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# MASTER'S THESIS FOR

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**DISCIPLINE: MATERIALS PHYSICS**

Norsk tittel: *"Atomkraftmikroskop studier av ullfiber-strukturer"*

English title: *"Atomic Force Microscopy Studies of Wool Fibre Structures"*

This work has been carried out at NTNU, under the supervision of Dag W. Breiby.

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Trondheim, 06.19.2009

Dag W. Breiby

Responsible supervisor

Associate Professor at Department of Physics



## **Abstract**

Wool is well suited to make clothes that are warm even in moist environments. In order to fully exploit the potential in wool it is important to understand the mechanisms in the wool fibre structure when it interacts with moisture. The atomic force microscope (AFM) is well suited for studying micro scale samples in moist conditions.

In this thesis, AFM techniques best suited to study the structure of wool fibres, and the response of wool fibres to moisture have been investigated. The wool fibres were studied dry and moistened, both the exterior and the internal structure. When the wool fibre was moistened the internal structure changed, not so much for small amounts of water, where it only looked bloated, but quite profoundly when submerged in water. Then the macrofibrils could no longer be recognized and the microfibrils had swollen.



## Sammendrag

Ull er godt egnet til å lage klær som er varme også i fuktige omgivelser. For å utnytte potensialet i ull fullt ut er det viktig å forstå mekanismene som finner sted i ullstrukturen når den utsettes for fuktighet. Atomkraftmikroskopet (AFM) er godt egnet til å studere prøver på mikroskala under fuktige omgivelser.

I denne oppgaven, blir AFM teknikker for å studere strukturen til ullfibre, og hvordan ullfibre oppfører seg i forbindelse med fuktighet studert. Ullfiberene har blitt studert ved tørre og fuktige omgivelser, både utside og tverrsnitt. Når ullfibrene ble fuktet ble det funnet at strukturen endret seg. Ikke så mye for små mengder vann, da så den bare oppsvulmet ut, men nedsunket i vann kunne ikke makrofibrilene gjenkjennes lenger og microfibrilene hadde utvidet seg.



## **Preface and Acknowledgements**

This master's thesis is written in the spring 2009 as the tenth semester of my master study in physics at the Norwegian University of Science and Technology (NTNU). I have studied the structure of wool fibers using the atomic force microscope.

I would like to thank my supervisor Dag W. Breiby and Arne Røyset for their guidance. Florian Mumm and Linh Hoang for helping with sample preparations, Yingda Yu for teaching and helping me with the SEM, and Janus for supplying the samples.

Mari Helene Farstad

Trondheim, 06.19.2008



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# 1 Introduction

This masters thesis is a part of the ColdWear project<sup>1</sup>. This project aims to provide the knowledge and scientific background needed to make smart work clothes for the High North.

In the effort of improving clothing for extreme conditions it is important to understand how the utilized material functions and reacts to changes in the surroundings. The fabric properties of the clothing affects the temperature and moisture distributions and thereby comfort of the clothing [1]. In order to obtain this knowledge it is necessary to study the structure of the material.

Wool has a reputation of keeping the wearer warmer than other textiles, it also has the most complex structure of all the textile fibers [2]. It is this structure, how moisture affects it, and how to study this that is studied in this thesis.

The atomic force microscope is a non destructive technique that can be applied multiple times to the same sample. It is flexible when it comes to the environment surrounding the sample, which is a great advantage, when the effect of moisture is investigated. In addition the atomic force microscope has the ability to distinguish different types of substances based on their material properties.

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<sup>1</sup>Project website: [www.sintef.no/Projectweb/ColdWear/](http://www.sintef.no/Projectweb/ColdWear/)

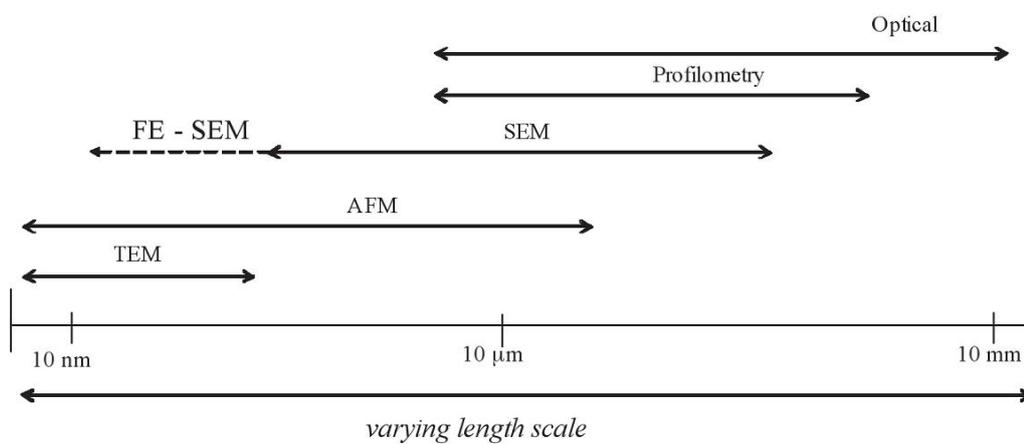
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## 2 Atomic force microscope

The atomic force microscope (AFM) is a scanning probe microscope (SPM). It has a small probe with which it scans the surface of the sample while monitoring the interaction forces between the sample and the probe. By adjusting the distance between probe and sample and thereby keeping the interaction force constant, the topography of the sample is recorded.

The AFM can scan structures of dimensions down to some nanometers, having a scan range up to about 100 microns. Figure 2.1 displays how the AFM compares to other normally used imaging techniques.



**Figure 2.1:** Length scale comparison of different imaging techniques. Only AFM and profiling gives Z-resolution. [3]

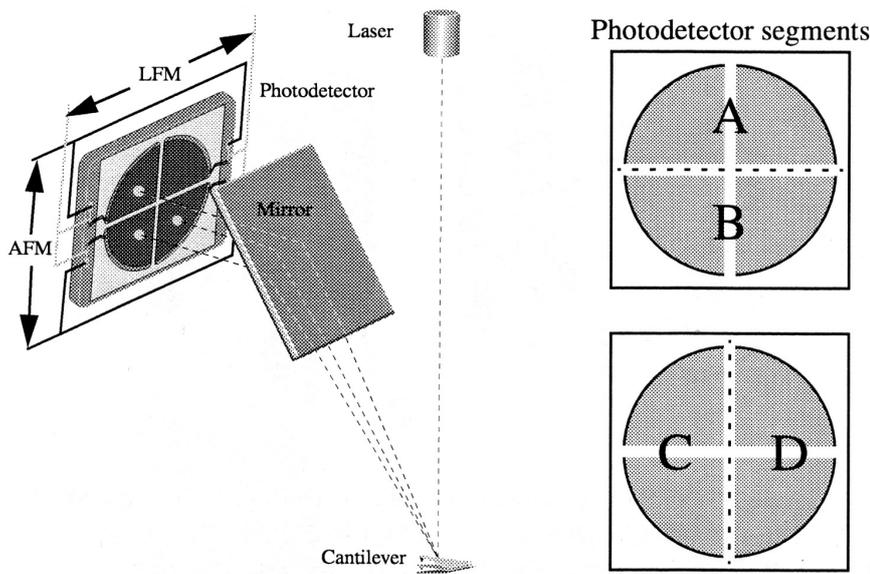
As can be seen from figure 2.1 transmission electron microscope (TEM) and scanning electron microscope (SEM) are the only alternatives in the highest resolution area. Both of these techniques demand high vacuum conditions. In addition the SEM requires a conducting sample surface, and the TEM requires samples that are maximum 100 nanometers thick at the area of interest and can only image areas of maximum  $0.6 \text{ mm}^2$  [4]. These sample preparations can damage or change the sample and prevent further investigations with other techniques. The AFM is much more flexible when it comes to imaging environment and sample preparations. It is normally operated at ambient pressure, but it can also be operated in fluids or in other controlled environments. The AFM is a non-destructive technique, the sample can usually be imaged as it is, as long as it can be mounted on the sample holder.

This chapter is based on information from [3] and [5] if nothing else is stated.

### 2.1 How it works

As mentioned the AFM scans the surface of the sample with a probe. This probe consists of a cantilever supporting a very sharp tip. When the tip is in

contact with the sample it will cause the cantilever to deflect. The deflection of the cantilever is monitored by a laser directed onto the end of the cantilever from which it is reflected onto a photodetector. This photodetector is divided into sections which makes it possible to determine the deflection of the cantilever. When the cantilever is deflected a signal is sent to the z-piezocontroller which then adjusts the height of the sample. The movement of the z-piezo together with the scanner position will then make an image of the surface. Figure 2.2 illustrates how the laser is reflected onto the detector when the cantilever is deflected.



**Figure 2.2:** Photodetector arrangement, sections A and B are used to measure topography, sections C and D are used in lateral force microscopy.

## 2.2 Hardware

In this section the main components of the AFM will be described. The AFM can be seen in figure 2.3. The main parts are the head and the scanner. The head holds the probe and measures the deflection of the cantilever, while the scanner moves the sample.

As can be seen in figure 2.4 the head provides adjustment knobs for the positioning of the probe over the sample, in addition to housing the detection system.

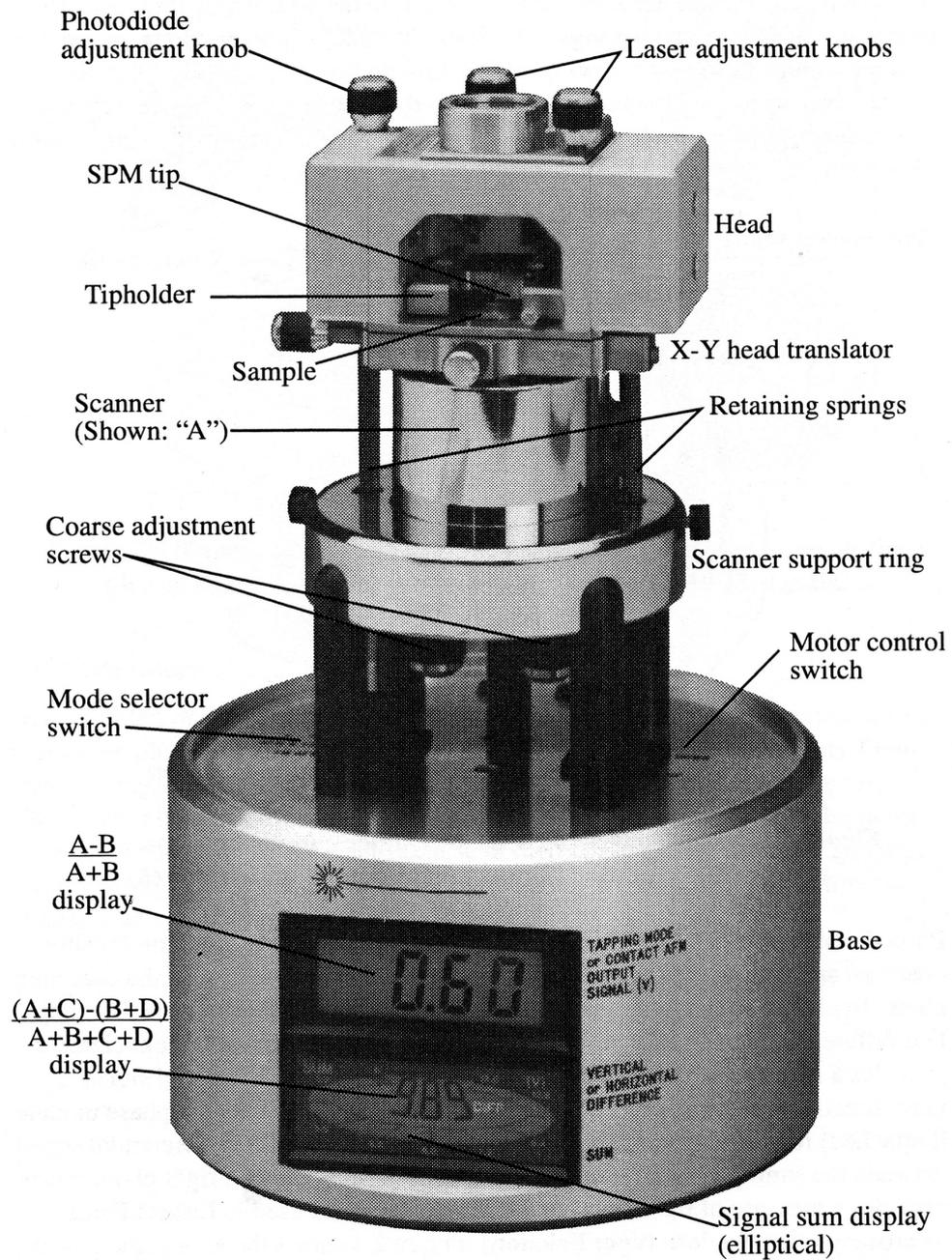
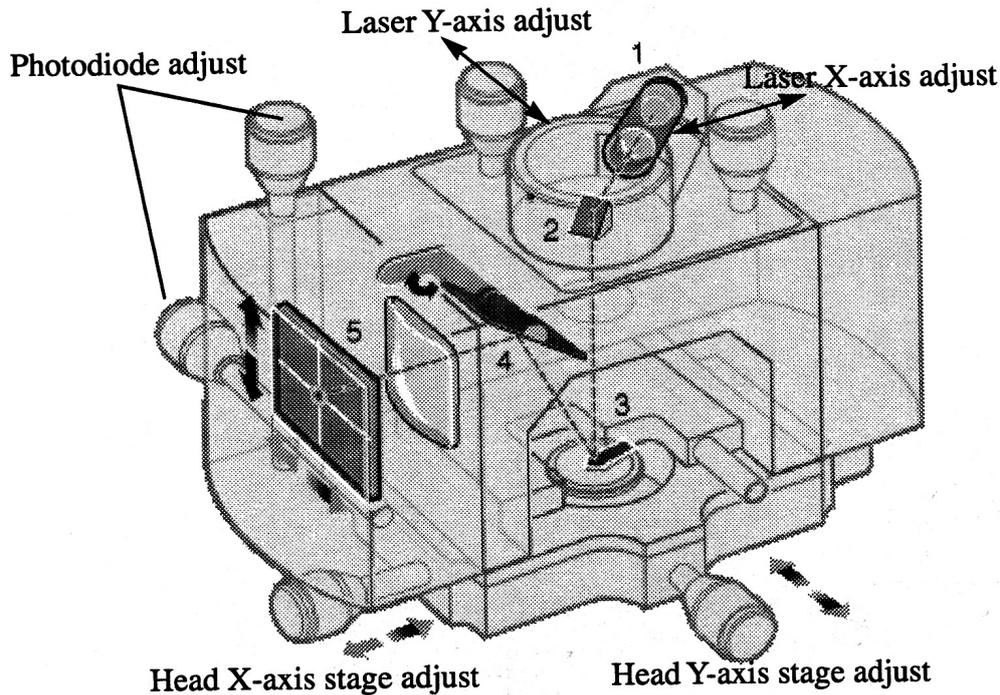


Figure 2.3: The AFM.



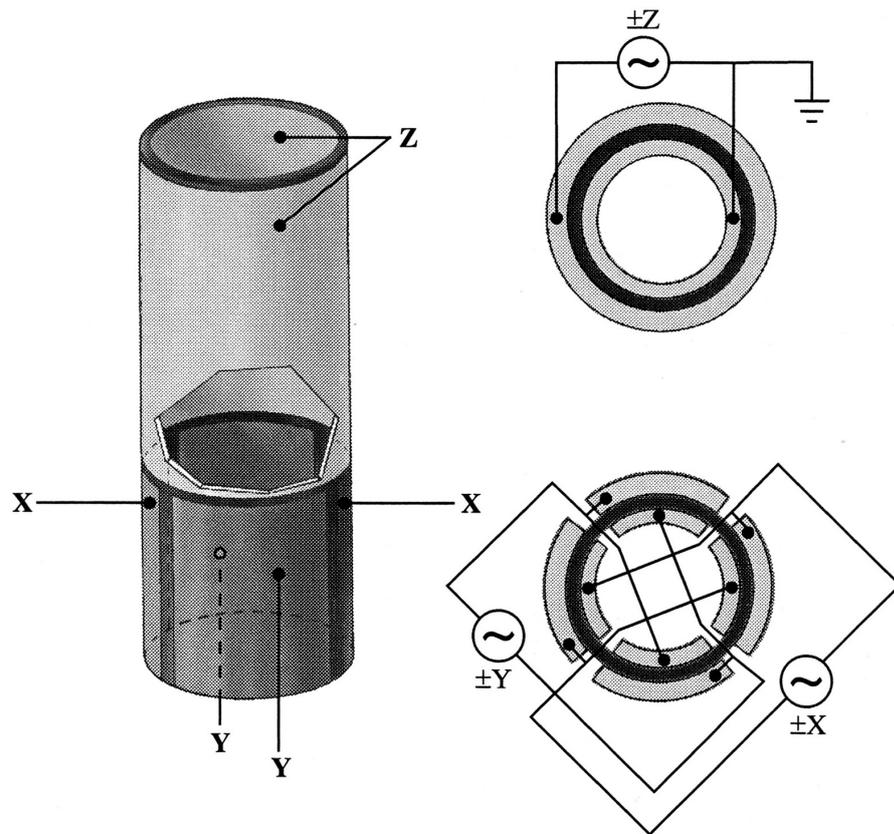
**Figure 2.4:** Multimode SPM head. 1) Laser, 2) mirror, 3) probe, 4) tilt mirror, 5) photodetector.

### 2.2.1 Scanner

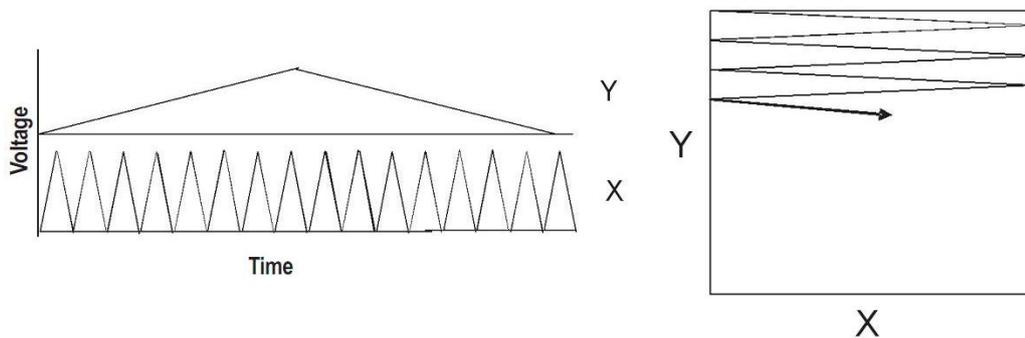
The scanner moves the sample and is thereby the limiting factor when it comes to scan size and height range of the image. Typical scan dimensions varies from  $0.4 \mu\text{m} \times 0.4 \mu\text{m}$  in the horizontal plane and  $0.4 \mu\text{m}$  vertically to  $200 \mu\text{m} \times 200 \mu\text{m}$  in the horizontal plane and  $8.0 \mu\text{m}$  vertically. The scanner consists of five piezoelectric parts, one that controls the Z-axis, and two for each of the the X- and Y-axis. The construction of the scanner is illustrated in figure 2.5.

The scanned z-range is limited to half the range of the piezo in each direction. That means that after engagement between the tip and the sample, can the sample be scanned half the z-range upwards and half the z-range downwards.

During scanning the sample is moved in a raster pattern with one fast scan axis and one slow scan axis. This is illustrated in figure 2.6. The fast and slow axes can be rotated relative to the X- and Y-axes, without any rotation the X-axis is the fast scan axis.



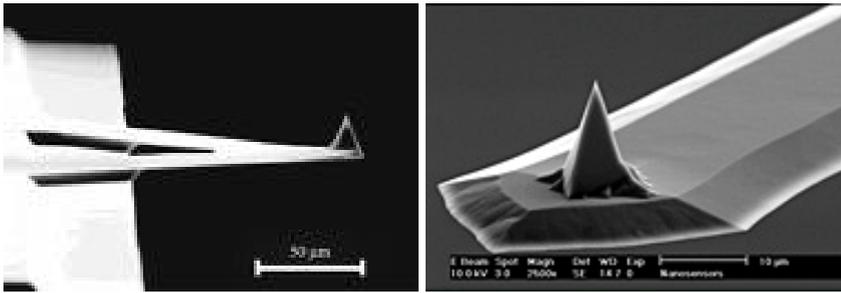
**Figure 2.5:** Typical scanner piezo tube and electrical configurations for X, Y and Z movement.



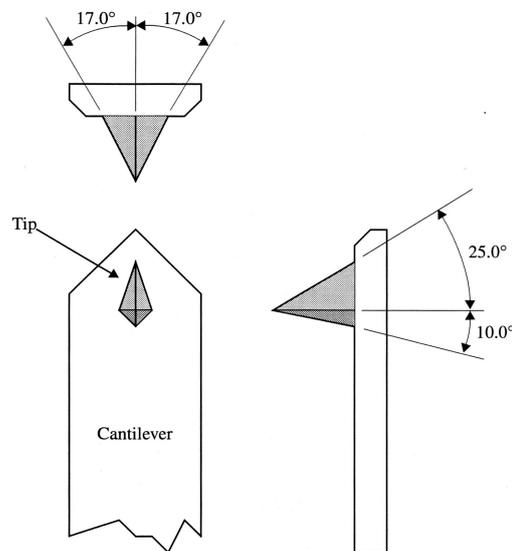
**Figure 2.6:** To the left is the signal output for driving the X and Y piezo elements and to the right is the resulting raster scan pattern.

### 2.2.2 The probe

The probe consists of a cantilever suspending an atomically sharp tip. Figure 2.7 shows two different types of probes, one with a triangular shaped cantilever and one with a straight cantilever. In figure 2.8 the realistic ideal design of a tip can be seen.

