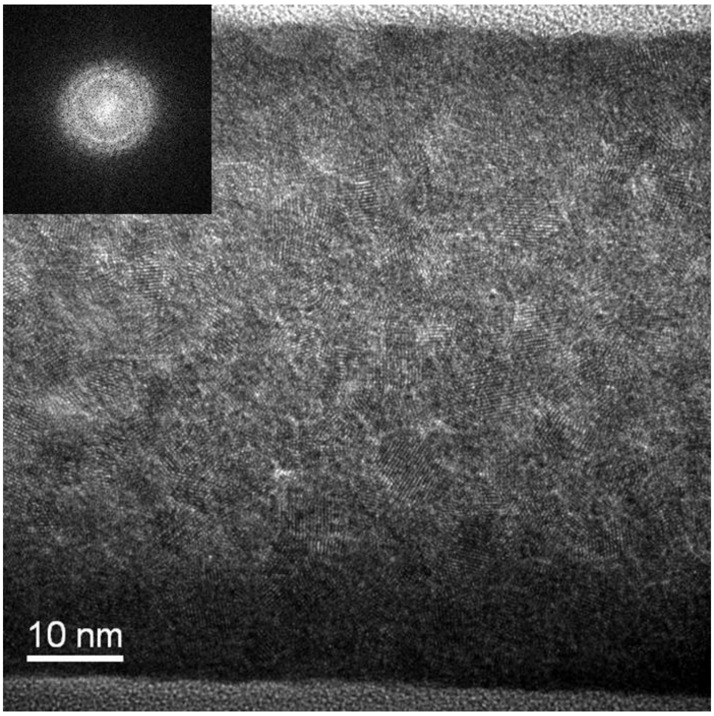


Figure 4.11: TEM high resolution bright field image and FFT of the polycrystalline tin oxide layer

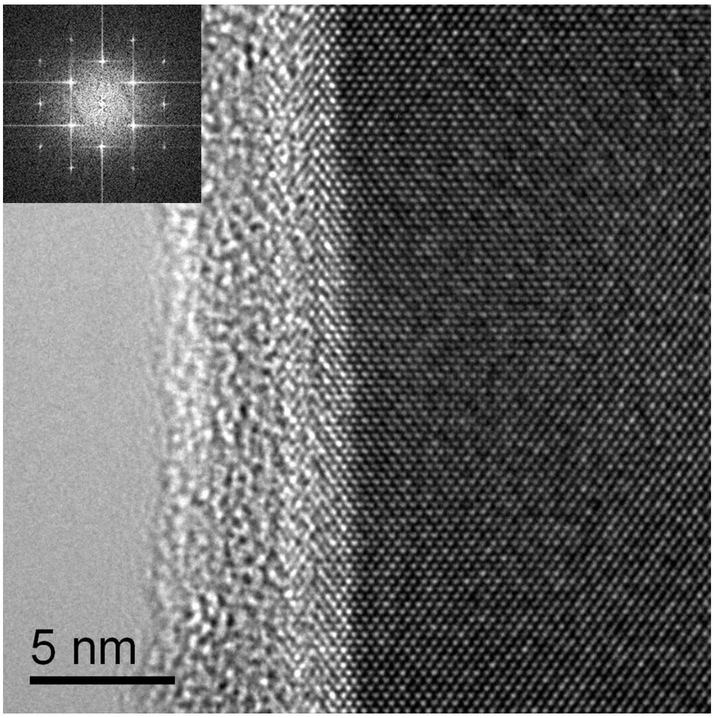


Figure 4.12: TEM high resolution image and FFT of the crystalline silicon substrate taken along [110] zone axis

The TEM investigation itself affected the sample. Due to impurities on sample and holder, or an insufficient vacuum, amorphous carbon layer grew where the electron beam hit the material. The growing was well observable at the wedge edge (see figure 4.13), but also deposition at top and bottom side occurs. This resulted in a lower image contrast (see section 2.4).

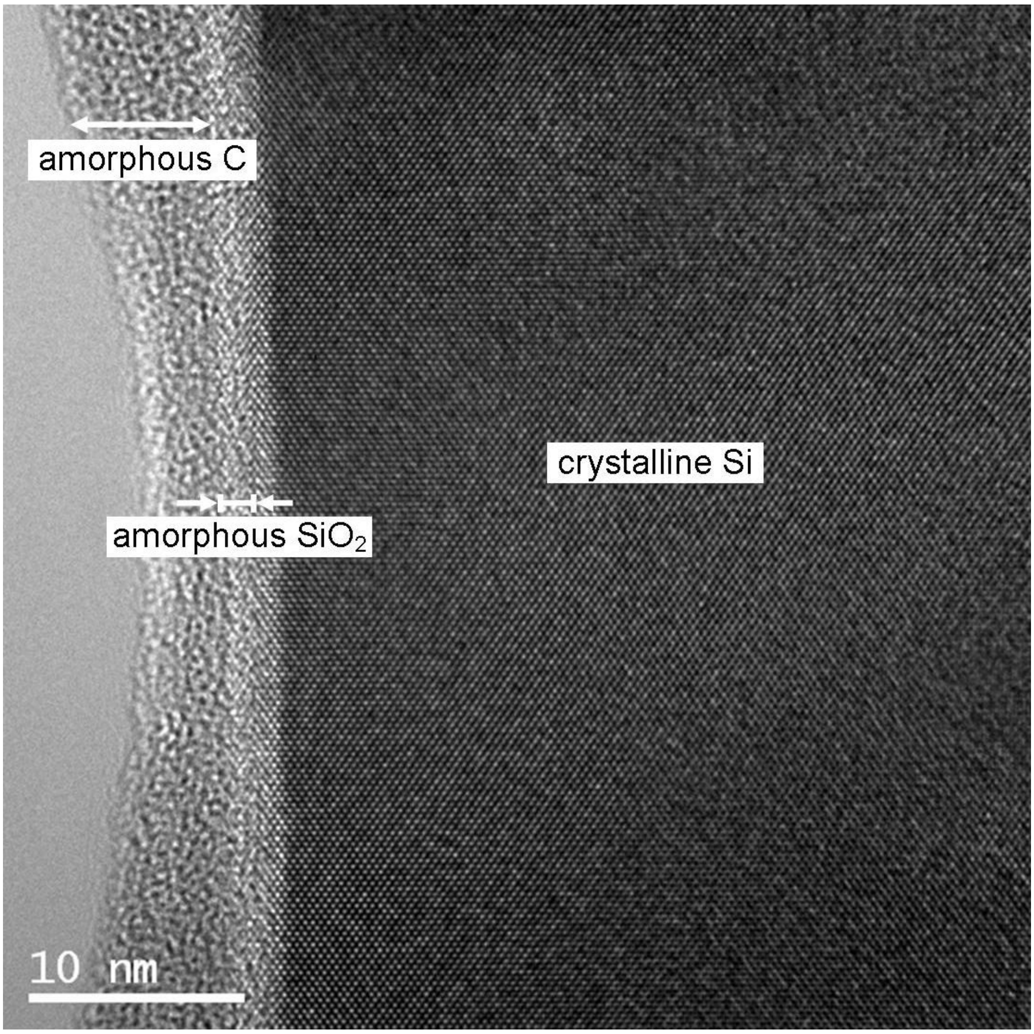


Figure 4.13: TEM high resolution image of the silicon tin oxide wedge taken along [110] zone axis: thick amorphous layer because of carbon deposition. The shape of the carbon layer is caused by unequal illumination.

4.2.4 Highly Doped Silicon

The basic material is an annealed silicon wafer that is highly doped with antimony. The concentration of Sb in the Si is inhomogeneous. It is only present within the first 50 nm of the sample.
Two platen of the wafer were glued together with the highly doped side face to face. This sample sandwich was fixed onto the pyrex so that the glue line is perpendicular to the pyrex front edge. Table 4.11 shows the general setting for this sample.

wedge angle:	2°
sample oscillation:	at speed 3 at each grain size at 1^{st} and 2^{nd} side
sample rotation:	none
adhesive sample-pyrex:	Loctite® 435™
adhesive sample-grid:	Loctite® 460™
unmount method sample-pyrex:	tilted pyrex
thickness control:	color optical fringes
subsequent treatment:	none

Table 4.11: Preparation details of Si (doped with Sb) sandwich

In table 4.12 the succession of the lapping films and the corresponding settings are listed. As the initial sample was very thick, also coarse grain sizes were applied. At second side polishing, which was performed according to table 4.13, the wedge was started at the 3 μm lapping film. This film was applied until the edge appeared red.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	0	50	distilled H_20	440 μm
15	0	50	distilled H_20	400 μm
6	0	50	distilled H_20	150 μm
3	0	50	distilled H_20	130 μm
1	0	50	distilled H_20	100 μm
0.5	0	30	Green Lube	50 μm
0.25	0	30	Green Lube	10 μm
0.1	0	30	Green Lube	5 μm
0.02 colloidal silica	0	80		2 min

Table 4.12: Succession of the first side polishing process of a highly doped silicon wedge

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
3	0	50	distilled H_20	400 μm
1	0	50	Green Lube	10 μm
0.5	0	30	Green Lube	5 min
0.25	0	30	Green Lube	5 min
0.1	0	30	Green Lube	5 min
0.02 colloidal silica	0	80		3 min

Table 4.13: Succession of the second side polishing process of a highly doped silicon wedge

Results

The fringes and color gradient (see figure 4.14) imply that the sample is of proper thinness over a large area. Nevertheless some parts of the wedge edge on the right hand side are teared off.

