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Atomic Force Microscopy
Building an AFM with a Piezoresistive Force
Measurement System

Bachelor thesis

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1 Motivation

Recently, the Institute of Solid State Physics got a new set of probes for electrical measurements in a SEM. It includes several micromanipulators and a piezoresistive force measurement system. With this force measurement system some interesting properties can be investigated, like the mechanical strength of nanowires or the conductance as a function of strain and of course atomic force microscopy (AFM) is possible. Furthermore it would be interesting to make measurements with a conducting piezoresisive cantilever. It is hard to contact small structures like carbon nanotubes electrically with probe tips because the only indication for electric contact is a current measured or not. Thus the nanotube might break because of pushing too hard. With a conducting cantilever the force-resistance relationship could be measured to determine the proper force for a good contact. Moreover conducting AFM measurements would be possible. So topographic imaging and potential mapping could be done at the same time. With this technique larger and rougher surfaces than with Kelvin Probe Force Microscopy could be scanned.

There was not much experience with the force measurement unit so it was decided to try to build an AFM with it.

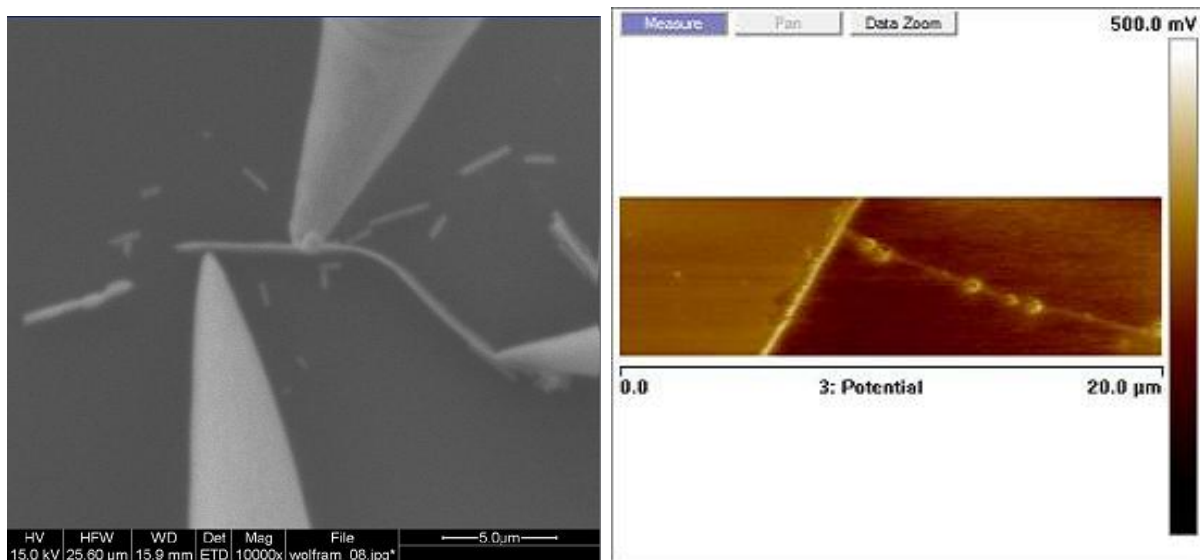


Figure 1: Small wire contacted with three probe tips and KPFM images of a carbon nanotube with Au particles

2 Atomic Force Microscopy

Atomic force microscopy (AFM) is a powerful tool for the examinations of surfaces. As shown in Fig. 2 the basic idea of AFM is to scan the sample's surface with a sharp tip of a cantilever. The interaction between tip and sample deflects the cantilever. This deflection is then measured by some convenient procedures, such as laser reflection or some other device and gives a 3 dimensional image of the surface. [2, 9, 10]

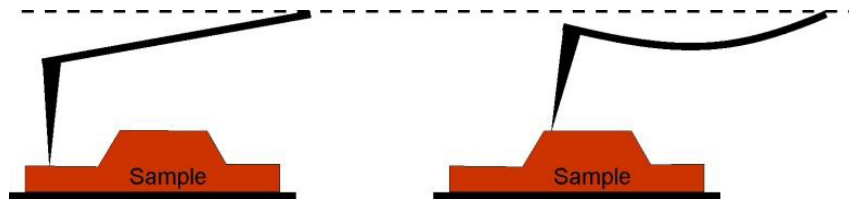


Figure 2: Principle of atomic force microscopy [10]

The driving motion of the cantilever is performed by piezoceramic actuators, which change their dimensions under applied voltage. The most frequently used driver is a hollow-tube scanner, which can move the tip or sample in all three mutually perpendicular directions. [9]

One of the main components of an AFM is the cantilever because from its deflections there can be drawn conclusions to the topography. According to Hooke's law $F = -x.k$ the force causing the deflection(x) is proportional to the force constant k. Cantilevers with different force constants are used for the different modes of AFM. In contact mode lower force constants than in tapping mode are used. [10]

As already mentioned the most common system for the deflection detection of the cantilever are optical laser detection systems (see Fig. 3). Therefore a laser is focused on the the end of the cantilever and reflected on a position sensitive detector(PSD). A force affecting the tip deflects the cantilever and the reflection on the PSD will be displaced. So vertical movement of the cantilever can be measured.

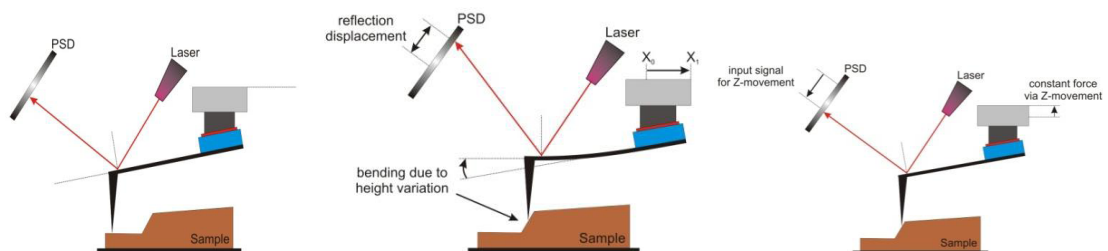


Figure 3: Principle of the laser detection system [10]

2.1 Contact mode

Depending on the setup, forces that are measured in AFM include mechanical contact force, Van der Waals forces, capillary forces, chemical bonding, electrostatic forces, magnetic forces, Casimir forces, solvation forces. Approaching the sample with the tip, first attractive Van der Waals forces dominate. As the tip gets closer to the surface repulsive force gets stronger. [9, 10]

There are many different AFM scanning modes. [2] Here the contact mode and tapping mode should be shortly introduced.

2.1 Contact mode

In contact mode the tip is in permanent contact with the sample. A certain force is chosen and tried to keep constant while scanning, i.e. the cantilever is permanent constantly deflected. This is achieved by a feedback-loop that moves the z axis until the chosen force is reached again (see Fig. 3). The advantages of contact mode are fast scanning and a good resolution (<3 nm lateral and < 0.2 nm vertical). [10]

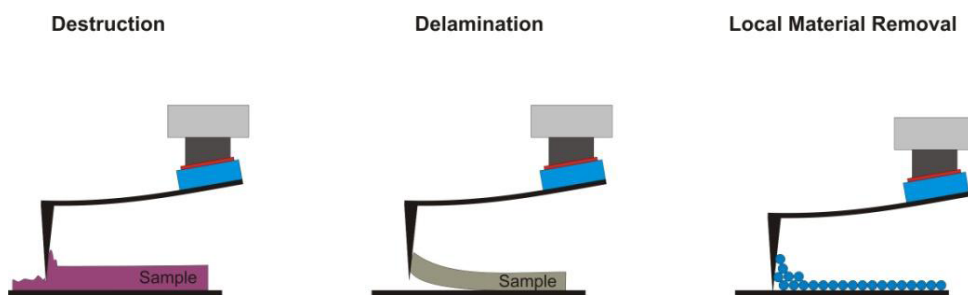


Figure 4: Problems in contact mode. [10]

As shown in Fig. 4 the disadvantages of contact mode AFM are that the hard and sharp tip can cause destruction, delamination or local material removal of soft parts of the sample.

2.2 Tapping mode

In tapping mode the tip is vertically oscillated at its resonance frequency. Interactions with the surface of the sample lowers the vibrational amplitude. This amplitude drop is used for feedback. There are two working regimes possible. In the non contact regime the tip never touches the sample and in the intermittent regime the tip is tapping on the surface of the sample. [9, 10]

3 Build an AFM

3.1 Idea

The idea is to use the Kleindiek force measurement system to build a constant force mode atomic force microscope. A Micromanipulator by Kleindiek is utilized to move the piezoresistive cantilever of the force measurement system. This micromanipulator is controlled by an Adwin system from Jäger Computergesteuerte Messtechnik. The ADwin system is used for data acquisition, scanning and feedback to keep the cantilever at a certain force.