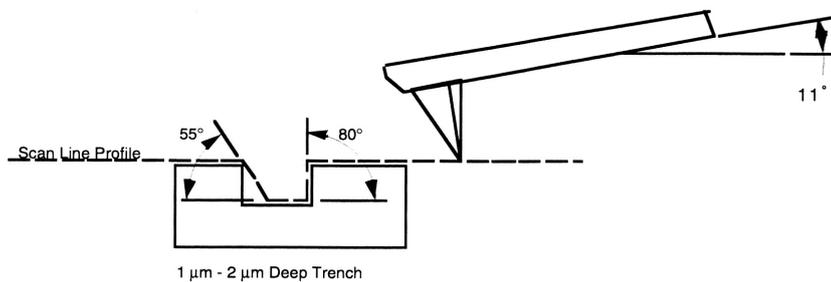


**Figure 2.7:** Two types of cantilevered probes, silicon nitride (left) and crystalline silicon (right).

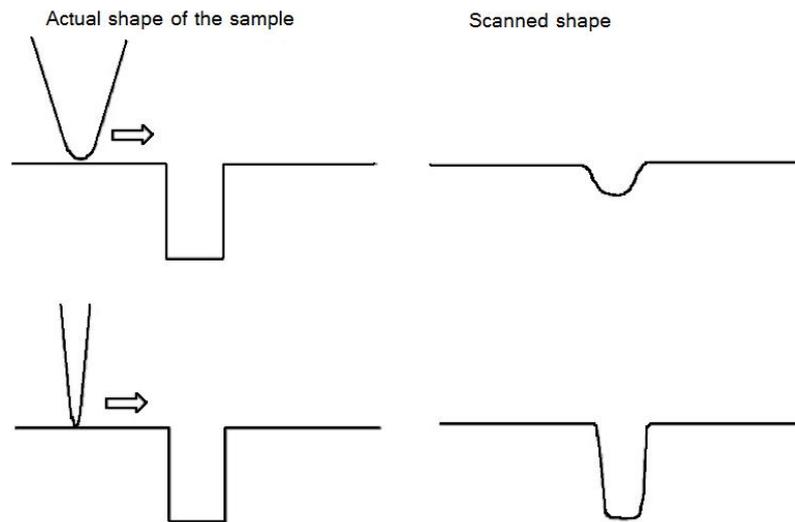


**Figure 2.8:** Theoretical shape of an etched silicon tip.

The shape of the tip limits the resolution of the scanned image and the angle of slopes in the topography, as illustrated in figure 2.9 and 2.10.

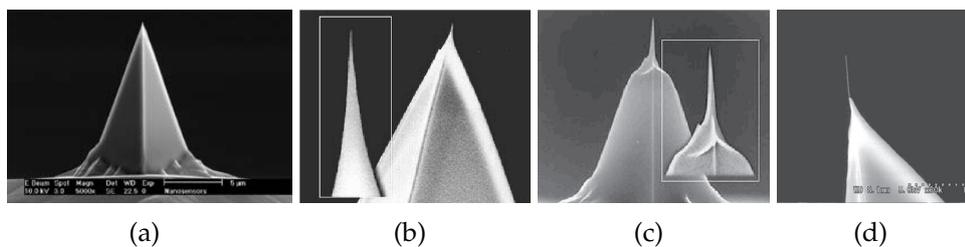


**Figure 2.9:** Artifact due to the shape of the tip. The steepest slope that can be traced properly has to be gentler than the corresponding tip angle.



**Figure 2.10:** The difference between a sharp and a dull tip. The sharp tip reaches the bottom, but not the corners, while the dull tip hardly reaches into the ditch.

The cantilever is normally made of silicon nitride or mono-crystalline silicon [6]. The silicon nitride cantilevers are physically much more flexible than the crystalline silicon cantilevers. There is a variety of probes to choose from, different sizes, flexibilities and coatings, which is the better one depends on the sample and operation mode. The tips can be extra sharpened by electrochemical etching, ion milling and by placing a carbon nanotube on the tip. Figure 2.11 displays tips resulting from different treatments.



**Figure 2.11:** Different tip preparations; a) normal tip, b) electrochemically etched tip, c) ion milled tip, d) carbon nanotube mounted on the tip.

## 2.3 Operating modes

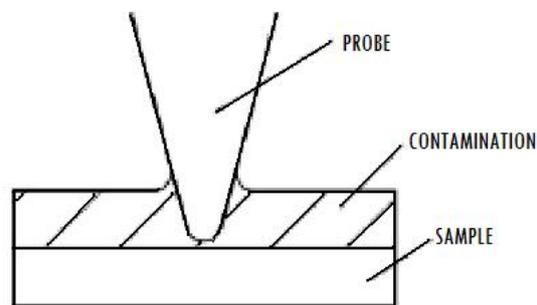
There are several ways to operate the AFM, including contact mode, non-contact mode and tapping mode including phase imaging. In addition, the different modes can be combined with a fluid cell which allows the sample to be immersed in a fluid.

### 2.3.1 Contact mode

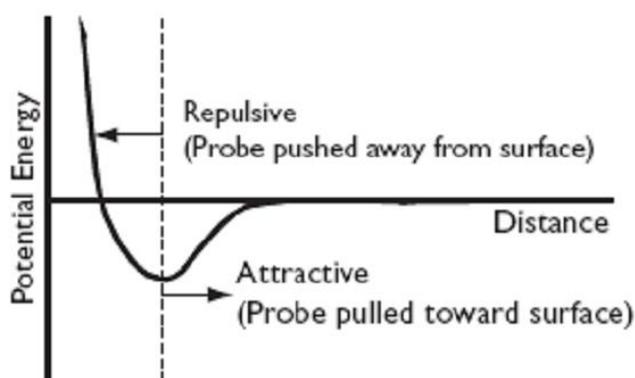
This is the original AFM method where the probe is sliding over the surface. In this mode there is a fairly strong interaction between the tip and the surface. The tip is pressed down onto the sample in order to maintain a constant deflection of the cantilever. Depending on the friction between probe and sample there may be lateral forces that influence the image or the sample. There is even a specialized technique based on measuring these forces called Lateral Force Microscopy.

When operating the AFM in ambient environments there will be a contamination layer consisting of water and miscellaneous hydrocarbons. The probe will have to go through this layer in order to get in contact with the sample, as illustrated in figure 2.12. When the probe is inside this layer it will experience capillary forces that pull the probe towards the sample, figure 2.13 displays a potential energy curve between probe and sample. Sometimes the capillary forces may cause the probe to be trusted into the sample causing damage to the sample, tip or both.

Because of the lateral forces and the capillary forces it can be very difficult to image soft samples. In order to avoid these problems the non-contact mode was developed. The non-contact mode scans the sample above the contamination layer, taking advantage of the attractive van der Waals forces. With this technique the resolution is much lower, and after the introduction of tapping mode it is rarely used.



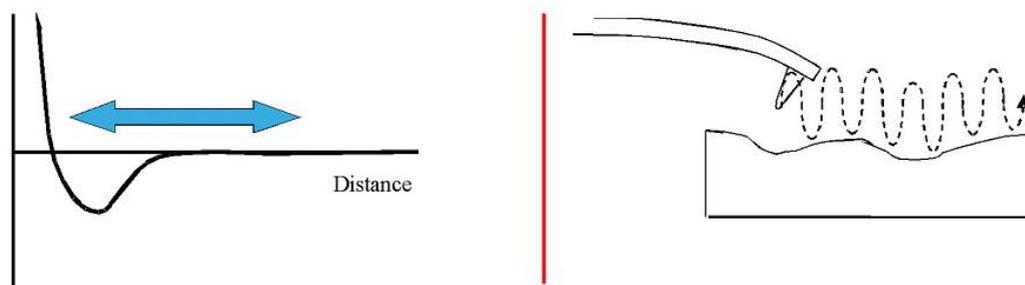
**Figure 2.12:** In ambient environments the tip has to pass through a contamination layer in order to get in contact with the sample.



**Figure 2.13:** Example of a potential energy diagram for probe and sample. The attractive forces are mainly capillary forces due to the contamination layer.

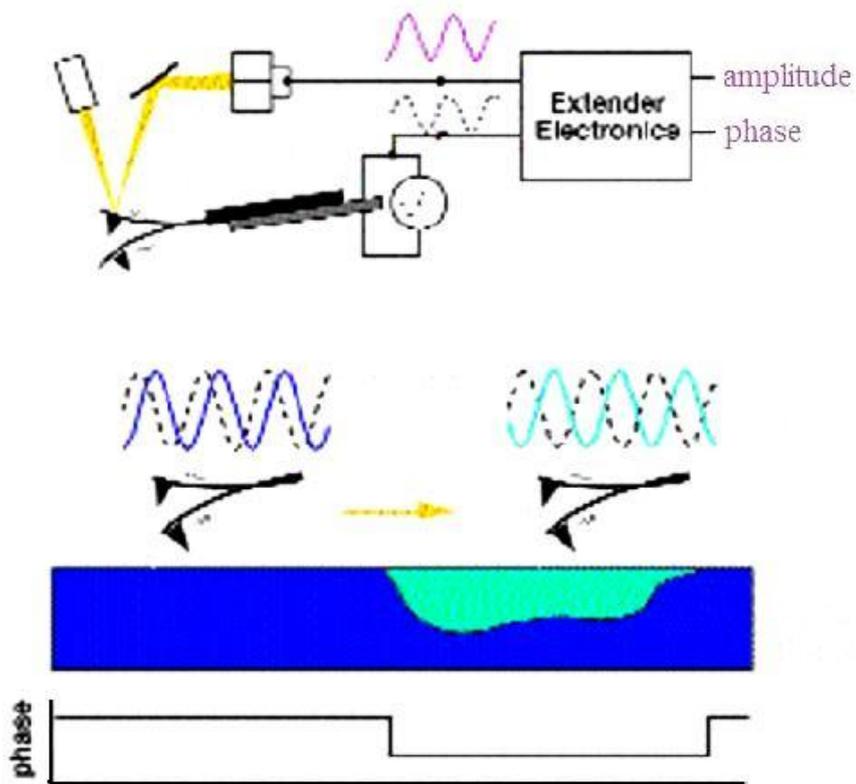
### 2.3.2 Tapping mode

In tapping mode the cantilever is oscillated while scanning the sample, as illustrated in figure 2.14. Because the tip is no longer dragged over the surface there are no lateral forces and the oscillations ensures that the cantilever has enough energy to break free of the contamination layer. This makes the tapping mode suitable for soft samples and high resolution measurements.

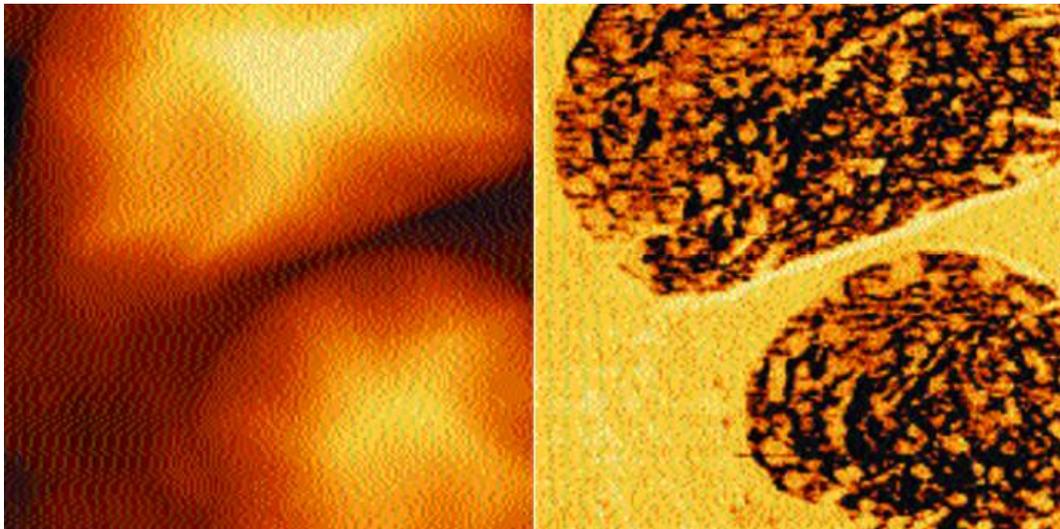


**Figure 2.14:** Potential diagram showing the motion area of tapping mode to the left, and a schematic illustrating the probe movement when scanning the sample to the right.

When the oscillating tip comes in contact with the sample it will affect the amplitude, the resonant frequency and the phase of the cantilever oscillations. In addition to information about the topography, the phase can give information about the material properties of the sample, this has been utilized in [7] and [8]. The phase imaging technique is illustrated in figure 2.15. In figure 2.16 a tapping mode image and a phase mode image of the same sample are displayed.



**Figure 2.15:** By measuring the phase lag of the cantilever relative to the piezo drive signal a phase image can be produced [9].



**Figure 2.16:** Both height image (left) and phase image (right) of a composite polymer embedded in a uniform matrix. The high resolution of the phase image highlights the two-component structure of the composite regions [9].

### 2.3.3 Fluid cell

In order to take pictures of samples submerged in water, specially adapted equipment is needed. The fluid cell provides a small container around the sample and probe which can be filled with liquid. The fluid cell is made of glass, it has an inlet and an outlet for the fluid and a groove for installing an o-ring which encloses the fluid over the sample, see figure 2.17. The glass allows the laser to be reflected from the cantilever without disturbance from the unstable fluid surface. In some cases when the sample is very hydrophobic it is possible to use the fluid cell without the o-ring, this would eliminate potential drift problems due to the lateral forces between the o-ring and the sample.

The fluid cell can be combined with both contact mode and tapping mode. The operational principles are the same as in air. Because of the extra mechanical resistance in the fluid compared to air, the cantilever should have a much lower resonance frequency than the cantilevers used in air.

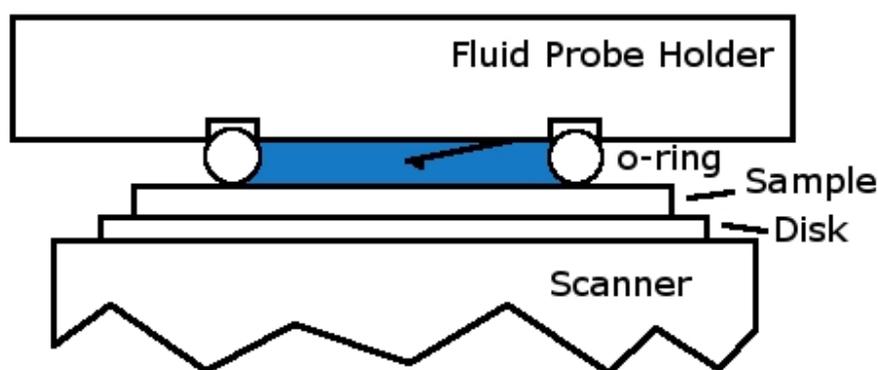


Figure 2.17: Cross section of a fluid cell over a sample.



### 3 Wool

Wool has some highly desirable properties when it comes to heat and moisture management. It is known for the ability to keep the wearer warm even though it is wet. These unique properties are a result of the complex structure of the wool fibre.

Up until now wool fibres have been studied with electron microscopy, x-ray scattering and chemical methods for studying proteins. This has led to a fairly good understanding of the internal structure of the fibre and its properties [2], [10], [11]. Recently there has been some studies of the surface properties, such as hardness and hydrophilicity, of the wool fibre, also in connection to moisture [7], [12], [13] and [14]. There has also been some studies of the cross section of the wool fibre [15], [16], but not with respect to moisture.

This section is based on [2] unless otherwise stated.

#### 3.1 Structure

The wool fibre has a very complicated structure compared to other textile fibres. It consists of two main structures, the cortex consisting of protein  $\alpha$ -helices also called keratin, and the cuticle, an outer shell encapsulating the cortex. The complete structure of wool is sketched in figure 3.1.

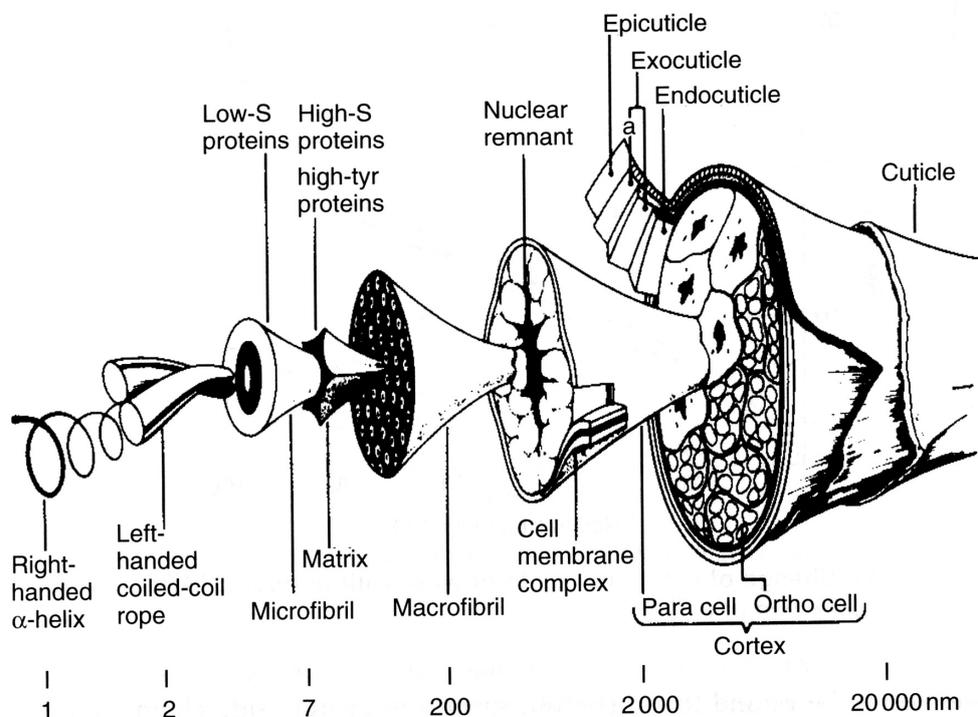
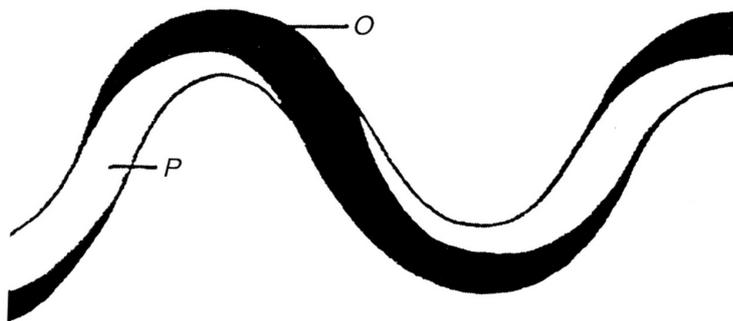


Figure 3.1: The structure of a wool fibre.

Wool fibres consist of carbon, hydrogen, oxygen, nitrogen and sulphur. These are, except for a high sulphur level, normal constituents in proteins. In addition to proteins the wool fibres contain lipids, both internal and external, the external lipids are known as woolgrease and are normally more or less removed during *scouring*. Wool fibres have a diameter between 15-40  $\mu\text{m}$  and a length of up to 50 cm. The cross section of a wool fibre is an approximate ellipse.

### 3.1.1 Cortex

The cortex amounts to 90% of the fibre. It consists of two types of cells, orthocortex and paracortex cells. Normally there are 60-90% orthocortex cells and 40-10% paracortex cells. The paracortex cells contain the larger amount of sulphur. Sulphur is the most important crosslinking agent in wool, which means that the paracortex cells are tougher and more highly cross-linked than the orthocortex cells. The cortex cells can be evenly distributed in the cortex or they can be sectioned into two parts. When the cortex is sectioned it results in a *crimp* where the paracortex cells always are situated at the inside of the crimp, as illustrated in figure 3.2. Fine wool types such as Merino wool usually have a sectioned cortex.



**Figure 3.2:** Organization of para- and orthocortex cells in a sectioned wool fibre.

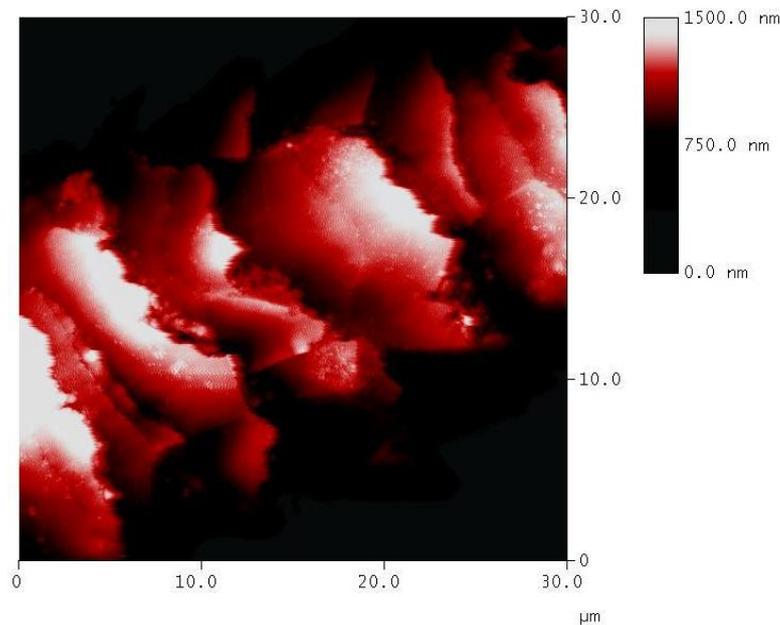
Both ortho- and paracortex cells consist of 5-20 macrofibrils containing 500-800 microfibrils. All of the fibril structures are embedded in their respective matrix. Figure 3.1 gives an overview of the structure of a paracortex cell.

### 3.1.2 Cuticle

The cuticle consists of scale-like cells wrapped around the cortex, each scale is about 10-30  $\mu\text{m}$  long and 400-800 nm thick. The scales partly overlap each other and merge together. The cuticle cells consist of at least four layers: epicuticle, exocuticle and endocuticle layer as indicated in figure 3.1. The resulting outer structure is displayed in figure 3.3. The different layers

consist of different compositions of lipids, proteins and carbohydrates. The endocuticle has the lowest sulphur level and is the most permeable layer.

It is the structure of the cuticle that is responsible for *felting*, which makes wool fabrics very sensitive to laundering, if care is not taken they will shrink considerably.



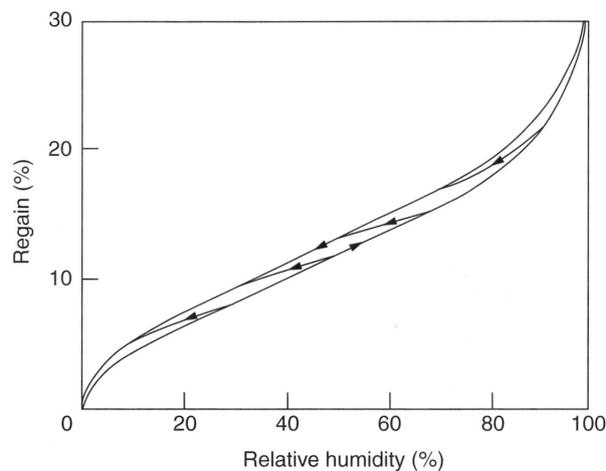
**Figure 3.3:** AFM height image of the outer structure of the hair of the author. The structure of human hair closely resembles that of wool.