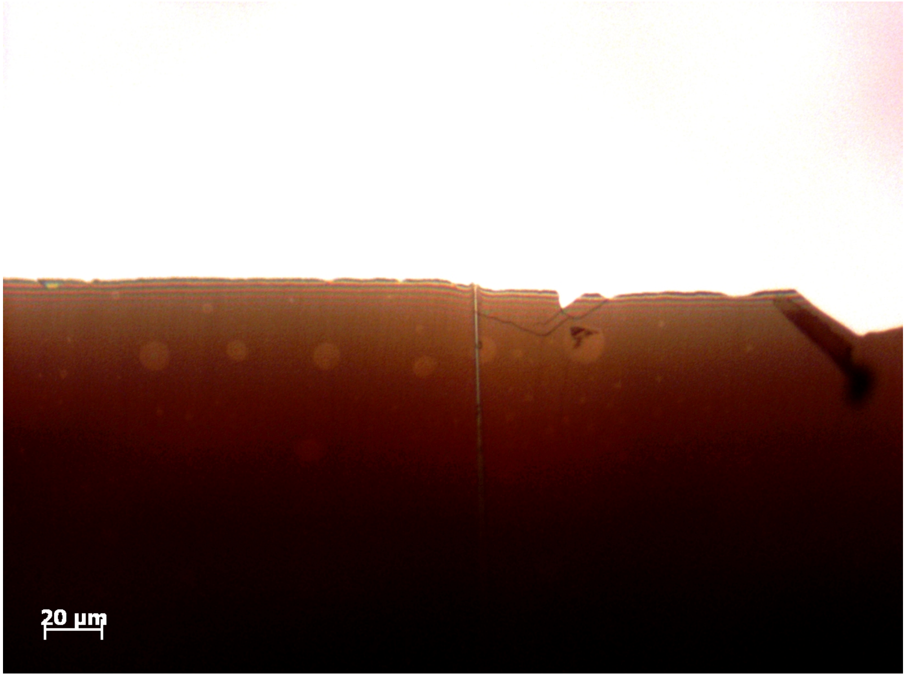


Figure 4.14: Visible light microscope photo of a silicon (doped with antimony) wedge at a magnification of 200×. The color gradient and the optical fringes indicate the thickness of the wedge.

Figure 4.15 shows a TEM bright field image of the sample. There are large very thin areas at the wedge edge, as depicted in the thickness map and the profile of thickness map 4.16. The probability for single scattering is very high for a region up to 1 μm from the edge. High resolution TEM investigation could be performed (see figure 4.17).

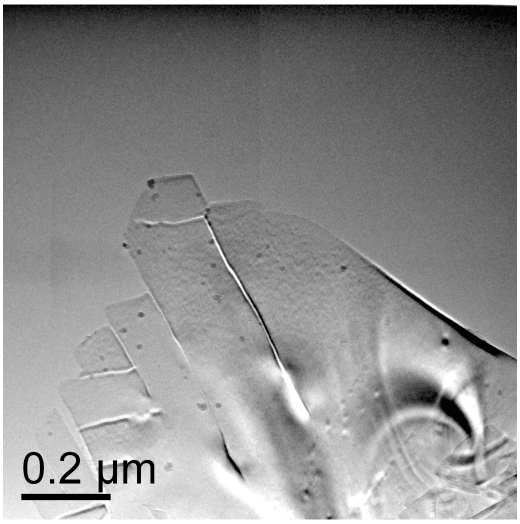
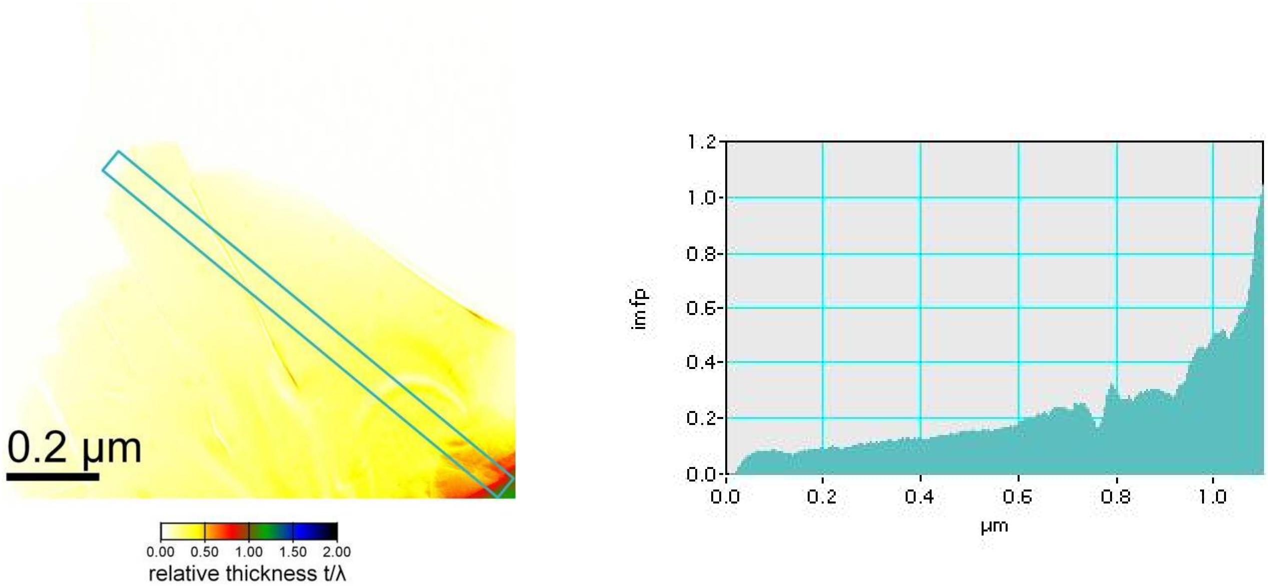


Figure 4.15: TEM bright field image of the silicon (doped with antimony) wedge



(a)

(b)

Figure 4.16: (a): Relative thickness map $\frac{t}{\lambda}$ of the silicon (doped with antimony) wedge. The box indicates the area where the profile was taken. (b): Profile of thickness map of the silicon (doped with antimony) wedge. The integration width is 100 pixels.

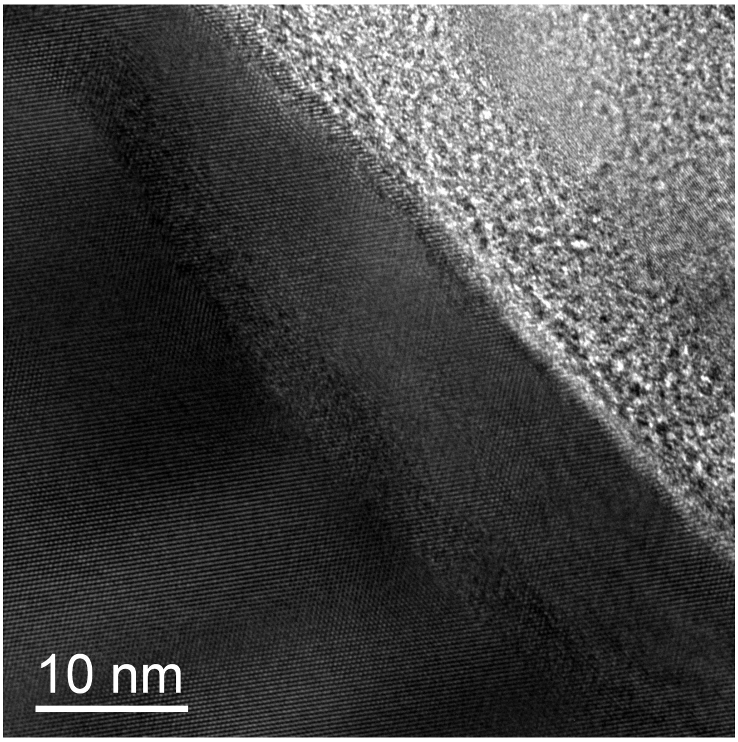


Figure 4.17: TEM high resolution image a silicon (doped with antimony) wedge taken along [110] zone axis. The bright region at the top on the right is the glue line.

4.3 Sapphire with Gallium Nitride Layer

Samples that mainly consist of sapphire are not easy to polish. They are very hard and brittle, which complicates the preparation procedure enormously. Many samples could not be thinned until electron transparency because the edge broke off before it was thin enough. When considering the mechanical instability and by polishing very gently, the procedure was very time consuming, which the adhesive between sample and pyrex often could not stand. The first samples were sandwiches of two Al_2O_3 plates with a GaN layer that were glued together face to face. Additionally they were protected with a glass cover slip on both sides. This increased polishing time, as also the glass had to be abraded.
After many unsuccessful attempts, a well working method for preparation of sapphire was found. It is described in the following subsections *4.3.1 Sapphire I* and *4.3.2 Sapphire II.* The glass cover slips were omitted, so that a sandwich only consisting of sapphire - gallium nitride - glue - gallium nitride - sapphire was obtained. The initial samples were about 400 μm in thickness, so that only a little bit of material had to be removed. It was mounted onto the pyrex with the glue line perpendicular to the pyrex front edge.

4.3.1 Sapphire I

The main setup for this sample is shown in table 4.14.

wedge angle:	4°
sample oscillation:	at speed 3 at each grain size at 1^{st} and 2^{nd} side
sample rotation:	limit rotation at speed 3 at each grain size at 1^{st} side
adhesive sample-pyrex:	mounting wax
adhesive sample-grid:	M-Bond 610
unmount method sample-pyrex:	tilted pyrex
thickness control:	infinite focus microscope optical fringes
subsequent treatment:	ion milling

Table 4.14: Preparation details of sapphire I wedge

First side polishing was performed as listed in table 4.15. The settings for second side polishing can be seen in table 4.16. As the sample was quite thin after first side polishing, the wedge angle was introduced with the first lapping film at a grain size of 6 μm. No colloidal silica was applied, to get a purely mechanical treatment.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
6	0	50	distilled H_20	80 μm
3	0	50	distilled H_20	30 μm
1	0	50	distilled H_20	60 μm
0.25	0	50	Green Lube	15 μm
0.1	0	50	Green Lube	1 min

Table 4.15: Succession of the first side polishing process of sapphire gallium nitride wedge I

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
6	0	50	distilled H_20	125 μm
1	0	50	Green Lube	9 μm
0.25	0	50	Green Lube	1.5 min
0.1	0	50	Green Lube	0.5 min

Table 4.16: Succession of the second side polishing process of sapphire gallium nitride wedge I

Results after Wedge Polishing

As the photo taken with the visible light microscope shows, the wedge edge does not appear very tattered, and the fringes indicate a low sample thickness (see figure 4.18).

