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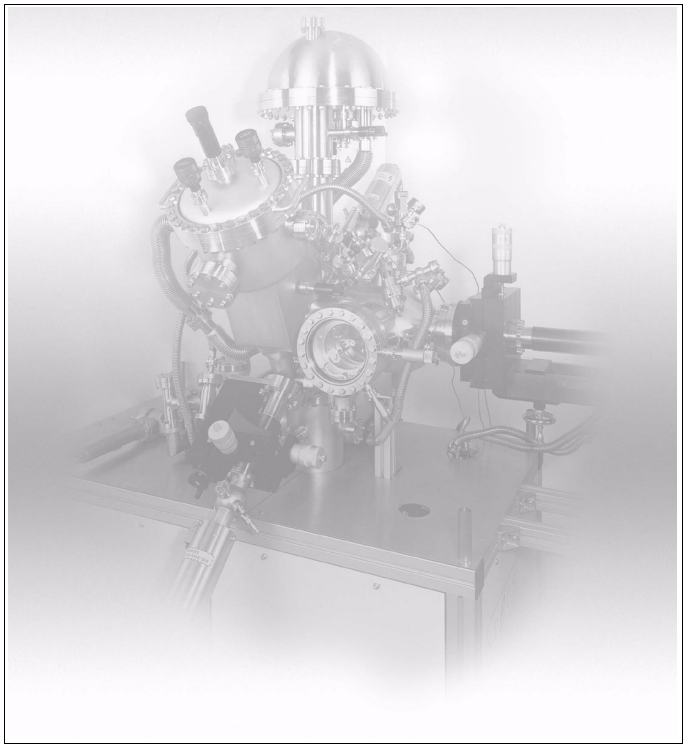

# STM 150

## Aarhus / USB

## User Manual

rev. 3.0

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User manual for the Aarhus / USB STM 150, USB version.

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# Chapter 1 Introduction

## 1.1 Introduction

The SPECS STM 150 Aarhus with ultimate atomic performance allows scientists to observe processes on surfaces at a scale of nanometers. The miniaturized design, with a smallest mechanical loop between tip and surface, results in extreme stability unique to the STM market. Fast scan rates are achieved by high resonant frequencies of this scanner head design. The SPECS STM 150 Aarhus is equipped with a fast approach mechanism for full approach speeds of more than 1 mm/min. The tip may be cleaned by parallel ion beam etching, field emission or short voltage pulses with no necessity for tip exchange as in other STMs.

The STM 150 Aarhus was designed by Professors Flemming Besenbacher, Erik Lægsgaard, and Ivan Stensgaard at the University of Aarhus, Denmark, and is being exclusively produced and marketed by SPECS GmbH.

## 1.2 Unpacking the Instrument

The SPECS STM 150 Aarhus consists of the following components:

**Components for mounting to the UHV-Chamber:**

1. STM 150 Aarhus head on a DN150CF (8'') conflat flange. The sample holder is mounted to a massive metal block (“cradle”). The scanning unit (“STM”) is located below

the sample holder. There are 3 feedthroughs on its back-side named A, B and C, see figure 12, “The STM flange from the back,” page 15.

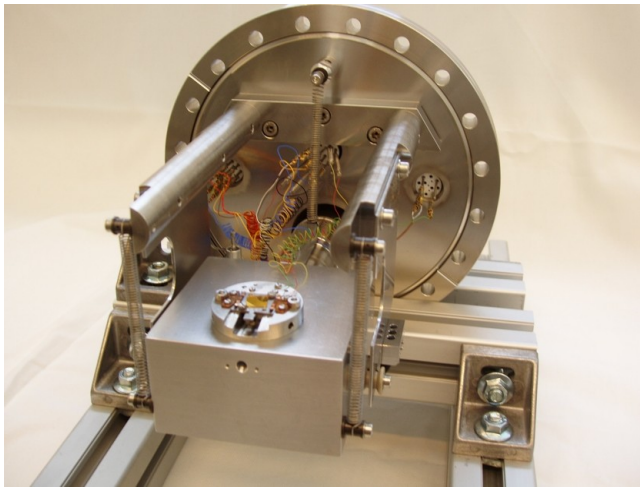


*Figure 1: The STM 150 Aarhus in a dedicated holder*

2. Set of M8 bolts and a CF150 copper gasket.
3. Magnetic transfer rod with sample transfer tool, including a port aligner, bolts, and a CF38 copper gasket.

**Electronics:**

4. Scanning probe controller SPC 260. Main controller of the electronics.

*Figure 2: The STM Controller, SPC 260*

5. Preamplifier. Amplifies the tunneling current in the nA range and converts it to voltages in a range of several volts.