## 3.2 Moisture absorption

A measure of moisture absorption is *moisture regain*, which is defined by

$$\text{Moisture regain} = \frac{\text{mass of absorbed water}}{\text{mass of dry fibre}}. \quad (3.1)$$

The moisture regain is dependent on the relative humidity, in figure 3.4 the moisture regain for wool with respect to relative humidity is displayed. As can be seen in figure 3.4, moisture absorption followed with moisture desorption creates hysteresis in the moisture regain.



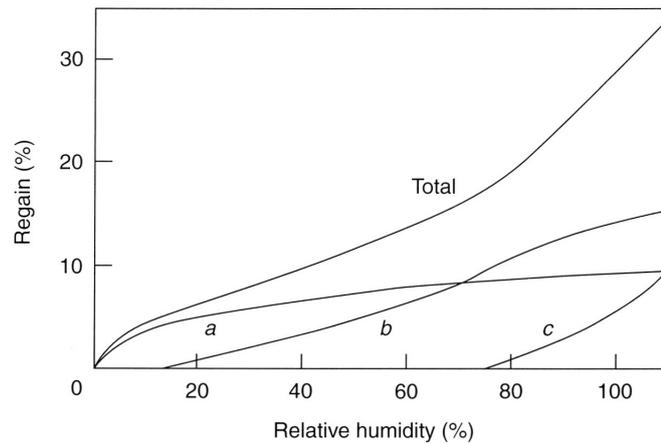
**Figure 3.4:** Moisture regain of wool with respect to relative humidity.

When a wool fibre absorbs moisture its physical properties change, which again influences further absorption. It is therefore difficult to tell exactly *how* the moisture is absorbed. There are several theories that try to explain this process. Speakman [17] has a theory from 1944 based on how the water is absorbed which fits very well with the experimental values. He divides the absorption into three types,

- a water binding to hydrophilic groups in the protein side chains,
- b water attaching to the main chain of the proteins, replacing hydrogen bounds,
- c capillary condensation.

The last point however, are more likely to be water adhering to already absorbed water in accordance to Peirce's theory from 1929 [10]. The contribution to the moisture regain from each type can be seen in figure 3.5. Type b has the most effect on the rigidity of the fibre.

Wool expands radially when it absorbs water, up to 16.3%. The initial increase in volume is less than the amount of water absorbed, whereas at about 15% regain the volumes become additive. It can be argued that this is because the initial water molecules fill up spaces in the protein molecules causing only a small expansion. When these spaces are all filled up the absorbed molecules will add more to the volume.



**Figure 3.5:** Absorption of water divided in three types. a) Hydrophilic groups, b) replacing hydrogen bounds, c) indirectly attached.

### 3.2.1 Heat of sorption

The amount of energy released when water is absorbed is described by the differential heat of sorption. The differential heat of sorption  $H$  for wool can be described by equation 3.2 which is based on Kirchoff's equation for dilution of solutions.

$$2.3 \log \left( \frac{h_1}{h_2} \right) = 9H \left( \frac{1}{T_2} - \frac{1}{T_1} \right) \quad (3.2)$$

$h_1$  and  $h_2$  are relative humidities and  $T_1$  and  $T_2$  are the corresponding temperatures at constant regain. The energy released from 1.0 kg of some textile material going from 40 to 80% relative humidity is listed in table 3.1. Wool clearly releases the largest amount of heat.

**Table 3.1:** Energy released from 1 kg of material going from 40 to 80% relative humidity. [10]

Material	Kilocalories
Wool	38
Cotton	20
Viscose rayon	40
Acetate	12
Nylon	10
Terylene	1



## 4 Experiments

In this section the sample preparations and imaging will be described. The fibres have been studied in both air and water.

### 4.1 The samples

The samples are obtained from four different qualities of merino wool cloth. They are all supplied from *Janus*. According to *Janus*, all of the samples are *Hercosett*-treated. The *Hercosett*-treatment consists of a chlorine-treatment which is neutralized with natriumbisulphite, followed by a nylon coating. This process is a shrink proofing process which allows the garment to be machine washed. According to [2], felting is prevented by scale masking done by the polymer.

The different qualities and their properties are listed in table 4.1.

**Table 4.1:** Properties of the samples and the cloth from which they were extracted.

Sample ID	Diameter [ $\mu\text{m}$ ]
405	20
410	17.5
432	20
453	20

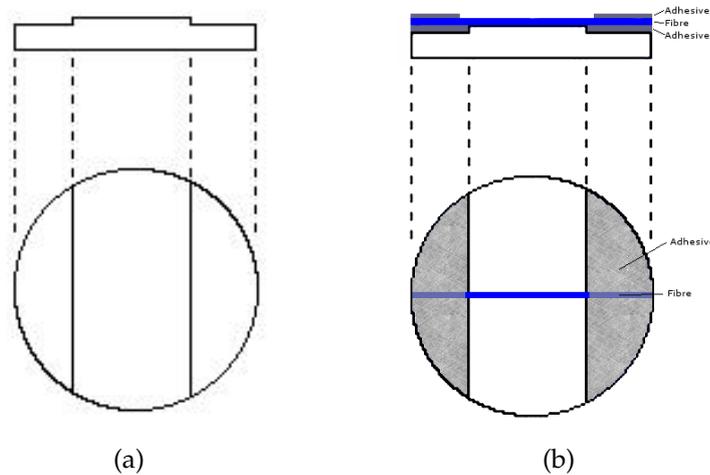
### 4.2 Sample preparations

Both the outside and the cross-section of the fibres have been studied. In order to image the samples in the AFM they have to be held steady onto the scanner. This is achieved by attaching the samples to a metal disk of 0.9 cm in diameter, which is then attached to the scanner by means of magnetism.

#### 4.2.1 Imaging of wool fibre exterior

The standard way of attaching the sample is to use double sided adhesive tape. This technique worked fairly well, but because of the crimp in the fibres and the small size they easily rolled over when pressed down, and were thereby contaminated by the adhesive. To avoid this, the adhesive was split in two parts leaving a clean area where the sample was to be scanned. This resulted in the fibre being suspended freely over the metal disk, but not sufficiently stable to be scanned. The solution to this was to make a disk where two opposing segments are slightly lower than the centre part, see illustration in figure 4.1. This holder worked fairly well, but because of the crimp in the fibres the adhesive was not enough to hold the fibres fully

stretched. This was solved by adding an extra layer of adhesive over the ends of the fibres.

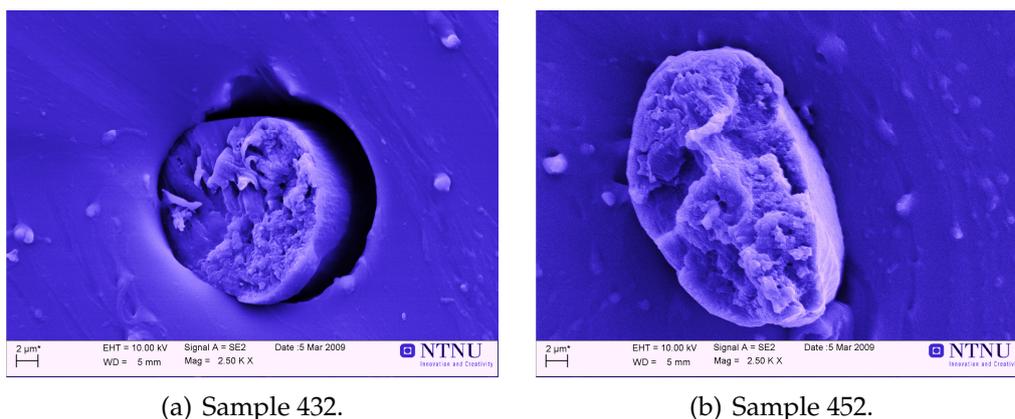


**Figure 4.1:** Modified sample holder disk (a), and sample mounting (b).

#### 4.2.2 Cross-section imaging

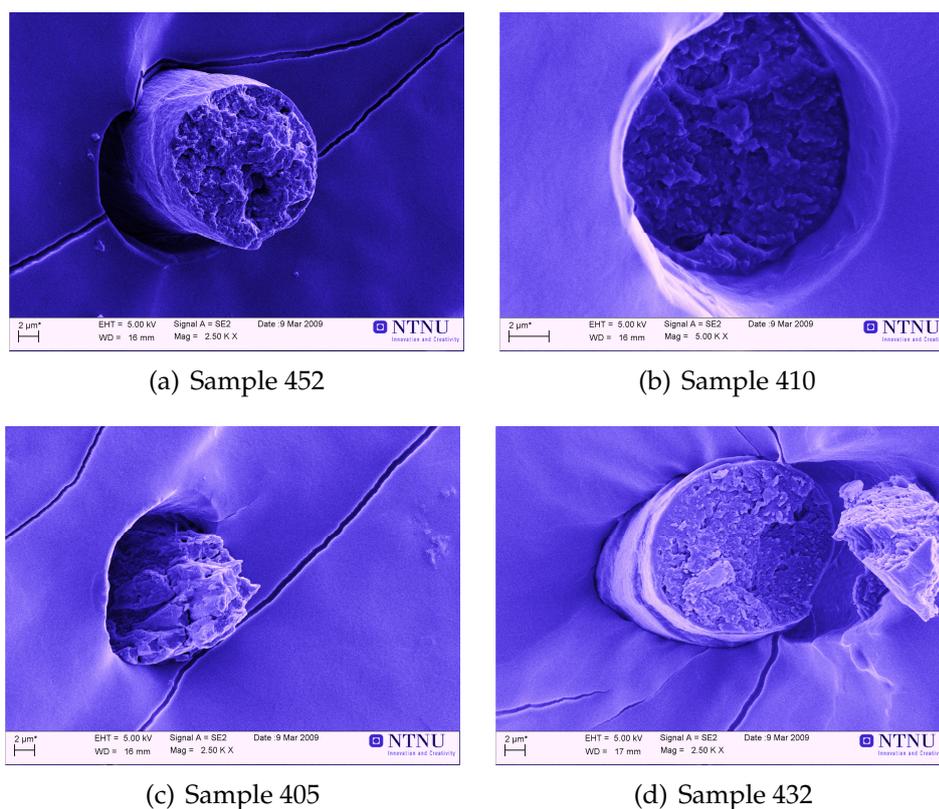
In order to study the cross section of the fibres they have to be embedded in a supporting “matrix” material. For imaging with the AFM the height variations of the sample can be up to  $5\ \mu\text{m}$ , but it is very difficult to get a good image if the entire z- range is in use.

Several possible techniques were tested. The fibres were embedded in PDMS (silicone). One part was then cut with a razor blade and another was broken after freezing in liquid nitrogen. The resulting cross sections were imaged using SEM, the results can be seen in figure 4.2 and 4.3.



**Figure 4.2:** SEM images of fibres embedded in PDMS and cut with a razor blade.

The SEM images clearly show that the fibres cut with a razor blade are likely to get deformed and quite rough, it can also be seen that the PDMS

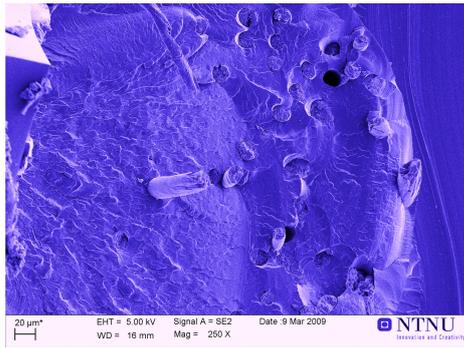


**Figure 4.3:** SEM images of fibres embedded in PDMS and broken in liquid nitrogen.

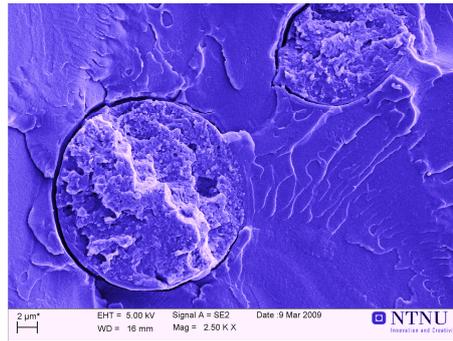
is not evenly attached to the fibre, in some of the pictures there is a gap between the fibre and the PDMS.

The fibres were also embedded in epoxy and broken in liquid nitrogen, figure 4.4 displays the results. This time multiple fibres were embedded together, out of practical reasons. It can clearly be seen that the fibres break randomly with a resulting rough cross-section, they also appear to have a random orientation coming out of the epoxy.

Cutting seems to be the most controllable method, however the razor blade is too blunt. The next option is then cutting with a microtome. In order to do this, the fibres has to be embedded in blocks of a supporting matrix e.g. epoxy resin, one called epon was used. The block dimensions are  $6 \times 4 \times 10 \text{ mm}^3$ , 10 mm being the height. The epoxy resin consists of three components, LX-112, DDSA and NMA, and a catalyst DMP-30. The data sheets can be found in appendix A.1. In order to embed the fibres in the desired blocks of epon, the following method was developed based on previous experiences. First the fibres are mounted slightly above an aluminium foil in order to be embedded in a thin layer of epon, this is in order to handle the fibres more easily. This first step should only cure for a few hours at  $60^\circ\text{C}$ , until it is no longer fluid, but not yet completely hard. Then a suitable piece is cut out and placed in the mould, which is then filled with epon and cured for 48 hours at  $60^\circ\text{C}$ . The resulting cross-sections can be seen in figure



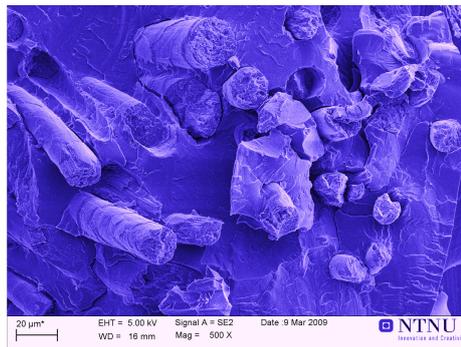
(a) Sample 452, overview.



(b) Sample 452, closer look at the fibres.



(c) Sample 410



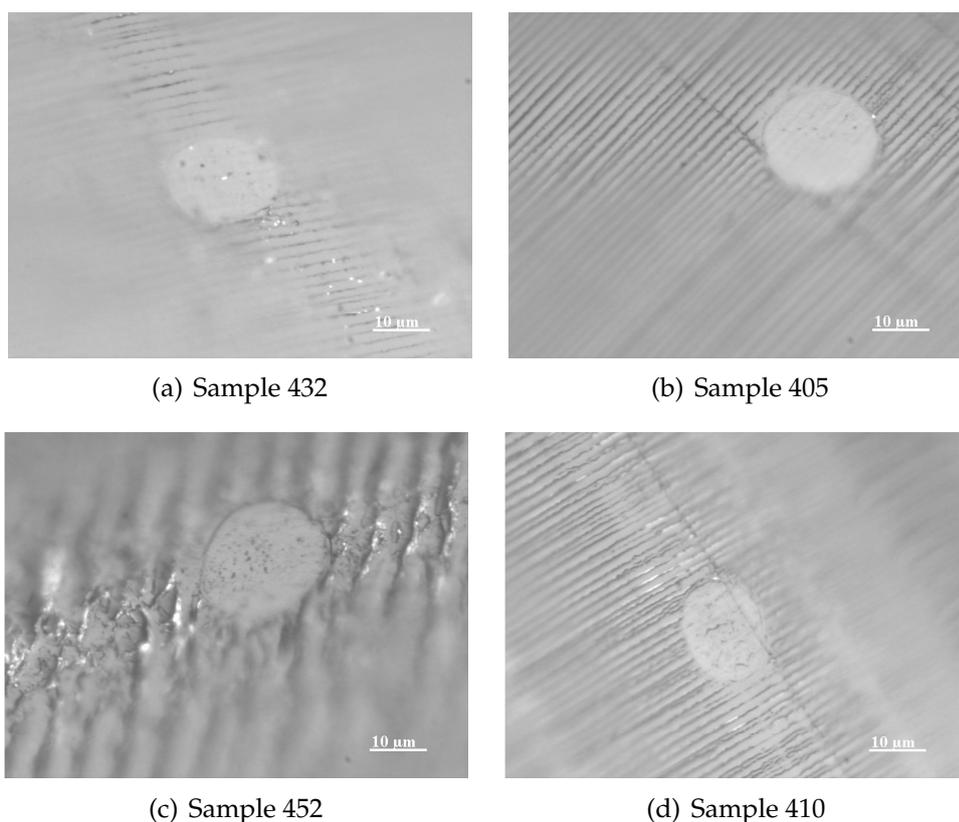
(d) Sample 432

**Figure 4.4:** SEM images of fibres embedded in epoxy and broken in liquid nitrogen. the cross sections appear to be quite rough.

4.5. One fibre was also dipped in agar prior to the embedding in epon in order to see if that would have any effect upon the adhesion between the epon and the fibres.

In figure 4.5 there are grooves in the surface of the epon. This is a result of the cutting process, however, they do not seem to affect the wool fibre structure.

In order to fit epon blocks onto the scanner they should not be higher than about 7 mm in tapping mode in air, with the fluid cell it has to be less than 4 mm. In order to achieve this, the block has to be shortened, this is done by sawing off the bottom part with a junior hacksaw. Care has to be taken to ensure that the top and bottom planes are parallel, some adjustment with a scalpel might be necessary.



**Figure 4.5:** Optical microscopy pictures of cross-sections of fibres embedded in epon. The stripes are an artifact from the cutting process.

### 4.2.3 Fluid Cell

When using the AFM with the fluid cell it is necessary to have a relatively flat surface, preferably hydrophobic. The fibres are located close to the edge of the epon block, which means that it will be hard to sustain a sufficiently large drop onto the block. Therefore it is necessary to build up the area around the block so that the surroundings of the sample have almost the same height as the sample. This was done by creating a circle of PDMS with an opening for the epon block. In order to prevent drift it is necessary to make the opening as small as possible, or make sure the epon block is securely fastened with something that does not react with water. A way to achieve this is to put the block into the PDMS before curing, making sure that the PDMS does not contact the upper surface and then cut out a circle around the block.

## 4.3 Experiments

Optical microscopy and AFM have been used to investigate wool fibres and their reaction to water. The samples have been studied dry, after they have absorbed a water droplet and submerged in water. The tips used for the

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AFM in air are from the NSC 15-series, with the fluid cell NP-S tips with a spring constant  $k=0.06$  and  $0.32$  was used.

### **4.3.1 External investigations**

The outer investigations of the fibre consist of preparing them as described in section 4.2.1, then studying them in an optical microscope to get familiar with the main features and thereby a better understanding of what to expect when using the AFM.

The effect of adding a drop of water onto the fibre was also studied, this was first done under the optical microscope, before it was attempted with the AFM. Attempts to study the exterior of the fibres with the fluid cell were also made.

### **4.3.2 Cross-section investigations**

Also the cross-sections were studied both with the optical microscope and the AFM. The optical microscope was used to get familiar with the sample and to get further credibility to some findings done with the AFM.

In addition SEM images were taken after the AFM investigations were done. This was done in order to compare the results of the AFM with another technique which has roughly the same resolution. Against what was expected the SEM images were made without any conducting coating. The images were instead taken with low electron high tension and a short distance to the detector.

### **4.3.3 Image analyses**

In the program that belongs to the AFM (Nanoscope 5.30r2) there are some analyzing and image treatment tools. The only treatment the images presented in this thesis has gone through are to adjust the height of the scan lines so that they make up a clear image without too much stripes. There is also a measurement tool, with which distances and angles in the images can be measured.

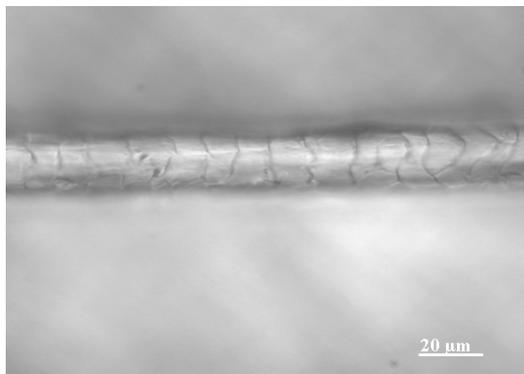
Some images have also been exported to MATLAB in order to get more accurate measurements due to the ability to enlarge the image and a tidier view of the image. The placement of the cursors are done with an uncertainty ranging from  $\pm 0.04$  to  $\pm 1.1$  of the respective unit dependent on the measurement technique. This will result in a negligible error when compared to other uncertainties such as instrumental error and environmental influences.

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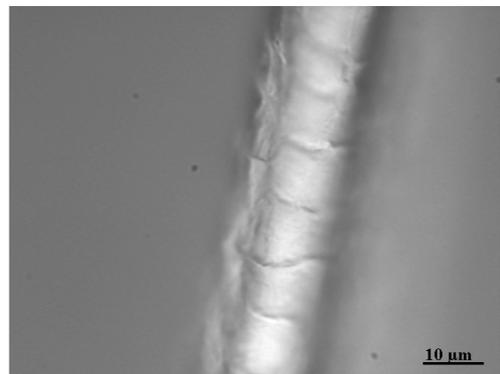
## 5 Results and discussion

### 5.1 External investigations

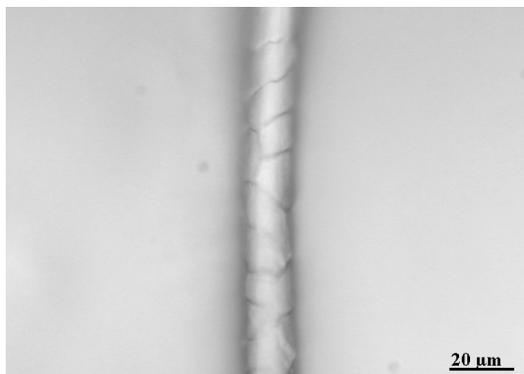
The structure of the cuticle is clearly visible in the optical microscope. It can however, be difficult to obtain larger areas in focus at the same time as the surface of the fibres is curved and does not have a constant diameter. Figure 5.1 displays images of the cuticle structure taken with an optical microscope. All of the fibres seems to have the same structure, the fibre in figure 5.1(d), is darker because of the colour, dark blue, which does not influence the structure of the fibre.