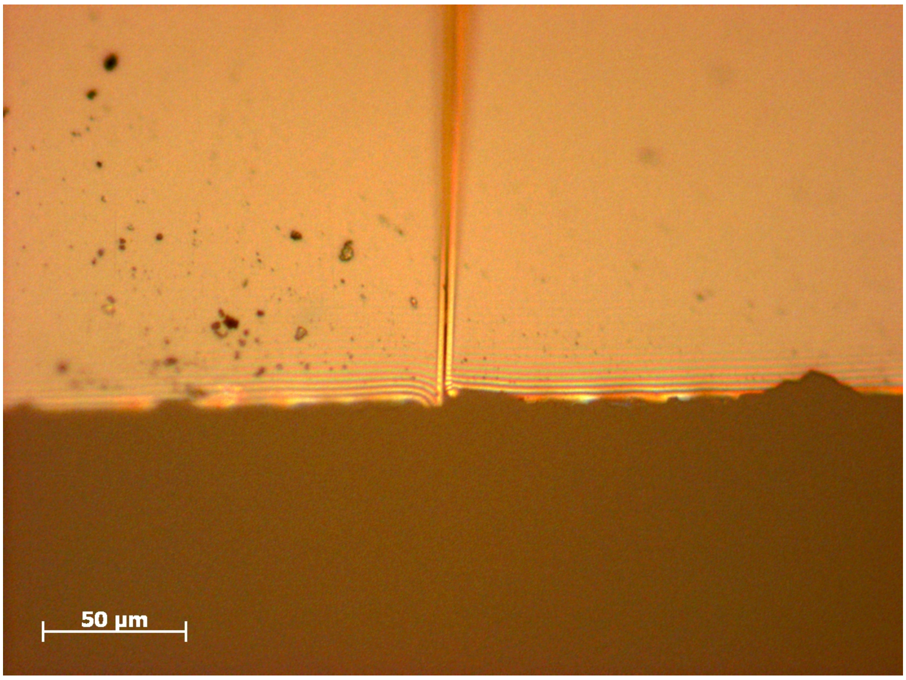


Figure 4.18: Visible light microscope photo of the sapphire gallium nitride wedge I at a magnification of 200×. The optical fringes indicate the low thickness of the edge.

Figure 4.19(a) shows a bright field image of sapphire and gallium nitride. In TEM investigation, the sample does not show good properties. The surface is rough (see figure 4.19(b)), which indicates a too short polishing time at fine grain sizes and that polishing with colloidal silica should not be omitted. Figure 4.20(a) shows the relative thickness map of the sapphire, and figure 4.20(b) the corresponding thickness map profile. It is obvious that the wedge angle of 4° is too high to obtain thin areas of adequate size and that the whole sample is too thick because of insufficient thinning. Fringes could be observed after polishing with the 1 μm lapping film.

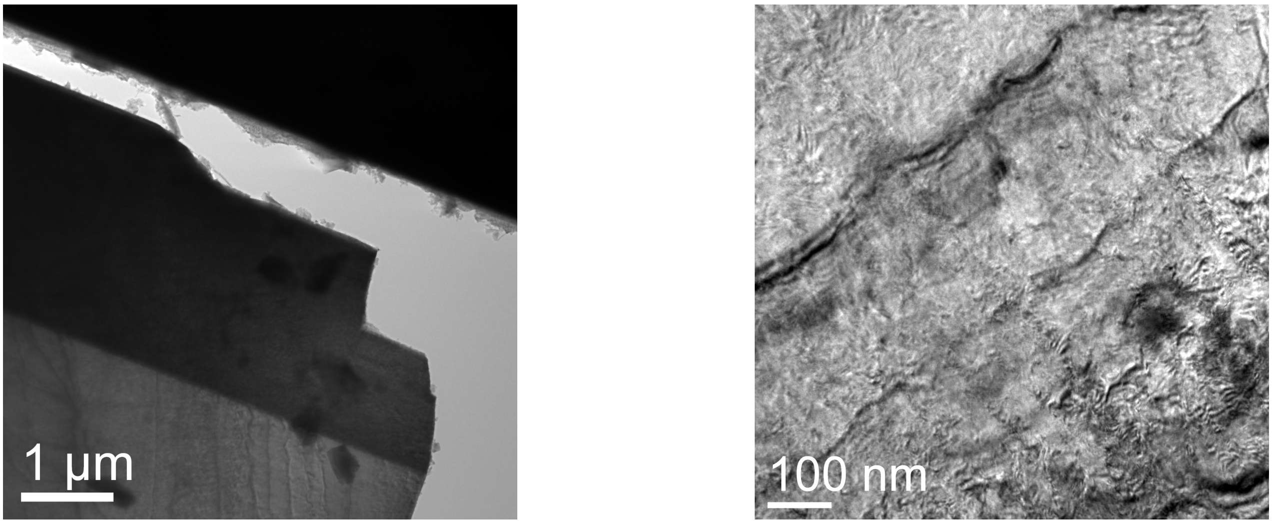
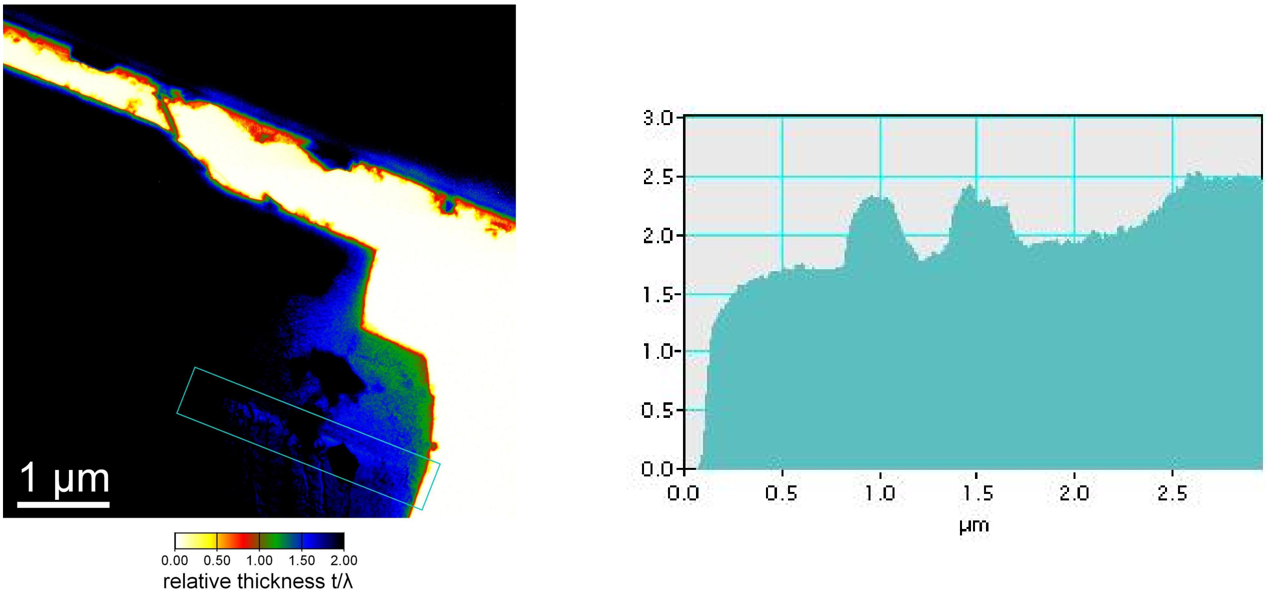


Figure 4.19: a): TEM bright field image of the sapphire I wedge before ion thinning. (b): TEM bright field image of the sapphire I wedge with higher resolution



(a)

(b)

Figure 4.20: (a): Relative thickness map $\frac{t}{\lambda}$ of the sapphire I wedge before ion thinning. The box indicates the area where the profile was taken. (b): Profile of thickness map of the sapphire I wedge before ion thinning. The integration width is 200 pixels.

Results after Ion Thinning

The sapphire I wedge was thinned with the instruments that are described in subsection 2.3.1:

- in the NEID for 100 min at 1.5 kV with an angle of ±7° (changed every 5 min)
- in the NEID for 120 min at 600 eV with an angle of ±10° (changed every 5 min)

In the unfiltered bright field image of the sample (see figure 4.21) the surface appears smoother than before ion thinning. The relative thickness map and its profile indicate a very thin areas over a wide area and a continuously increasing thickness (see figure 4.22). Therefore ion thinning highly improved the performance of the sample.