3.2 Components

3.2.1 Kleindiek FMS-EM Force measurement system



Figure 5: FMS controller and force measurement tip in holder mounted on a micromanipulator [8]

The force measurement system (FMS) consists of a cantilever, a resistor bridge, an amplifier, an analogue output, a loudspeaker and a display. The cantilever and the resistor bridge are integrated into the force measurement tip (FMT) and the other components are integrated into the FMS controller. The cantilever is covered with piezo-resistive material. When it is brought into contact with a sample (and is thus bent), this material changes its resistance. The material is one of the components of the resistor bridge, so that the resistance of the bridge changes when the cantilever is bent. By applying a voltage to the bridge, the change in resistance can be converted into a change in voltage. [8]

3.2 Components

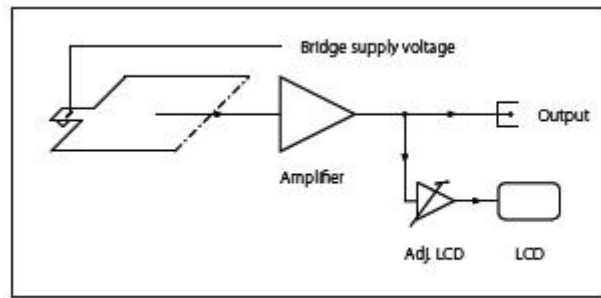


Figure 6: Principle of the FMS [8]

The changes in voltages are amplified and the amplified signal is output by the controller on a display in front, through an integrated loudspeaker and through a BNC plug on the rear of the FMS controller.

The FMS requires calibration which is done with a seesaw where you know how much force must be applied to lift it. The force shown on the display can be changed with regulators. It can also be used without first performing a calibration however then there is a large margin of error (approximately 40%). . In this work most of the time the FMS was used uncalibrated because there was no demand for the absolute force. We used the recommended setting for an uncalibrated FMS of LCD-Adj. 2.00 [8]

The force measurement tip is very fragile and therefore has to be handled very carefully. As you can see in Fig. 7 it consists of a cantilever with a sharp tip and of a smaller second one. The cantilever with the sharp tip is bending when performing force measurements and the small one is the reference piezoresistance of the resistor bridge. Here the Kleindiek FMT-120 sensor with the following technical specifications is used [3]:

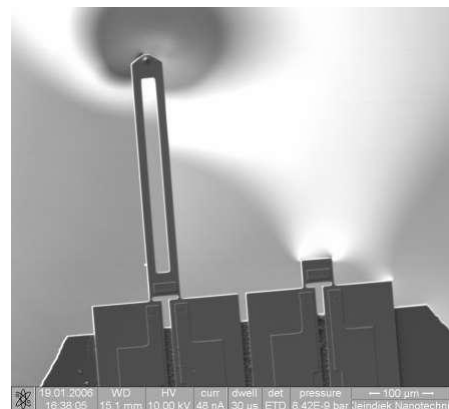


Figure 7: FMT-400 [3]

- Length 120 μm
- Width 50 μm
- Height 4 to 5 μm
- Tip radius < 20 nm

3.2 Components

- Tip height $> 5 \mu\text{m}$
- Tip force constant (calculation) 30 to 40 N/m
- Maximum tip force 360 μN
- Resistance 500 to 650 Ω
- Sensitivity $18.8 \times 10^{-3} \text{ mV/nm}$ at $V_{\text{bridge}} = 2.5 \text{ V}$
- Lowest pressure 10^{-7} mbar

3.2.2 Kleindiek MM3A-EM micromanipulator



Figure 8: Kleindiek MM3A-EM micromanipulator [3]

The Kleindiek MM3A-EM micromanipulator is a micromanipulator with subnanometer resolution (0.25nm) [3]. It can be used for nanomanipulations. For the AFM it is used to hold the force measurement tip and to move it, i.e. to increase or decrease the force (move z) and scan the sample (move x y). The principle behind the micromanipulator is a piezoelectric motor, the Nanomotor[®]. The Nanomotor[®] consists of a stator (piezo crystal) and a slider. The slider is held in place by friction i.e. the slider is touching the stator. The stator expands or contracts under electrical voltage and the Nanomotor[®] uses these movements in two different ways: fine mode and coarse mode. [7]

Fine mode is used to control the manipulator in extremely small steps for very fine work. In fine mode, the motions of the manipulator are created by deflection of the stator with a voltage between -80 V and +80 V. The total fine range of the linear axis is about $1\mu\text{m}$ and the total fine range of the rotational axes is about $20\mu\text{m}$.

In coarse mode, momentum is used to ratchet the motion of the manipulator. This is possible due to a frictional clutch between the stator and the slider. If the frictional

3.2 Components

force of this clutch is exceeded, the two parts will move independently. This principle also allows the manipulator to be moved by hand without damaging the stator. The idea behind the ratchet is that the slider is accelerated in the desired direction of motion using a force below the friction limit, but then the stator is very quickly deflected in the opposite direction such that the slider cannot follow without exceeding the maximum frictional force. Instead, it stays in place, permitting the stator to begin deflection in the desired direction once again. In this way, large motion in either direction is possible. [7]

The micromanipulator is controlled by the NanoControl electronics. The NanoControl contains a 12 bit digital analogue converter that allows the voltage to be applied in $4096(2^{12})$ steps. The NanoControl provides three different speeds in fine mode where a different number of finesteps are executed each time the NanoControl receives a command to move either by the knobs on the front of the NanoControl or a Sony PlayStation[®]2 DUALSHOCK[®]2 controller plugged into the back of the NanoControl.

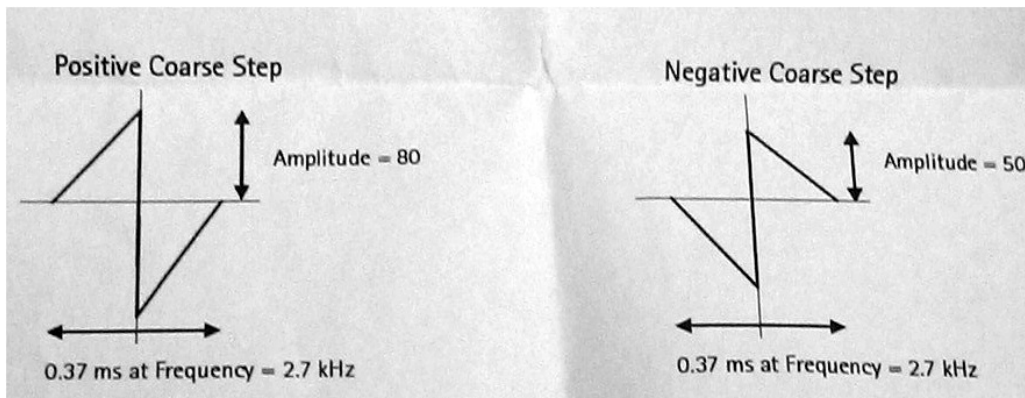


Figure 9: Voltage change to perform a coarse step [7]

In coarse mode, the momentum needed for the motion is created by a quick voltage change on the piezo crystal as shown in Fig. 9. Besides, there are two factors playing an important role in the execution of a coarse step, amplitude and frequency. The amplitude determines the size of the coarse step and the frequency affects the speed of the manipulator. The NanoControl also provides three different speeds in coarse mode, where the number of coarse steps that are executed each time a command to move is received is different. [7]

The micromanipulator can also be controlled by applying a voltage from an external analog digital converter directly to the NanoControl. However, it should not be both, the internal and the a external analog digital converter, used at the same time because the two voltages sum up and can exceed the maximum.

3.2 Components

3.2.3 ADwin system by Jäger Computergesteuerte Messtechnik



Figure 10: ADwin-Pro II by Jäger Computergesteuerte Messtechnik [6]

The ADwin-Pro II system is an external processing system with modular expansion options like analog and digital input and output modules. [6] The ADwin-Pro II system has a central processing unit (CPU), which executes all time-critical tasks like measurement data acquisition or analog signal generation in real-time. The ADwin-Pro II system is connected via ethernet with a computer. The processor of the ADwin system is programmed with a real-time development tool ADbasic. ADbasic is an integrated development environment under Windows that uses the BASIC command syntax which is expanded with more functions for accessing the inputs and outputs, controlling real-time processes and preparing the data exchange with the computer. [5]

ADbasic programs are created with the editor of the development environment and the compiler translates this source code into a executable process for the ADwin system. Each program must at a minimum have an EVENT: section. This is the main program section, which is called in regular time intervals until it is stopped. In ADbasic there are global variables that are already pre-defined and do not need to be declared. The global variables and arrays may be accessed from other processes and from other programs running on a PC like Labview. [5]

3.3 Realization

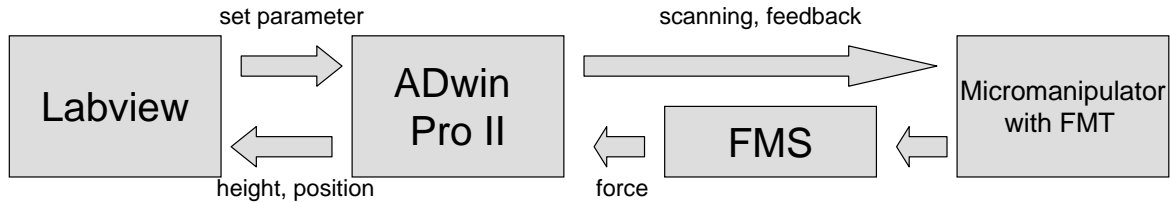


Figure 11: Block diagram of the atomic force microscope

As can be seen in Fig. 11 the control of the AFM consists of two parts:

- Labview program
- ADbasic programs for the ADwin system

Labview is used to set all scanning parameters like the x-y scanning range and stepsize, start the ADbasics programs and save height and position data of the scan in a proper format for further use.