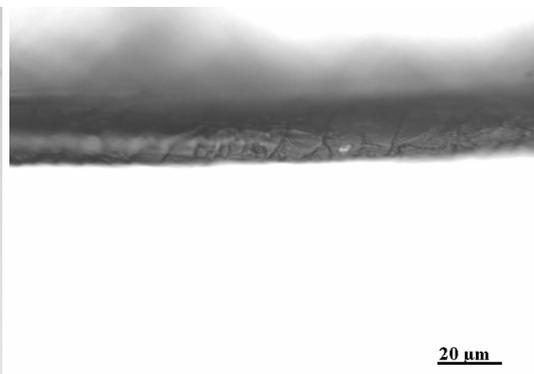
(a) Sample 405



(b) Sample 410

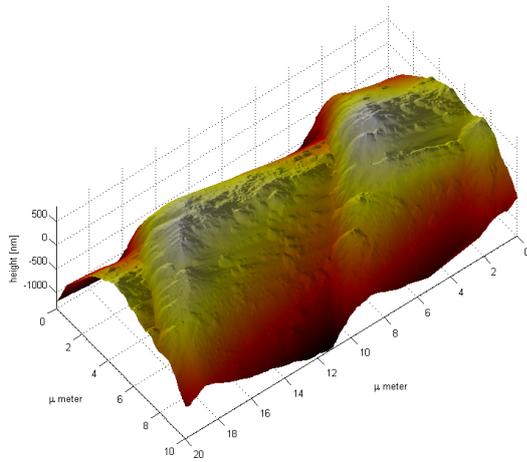


(c) Sample 432

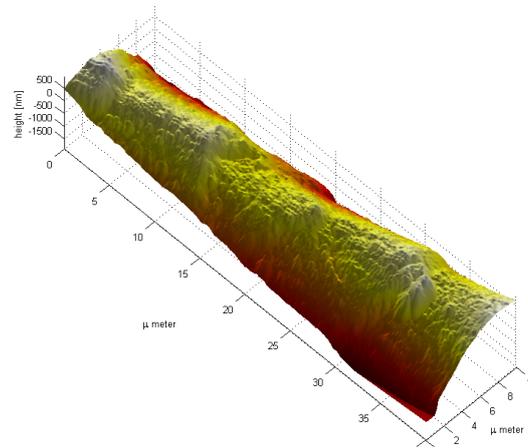


(d) Sample 452, this fibre is dark blue, there are therefore some shadow effects.

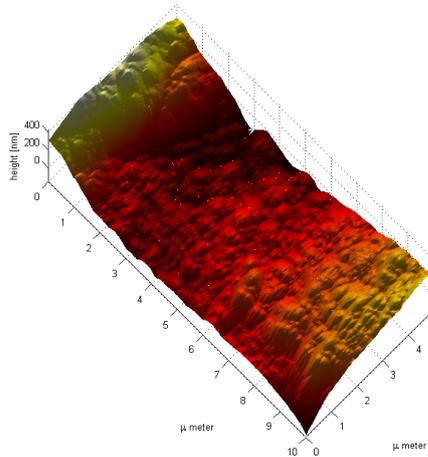
**Figure 5.1:** Images of the cuticle taken with an optical microscope.



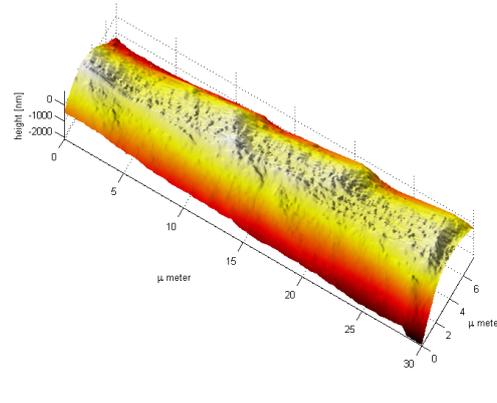
(a) Sample 405.



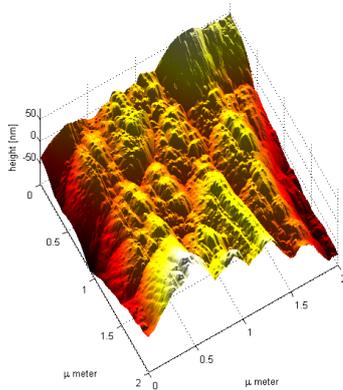
(b) Sample 410. The edge between two adjacent cuticle cells looks rounded.



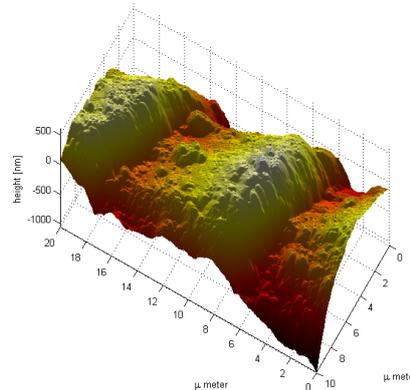
(c) The center cuticle cell from (b), the surface of the cuticle cell is quite rough.



(d) Sample 432.



(e) A segment of (c), grooves along the length (f) Sample 452. The edge between two adjacent cuticle cells looks rounded.

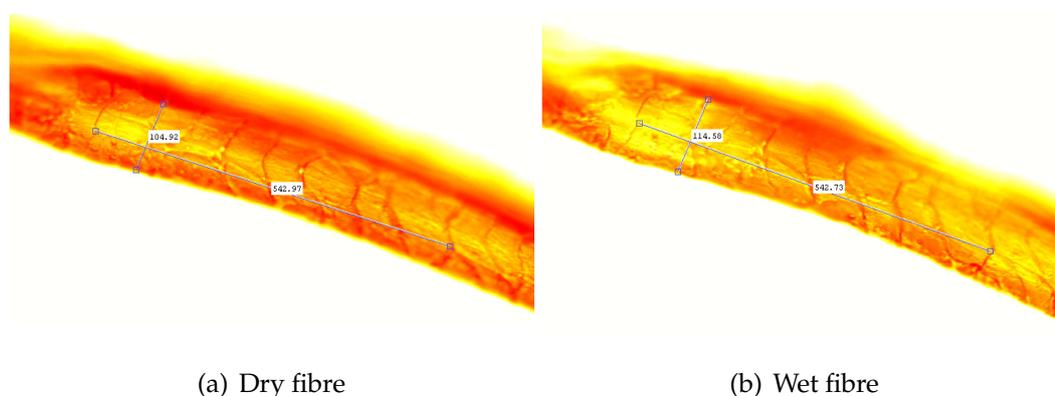


**Figure 5.2:** Images of the cuticle taken with AFM, all fibres were imaged dry. It can clearly be seen that the surface is uneven and at times there are distinctive grooves along the length of the fibre, seen for example in (b), (d) and (e).

Figure 5.2 displays images of the cuticle surface structure taken with the AFM. It is possible to image a width of about  $10\ \mu\text{m}$  of the fibre before the curvature becomes too large to image. This means that only the upper segment of the fibre is displayed in the image. Exactly how much of the fibre that can be scanned depends on the shape of the tip, see section 2.2.2, shape of the fibre (they are not entirely circular and not uniform) and where the tip engaged the fibre, determining relative the z-range. It is therefore important to look at clear features on the surface when comparing images from the same fibre in order to make sure it is exactly the same place, especially if the fibre has changed somewhat between each scan, for instance it might have expanded due to moistening.

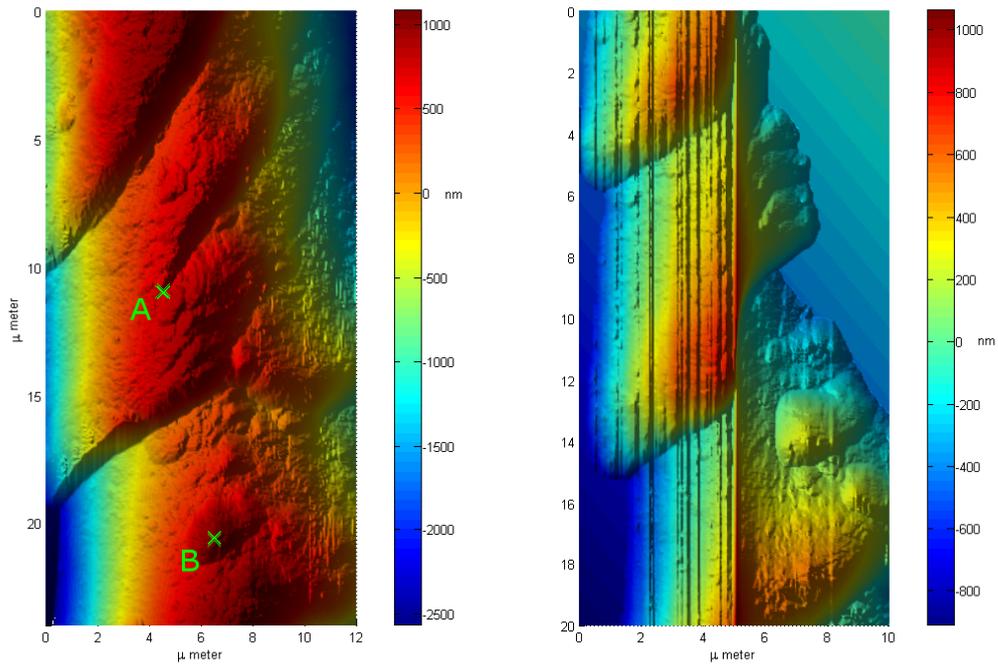
The surface structure of the cuticle cells is quite rough, at times there are grooves along the length of the fibre. When compared to my untreated human hair in figure 3.3, it is clear that the cuticle cells of the wool fibres have much smoother edges and a much rougher surface. This is probably due to the nylon coating from the Hercosett-treatment described in section 4.1.

Figure 5.3 displays the effect of wetting a fibre imaged with an optical microscope. The fibre was imaged dry and just after it had absorbed a water drop. The wetting resulted in a radial expansion estimated to 9%, and no significant longitudinal expansion.



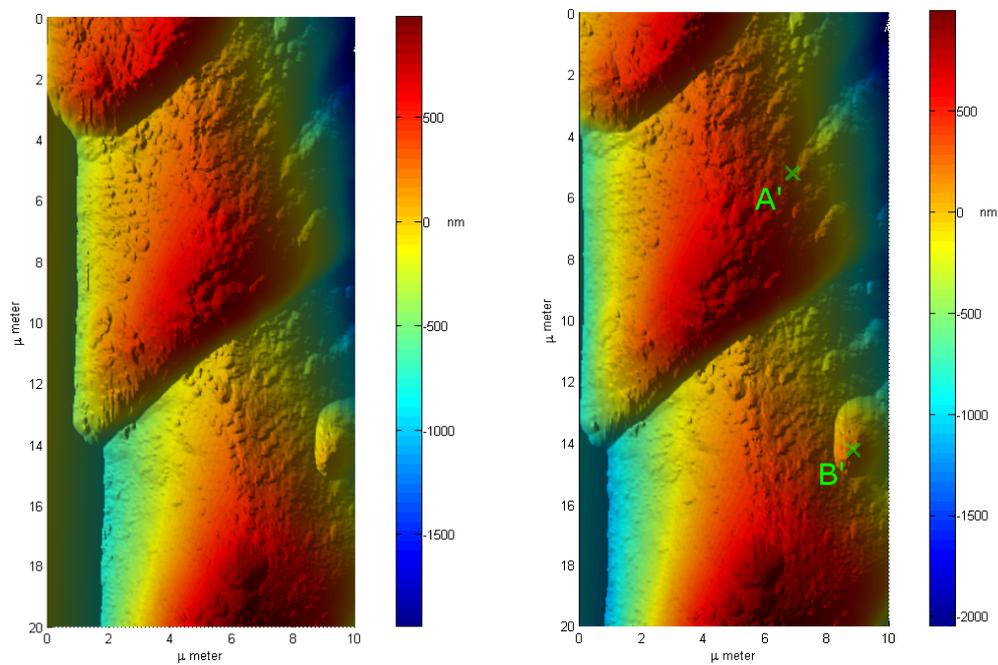
**Figure 5.3:** Optical microscope image before and after wetting of the fibre. The measurements gives a radial expansion of 9% and no longitudinal expansion.

In figure 5.4 the results of wetting a fibre scanned with the AFM can be seen. A drop of water was placed on the fibres a small distance away from the AFM cantilever, the imaging was started as the drop was absorbed by the fibre. During this first scan the fibre twisted a little, causing the shift in figure 5.4(b), this also made it hard to compare the images of before and after the wetting. In the two last pictures it is however possible to make some comparisons, revealing a radial expansion of 2%. It should be noted that the last scan was finished 30 min after the wetting, as each scan took 10 min.



(a) Dry fibre, just before wetting

(b) Wet, the fibre twisted during the scan.



(c) After 15 min

(d) After 25 min

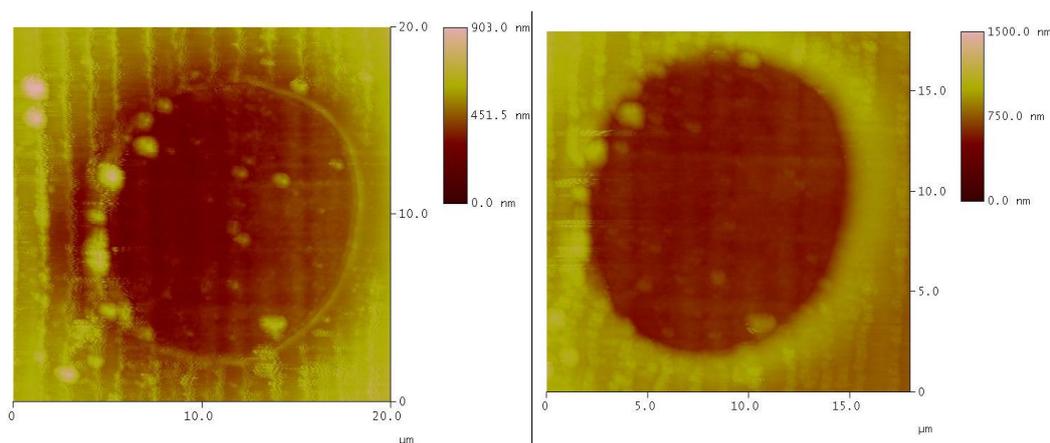
**Figure 5.4:** AFM images of sample 432. a) Before placing a drop of water onto the fibre, b) just after the drop has been absorbed, c) about 15 min later and d) about 25 min later. Points A and B in figures (a) and (d) show how the fibre twisted.

## 5.2 Cross-section investigations

When investigating cross-sections the optical microscope was mainly used to locate the fibre and to get an idea of its size and shape, as the fine structure of the fibre can not be seen in the optical microscope. The different samples are expected to have the same type of substructure.

With the AFM three samples, 432, 405 and 452, have been studied dry. Sample 432 has been studied moistened by placing a droplet of water onto the sample and waiting for it to be absorbed before imaging. Sample 405 and 452 have been studied submerged in water with the fluid cell. Sample 410 was attempted studied with the fluid cell but the elevation of the epon around the fibre prevented the tip from getting in contact with the fibre. Some of the smaller scale images, the height images in particular, are somewhat unclear. A better tip might yield more detailed images.

In figures 5.5(a) and 5.6 there is some contamination, the sample is therefore cleaned by rinsing it in distilled water and gently removing any excess water with blotting paper, the sample was left to dry over night. Figure 5.5(b) displays the cross section after cleaning.



(a) Before cleaning. The fibre is at level with the surrounding epon, and the cuticle is clearly visible.

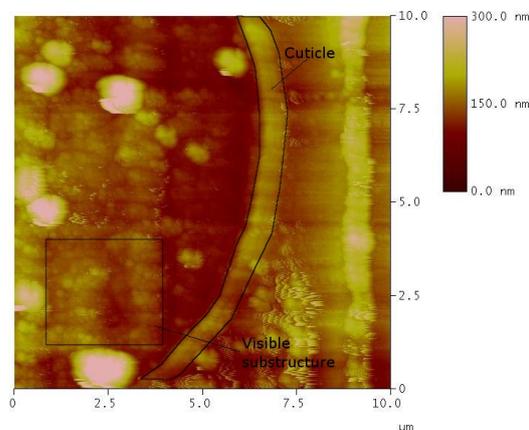
(b) Right after cleaning, most of the contaminations are gone, the fibre has retracted somewhat into the epon.

**Figure 5.5:** AFM images of cross-sections of sample 432, taken before and after cleaning.

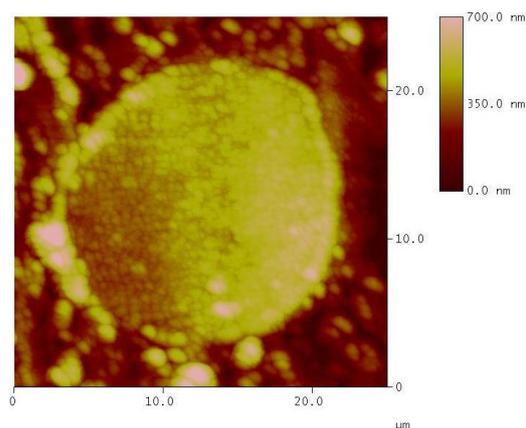
### 5.2.1 Studies of dry fibre cross sections

Figures 5.5 to 5.12 display selected results from the studies of dry fibres. In figure 5.5 and 5.7 the cross section of the entire fibre is imaged. In figure 5.5(a) the cuticle is clearly visible, and if looked closely some circular substructure may be distinguished, this is, however easier in figure 5.6. The grooves from the cutting can also be seen there. They do not seem to affect the sub structure which is much smaller, than the distance between the grooves.

In figure 5.7 the circular substructure is quite clear. It seems to stand out more clearly than for the other samples. This is the same sample as in figure 4.5(c), which seems to have been cut somewhat differently than the others.



**Figure 5.6:** AFM image of cross-section of sample 432, the image was made before cleaning while the sample was dry. The cuticle and the substructure can be distinguished, the slight grooves from the cutting does not influence the substructure.



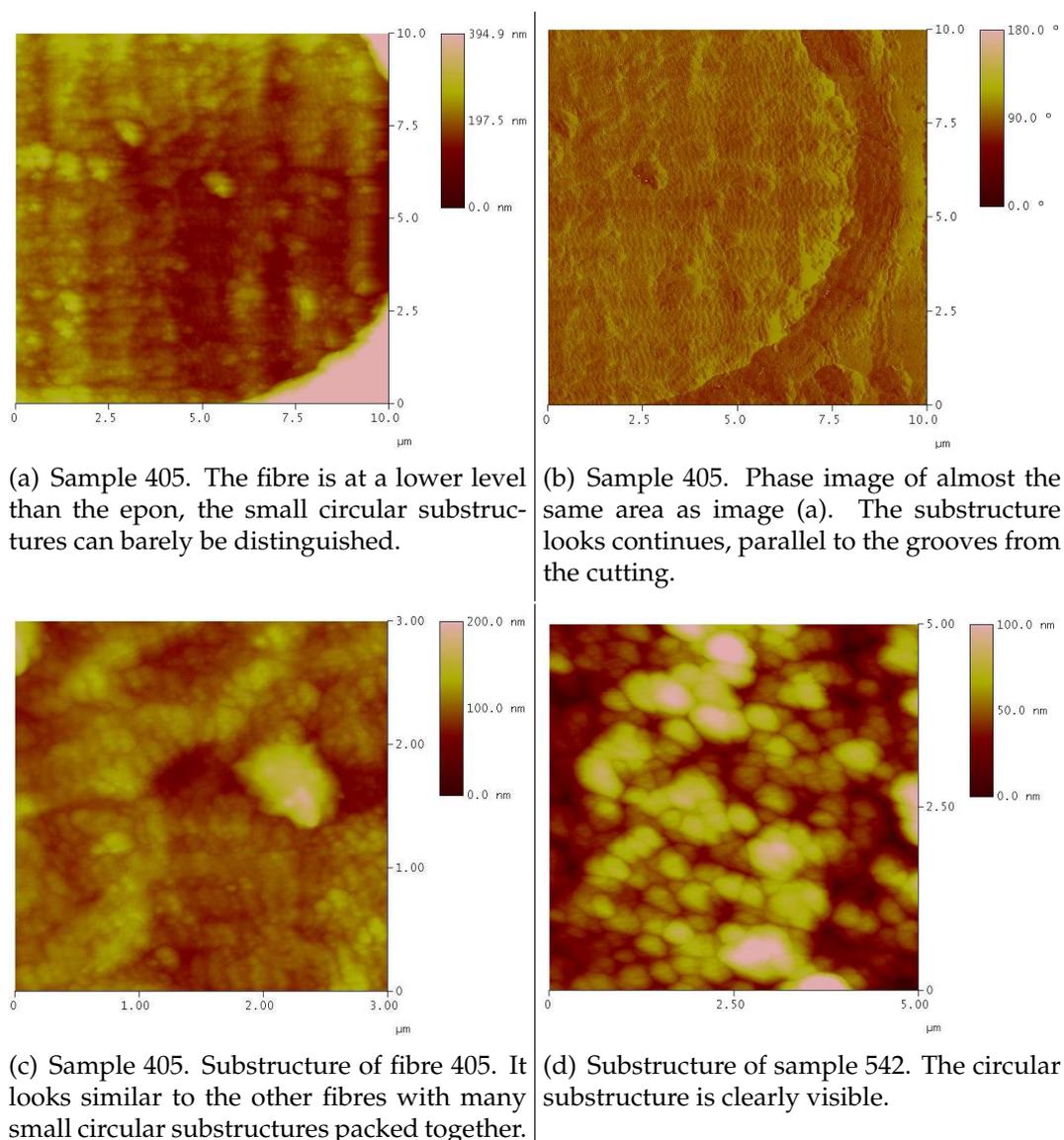
**Figure 5.7:** AFM image of cross-section of sample 452, the image was made when the fibre was dry. The fibre clearly consists of a circular substructure, with a diameter of about 0.5  $\mu\text{m}$ .

### The cortex

The fibres show a structure consisting of many irregular almost circular components, see for example figure 5.8, which when magnified turns out to consist of a smaller, but similar structure. This can be seen in figure 5.9 and 5.10. This structure fits the concept of having macrofibrils consisting of microfibrils. The observed macrofibrils have diameters ranging from 200 - 600 nm.