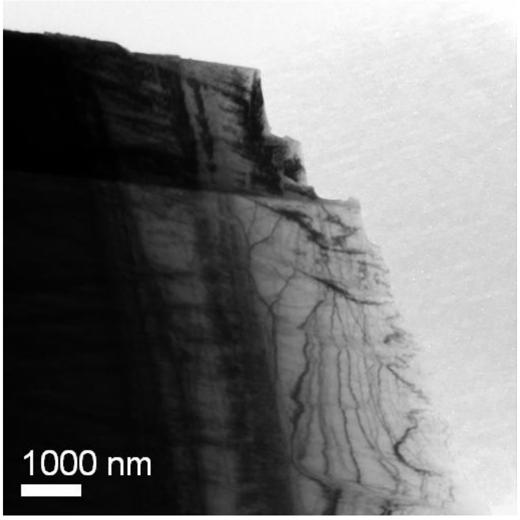
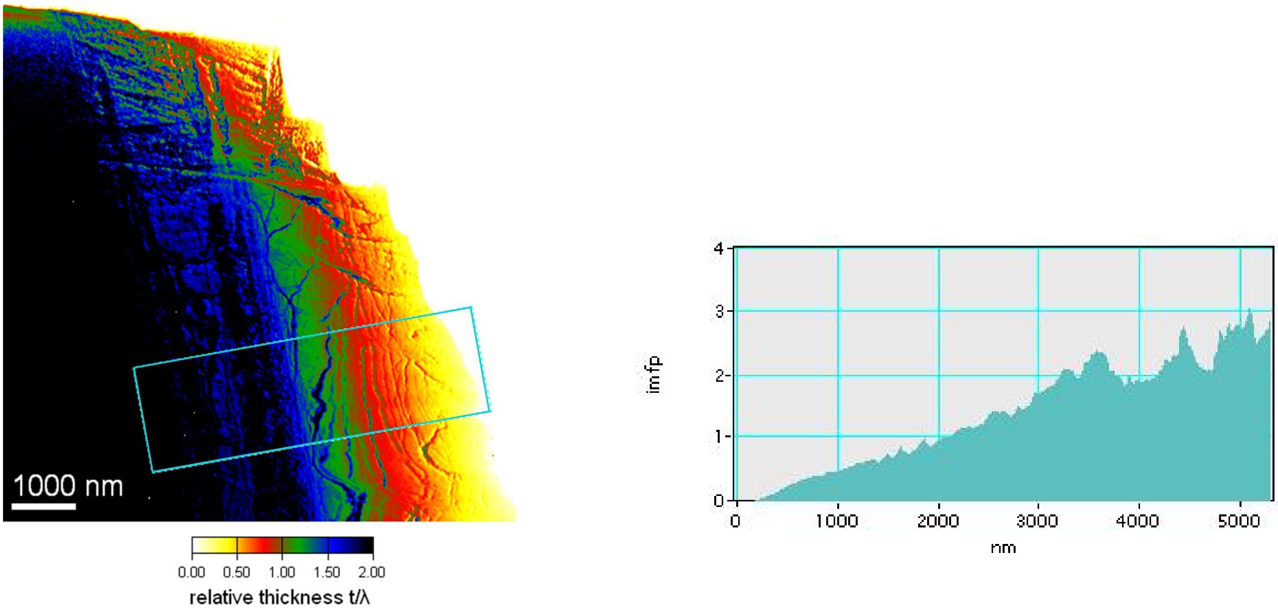


Figure 4.21: TEM bright field image of the sapphire I wedge with GaN layer after ion thinning



(a)

(b)

Figure 4.22: (a): Relative thickness map $\frac{t}{\lambda}$ of the sapphire I wedge with GaN layer after ion thinning. The box indicates the area where the profile was taken. (b): Profile of thickness map of the sapphire I wedge with GaN layer before ion thinning. The integration width is 100 pixels.

4.3.2 Sapphire II

Table 4.17 lists the preparation setup for the Al_2O_3 with GaN sample.

wedge angle:	3°
sample oscillation:	at speed 3 at each grain size at 1^{st} and 2^{nd} side
sample rotation:	none
adhesive sample-pyrex:	mounting wax at 1^{st} side Loctite® 435™ at 2^{nd} side
adhesive sample-grid:	M-Bond 610
unmount method sample-pyrex:	tilted pyrex
thickness control:	infinite focus microscope optical fringes
subsequent treatment:	none

Table 4.17: Preparation details of sapphire II wedge

The succession of first side polishing including the corresponding settings is listed in table 4.18. Table 4.19 shows how second side polishing was performed. As the sample thickness was very low, only fine lapping films were used. The wedge angle was introduced from the beginning of second side polishing at a grain size of 1 μm. This time colloidal silica also was applied. Fringes could first be observed at the end of polishing with a grain size of 1 μm.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
1	0	50	distilled H_20	40 μm
0.5	0	50	Green Lube	3 μm
0.25	0	50	Green Lube	3 min
0.1	0	50	Green Lube	4 min
0.02 colloidal silica	0	80		4 min

Table 4.18: Succession of the first side polishing process of sapphire gallium nitride wedge II

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
3	0	50	distilled H_20	100 μm
1	0	50	Green Lube	20 μm
0.1	0	50	Green Lube	2 min
0.02 colloidal silica	0	80		1 min

Table 4.19: Succession of the second side polishing process of sapphire gallium nitride wedge II

Results

The visible light microscope photo (see 4.23) of the sapphire wedge II shows a nearly smooth wedge edge and optical fringes which indicate thinness.

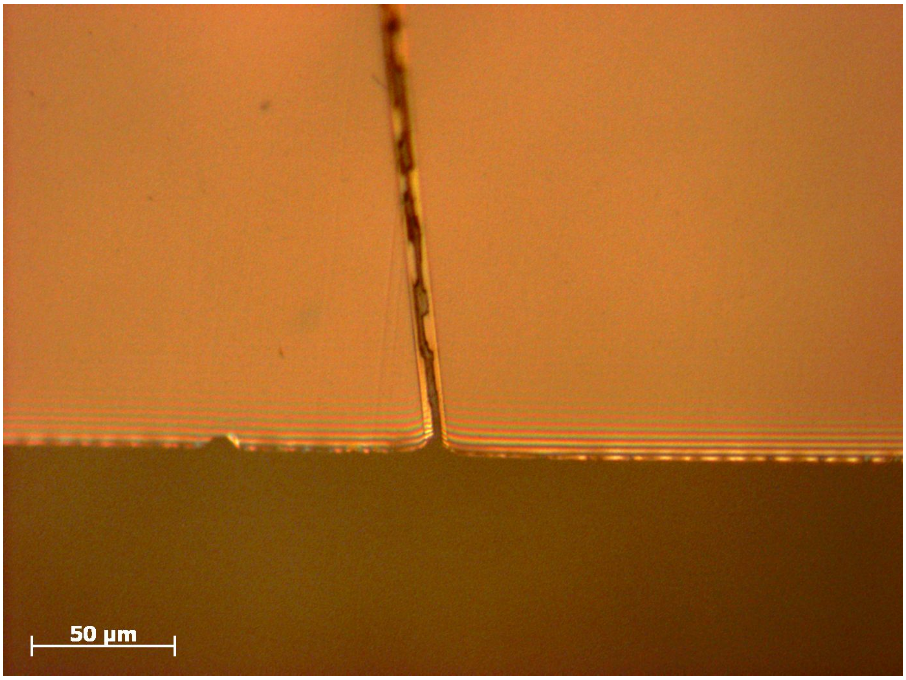


Figure 4.23: Visible light microscope photo of the sapphire gallium nitride wedge II at a magnification of 200×. The optical fringes indicate the low thickness of the edge.

In the TEM, the wedge edge appears more tattered and the surface shows inhomogeneities (see figure 4.24). Nevertheless the sample is of proper thinness in many areas of the sample. Figure 4.25(a) shows the thickness map of the GaN layer. The profile of the thickness map (see figure 4.25(b)) indicates that the gallium nitride can be investigated up to 1 μm distance from the wedge edge, without multiple scattering.

The TEM high resolution BF image (see fig 4.26) shows the crystallinity of the sample.

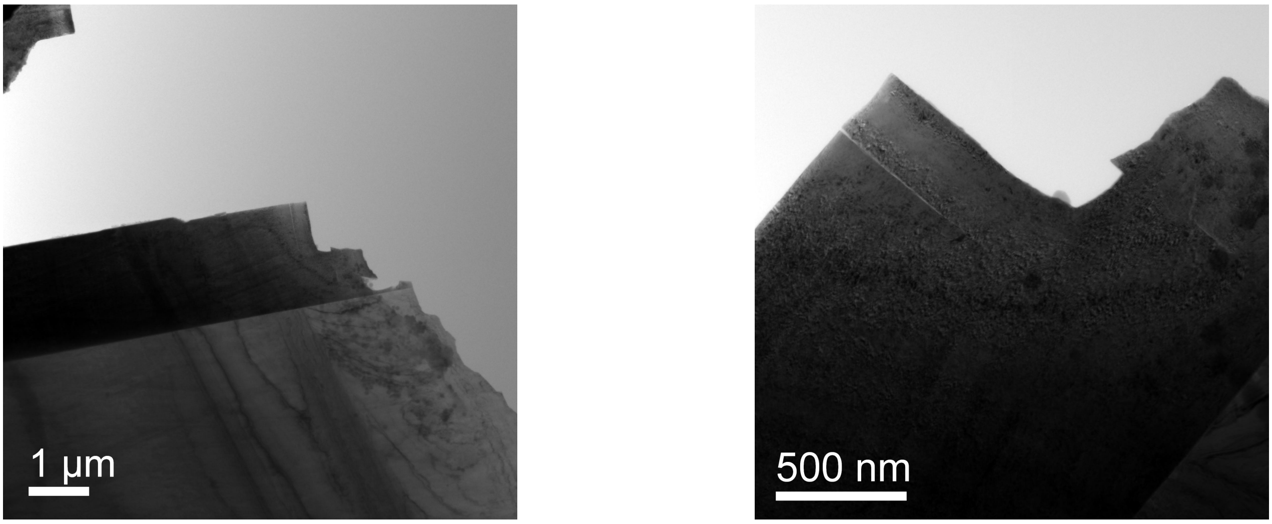
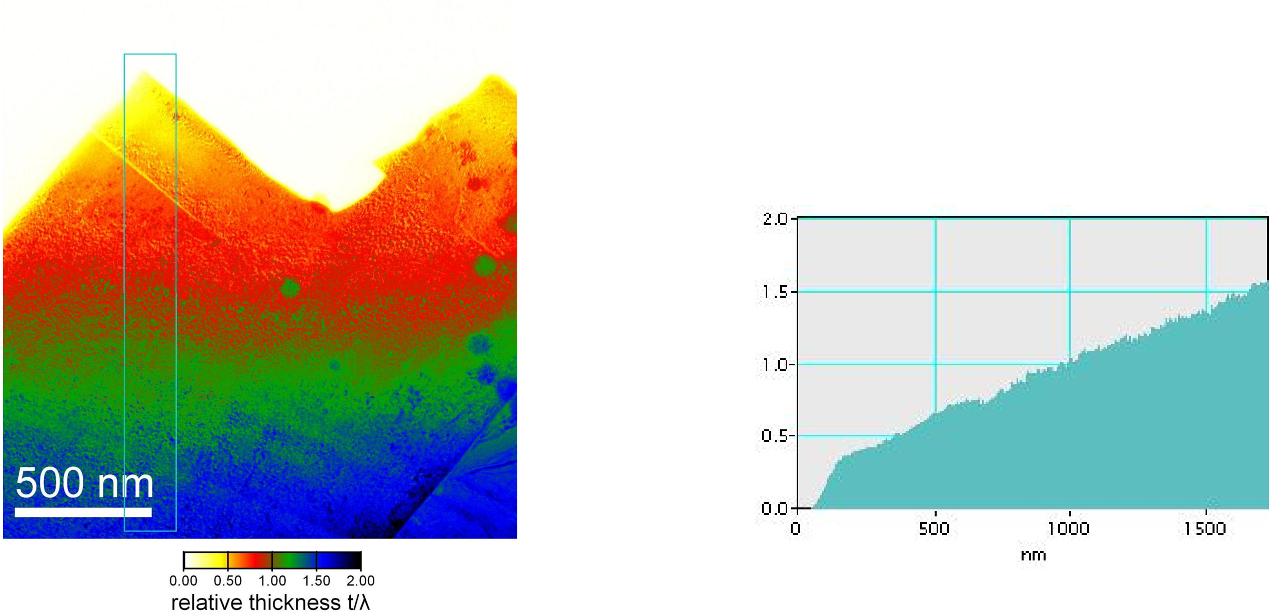