The ADwin system is used for feedback and scanning. Overall there are two ADbasic programs. One is for feedback and includes line scanning and the other one is for lateral scanning.

In the following, force and position stands for the voltage measured at the resistor bridge of the FMT corresponding to the force and the voltage applied to the piezo crystal to achieve deflection.

The feedback tries to keep the cantilever at a certain force by moving the z axis of the micromanipulator up or down. In the feedback program a reference force and a acceptable range can be chosen arbitrarily. If the measured force is between plus minus the value of the acceptable range around the reference force, no z movement will happen.

In the event section of the feedback and line scan program, ten force measurements are averaged to the feedback stable. Then this average value is compared to the reference force plus minus the range. If the average value is larger, the height(z axis) is increased and if it is smaller the height is decreased. This comparison takes place 500 times. The the current position and height is then saved into two two-dimensional arrays, the cantilever is moved one step to the side (x-axis) and the force feedback starts again. When the chosen maximum scan range of the x-axis is reached, the direction of the steps change

3.3 Realization

and the scan continues back to the origin where finally the program stops. So a line scan with trace and retrace is performed. For rougher surfaces the number of feedback cycles should be increased in order to assure the cantilever can follow the structure.

The second ADbasic program is just a small one. In fact it just moves the linear axis (y axis) of the micromanipulator to a desired position.

The order of both programs is controlled by a Labview program. The principle is easy: First the feedback line scan is performed. As it is finished the height and position data of the trace and retrace are transferred and collected together with the actual position of the linear axis in two arrays. Then the linear axis (y) moves one step and the line scan starts again. More precisely both programs are running consecutively in a loop that is executed as many times as needed to reach the maximum scan range of the y-axis. In the end the data is saved to text files, one for traces and one for retraces. These text files contains three columns, one for the x and one for the y positions and the last one for the corresponding heights.

For both, scans (x- and y- axis) and the height adjustment (z-axis) just the fine mode of the micromanipulator is used. Coarse motion, due to the fact that a quick change in momentum is used to achieve motion, is unsuitable because the very sensitive piezo-resistive force measurement system will be disturbed every time a coarse step is performed. Therefore the scan range is limited by the maximum piezo deflection which is for the rotational axes(x- and z-axis) about $20\mu\text{m}$ and for the linear(y-axis) about $1\mu\text{m}$. [7] In order to have the whole scanning range available the piezos have to be first deflected fully in one direction by applying the maximum or minimal voltage of $\pm 80\text{V}$. This should be done before starting a scan and is executed by another Adbasic program that ramps the voltage of the x- and y-piezo to almost -80V .

In Fig. 12 the experimental setup can be seen. Figure 9(a) shows the closed vacuum chamber in which the micromanipulators are mounted. On top of it is a microscope with a USB ccd-camera to film the sample inside the chamber. The microscope has a stative and can be displaced by two micrometer gauges. The chamber is located on a vibration isolated table. For all of the measurements described here, vacuum was never needed. In (b) the electronic rack can be seen. It contains the ADwin system, the FMS and the NanoControls and power supplies of the micromanipulators. (c) and (d) show the inside of the vacuum chamber. The micromanipulators can be seen. One is equipped with the force measurement tip. The sampleholder is a LT 6820 substage by Kleindiek that has a

3.3 Realization

orthogonal positioning system, thus can be moved with a NanoControl unit.

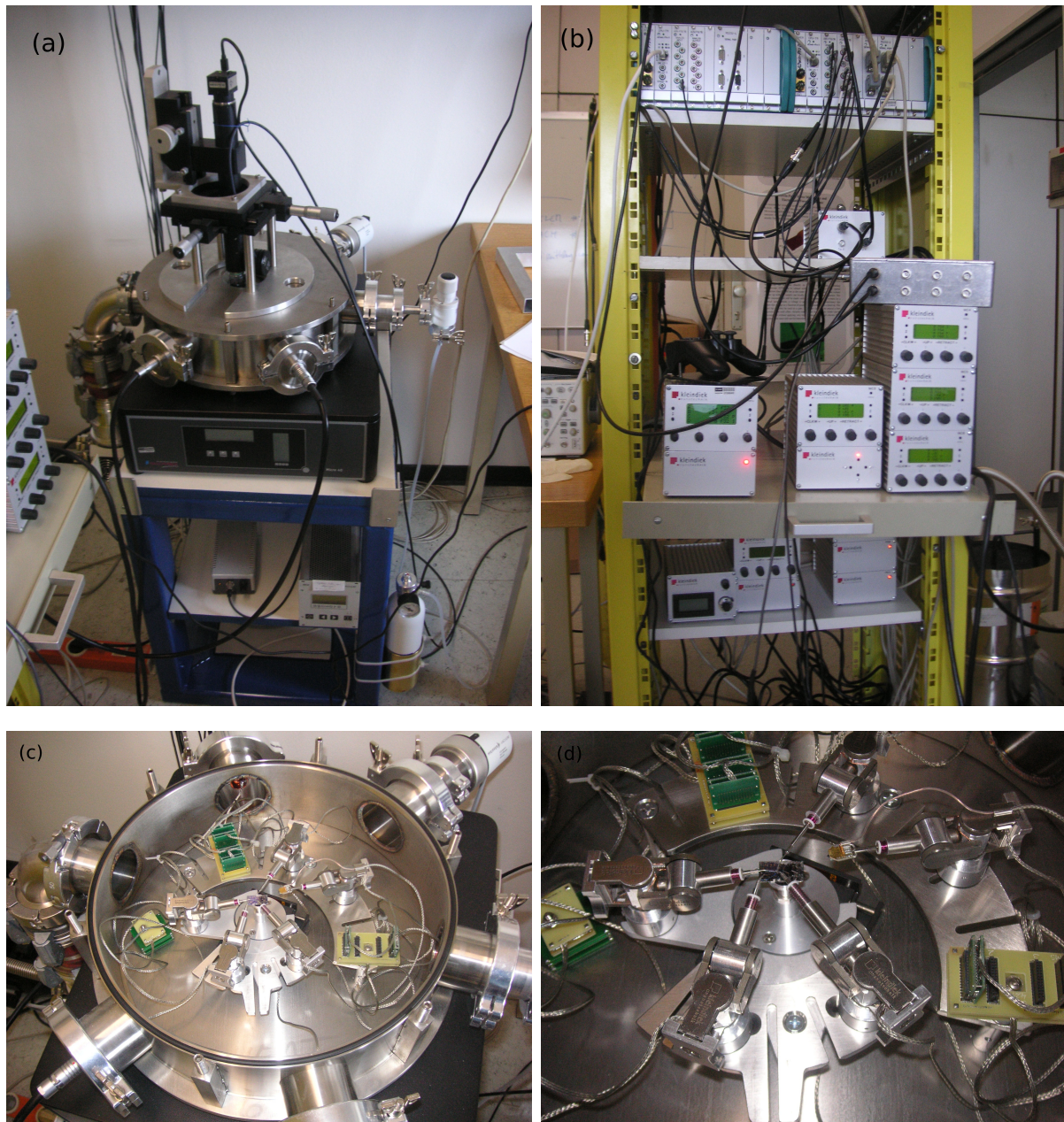


Figure 12: The experimental setup at the laboratory

3.4 Problems

3.4.1 Vibrations

Since the force measurement system is a very sensitive measurement unit, vibration isolation is required. Therefore the whole scanning set up is situated on the active vibration isolation system Micro 40 by Halcyonics. Initial tries without vibration isolation led to unusable measurement conditions because just vibrations were recorded as can be seen in Fig. 13.

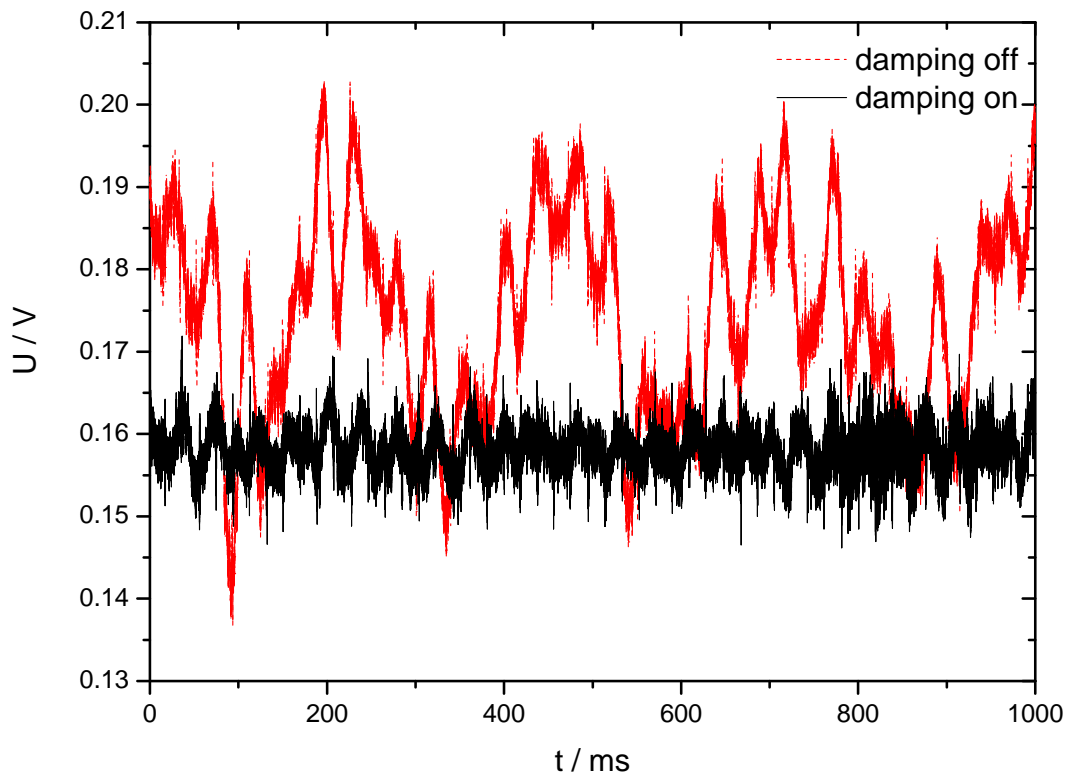


Figure 13: Output signal of the force measurement system with and without active vibration isolation