The macrofibrils appear almost blurred in the height images, see figure 5.9(a), while in the phase images, see figure 5.9(b), they have a defined border which has a lighter colour than the macrofibrils themselves. This border is likely to be either a thin membrane encapsulating the macrofibrils or

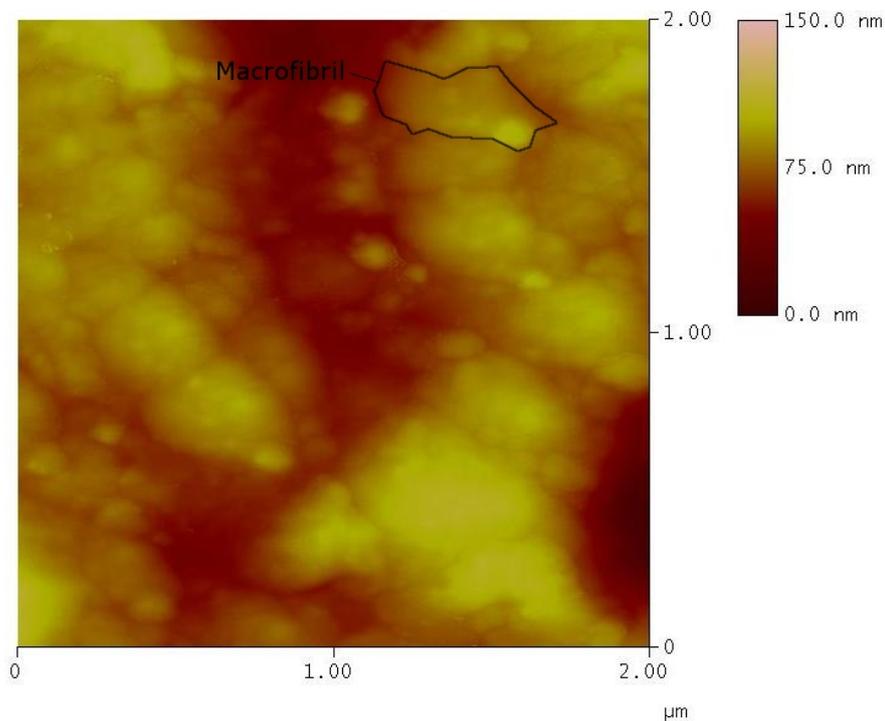
a tiny gap between each macrofibril. In the phase images, such as figure 5.9(b), the macrofibrils have a distinctive edge which has a lighter colour than the macrofibrils themselves.



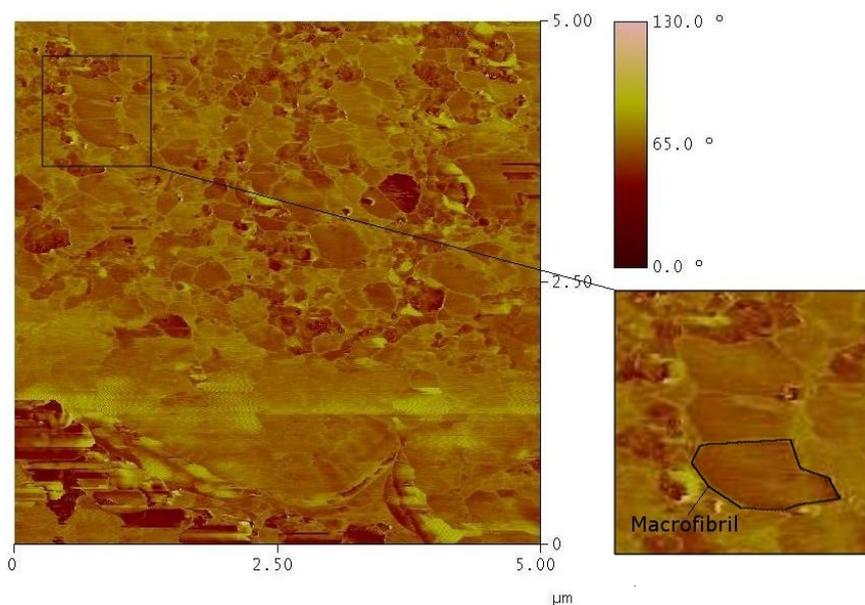
**Figure 5.8:** AFM images of the substructure of different samples.

In figure 5.9(b) there are also some darker areas which are not uniform like the other macrofibrils are, a smaller structure is visible. Figure 3.1 indicates that the orthocortex consists of a clearly circular structure, while the paracortex seems to have a more compact structure, which would be harder to resolve. This implies that the lighter uniform macrofibrils are part of the paracortex and the darker ones are part of the orthocortex. This means, however that the para- and orthocortex are not entirely separated which would be expected for merino wool, see section 3.1.1, and that the cell membrane surrounding the para-cells is just as thin as the separation of

the macrofibrils. This does not seem likely.

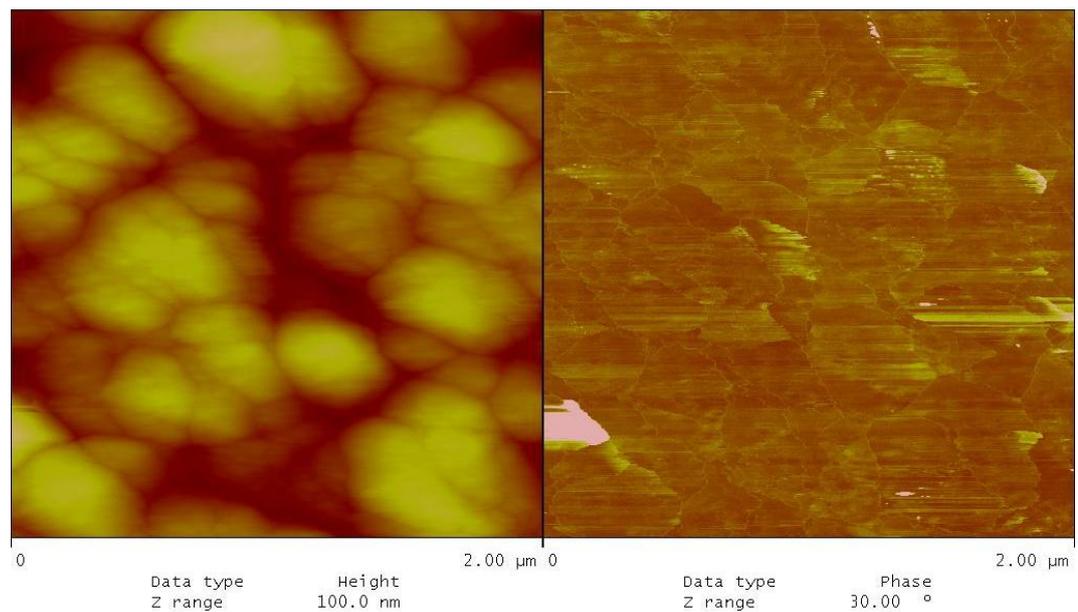


(a) After cleaning. Macrofibrils are visible, there are some uneven regions within the macrofibrils. These might be microfibrils.

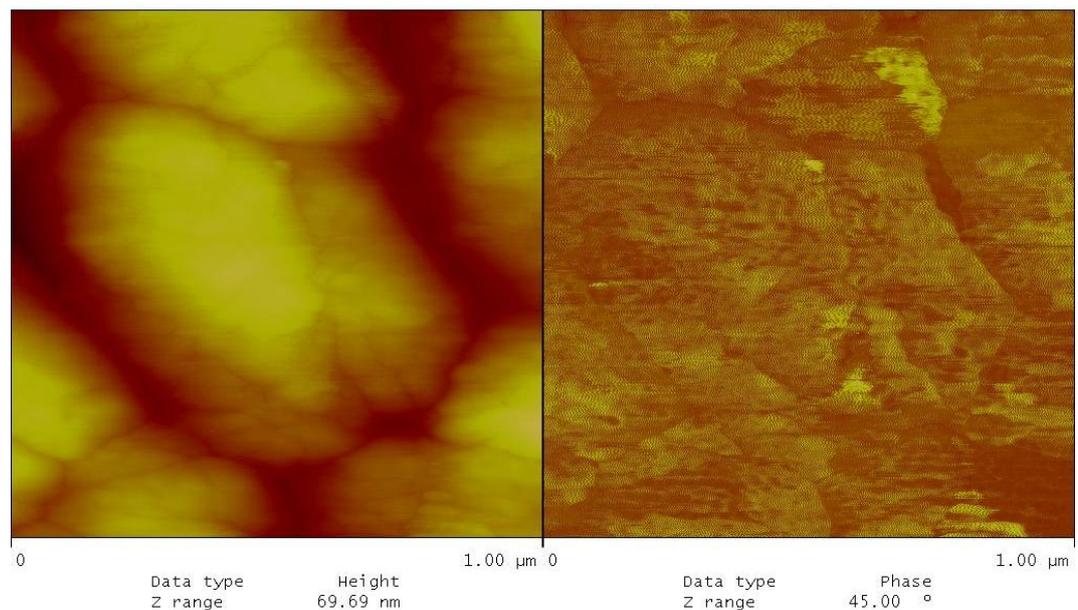


(b) Phase image corresponding to figure 5.12. The macrofibrils of the cortex are clearly visible.

**Figure 5.9:** AFM images of cross-sections of sample 432, all taken when the sample was dry. The indicated macrofibril in figure (a) is in the same group of macrofibrils as the one indicated in (b). They are respectively the upper and the lower of the three similar ones grouped together.



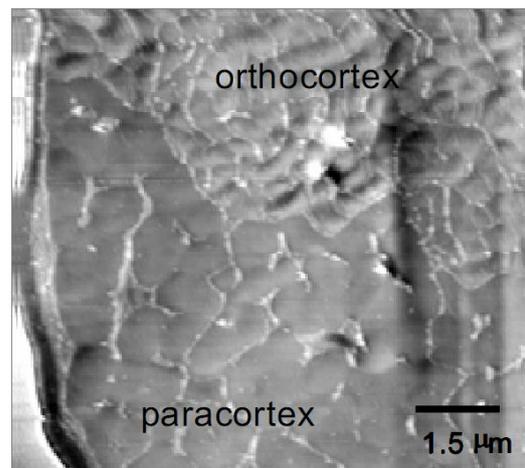
(a) Height image (left) and corresponding phase image(right). Macrofibrils are clearly visible.



(b) Height image (left) and corresponding phase image(right). The phase image reveals the presence of microfibrils within the macrofibril.

**Figure 5.10:** AFM height and phase images of the substructure of fibre 452. Macrofibrils and microfibrils can be distinguished.

Another possibility would be that the darker structures make up the cell membrane, this could fit for the right part of figure 5.9(b), but also there the membrane would at some places be very thin. In the other part of the image there is no continuous line that is long enough to resemble a membrane.



**Figure 5.11:** Electronic force mode image of a wool fibre cross section [18]. The ortho- and para-cortex have been identified, but there are no visible cell membranes present.

In figure 5.11 the para- and ortho-cortex were identified [18]. The cell membrane, however, is not visible there either. Figure 5.10(a) has the same kind of macrofibrils, but only the uniform type.

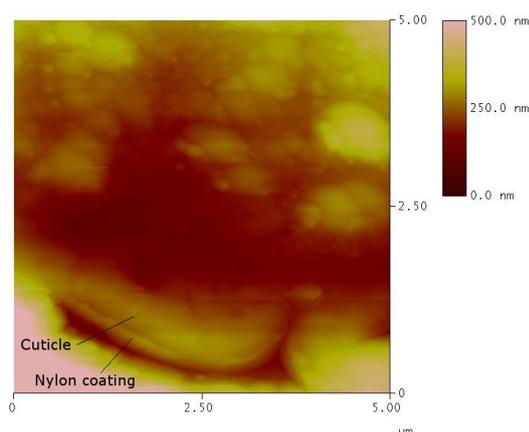
In figure 5.10(b) the phase image reveals a substructure in the macrofibrils, this structure corresponds to microfibrils in a matrix such as sketched in figure 3.1.

In figure 5.8(b) the substructure looks continuous parallel to the grooves from the cutting. Also in figure 5.8(c) it can be seen that the macrofibrils lay closer together in this direction.

### The cuticle

It is usually hard to get a good look at the structure of the cuticle, normally it follows the epon closely like in figure 5.6. It can be hard to determine exactly where the border between the epon and the cuticle is. In figure 5.12 there is an anomaly in the cuticle, it looks bent or broken. This results in the cuticle being separated from the epon, which makes it possible to get a closer look. This reveals a thin external layer which may either be the nylon coating or the epicuticle. The epicuticle is normally about 2.5 nm thick [2], whereas the imaged layer is about 80 nm thick and somewhat uneven. Therefore it is likely that this layer is the nylon coating.

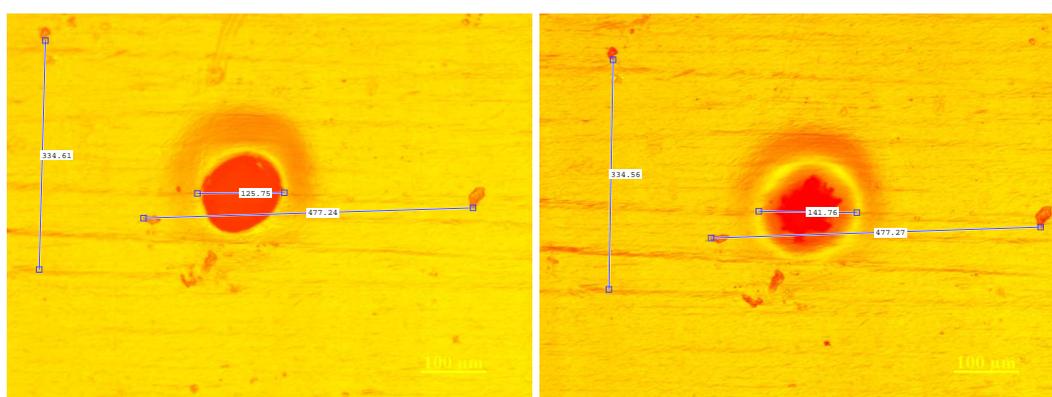
This anomaly might be due to the slight overlap of the cuticle cells which is not strictly along a straight line around the fibre but undulates somewhat, see figure 5.1. If the cross section is made over two different cuticle cells the result might be like this anomaly.



**Figure 5.12:** AFM image of cross-section of sample 405. The image was made after the sample had been cleaned and dried. The nylon coating is visible due to an anomaly in the cuticle.

### 5.2.2 Studies of wet fibre cross sections

In order to moisten the fibres embedded in epon a pipette was used to place a drop over the fibre ends. This drop will then mainly be absorbed by the fibre, but the epon is also likely to absorb some water. To see what happens to small holes in the epon when it is exposed to water, a drop of water was placed over an indentation made with a needle. The result is displayed in figure 5.13. It looks like if the epon absorbs more water in the indentation where the surface has been chipped. Where the surface is undamaged it remains the way it was before wetting, and in the indentation it expands. It may also be noted that in the process of making the indentation, an elevation of the epon around the indentation occurs.



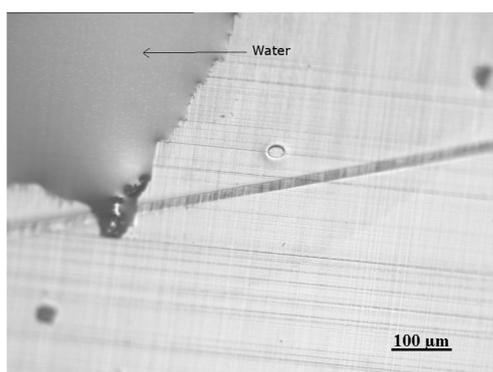
(a) Before wetting.

(b) After wetting.

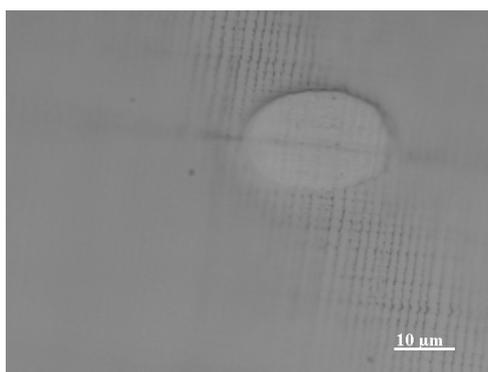
**Figure 5.13:** Images of the epon surface with an indentation before and after wetting. After wetting the diameter of the highest point has increased, but the insides of the indentation has swelled towards the centre.

When the fibres were moistened it resulted in an elevation of the epon around the fibre creating a “crater edge” surrounding the fibre. If this crater

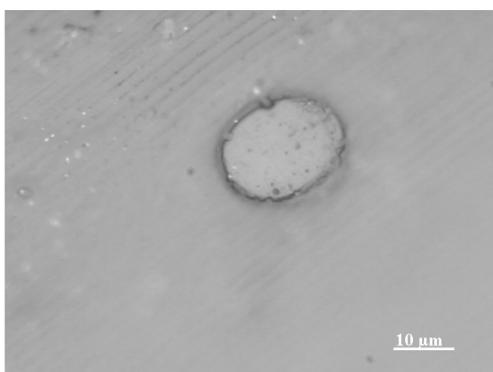
edge is too high it is not possible to image the fibre with the AFM, as the tip will not reach down to the fibre. Figure 4.5 displays fibres that never have been in contact with water, and figure 5.14 displays fibres that have been in contact with water. In figures 5.14(a) and 5.14(b) the epon is clearly elevated around the fibre. Figure 5.14(c) displays a fibre a day after it was moistened. It looks like if the fibre has retracted down into the epon, which is supported by measurements done with AFM. Figure 5.15 displays a section analysis of a fibre that has been left to dry over night.



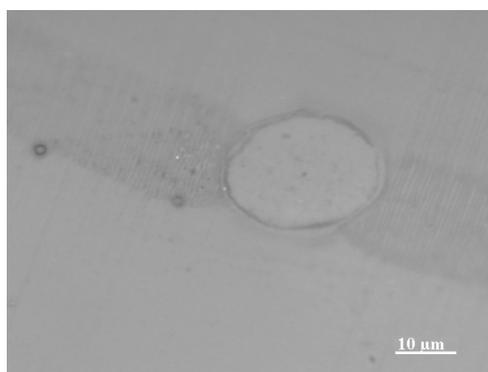
(a) Recently moistened fibre, there is still some water on the epon.



(b) Moistened fibre.



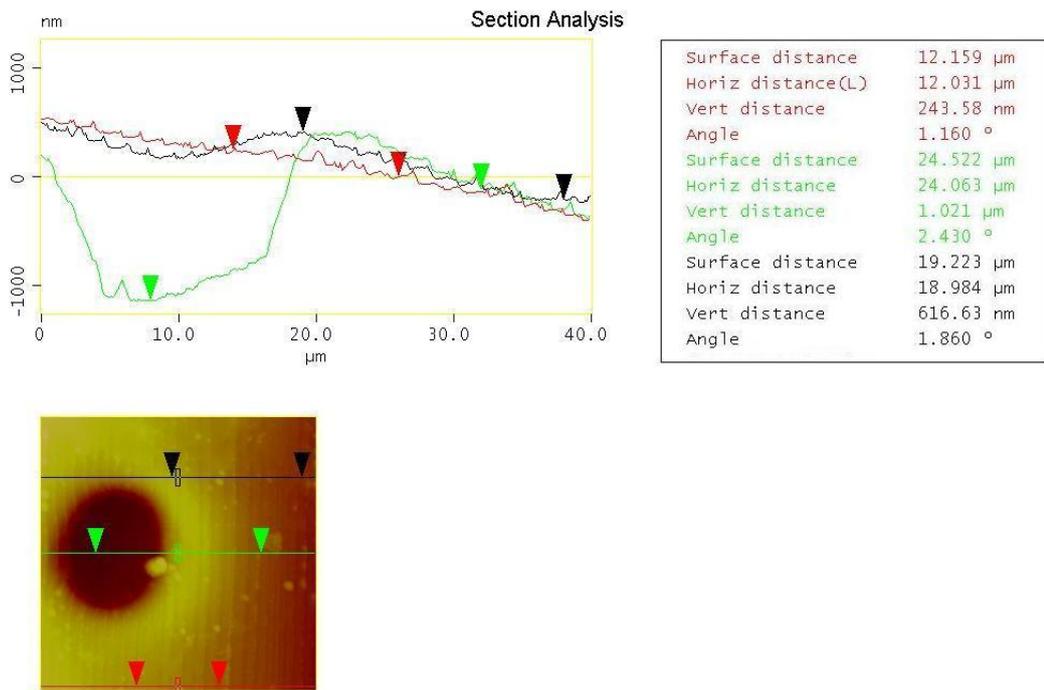
(c) fibre that has been moistened and dried.



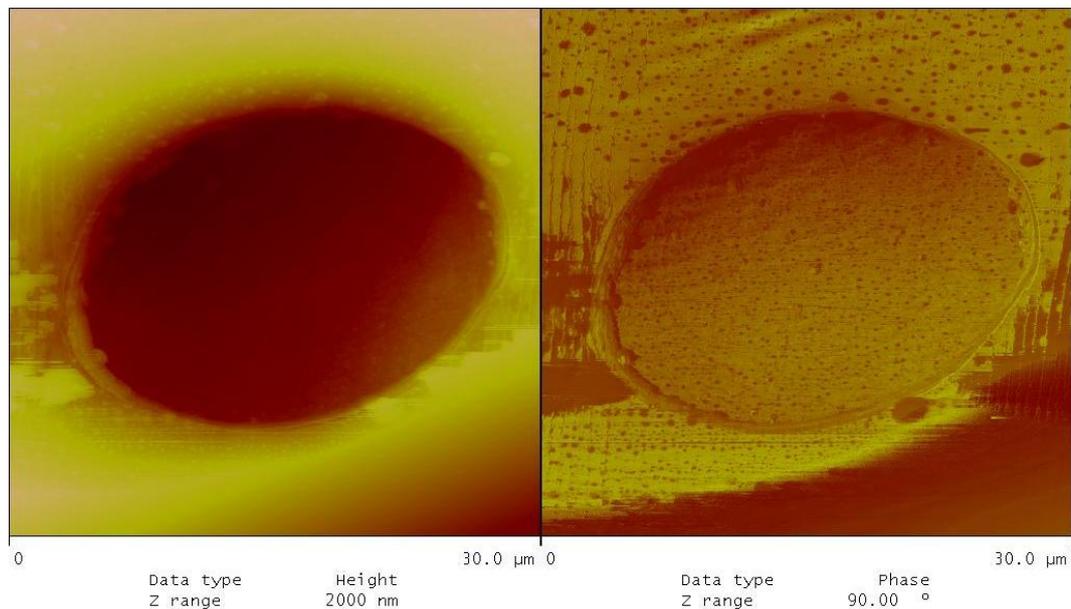
(d) Moistened fibre with agar.

**Figure 5.14:** Optical microscopy images of the effect of moistening embedded fibres. The thick dark line in (a) is the border between the small epon part, used to simplify handling the fibre, and the epon block. The same type of line is also present in image (d) where the fibre is located on this line.