Figure 4.24: (a): TEM bright field image of the sapphire II wedge with GaN layer. (b): TEM bright field image of the GaN layer



(a)

(b)

Figure 4.25: (a): Relative thickness map $\frac{t}{\lambda}$ of the GaN layer of the sapphire II wedge. The box indicates the area where the profile was taken. (b): Profile of thickness map of the GaN layer of the sapphire II wedge. The integration width is 200 pixels.

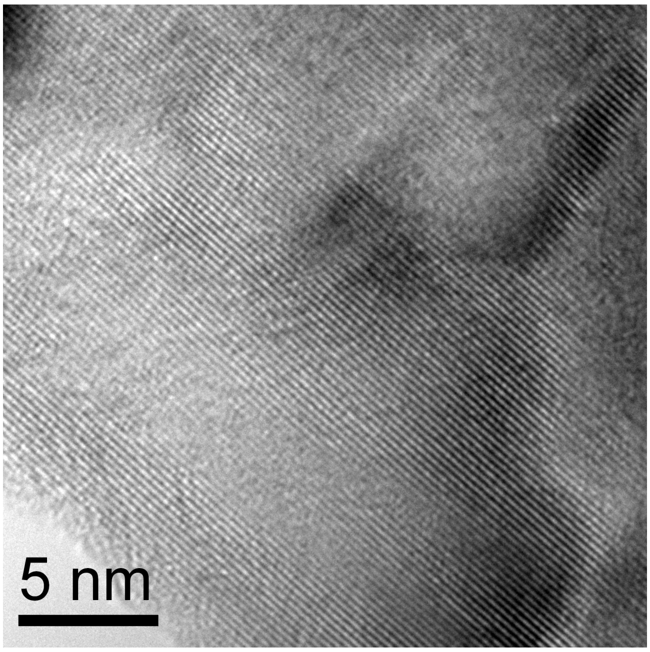


Figure 4.26: TEM high resolution bright field image of the sapphire II wedge

4.4 Tungsten Carbide with Cobalt

Tungsten Carbide (WC) is a dense, hard chemical compound. With cobalt (Co) as a binder, the properties of the material can be adjusted by the amount of cobalt. Strength, toughness and high hardness can be achieved. The prepared sample consists of WC grains of 300 to 500 nm in diameter which are distributed in Co.

4.4.1 Tungsten Carbide I

The general settings for the preparation of the WC-Co sample are shown in table 4.20.

wedge angle:	3°
sample oscillation:	none
sample rotation:	none
adhesive sample-pyrex:	mounting wax
adhesive sample-grid:	Loctite® 435™
unmount method sample-pyrex:	tilted pyrex
thickness control:	infinite focus microscope
subsequent treatment:	ion milling

Table 4.20: Preparation details of tungsten carbide I

In table 4.21 the setup for first side polishing is listed. Initially the sample was thinned with a grain size of 30 μm until a smooth, even surface was obtained. This was done with a sample load of “0” to avoid scratches on the lapping film due to the initially sharp-edged material. Table 4.22 shows the second side polishing process. With the the first lapping film, the sample was thinned until an even surface and a thickness of 500 μm was obtained. The wedge angle was introduced with the 15 μm lapping film.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	0	100	H_20	until even
15	1	80	H_20	260 μm
6	5	80	H_20	150 μm
3	5	60	H_20	70 μm
1	5	60	H_20	20 μm
0.5	5	50	Green Lube	8 μm
0.1	5	50	Green Lube	3 μm
0.02 colloidal silica	0	80		3 min

Table 4.21: Succession of the first side polishing process of tungsten carbide with cobalt wedge I

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
30	0	100	H_20	until 500 μm
15	0	80	H_20	200 μm
6	0	80	H_20	210 μm
3	0	50	H_20	20 μm
1	0	50	Green Lube	10 μm
0.5	0	30	Green Lube	3 min
0.1	0	30	Green Lube	2 min
0.02 colloidal silica	0	50		2.5 min

Table 4.22: Succession of the second side polishing process of tungsten carbide with cobalt wedge I

Results after Wedge Polishing

The photo, taken with the visible light microscope (see figure 4.27), shows that the surface of the sample is not completely smooth. There are many grooves which originate from polishing without sample oscillation. The wedge edge appears wavy.

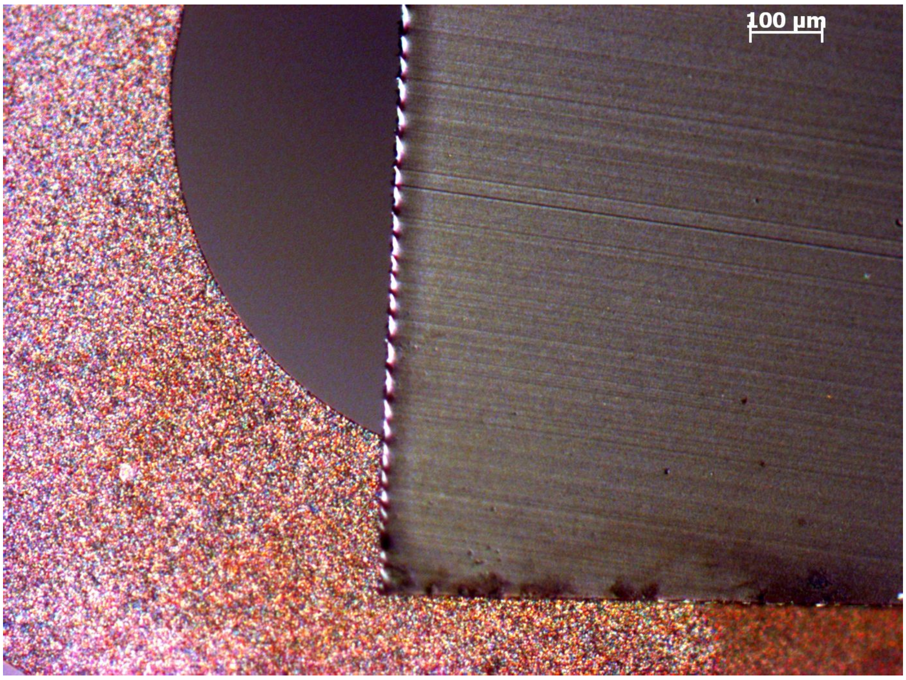


Figure 4.27: Visible light microscope photo of the tungsten carbide with cobalt wedge I at a magnification of 50×.

The WC-Co wedge I could be investigated via TEM properly as there were many thin areas over the whole wedge edge. The sample consists of different grains, with various crystal orientations. These grains tear off when the sample gets too thin. Therefore the wedge edge does not proceed straight, but along the former grain boundaries (see figure 4.28) Due to the different materials with differing abrasion properties, the wedge thickness does not decrease steadily and shows discontinuities in the relative thickness map (see figure 4.29(a)) and the profile of thickness map (see figure 4.29(b)). The thickness map and its profile also get influenced by the different mean free paths of the grains and the binder ($\lambda_{Co} \approx 1.2\lambda_{WC}$). Nevertheless this effect is small in comparison to the different abrasion rates. Figure 4.28 and 4.30 show that the WC-Co sample exhibits many defects where the crystalline structure is disturbed. It is very likely that these damages are due to preparation.