3.4 Problems

3.4.2 Drift

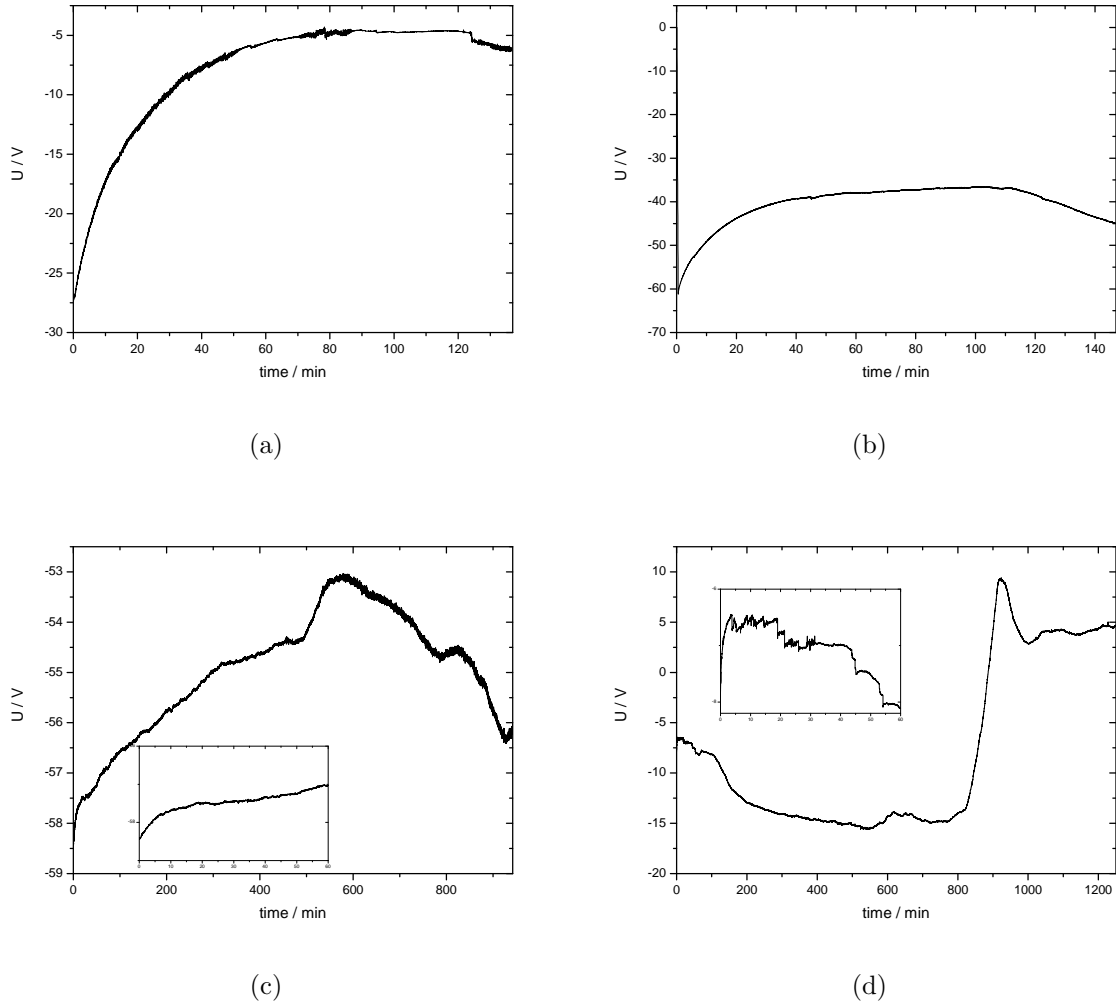


Figure 14: Applied voltage(height) to the z-axis-piezo while running the feedback to keep the cantilever at a constant force recorded over a long time

Every time the cantilever landed on the sample a drift in force was observed. The force started to change although the manipulator was not moved. The first thoughts were that piezo creep is responsible for the drift. Therefore long time measurements were started where the height of the z-axis piezo was recorded after the cantilever was landed and the feedback that keeps the cantilever at the same force started. This kind of measurement was chosen to not exceed the maximum tip force and break it. As you can see in Fig. 14 the first two measurements look like the typical logarithmic behavior of piezo drift (14(a),(b)) however after longer time unexpected behavior was discovered(14(c),(d)). The reason for it is not clear yet. Luckily as you can see in the graphs after about half

3.4 Problems

an hour the height approaches a limit. Therefore before starting a measurement it is required to wait for about half an hour until quite stable conditions are reached.

3.4.3 Dirt

AFM samples should be very clean because otherwise the cantilever could pick up dirt particles and the measurements will become inaccurate. A dirt particle sticking next to the cantilever's tip could act also as tip and the recorded images will show a distorted structure. Unfortunately, cleaning the cantilever is not so easy. Figure 15 shows a dirt particle sticking at the beginning of the cantilever. The attempt to clean it with another micromanipulator's tip was successful.

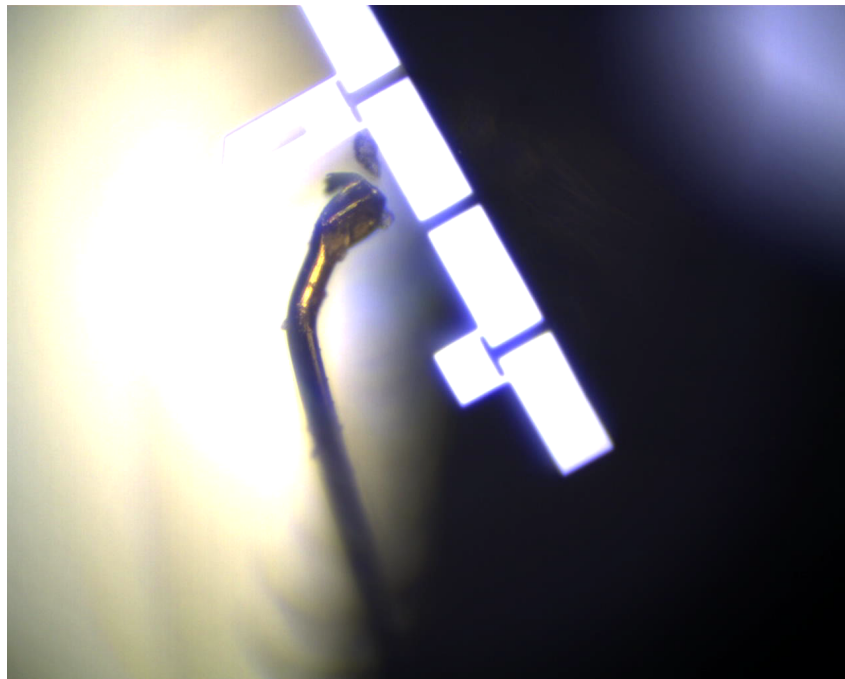


Figure 15: Cleaning a dirt particle off the cantilever with an other micromanipulator's tip

4 Scanning samples

As already mentioned, before scanning the x and y axis of the micromanipulator are deflected fully by starting the ADbasic program that ramps the piezo voltages to almost -80V. Next the cantilever is landed on the sample. The landing position can be determined by the LT 6820 substage with its orthogonal positioning system. Landing can be done by an ADbasic landing program that moves the z-axis of the micromanipulator downwards in coarse and fine mode until a force is measured. Afterwards the force feedback should be turned on until constant conditions are reached. The landing and feedback program are combined in one Labview program for easier execution. The scanning parameters are set with the first Labview program, whereas the value 65536 (2^{16}) corresponds to the maximum voltage output value since the Adwin system has a 16-bit digital analog converter. Thus 0 corresponds to -80V and 65536 to +80V. More precisely the range of the ADwin D/A converter is -10V to +10V but the voltage is amplified 8 times by an integrated amplifier in the NanoControl system of the micromanipulator. However there should be paid attention to the applied voltage value to deflect the piezo before scanning. Here $-79.756V (\hat{=}100)$ is chosen and therefore the maximum x and y scan range parameter can only be 65436 ($\rightarrow 65436 + 100 = 65536 \hat{=}80V$). In the following all the values of x,y and z axis position are given in the corresponding 16-Bit value of the applied piezo voltage like 65536 (2^{16}) for +80V or 0 for -80V.