Figure 5.16 displays the cross section of fiber 405, dipped in agar prior to embedding, submerged in water. There are many dots all over the surface. These have not been present on any of the other samples. Therefore it is likely that they are connected to the agar treatment.



**Figure 5.15:** Height profile of a fibre that has been moistened and dried over night. The red line shows that the entire epon block has a constant tilt, the black line indicates the shape of the “crater edge” close to the fibre, and the green line shows that the fibre is located below the normal height of the epon block (the red line) and that the fibre is tilted in a different direction than its surroundings. The data window gives information about the placement of the cursors.

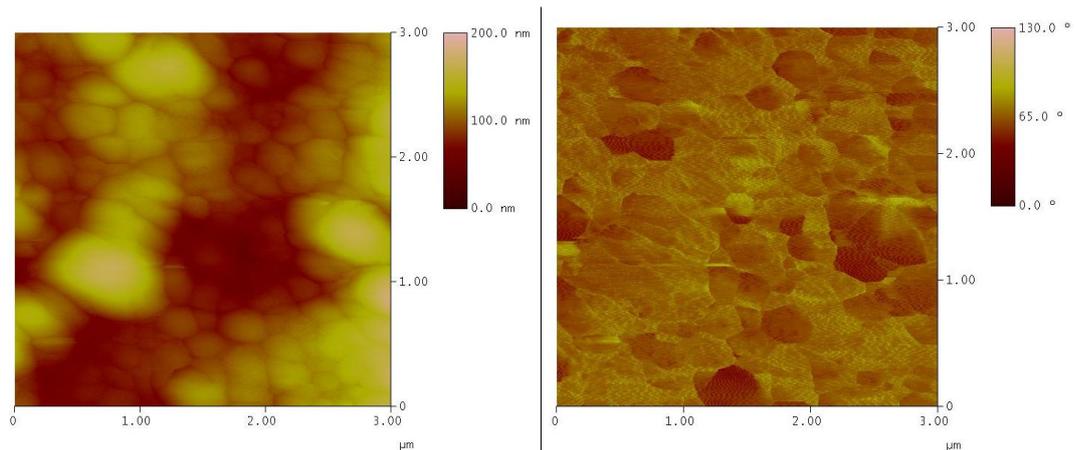


**Figure 5.16:** Height (left) and phase (right) image of the cross section of fiber 405, dipped in agar prior to the embedding, submerged in water. There are many small dots all over the surface.

## Structure analysis

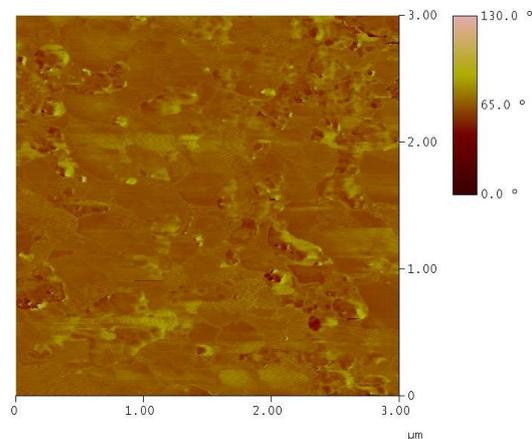
Some selected images from the studies of moist fibres are presented in figures 5.17, 5.19, 5.18 and 5.20.

In figure 5.5(b) the fibre has just been cleaned, most of the contaminations are gone, and the fibre has retracted somewhat into the epon, which has made it hard to see the cuticle. This is most likely caused by the steep edge down to the fibre.



(a) Substructure right after a droplet of water has been absorbed.

(b) Phase image corresponding to (a). The macrofibrils are clearly visible, some are darker than the rest.



(c) Phase image from a scan of the same area as (a) and (b) when the fibre was dry.

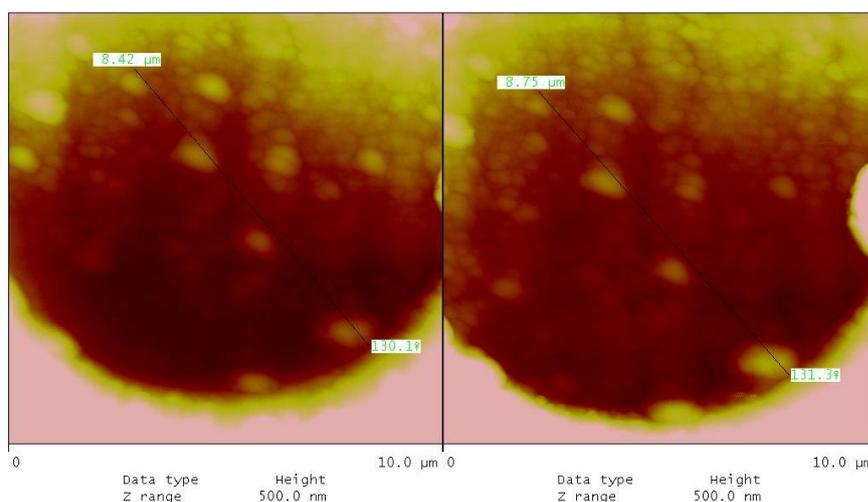
**Figure 5.17:** AFM images of cross-sections of sample 432. Most images are made after the sample was moistened, (c) are from dry fibre for comparison.

Figure 5.17(a) displays the height image of the macrofibrils of the recently moistened fibre, figure 5.17(b) the corresponding phase image and figure 5.17(c) the phase image of the same area from when the fibre was dry. The wet structure seems bloated. The smaller structures have disappeared, but the larger structures are not notably larger. They have, however

changed their shape, they now have softer edges and thereby get the bloated look. The smaller not uniform structures are gone, and replaced with dark macrofibrils that look the same as the lighter ones.

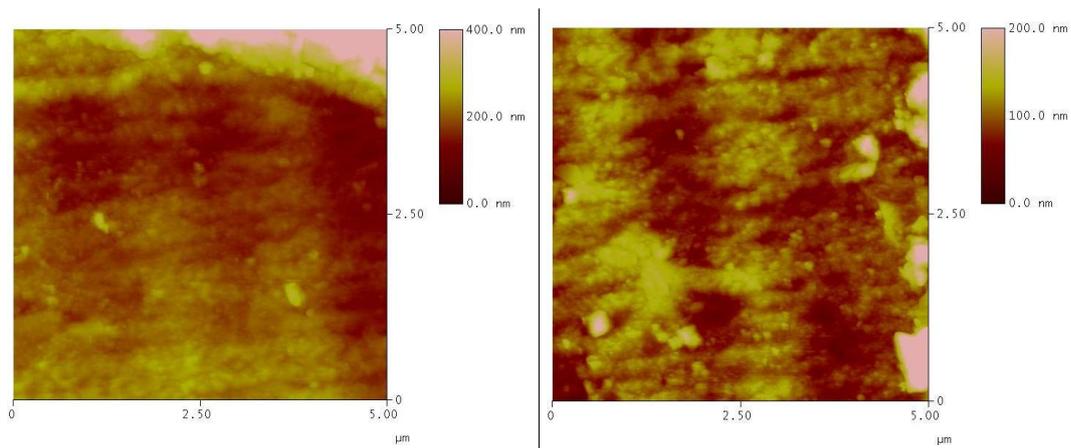
Even though each macrofibril has not changed its size much, there is a small change in the total size of the fibre, see figure 5.18. The measured length within the cross section has increased with 4%, this is much less than the 9% the fibre expanded under the optical microscope. Reasons for this are that the applied drop was smaller, and that the epon might have prevented the fibre from fully expanding.

Figure 5.19 and 5.20 display images with a different structure than the images in figure 5.17. This difference is caused by the wetting technique. The sample in figure 5.17 has been moistened with a droplet, while the other two have been completely submerged in water during the imaging.



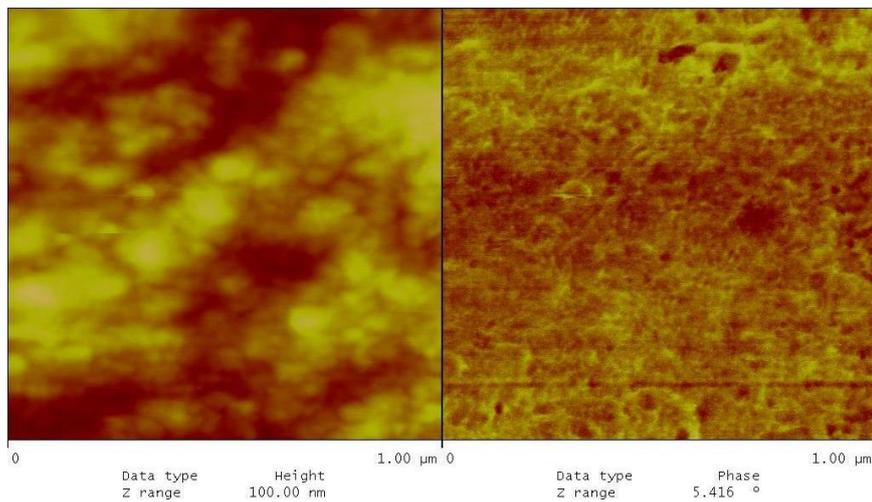
**Figure 5.18:** Comparison of dry (left) and moistened (right) fibre of sample 432. The images are taken right before and right after a droplet was applied. The measured length are 4% longer for the moistened fibre.

In figure 5.19 and 5.20 the structure of the macrofibrils seems to have been dissolved. In figure 5.19 there are instead many even smaller circular structures, diameters less than 70 nm. The phase images display a structure that resembles the structure inside the macrofibrils in the phase image in figure 5.10(b), i.e. the matrix around the microfibrils. Therefore it is likely that the structure seen in figure 5.19 is swollen microfibrils.

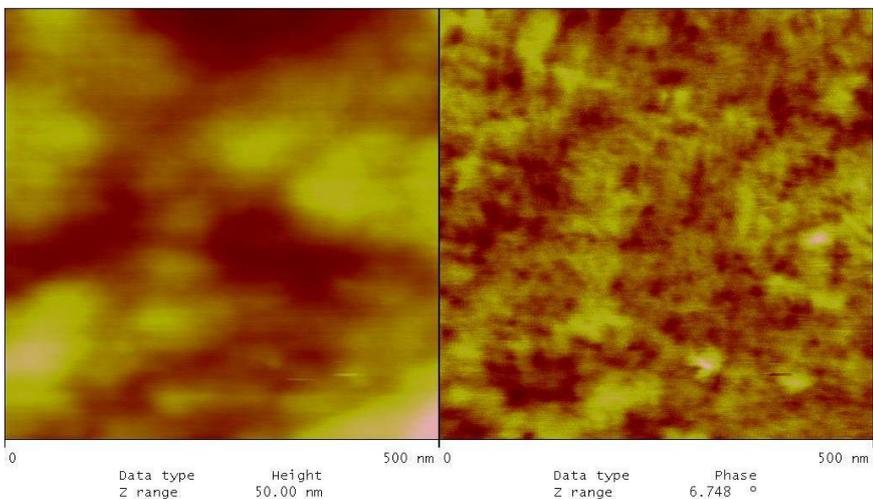


(a) The epon can barely be seen up in the right corner.

(b) The substructure has when submerged many small circular components with a diameter of  $< 70$  nm. There are some striping.

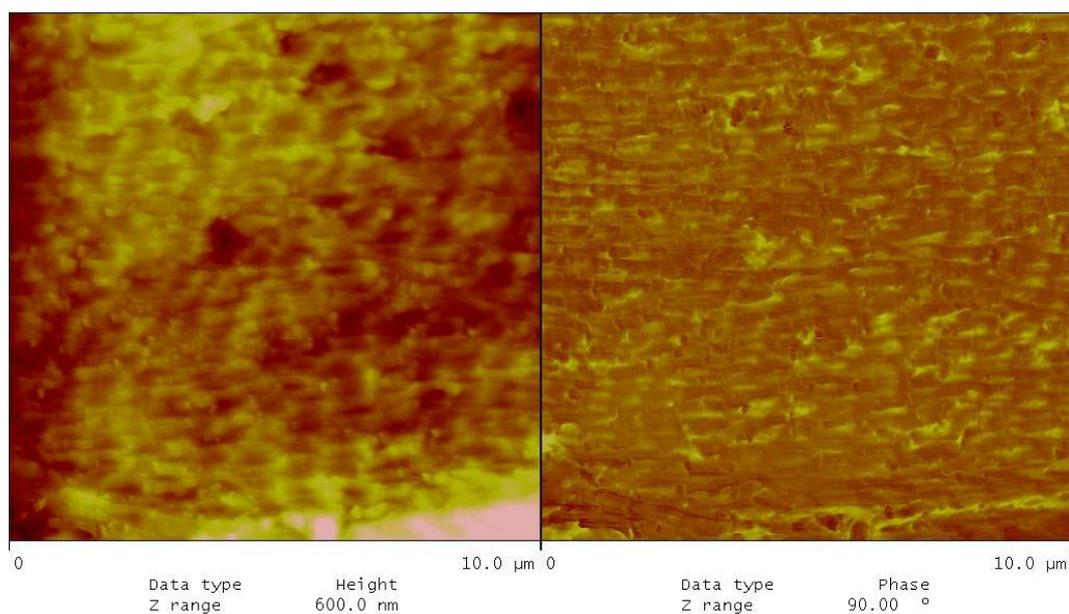


(c) Height image (left) and phase image (right). The phase image looks like a porous matrix structure.

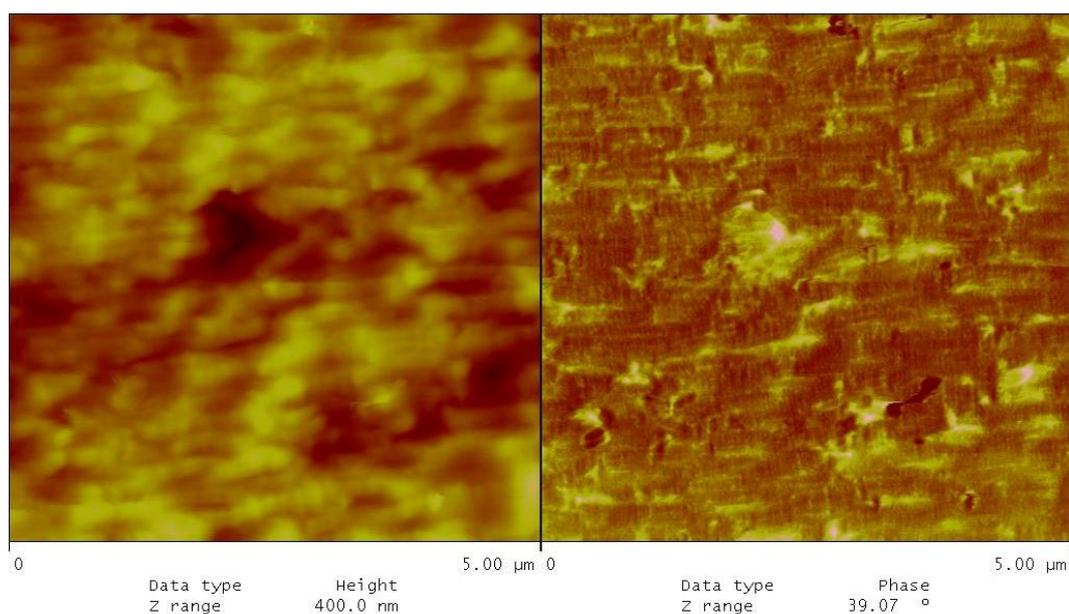


(d) Height image (left) and phase image (right). The phase image looks like a porous matrix structure.

**Figure 5.19:** AFM images of cross-sections of sample 405. All taken while the fibre was submerged in water.



(a) Height image (left) and phase image (right). The substructure comprises some circular structure and a rectangular mesh. The cuticle can just be seen at the bottom of the image.



(b) Height image (left) and phase image (right).

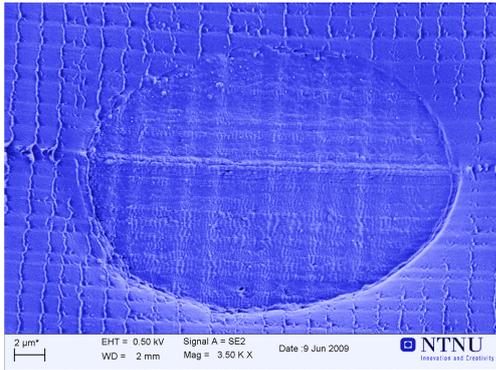
**Figure 5.20:** AFM images of cross-sections of sample 452. All taken while the fibre was submerged in water. The fibre substructure consists of some small circular elements and a rectangular mesh. The rectangles has a size of about  $150 \times 350$  nm.

In figure 5.20 there are only some circular structures, diameter  $< 200$  nm. They are larger than those in figure 5.19, but smaller than the macrofibrils in the dry fibre cross sections, figure 5.9. The rest of the structure makes up a pattern that looks like rectangles. The rectangles have a size of  $150 \times 350$  nm, which is close to the size of the macrofibrils. The origin of the

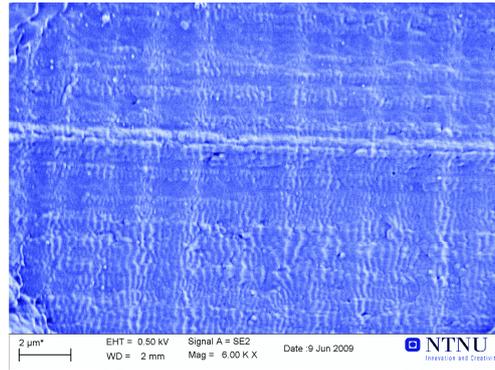
rectangular pattern is not known.

### 5.2.3 SEM

The results of the SEM imaging are presented in figure 5.21, 5.22 and 5.23. The SEM images display the same type of substructure as the AFM image in figure 5.8(b).

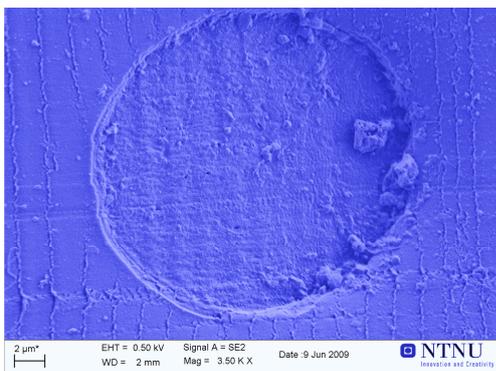


(a) Image of the entire fibre cross section. The double horizontal line crossing the cross section is probably the result of an artefact on the knife while cutting.

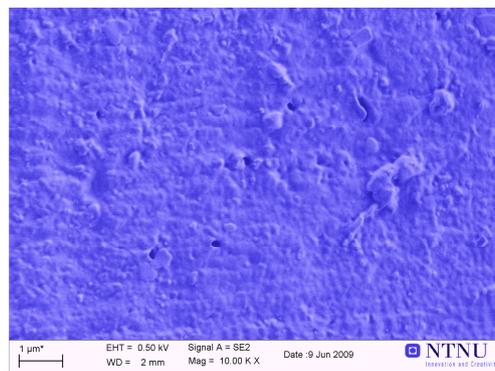


(b) The continuous substructure from figure 5.8(b) are clearly visible.

**Figure 5.21:** SEM images of sample 432. It has been moistened with a water droplet once, a long time before the picture was taken.

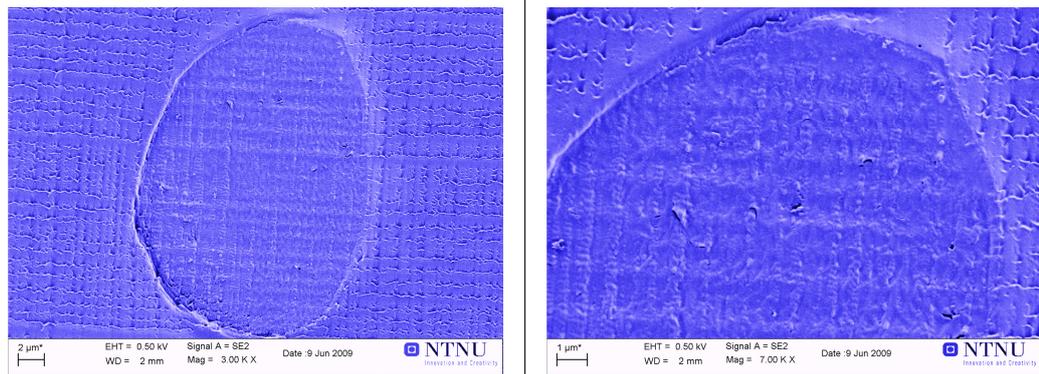


(a) Image of the entire cross section, even with the SEM it is possible to see that the fibre has retracted into the epon.



(b) The continuous substructure is only visible down in the right corner. There are some small holes.

**Figure 5.22:** SEM images of sample 432, this sample has been submerged in water and dried.



(a) Image of the entire cross section. The fibre looks to be more level with the epon. There are many stripes from the cutting in two directions, making it hard to see any structure.

(b) Some holes and partial bits of the continuous structure are visible.

**Figure 5.23:** SEM images of sample 405. This sample has never been in contact with water.

The SEM does not seem to be able to resolve the substructure of the fibre cross section. When the fibre were broken some structure could be seen, see section 4.2.2, but it could not get the same details as with the AFM.



## 6 Conclusion

Both the exterior and the cross section of the wool fibers have been studied using the AFM.

When the exterior of the fiber was imaged with the AFM, only a small part of the fiber was covered. This makes the AFM mainly suited to look at details on the surface of the exterior of the fiber. In cases where the whole fiber is of interest an optical microscope or SEM may be just as useful.

For preparing the cross section samples, the microtomography provided a suitable surface for imaging with AFM, although the grooves from the cutting should be minimized. The embedding material should preferably have as little influence on the expansion of the fiber as possible, epon is not an ideal choice.

It was possible to study the internal structure of the fiber, down to the microfibrils. The phase images proved helpful when studying smaller structures. The AFM provided far more details than the SEM. The structure changed when it was exposed to moisture. Not so much for small amounts of water, where it only looked bloated, but quite profoundly when submerged in water, then the macrofibrils could no longer be recognized and the microfibrils had swollen.

### 6.1 Future work

For future work it would be interesting to study how the internal structure changes as a function of relative humidity. This could be done with a container similar to that in [13]. This might also help in understanding the origin of the structure of the submerged fiber, which also should be studied further. It would also be interesting to study the effects of the *Hercosett*-treatment with respect to the moisture handling properties of the wool fibre.

A better alternative for studying the effect of moistening the exterior of the wool fibers could be to use an environmental scanning electron microscope (ESEM) such as described in [19].