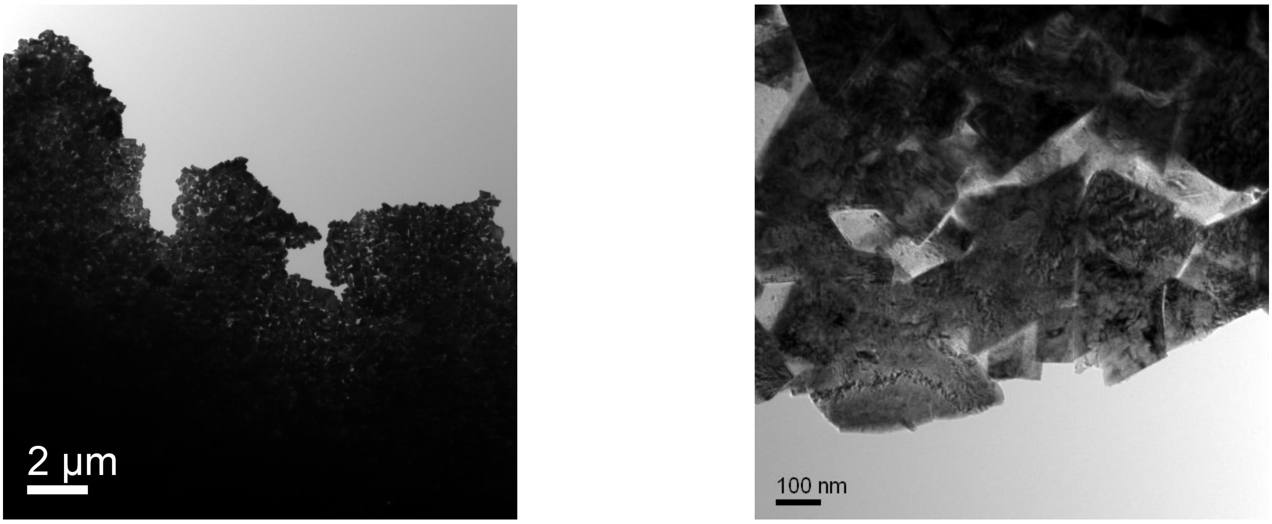
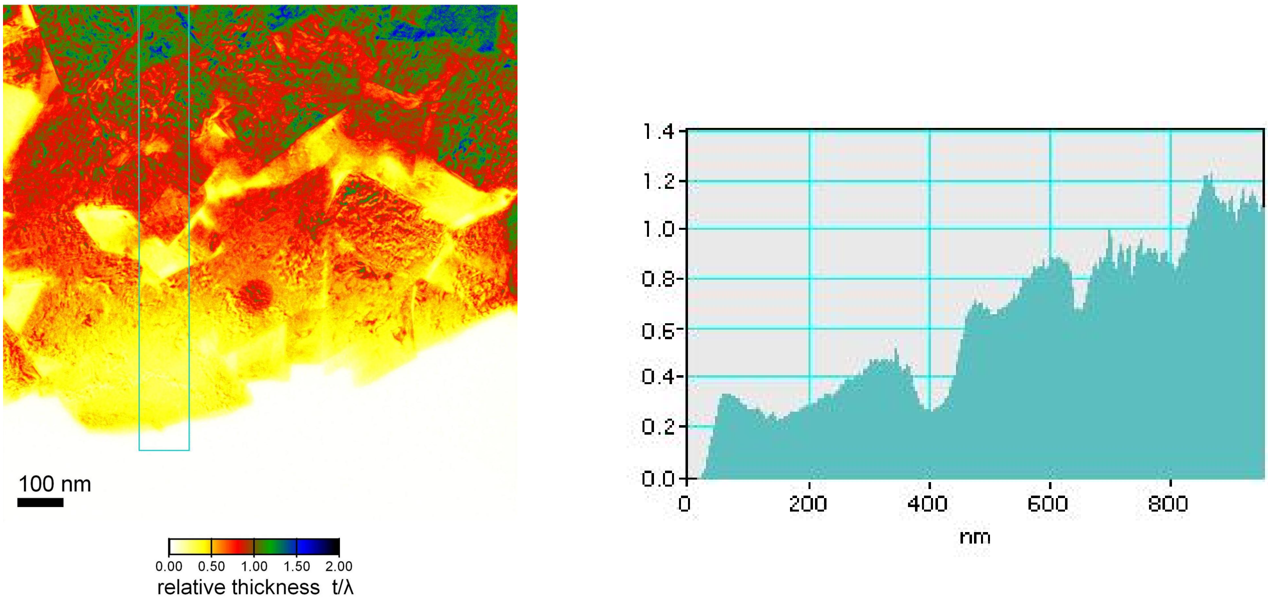


Figure 4.28: (a): TEM bright field overview image of the tungsten carbide with cobalt wedge I. (b): TEM bright field image of the tungsten carbide with cobalt wedge I



(a)

(b)

Figure 4.29: (a): Relative thickness map $\frac{t}{\lambda}$ of the tungsten carbide with cobalt wedge I before ion thinning. The box indicates the area where the profile was taken. (b): Profile of thickness map of the tungsten carbide with cobalt wedge I before ion thinning. The integration width is 200 pixels.

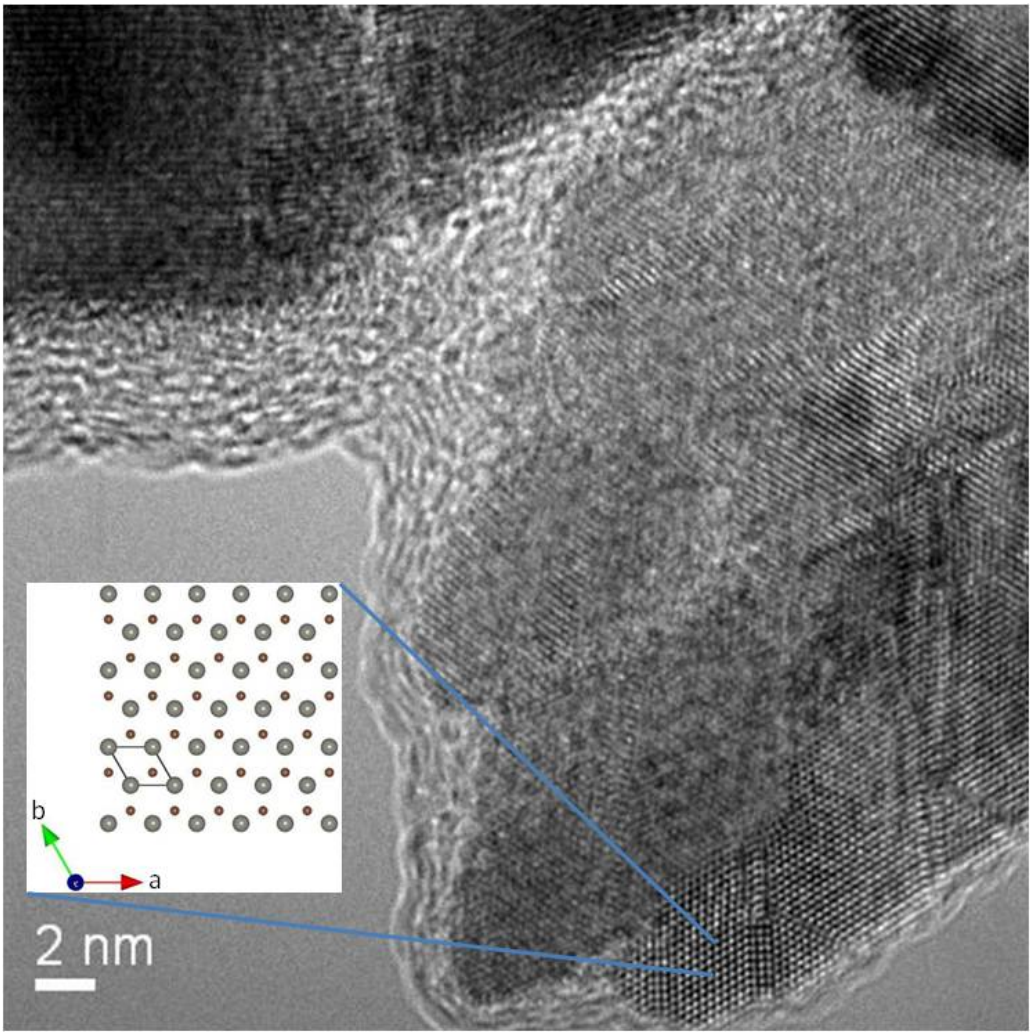


Figure 4.30: TEM high resolution bright field image of the tungsten carbide with cobalt wedge I taken along [001] zone axis. A schematic picture depicts the hexagonal crystalline structure of the marked area within the grain.

Results after Ion Thinning

The WC-Co wedge was thinned with two instruments that are described in subsection 2.3.1:

- in the PIPS for 2 h at 4 kV with angles of 4° from above and 6° from beneath
- in the NEID for 40 min at 600 eV with an angle of ±7° (changed every 5 min) and a rotation of 60°

Figure 4.31 shows a bright field TEM image of the sample. Ion thinning decreased the thickness of the wedge significantly (see 4.32(a)). The profile of the thickness map indicates that the sample can be investigated up to 3.5 μm with a low probability of multiple scattering (see figure 4.32(b)). As the TEM bright field image of medium resolution depicts, the areas with crystal defects are sputtered away (see figure 4.33). In comparison with the images before ion thinning, the quality has increased because the sample is thinner and shows less defects. This is indicated by the tungsten carbide grains, which showed inhomogeneities due to a changing orientation contrast, while after ion thinning the grains appeared more homogeneously. The disadvantage of the ion thinned WC-Co wedge I is, that its outside margin bends and curls during irradiation with the electron beam, and that the sample shows a comparable large amorphisation layer.