4.1 WSxM software for data visualization by Nanotec Electrónica

WSxM is a free software for data visualization and processing of SPM images developed by Nanotec Electrónica. [1, 4] Here the WSxM_SPMAGE09 version of this software was used. The data files of the scans saved with Labview have already the right format of a "ASCII XYZ file" just a simple header has to be added.

example file :

WSxM file

WSxM ASCII XYZ file

X[a.u.] Y[a.u.] Z[a.u.]

```
x1      y1      z
x2      y1      z
.       .       .
```


4.2 Images

. . .
. . .
x1 y2 z
x2 y2 z
. . .
. . .
. . .

4.2 Images

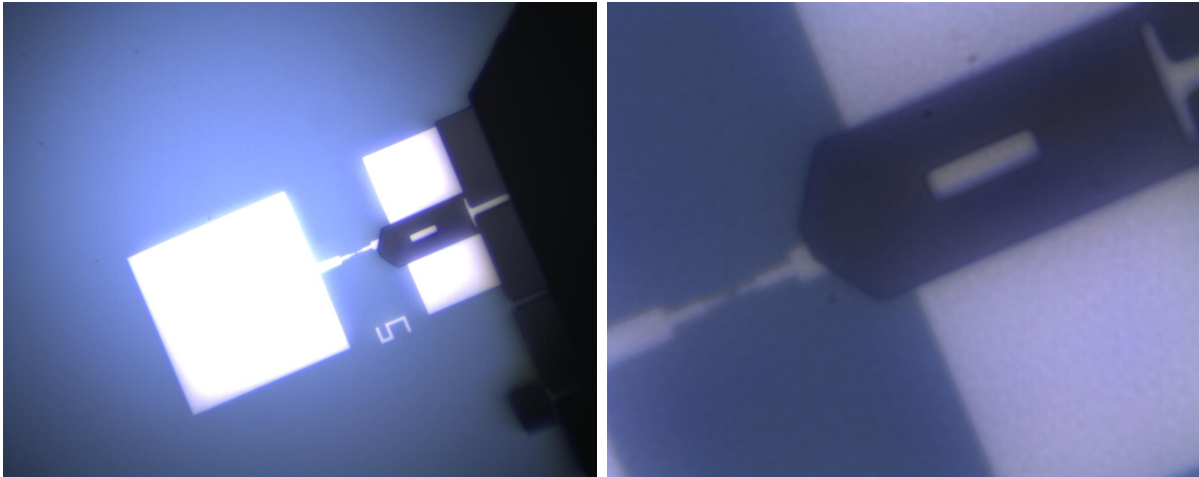


Figure 16: Cantilever next to a structure of a single electron transistor device

The first scan was the structure of a single electron transistor device. As you can see in Fig. 16 it is hard to figure out where the tip of the cantilever is and thus the scan starts. Following scan parameter were chosen:

- Maximum x-axis range 64000 with a step size of 200
- Maximum y-axis range 60000 with a step size of 3000

First the bigger line was scanned. The AFM images are shown in Fig. 17 whereas the left side (a),(c) corresponds to the trace and the right side (b),(d) to the retrace. The 21 horizontal lines in the images are a result of the 21 linescans performed because of the step size and scan range of the y-axis. The reason for the whole deformation of the images is probably piezo drift because it took about 2 hours to scan the sample. Later this problem was reduced by accelerating the scanning program. The acceleration was possible because

4.2 Images

the CPU of the ADwin system had enough power to run the programs faster, i.e. with a reduced global delay. Another reason could be that the force is increased when scanning forward because the linear axis is a little bit tilted and therefore the forward motion is actually a forward and down motion.

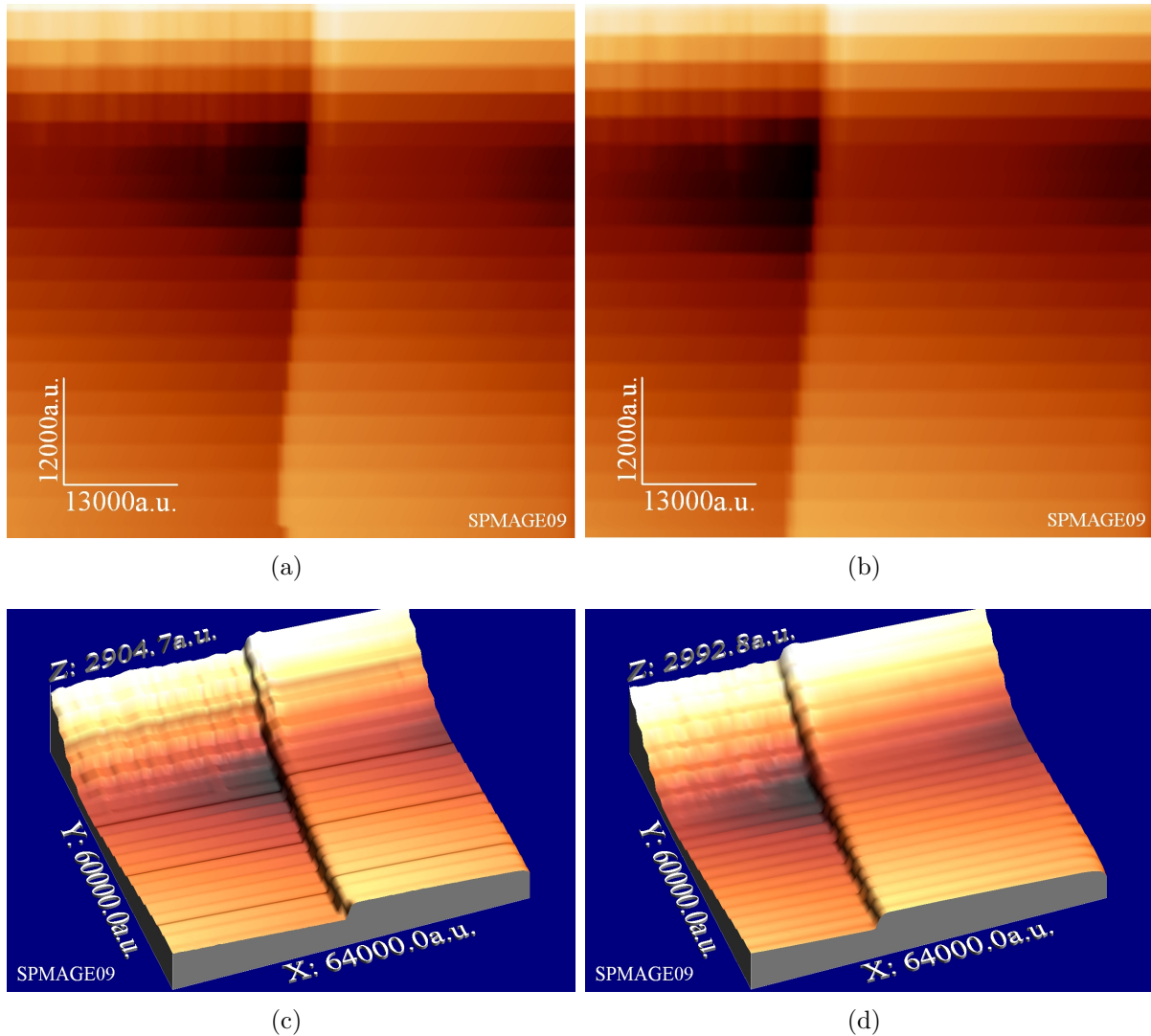


Figure 17: 2d and 3d AFM images of an electronic structure- (a),(c) corresponds to trace and (b),(d) to retrace

Looking at the AFM images there has always to be in one's mind that the x and y dimensions are not the same due to the fact that the linear axis (y-axis) of the micromanipulator has just a total fine range of about $1\mu\text{m}$ and the rotational axis' (x-axis) total range is $20\mu\text{m}$. Here in all images the axis are not scaled and thus both seemed to be the same size.