When studying the cross section with respect to moisture it would be interesting to try different embedding materials, mainly more pliable materials. Embedding the fibers while they are wet could also be considered. Also different types of tips for the AFM may be tested to see if it is possible to obtain even more details.

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## References

- [1] S. X. Wang et al. Effect of moisture management on functional performance of cold protective clothing. *Textile Research Journal*, 77:968–980, 2007.
  - [2] W. S. Simpson and G. H. Crawshaw. *Wool: Science and technology*. Woodhead Publishing Ltd, Cornwall, England, 2002.
  - [3] Paul E. West. *Introduction to atomic force microscopy*. Pacific Nanotechnology, 2007.
  - [4] David B. Williams and C. Barry Carter. *Transmission Electron Microscopy - Basics*. Plenum Press, 1996.
  - [5] *Multimode SPM Instruction manual*, 1996.
  - [6] MicroMasch. [www.spmtips.com](http://www.spmtips.com).
  - [7] M. Huson, D. Evans, J. Church, S. Hutchinson, J. Maxwell, and G. Corino. New insights into the nature of the wool fibre surface. *Journal of Structural Biology*, 163:127–136, 2008.
  - [8] I. Schmitz, M. Schreiner, G. Friedbacher, and M. Grasserbauer. Phase imaging as an extension to tapping mode afm for the identification of material properties on humidity-sensitive surfaces. *Applied Surface Science*, 115:190–198, 1997.
  - [9] K. L. Babcock and C. B. Prater. Phase imaging: Beyond topography. *AFM usermeeting*, 2008.
  - [10] W. E. Morton and J. W. S. Hearle. *Physical properties of textile fibres*. Butterworths & Co. Ltd, The Textile Institute, 1975.
  - [11] H. Zahn, F.J. Wortmann, and H. Höcker. Chemie und aufbau der wolle. *Chemie in unserer zeit*, 6:280–290, 1997.
  - [12] J. M. Maxwell and M. G. Huson. Scanning probe microscopy examination of the surface properties of keratin fibres. *Micron*, 36:127–136, 2005.
  - [13] J. M. Maxwell and M. G. Huson. Using the scanning probe microscope to measure the effect of relative humidity on sample stiffness. *Rev. Sci. Instrum.*, 73:3520–3524, 2002.
  - [14] J. A. A. Crossley, C. T. Gibson, L. D. Mapledoram, M. G. Huson, S. Myhra, D. K. Pham, C. J. Sofield, P. S. Turner, and G.S. Watson. Atomic force microscopy analyses of wool fibre surfaces in air and under water. *Micron*, 31:659–667, 2000.
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- [15] L. A. Titcombe, M. G. Huson, and P. S. Turner. Imaging the internal cellular structure of merino wool fibers using atomic force microscopy. *Micron*, 28:69–71, 1997.
- [16] A. N. Parbhu, W. G. Bryson, and R. Lal. Disulfide bounds in the outer layer of keratin fibers confer higher mechanical rigity: Correlative nano-indentation and elasticity measurement with an afm. *Biochemistry*, 38:11755–11761, 1999.
- [17] J. B. Speakman. An analysis of water adsorption isotherm of wool. *Trans. Faraday Soc.*, 40:6–10, 1944.
- [18] J. M. Maxwell and M. G. Huson. Scanning probe microscopy of crosslinked proteins: from textiles to rubbers. *Microsc Microanal*, 11:350–351, 2005.
- [19] Q. F. Wei, X.Q. Wang, and W.D. Gao. Afm and esem characterisation of functionally nanostructured fibers. *Applied Surface Science*, 326:456–460, 2004.
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## **A Data sheets**

### **A.1 Epoxy resin**

Ikke klassifisert som brann- eller eksplosjonsfarlig.

MILJØ:

Ikke klassifisert som miljøfarlig.

#### 4. Førstehjelpstiltak

Generelt	Flytt pasienten vekk fra eksponeringskilden snarest mulig. Sørg for ro, varme og frisk luft. Hvis pasienten er bevisstløs, men puster selv, sørg for frie luftveier og legg i stabil sideleie. Gi kunstig åndedrett ved åndedrettsstans.
Innånding	Flytt straks til frisk luft. Kontakt lege ved vedvarende ubehag.
Hudkontakt	Skyll straks huden grundig med mye vann. Fjern tilsølte klær og skyll huden under. Kontakt lege ved vedvarende ubehag.
Øyekontakt	Skyll straks øyet grundig med mye vann mens øyelokket løftes. Kontakt lege snarest. Fortsett å skylle til medisinsk kyndig person kan overta behandlingen.
Svelging	Gi straks mye vann å drikke. Fremkall ikke brekninger. Kontakt lege omgående.
Annen informasjon	NØDTELEFON: Giftnformasjonssentralen - 22 59 13 00. Medisinsk nødhjelp - 113.

#### 5. Tiltak ved brannslukking

Passende brannslukningsmiddel	Karbondioksid (CO <sub>2</sub> ). Skum. Pulver.
Brann- og eksplosjonsfarer	Brennbar. Ved brann kan det dannes farlige damper/gasser av: Nitrogenoksider (NOx). Ved sterk oppvarming kan det dannes brannfarlige damper/gasser.
Personlig verneutstyr	Evakuer alt personell, benytt verneutstyr for brannslukking.
Annen informasjon	Fjern branntruede beholdere hvis det er mulig uten risiko. Flammesattsatte beholdere kjøles med vann inntil brann er slukket. NØDTELEFON: Brann - 110. Polit - 112.

#### 6. Tiltak ved utilsikket utslipp

Sikkerhetstiltak for å beskytte personell	Sørg for effektiv ventilasjon. Unngå direktekontakt med produktet. Unngå innånding av gass/damp. Benytt egnet verneutstyr.
Sikkerhetstiltak for å beskytte ytre miljø	Forhindre spredning til omgivelsene.
Metoder til opprydding og rengjøring	Spill samles opp med et inaktivt absorpsjonsmateriale, f.eks. Vermikulitt. Oppsamlet materiale lagres på tette, merkede beholdere og behandles som angitt under pkt. 13 - "Fjerning av kjemikalievfall". Små mengder kan destrueres ved brenning.

#### 7. Håndtering og lagring

Håndtering	Unngå søl, hud- og øyekontakt. Unngå sterk oppvarming.
Oppbevaring	Lagres på et tørt, godt ventilert sted, i tett lukket beholder. Anbefalt lagringstemperatur: +15 - +25 C.

#### 8. Eksponeringskontroll / personlig verneutstyr

<b>Administrative normer</b>					
CAS-nr.	EC-nr.	Komponentnavn	8 t. normverdi	ppm/mg/m <sup>3</sup> Kort normverdi	ppm/mg/m <sup>3</sup> Norm år
90-72-2	202-013-9	2,4,6-			

Dette Sikkerhetsdatabladet er utarbeidet i ECO Publisher (ECOonline)

## SIKKERHETSDATABLAD DMP-30

### 1. Identifikasjon av stoffet / produktet og av selskapet / foretaket

Utgitt dato	28.12.2005
Kjemikaliet navn	DMP-30
Kjemisk navn	2,4,6-Tris(dimetylaminometyl)fenol
Synonymer	2,4,6-Tris(dimetylaminometyl)fenol; 2,4,6-Tris(dimethylaminomethyl)phenol;
Artikkelnr.	13600, 21370
CAS-nr.	90-72-2
EC-nr.	202-013-9
Indeksnr.	603-069-00-0
Formel	C15H27N3O
Produktgruppe	Organisk væske.
Kjemikaliet bruksområde	Laboratoriekjemikalie.
Firmanavn	Chem-Teknik as
Besøksadresse	Tvetenveien 30
Postadresse	Tvetenveien 30
Postnr.	0666
Poststed	Oslo
Land	Norge
Telefon	22654100
Telefaks	22657701
E-post	chemi-teknik@chemi-teknik.no
Kontaktperson	Sten Kongsgaarden

### 2. Stoffblandingers sammensetning og stoffenes klassifisering

CAS-nr.	EC-nr.	Komponentnavn	Innhold	Merking/klassifisering	Anm.
90-72-2	202-013-9	2,4,6-Tris(dimetylaminometyl)fenol	85 - 98 %	Xn, R22, R36/38	
Kolonnetilblåring				CAS-nr. = Chemical Abstracts Service; EU (Einecs- eller Elincsnr) = European inventory of Existing Commercial Chemical Substances; Ingrediensnavn = Navn iflg. stoffliste (stoffer som ikke står i stofflisten må oversettes hvis mulig). Innhold oppgitt i: %, %vkt/vkt, %vol/vkt, %vol/vol, mg/m <sup>3</sup> , ppb, ppm, vekt%, vol%	
FH/FB/FM				T+ = Meget giftig, T = Giftig, C = Etsende, Xn = Helsekadelig, Xi = Irriterende, E = Eksplosiv, O = Okstiderende, F+ = Ekstremt brannfarlig, F = Meget brannfarlig, N = Miljøskadelig.	

### 3. Viktigste faremomenter



Helsekadelig

Farebeskrivelse

HELSE:

Farlig ved sveiging.  
Irriterer øynene og huden.

BRANN OG EKSPLOSION:

Dette Sikkerhetsdatabladet er utarbeidet i ECO Publisher (ECOonline)

### 13 Fjerning av kjemikalieavfall

NORSAS	7152
Produktet er klassifisert som farlig avfall	Ja
Egnede metoder til fjerning av kjemikallet	Behandles som farlig avfall. Spill/rester leveres godkjent mottaksstasjon for destruksjon.
Annen informasjon	NORSAS avfallsgruppe: 7152 - Organisk avfall uten halogen.

### 14. Opplysninger om transport

Andre relevante opplysninger Ikke transportklassifisert.

### 15. Opplysninger om lover og forskrifter

#### Faresymbol



Helskadelig	
Sammensettning på merkeetiketten	2,4,6-Tri(dimetylaminometyl)fenol: 85 - 98 %
EC-nr.	202-013-9
R-setninger	R22 Farlig ved sveiging. R36/38 Irriterer øynene og huden.
S-setninger	S26 Får man stoffet i øynene: skyl straks grundig med store mengder vann og kontakt lege. S28 Får man stoff på huden, vask straks med store mengder vann.
Referanser (LowerForskrifter)	S28 Får man stoffet i øynene: skyl straks grundig med store mengder vann. Forskrift om helsefare-, miljøfare-, brannfare- og eksplosjonsfaremerking. Forskrift om stoffliste. Administrative normer for forurensning i arbeidsatmosfære, 2003. Forskrift om avfall. Transport av farlig gods: ADR, RID, IMDG, IATA.

### 16 Andre opplysninger av betydning for helse, miljø og sikkerhet

Erstatter Sikkerhetsdatablad av seksjon 2)	22.02.2005
Liste over relevante R-setninger (i viktigste kilder ved utarbeidelsen av Sikkerhetsdatabladet (ikke norske)	R22 Farlig ved sveiging. R36/38 Irriterer øynene og huden. Datablad fra leverandør/produzent. Arbeidsmiljøseniterets hanskeguide. Arbeidstilsynets brosjyrer om verneutstyr. Hva du må vite når du bruker åndedrettsvern (Orientering, best.nr. 539, Arbeidstilsynet).
Opplysninger som er nye, slettet eller revidert	REVISJONSOVERSIKT: ----- 20.02.91 - Utgitt 21.03.01 - Generell oppdatering. 22.02.05 - Generell oppdatering. 28.12.05 - Pkt 1.

Tri(dimetylaminometyl)fenol

### Eksponeringskontroll

Begrensning av eksponering på arbeidsplassen	Sørg for effektiv ventilasjon. Arbeid i avtrekkskap. Vask hender og ansikt grundig etter arbeid med stoffet. Skift straks forurensete klær. Øyedusj skal finnes på arbeidsplassen.
Åndedrettsvern	Ved utilstrekkelig ventilasjon må det brukes egnet åndedrettsvern. Gassfilter A (organiske stoffer, brunt).
Håndvern	Benytt egnede vernehansker ved fare for hudkontakt. Bruk hansker av: Butyl, Nitril.
Øyevern	Bruk godkjente vernebriller ved fare for øyekontakt.
Annet hudvern enn håndvern	Bruk egnede verneklær ved fare for direkte kontakt med produktet.

### 9. Fysiske og kjemiske egenskaper

Tilstandsform	Væske.
Lukt	Karakteristisk.
Farge	Brunaktig.
Løselighet i vann	Blandbar.
Relativ tetthet	Værdi: 0.973 g/cm3
Kokepunkt/ kokepunktintervall	Værdi: 316 °C
Flammepunkt	Værdi: ~ 160 °C
Damptrykk	Værdi: < 0.01 mmHg (21 C)
Damptetthet	Værdi: > 1
Referanse-gass: (luft=1)	
Andre fysiske og kjemiske egenskaper	Molekylvekt: 265.40

### 10. Stabilitet og reaktivitet

Forhold som skal unngås	Sterk oppvarming.
Materialer som skal unngås	Sterkt oksiderende stoffer. Sterkt reduserende stoffer.
Farlige spaltningsprodukter	Nitrose gasser (NOX).
Stabilitet	Stabil ved normal håndtering.

### 11 Opplysninger om helsefare

#### Øvrige helsefareopplysninger

Generelt	Farlig ved sveiging.
Innånding	Kan forårsake: Irritasjon av luftveiene. Hoste. Pustebesvær.
Hudkontakt	Kan forårsake: Sterk hudirritasjon.
Øyekontakt	Kan forårsake: Sterk øyeirritasjon. Øyeskade.
Sveiging	Kan forårsake: Sterk innvendig irritasjon. Kvalme. Oppkast.
Kroniske effekter	Andre helseskadelige effekter kan ikke utelukkes.

### 12. Miljøopplysninger

#### Toksikologisk informasjon

Akvatisk kommentarer Kvantitative data for akvatisk toksisitet er ikke tilgjengelig.

#### Øvrige miljøopplysninger

Økotoksitet	Miljøskadelige effekter er ikke forventet, men kan ikke utelukkes.
Mobilitet	Produktet er oppløselig i vann.
Persistens og nedbrytbarhet	Informasjon om biologisk nedbrytbarhet er ikke tilgjengelig.
Bioakkumulasjonspotensial	Informasjon om bioakkumulasjonspotensialet er ikke tilgjengelig.
Andre skadevirkninger / annen informasjon	Produktet må behandles med varsomhet og utslipp til miljøet unngås.

Dette Sikkerhetsdatablad er utarbeidet i ECO Publisher (ECOonline)

Dette Sikkerhetsdatablad er utarbeidet i ECO Publisher (ECOonline)

## MILJØ:

Ikke klassifisert som miljøfarlig.

#### 4. Førstehjelpstiltak

Generelt	Flytt pasienten vekk fra eksponeringskilden snarest mulig. Sørg for ro, varme og frisk luft. Hvis pasienten er bevisstløs, men puster selv, sørg for frie luftveier og legg i stabilt sideleie. Gi kunstig åndedrett ved åndedrettsstans.
Innånding	Flytt straks til frisk luft. Kontakt lege ved vedvarende ubehag.
Hudkontakt	Skyll straks huden grundig med mye vann. Fjern tilsette klær og skyll huden under. Kontakt lege ved vedvarende ubehag.
Øyekontakt	Skyll straks øyet grundig med mye vann mens øyelokket løftes. Fortsett å skylle i minst 15 minutter. Kontakt lege.
Svelging	Fremkall ikke brekninger. Kontakt lege ved vedvarende ubehag.
Annenn informasjon	NØDTELEFON: Giftnformasjonssentralen - 22 59 13 00. Medisinsk nødhjelp - 113.

#### 5. Tiltak ved brannslukking

Passende brannslukningsmiddel	Vann. Karbondioksid (CO2). Skum. Pulver.
Brann- og eksplosjonsfarer	Brennbar. Ved sterk oppvarming kan det dannes brannfarlige damper/gasser.
Personlig verneutstyr	Evakuer alt personell, benytt verneutstyr for brannslukking.
Annenn informasjon	Fjern branntruede beholdere hvis det er mulig uten risiko. Flammeutsatte beholdere kjøles med vann inntil brann er slukket. NØDTELEFON: Brann - 110. Politte - 112.

#### 6. Tiltak ved utilsiktet utslipp

Sikkerhetstiltak for å beskytte personell	Sørg for effektiv ventilasjon. Unngå direktekontakt med produktet. Unngå innånding av gass/damp. Benytt egnet verneutstyr.
Sikkerhetstiltak for å beskytte ytre miljø	Forhindre spredning til omgivelse.
Metoder til opprydding og rengjøring	Spill samles opp med et inaktivt absorpsjonsmateriale, f.eks. Vermikulitt. Oppsamlet materiale lagres på tette, merkede beholdere og behandles som angitt under pkt. 13 - "Fjerning av kjemikalieavfall". Rengjør forurenset område. Små mengder kan destrueres ved brenning.

#### 7. Håndtering og lagring

Håndtering	Unngå søl, hud- og øyekontakt. Unngå sterk oppvarming.
Oppbevaring	Lagres på et tørt, godt ventilert sted, i tett lukket beholder. Anbefalt lagringstemperatur: +5 - +30 C.

#### 8. Eksponeringskontroll / personlig verneutstyr

CAS-nr.	EC-nr.	Komponentnavn	8 t. normverdi	ppm/mg/m3	Kort normverdi	ppm/mg/m3	Norm år
25377-73-5	246-917-1	Dodecenylosuccinicanhydrid					

#### Eksponeringskontroll

Begrensning av eksponering på arbeidsplassen  
Sørg for effektiv ventilasjon. Vask hender og ansikt grundig etter arbeid med stoffet. Skift straks forurensete klær. Øyedusj skal finnes på arbeidsplassen.

Dette Sikkerhetsdatabladet er utarbeidet i ECO Publisher (ECOonline)

# SIKKERHETSDATABLAD

## DDSA

### 1. Identifikasjon av stoffet / produktet og av selskapet / foretaket

Utgitt dato	28.12.2005
Kjemikaliets navn	DDSA
Kjemisk navn	Dodecenylosuccinicanhydrid
Synonymer	Dodecenylosuccinic anhydride; Dodecenylobersteinsyreanhydrid; 3-(2-Dodecylen-1-yl)-dihydro-2,5-furandione;
CAS-nr.	25377-73-5
EC-nr.	246-917-1
Formel	C16H26O3
Produktgruppe	Organisk væske.
Kjemikaliets bruksområde	Laboratoriekjemikalie.
<b>Importør/Omsetter</b>	
Firmanavn	Chemi-Teknik as
Besøksadresse	Tvetenveien 30
Postadresse	Tvetenveien 30
Postnr.	0666
Poststed	Oslo
Land	Norge
Telefon	22654100
Telefaks	22657701
E-post	chemi-teknik@chemi-teknik.no
Kontaktperson	Sten Kongsgaarden

### 2. Stoffblandingers sammensetning og stoffenes klassifisering

CAS-nr.	EC-nr.	Komponentnavn	Innhold	Merking/klassifisering	Anm.
25377-73-5	246-917-1	Dodecenylosuccinicanhydrid	70 - 100 %	Xi; R36/38	
Kolonnetforklaring					
CAS-nr. = Chemical Abstracts Service; EU (Einecs- eller Elincnummer) = European inventory of Existing Commercial Chemical Substances; Ingrediensnavn = Navn iflg. stoffliste (stoffer som ikke står i stofflisten må oversettes hvis mulig). Innhold oppgitt i %, %vkt/vkt, %vol/vol, mg/m3, ppb, ppm, vekt%, vol%					
FH/FB/FM					
T+ = Meget giftig, T = Giftig, C = Etsende, Xn = Helsekadelig, Xi = Irriterende, E = Eksplosiv, O = Okksiderende, F+ = Ekstremt brannfarlig, F = Meget brannfarlig, N = Miljøskadelig.					

### 3. Viktigste faremomenter



Inferierende

Farebeskrivelse

HELSE:  
Irriterer øynene og huden.

BRANN OG EKSPLOSJON:  
Ikke klassifisert som brann- eller eksplosjonsfarlig.

Dette Sikkerhetsdatabladet er utarbeidet i ECO Publisher (ECOonline)

Egnede metoder til fjerning av kjemikaliet

Behandles som farlig avfall. Spill/rester leveres godkjent mottaksstasjon for destruksjon.

Annenn informasjon

NORSAS avfallsgruppe: 7152 - Organisk avfall uten halogen.

## 14. Opplysninger om transport

Andre relevante opplysninger

Ikke transportklassifisert.

## 15. Opplysninger om lover og forskrifter

### Faresymbol



Inferierende

Sammensetning på merkeetiketten

Dodeceny Succinicanhydrid: 70 - 100 %

EC-nr.

246-917-1

R-setninger

R36/38 Irriterer øynene og huden.

S-setninger

S26 Får man stoffet i øynene; skylk straks grundig med store mengder vann og kontakt lege.

Referanser (Lover/Forskrifter)

Forskrift om helsefare-, miljøfare-, brannfare- og eksplosjonsfaremerking.

Forskrift om stoffliste.

Administrative normer for forurensning i arbeidsatmosfære, 2003.

Forskrift om avfall.

Transport av farlig gods: ADR, RID, IMDG, IATA.

## 16 Andre opplysninger av betydning for helse, miljø og sikkerhet

Erstatter Sikkerhetsdatablad av 13.05.2002

Liste over relevante R-setninger (i seksjon 2)

R36/38 Irriterer øynene og huden.

Viktigste kilder ved utarbeidelsen av Sikkerhetsdatabladet (ikke norske)

Datablad fra leverandør/produzent.

Arbeidsmiljøsementerets hanskeguide.

Arbeidstilsynets brosjyrer om verneutstyr.

Hva du må vite når du bruker åndedrettsvern (Orientering, best.nr. 539, Arbeidstilsynet).

Opplysninger som er nye, slettet eller revidert

REVISJONSOVERSIKT:

13.05.02 - Utgitt

28.12.05 - Generell oppdatering.

Åndedrettsvern

Bruk egnet åndedrettsvern ved fare for innånding av damp/gass. Gassfilter: A (organiske stoffer, brunt).

Håndvern

Benytt egnede vernehansker ved fare for hudkontakt. Bruk hansker av: Butyl, Nitril.

Øyevern

Bruk godkjente vernebriller ved fare for øyekontakt.

Annet hudvern enn håndvern

Bruk egnede verneklær ved fare for direkte kontakt med produktet.

## 9. Fysiske og kjemiske egenskaper

Tilstandsform

Væske.

Lukt

Karakteristisk.

Farge

Lys gul.

Løselighet i vann

Ikke blandbar.

Løselighet i fett

Acelon. Etanol.