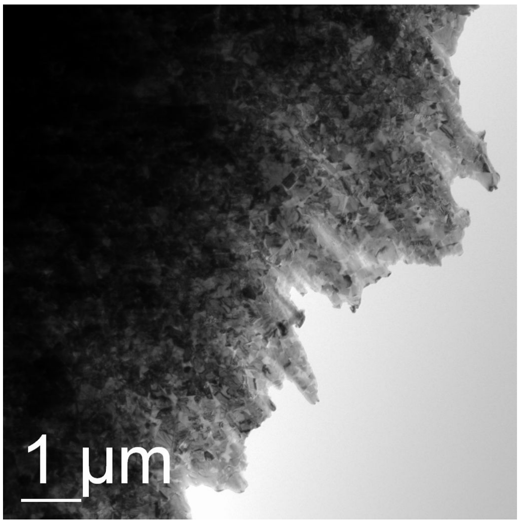
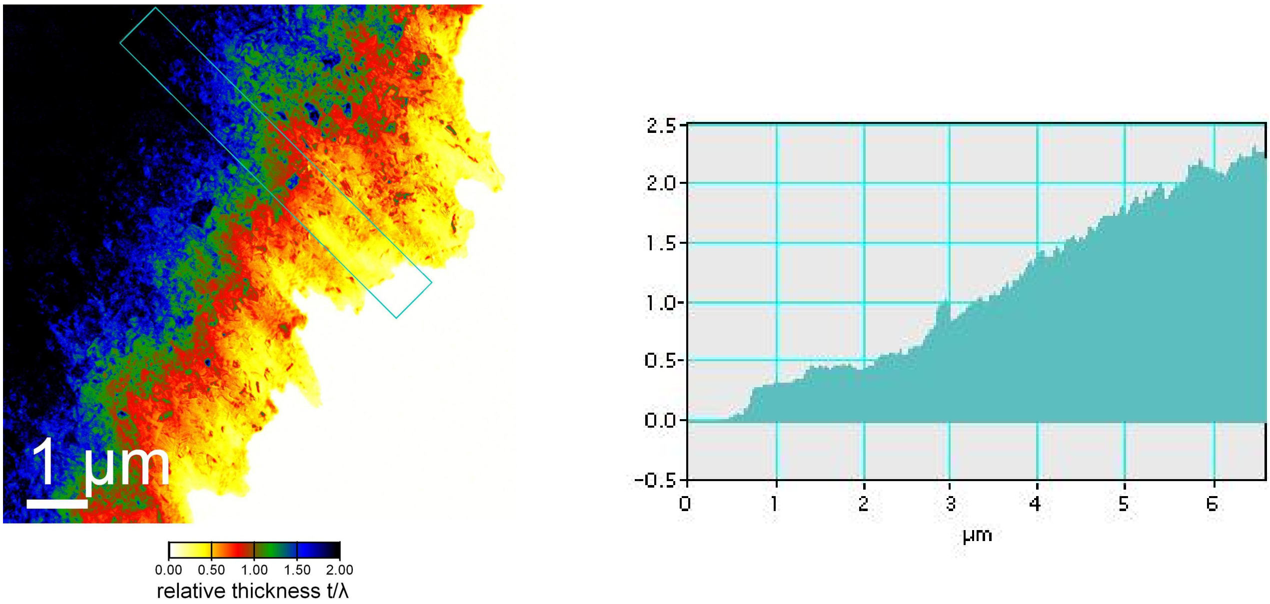


Figure 4.31: TEM bright field image of the ion thinned tungsten carbide with cobalt wedge I



(a)

(b)

Figure 4.32: (a): Relative thickness map $\frac{t}{\lambda}$ of the tungsten carbide with cobalt wedge I after ion thinning. The box indicates the area where the profile was taken. (b): Profile of thickness map of the tungsten carbide with cobalt wedge I after ion thinning. The integration width is 200 pixels.

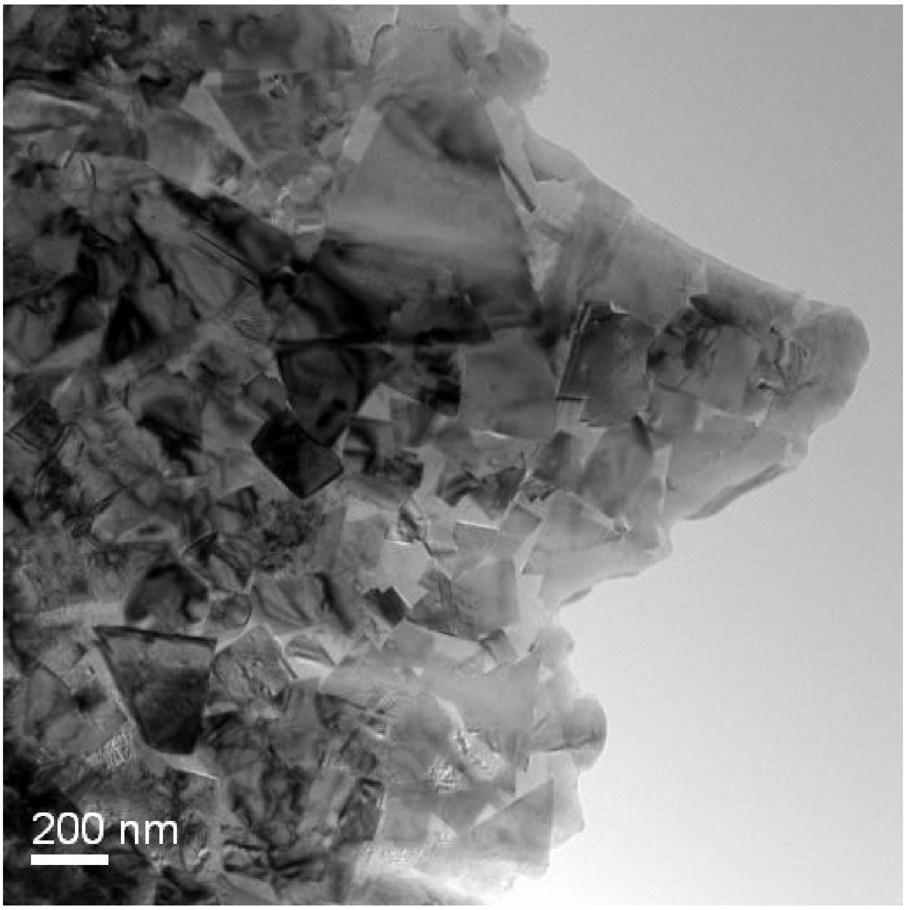


Figure 4.33: TEM bright field image of the ion thinned tungsten carbide with cobalt wedge I

4.4.2 Tungsten Carbide II

Tungsten carbide sample II was polished with a procedure that was similar to the one used for sapphire, because the two materials show a comparable hardness. The general setup is shown in table 4.23.

wedge angle: 2°

sample oscillation: at speed 3
at each grain size
at 1^{st} and 2^{nd} side

sample rotation: none

adhesive sample-pyrex: mounting wax at 1^{st} side
Loctite® 435™ at 2^{nd} side

adhesive sample-grid: Loctite® 435™

unmount method sample-pyrex: tilted pyrex

thickness control: infinite focus microscope

subsequent treatment: none

Table 4.23: Preparation details of tungsten carbide II

The first side polishing conditions are shown in table 4.24. Again the procedure was started at load “0” to prevent the lapping film from scratching due to the initially sharp-edged material. The second side polishing procedure and its corresponding settings are listed in table 4.25. Initially the sample was thinned to approximately 500 μm. The wedge angle was introduced at a grain size of 6 μm.

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
15	0	80	distilled H_20	200 μm
6	3	80	distilled H_20	50 μm
3	3	60	distilled H_20	20 μm
1	5	60	distilled H_20	30 μm
0.5	3	50	Green Lube	3 μm
0.1	3	50	Green Lube	10 min
0.02 colloidal silica	0	80		3 min

Table 4.24: Succession of the first side polishing process of tungsten carbide with cobalt wedge II

grain size [μm]	load	rotating speed [rpm]	lubricant	abrasion
15	0	80	distilled H_20	350 μm
6	0	80	distilled H_20	200 μm
3	0	60	distilled H_20	20 μm
1	0	60	distilled H_20	15 μm
0.5	0	30	Green Lube	1 μm
0.25	0	50	Green Lube	4 min
0.1	0	30	Green Lube	5 min
0.02 colloidal silica	0	80		3 min

Table 4.25: Succession of the second side polishing process of tungsten carbide with cobalt wedge II

Results

A smooth, homogeneous surface was obtained by applying sample oscillation (see figure 4.34). Nevertheless the wedge edge appears corrugated again.

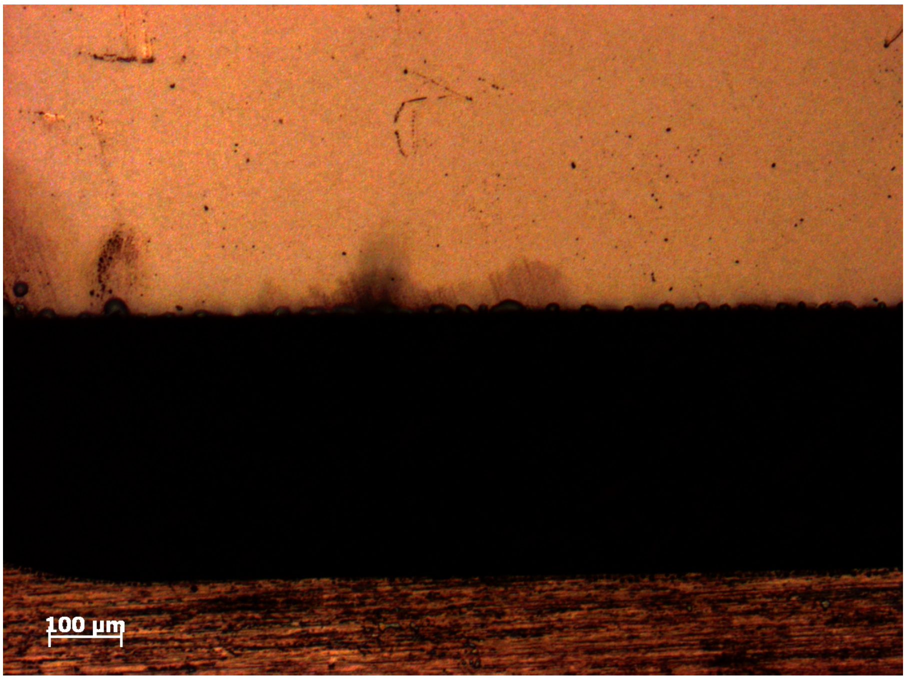


Figure 4.34: Visible light microscope photo of the tungsten carbide with cobalt wedge II at a magnification of 50×.

The TEM bright field images of the WC-Co II sample (see figure 4.35) show many defects of the crystal structure of the individual grains. They are supposed to be induced by the polishing procedure. Though polished with a lower wedge angle, the tungsten carbide wedge II shows a smaller thin area than wedge I (see figure 4.36(a)). The grains tend to break off more easily at a wedge angle of 2°. As with tungsten carbide I, the grains also cause a discontinuous profile in the thickness map (see 4.36(b)) and unsteady wedge edges.