4.2 Images

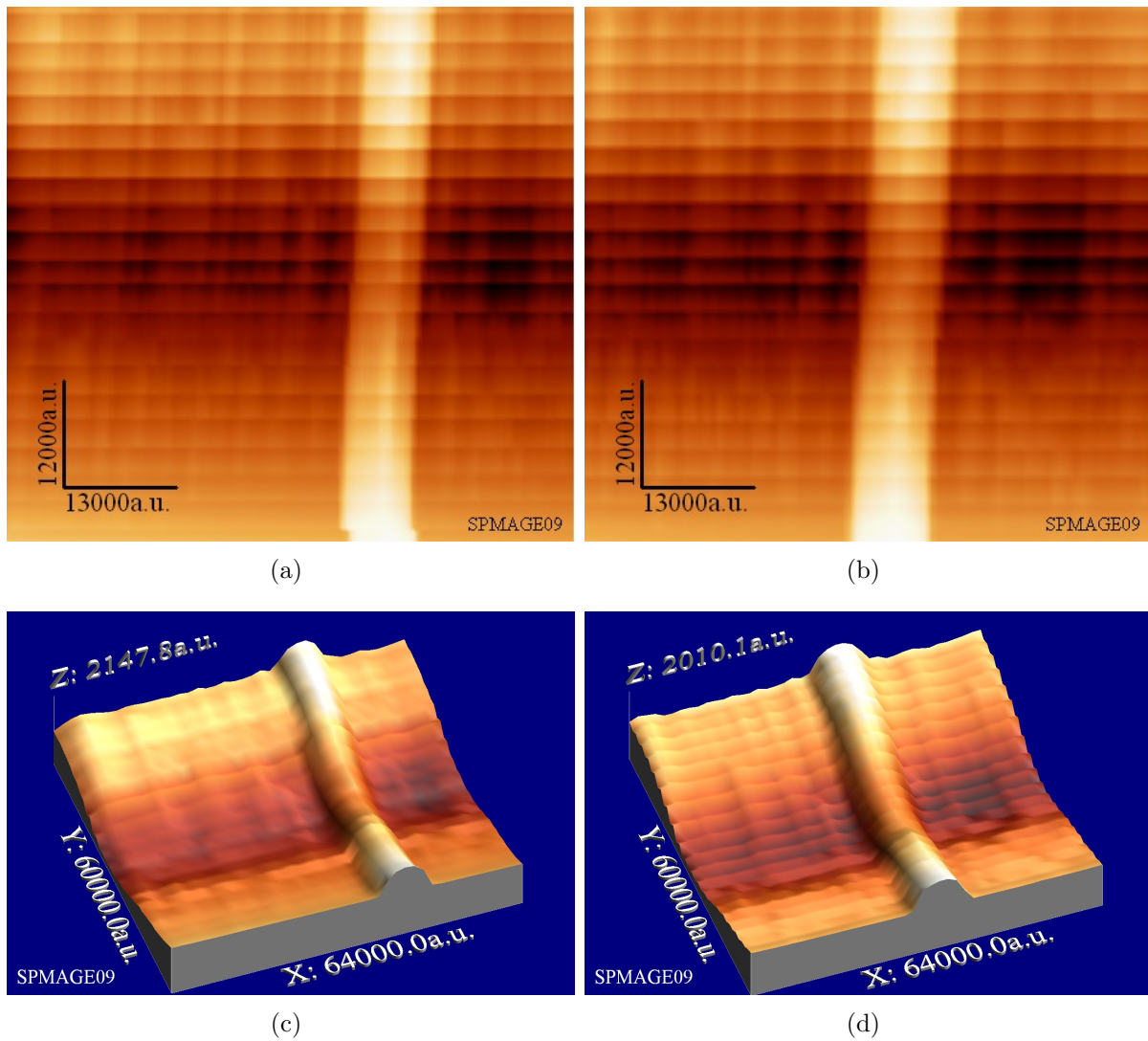


Figure 18: 2d and 3d AFM images of a smaller electronic structure- (a),(c) corresponds to trace and (b),(d) to retrace

The smaller line of the electronic structure was also scanned. The scanning parameters are the same as before and the deformation is again a result of drift.

4.2 Images

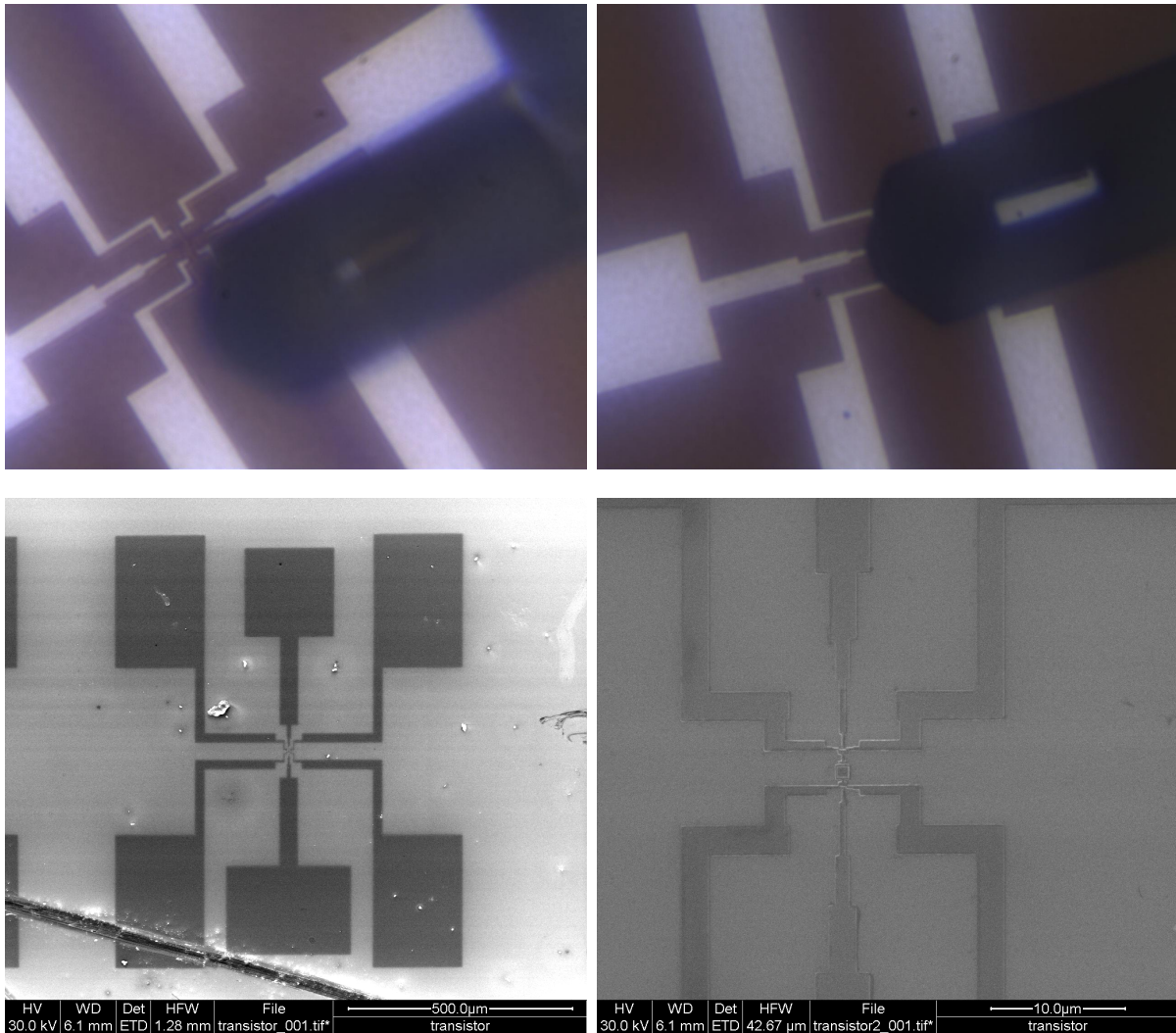


Figure 19: Images of the cantilever next and over the structure of an electronic device taken under the light microscope and SEM images of the structure

Next an electronic device with some more structure was scanned. Therefore, as shown in Fig. 19, the cantilever was landed somewhere in the middle where structure was assumed. The scan parameters were the same as before:

- Maximum x-axis range 64000 with a step size of 200
- Maximum y-axis range 60000 with a step size of 3000

There were several scans made of the structure. After each scan the start position of the new scan was changed by moving the sample with the substage to a desired position. The first images 20(a),(b) was recorded with the slow feedback and linescan program. Afterwards this program was accelerated by reducing the global delay. Now the scanning

4.2 Images

time was reduced from about 2 hours to about 12 minutes. As can be seen from Fig. 20(c)-(f) the images are not so deformed anymore because the scantime reduction reduced also the drift.