Relativ tetthet

Verdi: 1,01 g/cm3

Kokepunkt/ kokepunktintervall

Verdi: 200 °C

Flammepunkt

Verdi: 178 °C

Viskositet

Testmetode: o.c.

Verdi: 85 mPas (20 C)

## Andre fysiske og kjemiske egenskaper

Fysiske og kjemiske egenskaper

Molekylvekt: 266.38

## 10. Stabilitet og reaktivitet

Forhold som skal unngås

Sterk oppvarming.

Materialer som skal unngås

Sterkt oksiderende stoffer.

Stabilitet

Stabil ved normal håndtering.

## 11 Opplysninger om helsefare

### Toksikologiske data fra komponenter

#### Øvrige helsefareopplysninger

Innånding

Kan forårsake: Lett irritasjon av luftveiene.

Hudkontakt

Kan forårsake: Hudirritasjon.

Øyekontakt

Kan forårsake: Øyeirritasjon.

Kroniske effekter

Andre helsefarlige effekter er ikke forventet, men kan ikke utelukkes.

## 12. Miljøopplysninger

### Toksikologisk informasjon

Akvatisk kommentarer

Kvantitative data for akvatisk toksisitet er ikke tilgjengelig.

### Toksikologiske data fra komponenter

#### Øvrige miljøopplysninger

Økotoksisitet

Miljøskadelige effekter er ikke forventet, men kan ikke utelukkes.

Mobilitet

Produktet er uoppløselig i vann.

Persistens og nedbrytbarhet

Informasjon om biologisk nedbrytbarhet er ikke tilgjengelig.

Bioakkumulasjonspotensial

Informasjon om bioakkumulasjonspotensialet er ikke tilgjengelig.

Andre skadevirkninger / annen informasjon

Produktet må behandles med varsomhet og utslipp til miljøet unngås.

## 13 Fjerning av kjemikalieavfall

NORSAS

7152

Produktet er klassifisert som farlig avfall

Ja

#### 4. Førstehjelpstiltak

Generelt	Flytt pasienten vekk fra eksponeringskilden snarest mulig. Sørg for ro, varme og frisk luft. Hvis pasienten er bevisstløs, men puster selv, sørg for frie luftveier og legg i stabilt sideleie. Gi kunstig åndedrett ved åndedrettslans.
Innånding	Flytt straks den eksponerte til frisk luft. Kontakt lege ved vedvarende ubehag.
Hudkontakt	Skyl straks huden grundig med mye vann. Fjern tilsette klær og skyl huden under. Kontakt lege ved vedvarende ubehag.
Øyekontakt	Skyl straks øyet grundig med mye vann mens øyelokket løftes. Fortsett å skylle i minst 15 minutter. Kontakt lege.
Svelging	Gi straks mye vann å drikke. Fremkall ikke brekninger. Kontakt lege ved vedvarende ubehag.
Annen informasjon	NØDTELEFON: Giftnformasjonssentralen - 22 59 13 00. Medisinsk nødhjelp - 113.

#### 5. Tiltak ved brannslukking

Passende brannslukningsmiddel	Karbondioksid (CO <sub>2</sub> ). Pulver.
Brennbar- og eksplosjonsfarer	Brennbar. Ved brann kan det utvikles farlige damper/gasser.
Personlig verneutstyr	Evakuer alt personell, benytt verneutstyr for brannslukning.
Annen informasjon	Fjern branntruede beholdere hvis det er mulig uten risiko. Flammeutsatte beholdere kjøles med vann inntil brann er slukket. NØDTELEFON: Brann - 110. Politi - 112.

#### 6. Tiltak ved utilsiktet utslipp

Sikkerhetsiltak for å beskytte personell	Sørg for effektiv ventilasjon. Unngå direktekontakt med produktet. Unngå innånding av gass/damp. Benytt egnet verneutstyr.
Sikkerhetsiltak for å beskytte ytre miljø	Forhindre spredning til omgivelse.
Metoder til opprydding og rengjøring	Spill samles opp med et inaktivt absorpsjonsmateriale, f.eks. Vermekullit. Oppsamlet materiale lagres på tette, merkede beholdere og behandles som angitt under pkt. 13 - "Fjerning av kjemikalievfall". Rengjør forurenset område.
	Små mengder kan løses i mye vann og spyles til avløp.

#### 7. Håndtering og lagring

Håndtering	Unngå søl, hud- og øyekontakt. Unngå sterk oppvarming.
Oppbevaring	Lagres på et tørt, godt ventilert sted, i tett lukket beholder. Oppbevares ved romtemperatur.

#### 8. Eksponeringskontroll / personlig verneutstyr

Eksponeringskontroll	Sørg for effektiv ventilasjon. Arbeid i avtrekkskap. Vask hender og ansikt grundig etter arbeid med stoffet. Skift straks forurensete klær. Øyedeusj skal finnes på arbeidsplassen.
Åndedrettsvern	Ved utilsikkelig ventilasjon må det brukes egnet åndedrettsvern. Gassfilter A (organiske stoffer, brunt).
Håndvern	Benytt egnete vernehansker ved fare for hudkontakt. Bruk hansker av: Butyl, Nitril.
Øyevern	Bruk godkjente vernebriller / ansiktsskjerm ved fare for øyekontakt.

Dette Sikkerhetsdatablad er utarbeidet i ECO Publisher (ECOonline)

## SIKKERHETSDATABLAD LX-112 EPOXY RESIN

### 1. Identifikasjon av stoffet / produktet og av selskapet / foretaket

Utgitt dato	04.05.2005
Kjemikaliets navn	LX-112 EPOXY RESIN
Synonymer	Epoksyharpiks;
Artikkelnr.	Ladd 21310
Produktgruppe	Organisk væske.
Kjemikaliets bruksområde	Imstøpningsmiddel for elektronmikroskopi.
<b>Importør/Omsetter</b>	
Firmanavn	Cheml-Teknik as
Besøksadresse	Tvetenveien 30
Postadresse	Tvetenveien 30
Postnr.	0666
Poststed	Oslo
Land	Norge
Telefon	22654100
Telefaks	22657701
E-post	chemi-teknik@chemi-teknik.no
Kontaktperson	Sten Kongsgaarden

### 2. Stoffblandingers sammensetning og stoffenes klassifisering

CAS-nr.	EC-nr.	Komponentnavn	Innhold	Merking/klassifisering	Anm.
25038-04-4		Glyceroipolymer med klormetyloxiran	100 %	Xi; R36/37/38	
Kolonneforklaring					
				CAS-nr. = Chemical Abstracts Service; EU (Einecs- eller Elinesnummer) = European inventory of Existing Commercial Chemical Substances;	
				Ingrediensnavn = Navn iflg. stoffliste (stoffer som ikke står i stofflisten må oversettes hvis mulig). Innhold oppgitt i: %, %vkt/vkt, %vol/vkt, %vol/vol, mg/m3, ppb, ppm, vekt%, vol%	
FH/FB/CM				T+ = Meget giftig, T = Giftig, C = Etsende, Xn = Helsekadelig, Xi = Irriterende, E = Eksplosiv, O = Oksiderende, F+ = Ekstremt brannfarlig, F = Meget brannfarlig, N = Miljøskadelig.	

### 3. Viktigste faremomenter

	Inferende
Farebeskrivelse	HELSE: Irriterer øynene, luftveiene og huden.  BRANN OG EKSPLOSJON: Ikke klassifisert som brann- eller eksplosjonsfarlig.  MILJØ: Ikke klassifisert som miljøfarlig.

Dette Sikkerhetsdatablad er utarbeidet i ECO Publisher (ECOonline)

**Faresymbol**

Innløpende

Sammensetning på merkeetiketten

Glycerolpolymer med klormetyloxyiran: 100 %  
R36/37/38 Irriterer øynene, luftveiene og huden.

R-setninger

S23 Unngå innånding av gass/damp.

S-setninger

S24/25 Unngå kontakt med huden og øynene.

S26 Får man stoffet i øynene: skyl straks grundig med store mengder vann og kontakt lege.

S37/39 Bruk egnete vernehansker og vernebriller/ansiktskjermer.

Referanser (Lower/Forskrifter)

Forskrift om helsefare-, miljøfare-, brannfare- og eksplosjonsfaremerking.

Forskrift om stoffliste.

Administrative normer for forurensning i arbeidsatmosfære, 2001.

Forskrift om spesialavfall.

Transport av farlig gods: ADR, RID, IMDG, IATA.

**16 Andre opplysninger av betydning for helse, miljø og sikkerhet**

Erstatter Sikkerhetsdatablad av

14.05.2000

Liste over relevante R-setninger (i

seksjon 2)

Viktigste kilder ved utarbeidelsen av

Sikkerhetsdatabladet (ikke norske)

Arbeidsmiljøsentrets hanskeguide.

Arbeidstilsynets brosjyrer om verneutstyr.

Hva du må vite når du bruker åndedrettsvern ( Orientering, best.nr. 539,

Arbeidstilsynet).

REVISJONSOVERSIKT:

14.05.00 - Utgitt

04.05.05 - Generell oppdatering.

Annet hudvern enn håndvern

Bruk egnete verneklær ved fare for direkte kontakt med produktet.

**9. Fysiske og kjemiske egenskaper**

Tilstandsform

Væske.

Lukt

Svak.

Farge

Fargeløs. Lys gul.

Løselighet i vann

Lett blandbar.

Løselighet i fett

Aceton, Metanol.

Relativ tetthet

Verdi: 1,23 g/cm3

Flammepunkt

Verdi: 76 °C

Damptrykk

Testmetode: o.c.

Verdi: 1 mm Hg (25 C)

**10. Stabilitet og reaktivitet**

Forhold som skal unngås

Sterk oppvarming.

Materialer som skal unngås

Sterkt oksiderende stoffer. Sterke syrer. Sterke baser.

Stabilitet

Stabil ved normal håndtering.

**11 Opplysninger om helsefare****Toksikologisk informasjon**

Andre toksikologiske data

Kvantitative data for toksikologisk effekt er ikke tilgjengelig.

**Øvrige helsefareopplysninger**

Innånding

Kan forårsake: Irritasjon av luftveiene.

Hudkontakt

Kan forårsake: Hudirritasjon. Virker avfettende / uttørrkende på huden.

Øyekontakt

Kan forårsake: Øyeirritasjon.

Sveiging

Kan forårsake: Innvendig irritasjon. Kvalme. Oppkast. Diare.

Kroniske effekter

Andre helsefarlige effekter er ikke forventet, men kan ikke utelukkes.

**12. Miljøopplysninger****Toksikologisk informasjon**

Kvalitativ kommentarer

Kvantitative data for akuttisk toksisitet er ikke tilgjengelig.

**Øvrige miljøopplysninger**

Økotoksisitet

Miljøskadelige effekter er ikke forventet, men kan ikke utelukkes.

Mobilitet

Produktet er lett oppløselig i vann.

Peristens og nedbrytbarhet

Produktet er biologisk lett nedbrytbart.

Bioakkumulasjonspotensial

Bioakkumulering er ikke forventet.

Andre skadevirkninger / annen

informasjon

Produktet må behandles med varsomhet og utslipp til miljøet unngås.

**13 Fjerning av kjemikalieavfall**

NORSAS

7151 - Organisk avfall med halogen

Produktet er klassifisert som farlig

Ja

avfall

Egnede metoder til fjerning av

Behandles som spesialavfall. Spill/resser leveres godkjent mottaksstasjon for

kjemikaliet

destruksjon.

**14. Opplysninger om transport**

Andre relevante opplysninger

Ikke transportklassifisert.

**15. Opplysninger om lover og forskrifter**

**BRANN OG EKSPLOSJON:**

Brennbar.

**MILJØ:**

Ikke klassifisert som miljøfarlig.

**4. Førstehjelpstiltak**

Generelt	Flytt pasienten vekk fra eksponeringskilden snarest mulig. Sørg for ro, varme og frisk luft. Hvis pasienten er bevisstløs, men puster selv, sørg for frie luftveier og legg i stabilt sideleie. Gi kunstig åndedrett ved andedrettsstans.
Innånding	Flytt straks til frisk luft. Kontakt lege ved vedvarende ubehag.
Hudkontakt	Skyll straks huden grundig med mye vann. Fjern tilsette klær og skyll huden under. Kontakt lege ved vedvarende ubehag.
Øyekontakt	Skyll straks øyet grundig med mye vann mens øyelokket løftes. Fortsett å skylle i minst 15 minutter. Kontakt lege hvis ikke alt ubehag gir seg.
Svelging	Gi straks mye vann å drikke. Fremkall ikke brekninger. Kontakt lege omgående.
Annen informasjon	NØDTELEFON: Giftnormasjonssentralen - 22 59 13 00. Medisinsk nødhjelp - 113.

**5. Tiltak ved brannslukking**

Passende brannslukningsmiddel	Skum, Karbondioksid (CO <sub>2</sub> ).
Brennbar- og eksplosjonsfarer	Brennbar. Dampene kan danne eksplosive blandinger med luft.
Personlig verneutstyr	Evakuer alt personell, benytt verneutstyr for brannslukking.
Annen informasjon	Fjern branntruede beholdere hvis det er mulig uten risiko. Flammeutsatte beholdere kjøles med vann inntil brann er slukket. NØDTELEFON: Brann - 110. Polit - 112.

**6. Tiltak ved utilsikket utslipp**

Sikkerhetstiltak for å beskytte personell	Sørg for effektiv ventilasjon. Unngå direktekontakt med produktet. Unngå innånding av gass/damp. Benytt egnet verneutstyr.
Sikkerhetstiltak for å beskytte ytre miljø	Forhindre spredning til omgivelsene.
Metoder til opprydding og rengjøring	Spill samles opp med et inaktivt absorpsjonsmateriale, f.eks. Vermekulitt. Oppsamlet materiale lagres på tette, merkede beholdere og behandles som angitt under pkt. 13 - "Fjerning av kjemikalieavfall". Rengjør forurenset område. Små mengder kan destrueres ved brenning.

**7. Håndtering og lagring**

Håndtering	Unngå søl, hud- og øyekontakt. Unngå sterk oppvarming. Unngå fuktighet/lufteksponering.
Oppbevaring	Lagres på et tørt, godt ventilert sted, i tett lukket beholder. Anbefalt lagringstemperatur: +15 - +25 C.

**8. Eksponeringskontroll / personlig verneutstyr**

<b>Administrative normer</b>			
CAS-nr.	EC-nr.	Komponentnavn	8 t. normverdi ppm/mg/m <sup>3</sup> Kort
			ppm/mg/m <sup>3</sup> Norm år

Dette Sikkerhetsdatablad er utarbeidet i ECO Publisher (ECOonline)

# SIKKERHETSDATABLAD

## NMA, LADD 21350

**1. Identifikasjon av stoffet / produktet og av selskapet / foretaket**

Utgitt dato	10.02.2005
Kjemikaliet navn	NMA, LADD 21350
Kjemisk navn	Metylnorbornen-2,3-dikarboxysyreanhydrid
Synonymer	Metylnorbornene-2,3-dicarboxylic anhydride; Metylnorbornen-2,3-dikarboxysyreanhydrid; Nadic methyl anhydride; Methyl nadic anhydride, 1,2,3,6-tetrahydro methyl-3,6-methanophthalic anhydride;
CAS-nr.	25134-21-8
EC-nr.	246-644-8
Formel	C10H10O3
Produktgruppe	Organisk væske.
Kjemikaliet bruksområde	Laboratoriekjemikalie.
Firmanavn	Chemi-Teknik as
Besøksadresse	Tvetenveien 30
Postadresse	Tvetenveien 30
Postnr.	0666
Poststed	Oslo
Land	Norge
Telefon	22654100
Telefaks	22657701
E-post	chemi-teknik@chemi-teknik.no
Kontaktperson	Sten Kongsgaarden

**2. Stoffblandingers sammensetning og stoffenes klassifisering**

CAS-nr.	EC-nr.	Komponentnavn	Innhold	Merking/klassifisering	Anm.
25134-21-8	246-644-8	Metylnorbornen-2,3-dikarboxysyreanhydrid	> 99 %	Xn; R22, R36/37/38, R42	
Kolonnetforklaring					CAS-nr. = Chemical Abstracts Service; EU (Einecs- eller Elincsnnummer) = European inventory of Existing Commercial Chemical Substances; Ingrediensnavn = Navn iflg. stoffliste (stoffer som ikke står i stofflisten må oversettes hvis mulig). Innhold oppgitt i: %, %vkt/vkt, %vol/vol, mg/m <sup>3</sup> , ppb, ppm, vekt%, vol% T+ = Meget giftig, T = Giftig, C = Ettsende, Xn = Helsekadelig, Xi = Irriterende, E = Eksplosiv, O = Oksiderende, F+ = Ekstremt brannfarlig, F = Meget brannfarlig, N = Miljøskadelig.
FH/FB/FM					

**3. Viktigste faremomenter**

Helsekadelig

**Farebeskrivelse**

HELSE:  
Farlig ved sveiging.  
Irriterer øynene, luftveiene og huden.  
Kan gi allergi ved innånding.

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Bioakkumulasjonspotensial	Informasjon om bioakkumulasjonspotensialet er ikke tilgjengelig.
Miljøopplysninger, konklusjon	Produktet må behandles med varsomhet og utslipp til miljøet unngås.

### 13 Fjerning av kjemikalieavfall

NORSAS	7152 - Organisk avfall uten halogen
Produktet er klassifisert som farlig avfall	Ja
Egnede metoder til fjerning av kjemikaliet	Behandles som spesialavfall. Spillrester leveres godkjent mottaksstasjon for destruksjon.

### 14. Opplysninger om transport

Proper Shipping Name	CORROSIVE LIQUID, ACIDIC, ORGANIC, N.O.S.
Varenavn (nasjonalt)	ETSENDE VÆSKE, SUP, ORGANISK, N.O.S.
UN-nr.	3265
Farlig gods ADR/RID	Ja, Klasse:8 Fare nr.:80 Bokstav:C3
Farlig gods IMDG	Ja, Klasse:8 Emballasjegruppe:I
Farlig gods ICAO/IATA	Ja, Klasse:8 Emballasjegruppe:I
Fareseddel	8
Andre relevante opplysninger	Begrenset mengde ADR: LQ22

### 15. Opplysninger om lover og forskrifter

#### Faresymbol



Sammenhengning på merkeetiketten	Metylnorbomen-2,3-dikarboksyreanhydrid: > 99 %
EC-nr.	246-644-8
R-setninger	R22 Farlig ved sveiging. R36/37/38 Irriterer øynene, luftveiene og huden. R42 Kan gi allergi ved innånding.
S-setninger	S39 Bruk vernebriller/ansiktskjermer.
Referanser (Lower/Forskrifter)	Forskrift om helsefare-, miljøfare-, brannfare- og eksplosjonsfaremerking. Forskrift om stoffliste. Administrative normer for forurensning i arbeidsatmosfære, 2001. Forskrift om spesialavfall. Transport av farlig gods: ADR, RID, IMDG, IATA.

### 16 Andre opplysninger av betydning for helse, miljø og sikkerhet

Erstatter Sikkerhetsdatablad av	05.04.2001
Liste over relevante R-setninger (i seksjon 2)	R22 Farlig ved sveiging. R36/37/38 Irriterer øynene, luftveiene og huden. R42 Kan gi allergi ved innånding.
Viktigste kilder ved utarbeidelsen av Sikkerhetsdatabladet (ikke norske)	Datablad fra leverandør/produzent. Arbeidsmiljøsementerets hantskeguide. Arbeidstilsynets brosjyrer om verneutstyr. Hva du må vite når du bruker åndedrettsvern (Orientering, best.nr. 539, Arbeidstilsynet).
Opplysninger som er nye, slettet eller revidert	REVISJONSOVERSIKT: 05.04.01 - Utgitt.

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25134-21-8	246-644-8	Metylnorbomen-2,3-dikarboksyreanhydrid	normverdi
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### Eksponeringskontroll

Begrensning av eksponering på arbeidsplassen	Sørg for effektiv ventilasjon. Arbeid i avtrekkskap. Vask hender og ansikt grundig etter arbeid med stoffet. Skift straks forurensete klær. Øyedusj skal finnes på arbeidsplassen.
Åndedrettsvern	Ved utilstrekkelig ventilasjon må det brukes egnet åndedrettsvern. Gassfilter A (organiske stoffer, brunt).
Håndvern	Benytt egnede vernehansker ved fare for hudkontakt. Bruk hansker av: Neopren.
Øyevern	Bruk godkjente vernebriller ved fare for øyekontakt.
Annert hudvern enn håndvern	Bruk egnede verneklær ved fare for direkte kontakt med produktet.

### 9. Fysiske og kjemiske egenskaper

Tilstandsform	Væske.
Lukt	Karakteristisk.
Farge	Gulaktig.
Løselighet i vann	Ikke blandbar.
Løselighet i fett	Aceton. Benzen. Xylen.
Relativ tetthet	Verdi: 1,245 g/cm3
Kokepunkt/ kokepunktintervall	Verdi: 140 °C
Flammepunkt	Verdi: 135 °C
Damptrykk	Verdi: 5 mmHg (120 C)
Damptetthet	Verdi: 6.1
Referanse-gass: (luft=1)	
Fysiske og kjemiske egenskaper	Molekylvekt: 178,19

### Andre fysiske og kjemiske egenskaper

Fysiske og kjemiske egenskaper

### 10. Stabilitet og reaktivitet

Forhold som skal unngås	Sterk oppvarming. Fuktighet / lufteksponering.
Materialer som skal unngås	Vann. Oksiderende stoffer. Sterke baser. Sterke syrer.
Stabilitet	Stabil ved normal håndtering.

### 11 Opplysninger om helsefare

#### Øvrige helsefareopplysninger

Innånding	Kan forårsake: Irritasjon av luftveiene. Hoste. Pustebesvær. Allergiske reaksjoner.
Hudkontakt	Kan forårsake: Hudirritasjon.
Øyekontakt	Kan forårsake: Øyeirritasjon.
Sveiging	Kan forårsake: Innvendig irritasjon. Kvalme. Oppkast. Magesmerter.
Kroniske effekter	Andre helseskadelige effekter kan ikke utelukkes.
Allergi	Kan gi allergi ved innånding.

### 12. Miljøopplysninger

#### Toksikologisk informasjon

Akvatisk kommentarer Kvantitative data for akvatisk toksisitet er ikke tilgjengelig.

#### Øvrige miljøopplysninger

Økotoksisitet	Informasjon om mulige miljøskadelige effekter er ikke tilgjengelig.
Mobilitet	Produktet er uoppløselig i vann.
Persistens og nedbrytbarhet	Informasjon om biologisk nedbrytbarhet er ikke tilgjengelig.

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