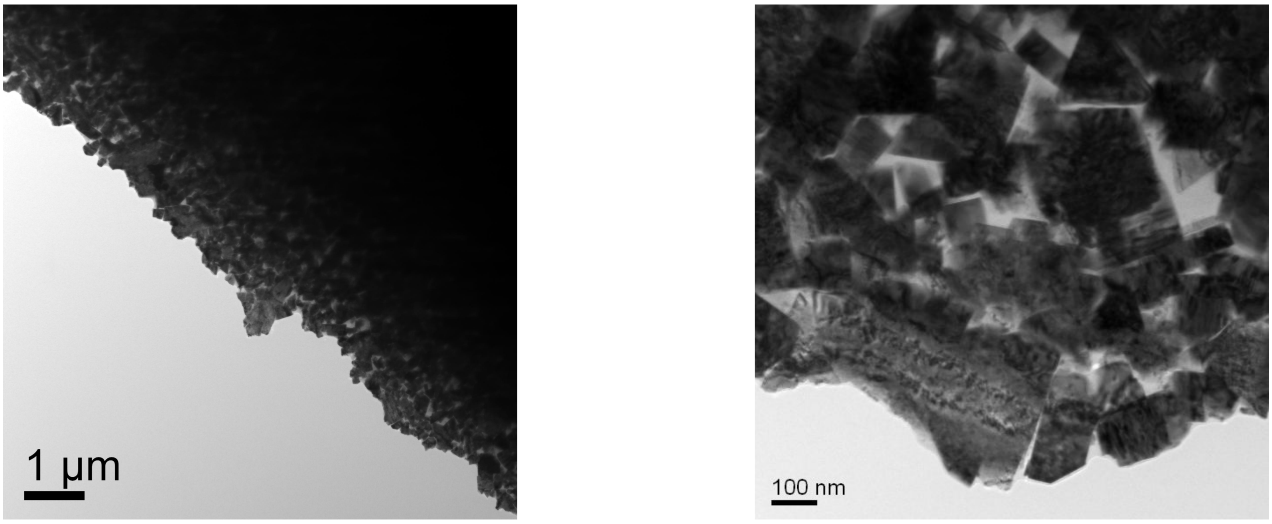
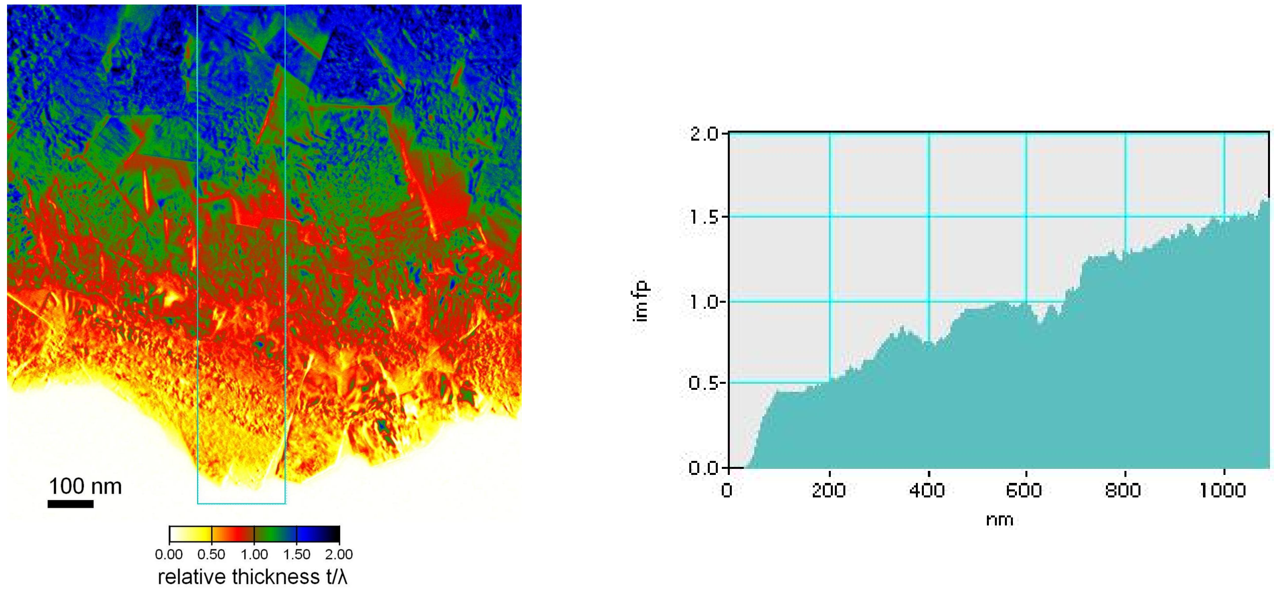


Figure 4.35: (a): TEM bright field overview image of the tungsten carbide with cobalt wedge II. (b): TEM bright field image of the tungsten carbide with cobalt wedge II



(a)

(b)

Figure 4.36: (a): Relative thickness map $\frac{t}{\lambda}$ of the tungsten carbide with cobalt wedge II. The box indicates the area where the profile was taken. (b): Profile of thickness map of the tungsten carbide with cobalt wedge II. The integration width is 200 pixels.

5 Conclusion

Wedge polishing as a sample preparation method can be applied to a variety of materials from very hard to medium hardness (see subsection 2.3.9) so that the final samples fulfill the conditions according to the methodology of TEM (see section 2.1). They may consist of bulk material and mono- or multilayer systems, but also of fine particles, which have to be embedded previously. Wedge polishing is only unsuitable if the sample consists of soft, ductile or biological material. The preparation time is about several hours. In some cases, subsequent ion thinning is recommendable.

Preparation induced amorphous layers occur due to mechanical stress during the polishing process (see subsection 2.4.1). Their thickness is proportional to the grain size of the abrasive, and they can be removed with a finer lapping film plus a last chemical-mechanical polishing step. This way the amorphous layer of the final sample can be reduced to a minimum.

The polishing behavior of, and therefore the polishing procedure for a sample, depends on its main component. In this thesis, samples which mainly consist of silicon, sapphire and tungsten carbide were prepared with the semiautomatic wedge polishing instrument *Multiprep*^{TM} *System* from *Allied High Tech Products, Inc.*

The quality of all samples was checked in the TEM by images and relative thickness maps, which depict the sample thickness in units of the mean free path ($\frac{t}{\lambda}$) in dependence of the spatial coordinates. In some cases also high resolution investigations could be performed.

Silicon (Si)

Silicon is highly suitable for wedge polishing. The most successful polishing procedure was applied to a silicon substrate with a tin oxide (SnO_2) layer and is described in subsection 4.2.3. For the silicon area, $\frac{t}{\lambda}<1$ at positions up to 4 μm from the edge. But also the tin oxide layer was suitably thin and therefore high quality high resolution images of both, the crystalline Si and the polycrystalline SnO_2 layer could be obtained. The important points of the polishing procedure are to apply sample oscillation and a load of "0", as well as to use Green Lube as lubricant at medium and small grain sizes. Due to sample oscillation, the surface gets homogeneously smooth and the abrasion rate is increased. As adhesive Loctite® 435$^{\text{TM}}$, which is very reliable, was used during the polishing process.

Also the procedures developed for a silicon wafer wedge (see subsection 4.2.2) and for silicon doped with antimony (see subsection 4.2.4) led to samples that were suitable for high resolution TEM investigations. The final amorphisation layer was very thin (<1.5 nm) for all silicon samples.

Sapphire (Al_2O_3)

Samples with Sapphire as main component are difficult to polish due to the high hardness and brittleness of this material. The polishing time is long because despite of the hardness, only low loads can be applied to avoid breaking. This is a great challenge for the adhesive that was used for mounting the sample onto the pyrex. In addition the wedge edge tends to tear off before electron transparency is obtained. Subsection 4.3.2 shows the procedure for the sapphire II wedge, which gave the best polishing results for this material. The Al_2O_3 sandwich with gallium nitride (GaN) layers was polished at medium speed with sample oscillation and zero load. At smaller grain sizes Green Lube was applied as lubricant. It is important to reduce the polishing time by starting with a sample of a small size, so that not too much material has to be removed. Sample oscillation provides a homogeneous smooth surface and increases the abrasion rate. The reliable adhesive Loctite® 435™ was used at second side polishing. As opposed to sapphire wedge I (see subsection 4.3.1), the wedge angle of 3° at sapphire II provided electron transparent areas with a sufficient size. The GaN layer could be investigated up to 1 μm distance from the wedge edge with $\frac{t}{\lambda}<1$. The high resolution image showed very good crystallinity of the sample.

Ion thinning can be applied as a subsequent treatment if the electron transparent area is not large enough. To avoid thick amorphisation layers, it is recommendable to perform ion polishing at low energies.

Tungsten Carbide in Cobalt (WC-Co)

The prepared samples consist of tungsten carbide grains in a cobalt binder. WC-Co is a composite material that is characterised by its strength, toughness and high hardness. This material could be polished so that a sufficiently large electron transparent area was obtained. The polishing results of WC-Co I (see subsection 4.4.1) and WC-Co II (see subsection 4.4.2) are very similar. Due to the different grains with various crystal orientations and the different abrasion rates of the WC and Co, the wedge edge and sample surface proceed along former grain boundaries. Though polished with a higher wedge angle, the tungsten carbide wedge I shows a larger thin area than wedge II. The distance from the wedge edge where $\frac{t}{\lambda}<1$, is 800 nm for the first, and 700 nm for the second sample. Therefore the grains tend to tear off less easily at a wedge angle of 3° than at 2°. Sample oscillation and Green Lube was applied during preparation and mounting wax was used for mounting the sample onto the pyrex. In the TEM investigation, both WC-Co samples showed crystal defects which are likely caused by polishing. With subsequent ion thinning of tungsten carbide I, the defect regions could be sputtered away and also the electron transparent area was increased. Nevertheless, the ion thinned sample shows comparably large amorphised layer. Therefore it is recommendable to remove the defect areas by low energy ion polishing.

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