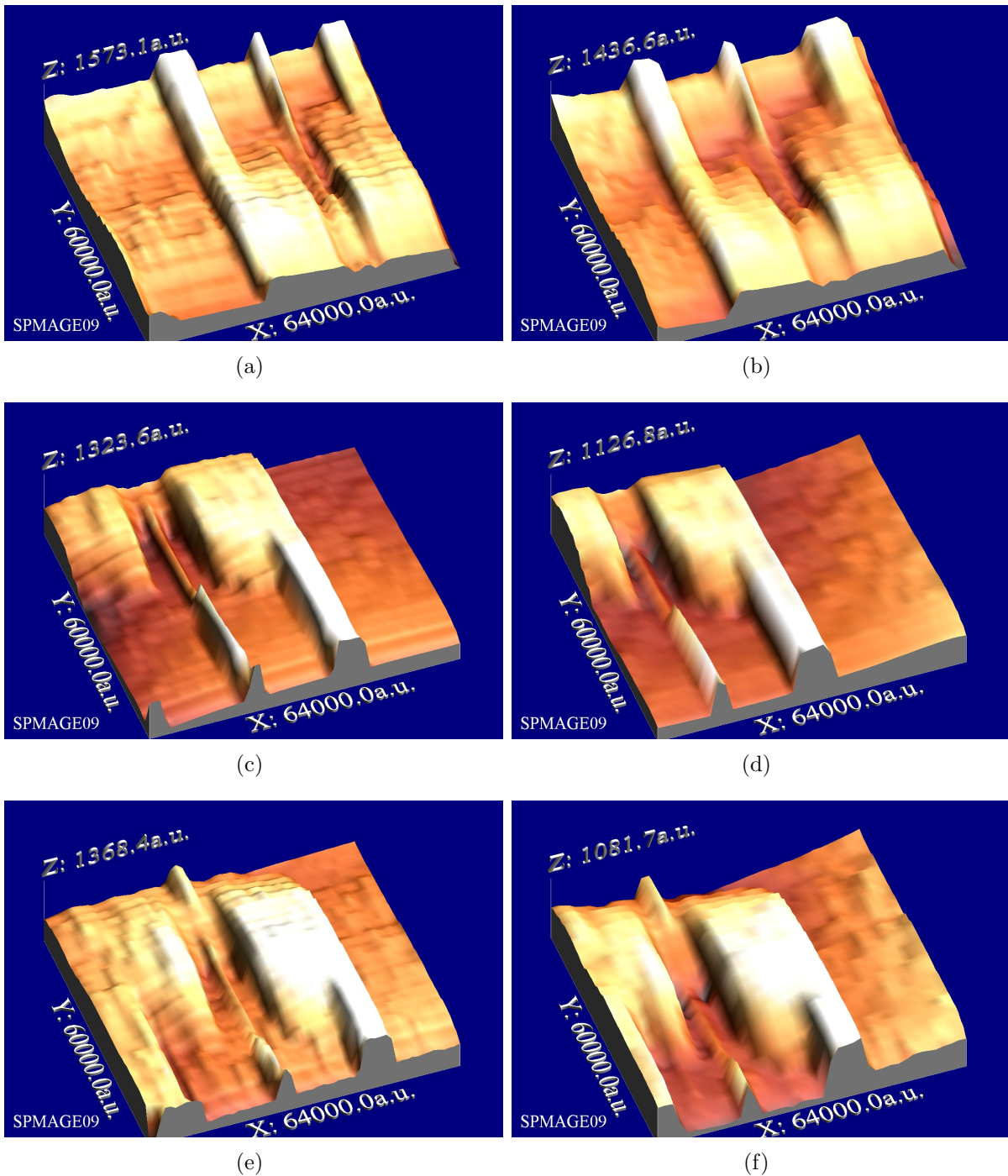


Figure 20: 3d AFM images of a structure of an electronic device- (a),(c),(e) corresponds to trace and (b),(d),(f) to retrace

4.2 Images

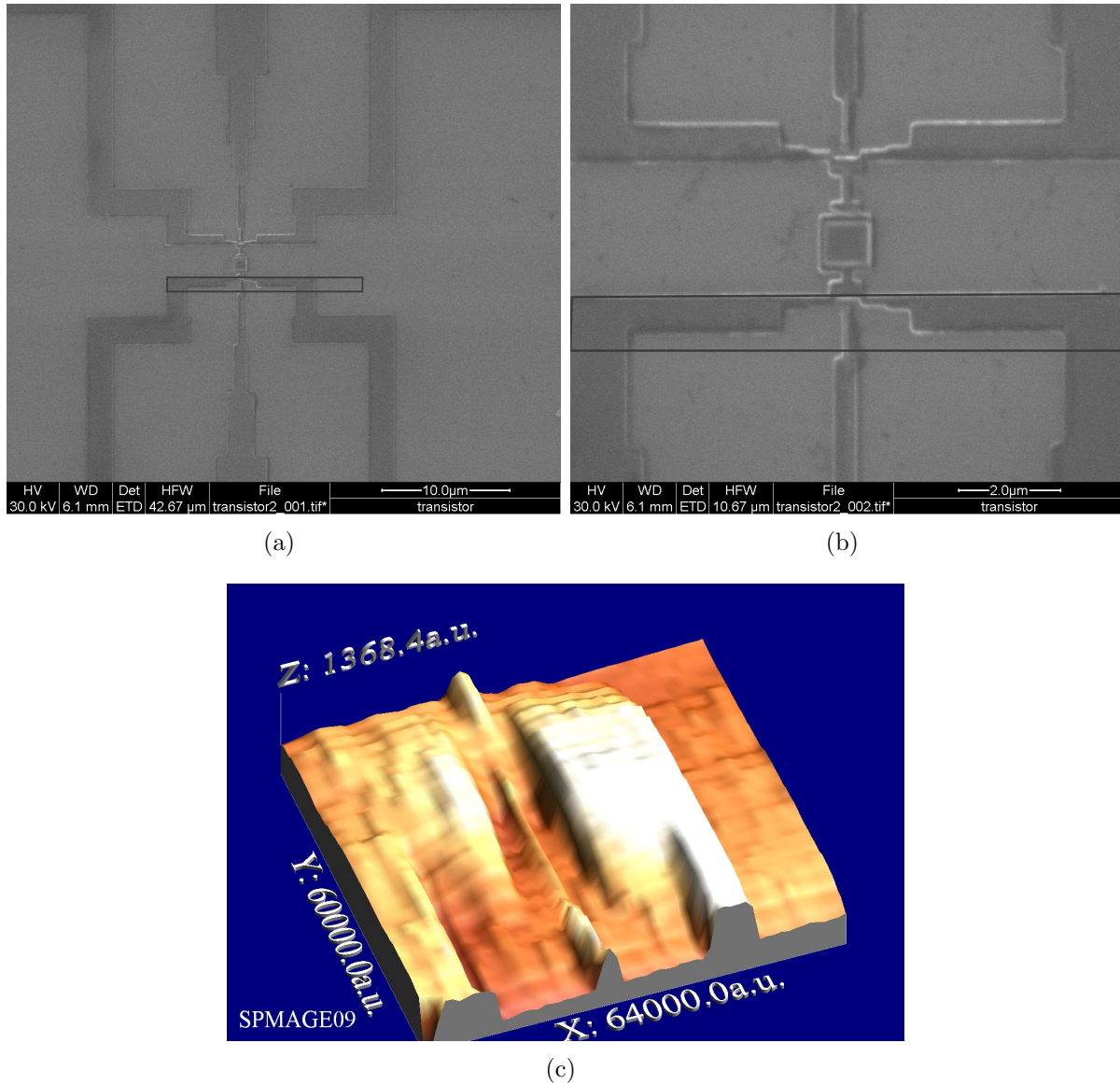


Figure 21: Estimated area scanned

In Fig. 21 the estimated scanning area of one AFM scan is marked. The two different scanning ranges of x and y axis are clearly recognizable. In fact the SEM images 21(a),(b) are not part of this particularly scanned device but of a similar one. The original structure can be seen in Fig. 22. Remarkable are the small spots all over the scanned area. First assumptions were that the sharp tip causes small indentations. There were also taken images of these samples with a commercial AFM in tapping mode (see Fig.23). In Fig.23a) it can clearly be seen that the spots are extra material and not indentations. The Assumption is that photo resist was still on the sample (bright square in the middle of Fig.23b)). Scanning the sample in contact mode dispersed it all over the scanned area.

4.2 Images

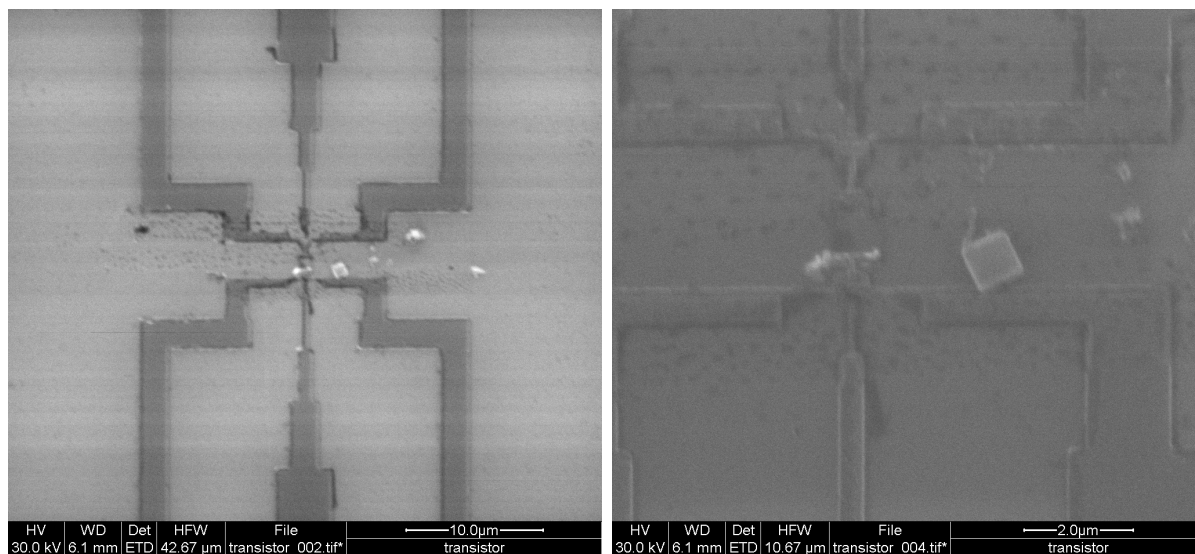


Figure 22: Structure of an electronic device after scanning - small spots can be seen all over the scanned area

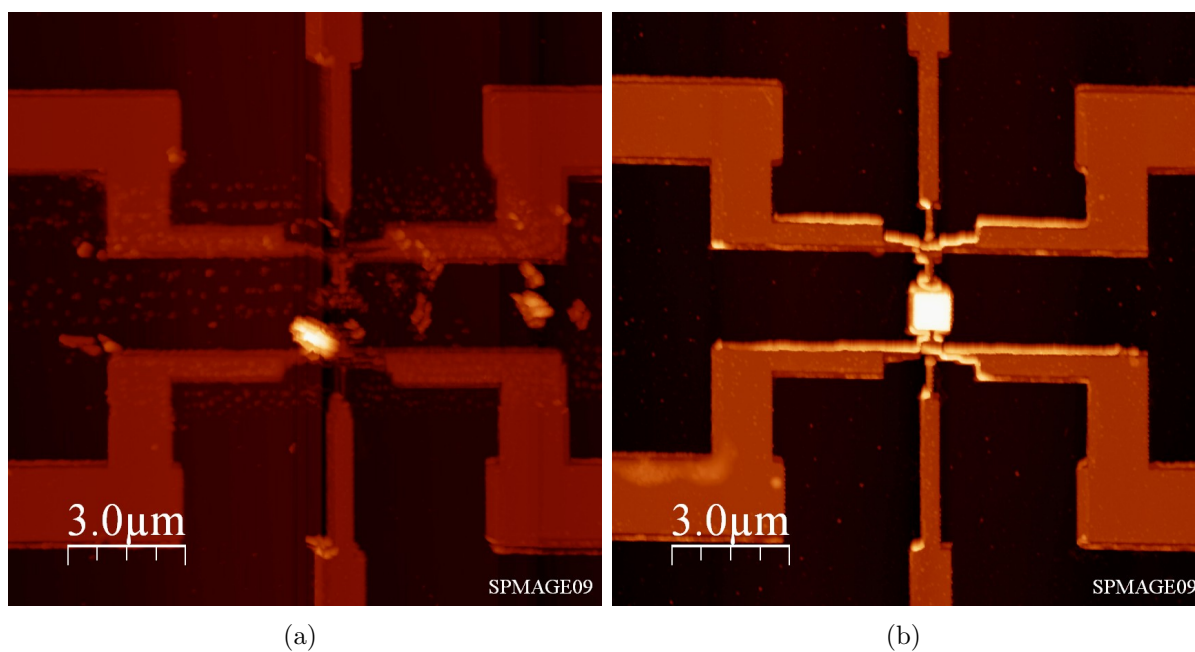


Figure 23: 2d tapping mode AFM images of the Structure of an electronic device- a)first in contact mode scanned sample b)not scanned sample (dark color means low; bright color high)

5 Scaling

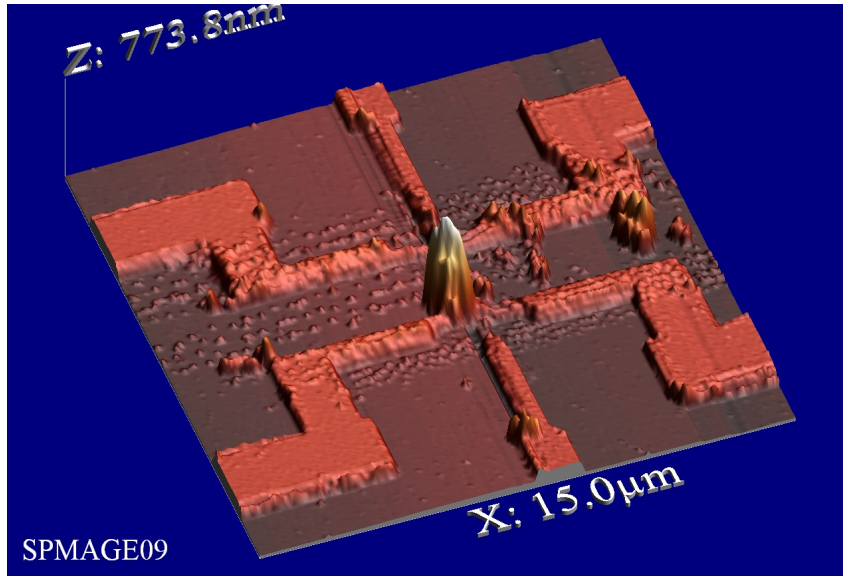


Figure 24: 3d tapping mode AFM image of the scanned structure

With the AFM images of the samples taken by a commercial AFM, it was possible to scale the x,y and z axes of the micromanipulator. A linear relationship between applied piezo voltage and deflection is assumed. Distinct distances were measured in both AFM images (Fig.24 and Fig.20) and compared. So a relationship between applied voltage and deflection could be calculated for all the three axes.

Table 1: Relationship between applied voltage and deflection for x-axis

d_{ref} ...reference distance

U...applied voltage

$d_{ref} / \mu\text{m}$	U / V	$d_{ref}/U / \mu\text{m}\cdot\text{V}^{-1}$
1.86	8163	0.000227
1.86	8126	0.000229
1.85	8200	0.000226
1.87	8201	0.000228
2.56	9183	0.000279
8.50	33870	0.000251

For the x-axis the calculated relation between applied voltage and deflection in nm is $d_x = (0.24 \pm 0.02) \cdot U$ where U is the applied voltage between -80V and +80V as 16 bit value. There should be mentioned that the real error is probably much larger than the

5 Scaling

standard deviation because linearity was assumed and the accuracy of the measurement was not so good.

Table 2: Relationship between applied voltage and deflection for y-axis

d_{ref} ...reference distance

U ...applied voltage

$d_{ref} / \mu\text{m}$	U / V	$d_{ref}/U / \mu\text{m}\cdot\text{V}^{-1}$
1.00	51250	0.0000195
0.32	14340	0.0000222

$$d_y = (0.020 \pm 0.002) \cdot U$$

Table 3: Relationship between applied voltage and deflection for z-axis

d_{ref} ...reference distance

U ...applied voltage

d_{ref} / nm	U / V	$d_{ref}/U / \text{nm}\cdot\text{V}^{-1}$
90	750	0.120
89	685	0.130
89	700	0.127
90	677	0.133
90	710	0.127
92	716	0.128
91	733	0.124

$$d_z = (0.127 \pm 0.004) \cdot U$$

With the relationships between applied voltage and deflection for all 3 axes Fig.20 was scaled. The comparison between the images of the commercial AFM and the build piezoresistive AFM can be seen in Fig.25.

6 Conclusion

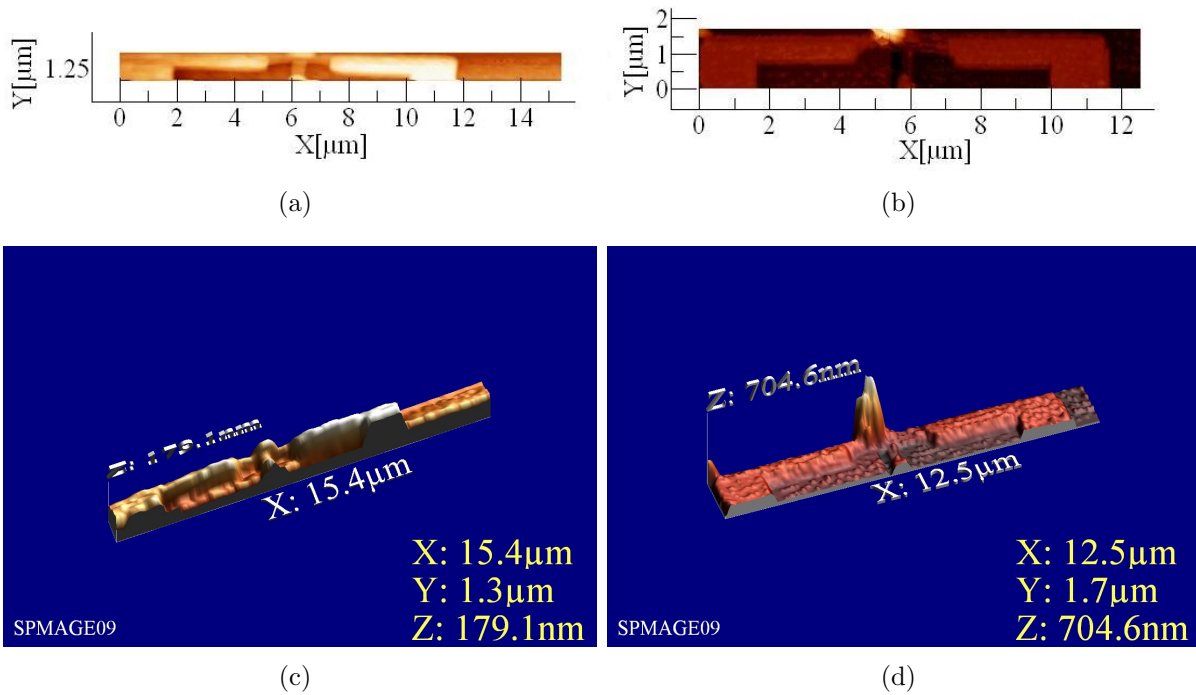


Figure 25: Comparison between commercial AFM and the build piezoresistive AFM - (a),(c) build AFM (b),(d) commercial AFM

6 Conclusion

On the whole the AFM we build with a piezoresistive force measurement system can be used to make 3 dimensional images. However, the resolution is not as good as the resolution of a commercial AFM. It was not the main point to build a perfect AFM but to get more experience with the piezoresistive force measurement system. Furthermore it would be interesting to use conduction cantilevers for electrical measurements. Equipped with a conducting cantilever this simple system would give the opportunity to do both, electrical measurements and topographic imaging at the same time.

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