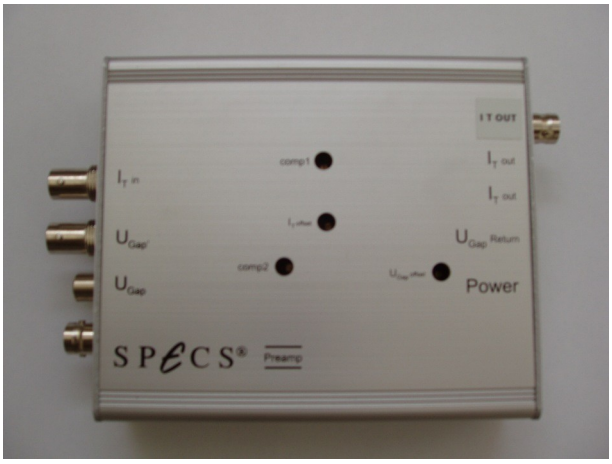



*Figure 3: The STM Preamplifier*

6. Variable temperature controller VTC 20. The temperature controller VTC 20 has two independent channels for heating the STM and the sample.

*Figure 4: The Temperature Controller VTC 20*

7. (Available March 2009) Digipot controller (see figure 5). The digital potentiometer-knobs in the top side offer an alternative way to easily offset the tip in x/y in an "analog fashion" (the same can be done by computer, too).

**(Available March 2009)**

*Figure 5: The Monitor-Digipot Controller*

10. Two wireless headphones with accessories.

**Wiring:**

12. 2-wire cable for feedthrough A on STM flange (0.15 m long).
13. 4-wire cable for feedthrough B on STM flange (2 m long).
14. 15-wire cable for feedthrough C on STM flange (4 m long).
15. A green 10-pin Amphenol plug for shortening feedthrough C on STM flange (for use during bake-out or tip-sputtering, only).
16. 3 mains voltage cables.
17. 2 USB cables

**STM accessories:**

20. Tips: 5 tungsten tips (not yet etched); 5 Pt/Ir tips (not yet cut); 3 etched tungsten tips; 15 bare tip plates.
21. Sample holders (metal plates acting as carriers for samples): 2 holders with rectangular window; 1 holder without window.
22. Sputter plate for shielding the STM while sputtering (only if a sputter gun has been delivered). In high temperature STMs, the high temperature adapter serves as a sputter plate.
23. Dedicated STM holder, see figure 1. Use for tip replacement.
24. A transport hood (steel). It can be used for shielding acoustic noise while tunneling in ambient air.

## 1.3 Installation of the STM 150

Before mounting the STM into the chamber it is recommended to prepare the STM by inserting a new tip and choosing the correct damping of the spring-suspended massive metal block housing the scanner. But first of all get a quick overview of the components and the wiring.

### 1.3.1 Quick Overview of the STM 150 Components

The STM head is mounted on a DN150CF (8'') conflat flange with all the necessary feedthroughs. Opposite the STM flange the transfer rod with the sample transfer tool has to be installed on a DN38CF (2.75'') flange facing the centre of the STM flange.

The scanning probe controller SPC 260 supplies all voltages to the STM head and reads in tunnelling current and voltage. It also powers the preamplifier, which is directly mounted near the STM flange.

The computer controls the SPC 260 electronics via a USB interface.

The Digipot serves to set the tip position offset with digital potentiometers. It is connected to the PC via the USB Interface.  (Digipot available March 2009.)

The temperature controller VTC 20 serves for heating the sample and the STM body. It need not be installed or can stay switched off for working at room temperature.

The tunnelling current can be monitored via wireless headphones. The headphone base station is connected to the SPC 260 via a standard stereo headphone connector. In addition, the tunnelling current can be monitored on an oscilloscope using the BNC cable coming from the preamplifier. Both options are not needed for operation, but are very useful.

### 1.3.2 Wiring

Figure 6 schematically shows the wiring diagram of the STM 150 Aarhus. The 15 wire cable does all the connection to the STM by LEMO plugs on the SPC 260 side and Plug C

and LEMO cables on the STM side. The LEMO cables on the STM side connect the preamplifier. The preamplifier itself is connected to the STM at Plug A via a short 2 wire BNC cable.

To connect the SPC 260 and the STM (or the Preamplifier) using the 15 wire cable, simply follow the tags on each side of the plugs, and connect according to the following scheme:

| Plug A | Preamp | Multi-pin LEMO Cables | SPC 260 |
|---|---|---|---|
| IT | $I_T$in | | |
| UGap | $U_{Gap}$' | | |
| | $U_{Gap}$ | UGap | VT out |
| | $I_T$out | IT | |
| | $I_T$out | IT | IT in |
| | $U_{Gap}$return | UGap Ret | VT in |
| | POWER | PP | P-Amp Power |

Table 1: Naming Conventions of the Cables / Connectors (1)

| Single-pin LEMO Cables | SPC 260 |
|---|---|
| +x | X |
| -x | $\overline{X}$ |
| +y | Y |
| -y | $\overline{Y}$ |
| z | Z |
| U | U |
| L | L |
| C | C |
| S | S |
| | 3pin 100mm Lemo from Z to VZ in |

*Table 2: Naming Conventions of Cables / Connectors (2)*

The Digipot gets its power by the computer and is connected to the PC via USB. Likewise, the SPC 260 is also connected to the PC via USB.

If you intend to control the temperature of the cradle and of the STM housed in the cradle, connect the temperature controller VTC 20 to Plug B at the STM flange.
**Remark:** Please make sure not to mix up Plug C and Plug B, the connectors fit to both plugs.

For monitoring the tunneling current via headphones, please use the standard headphone output.

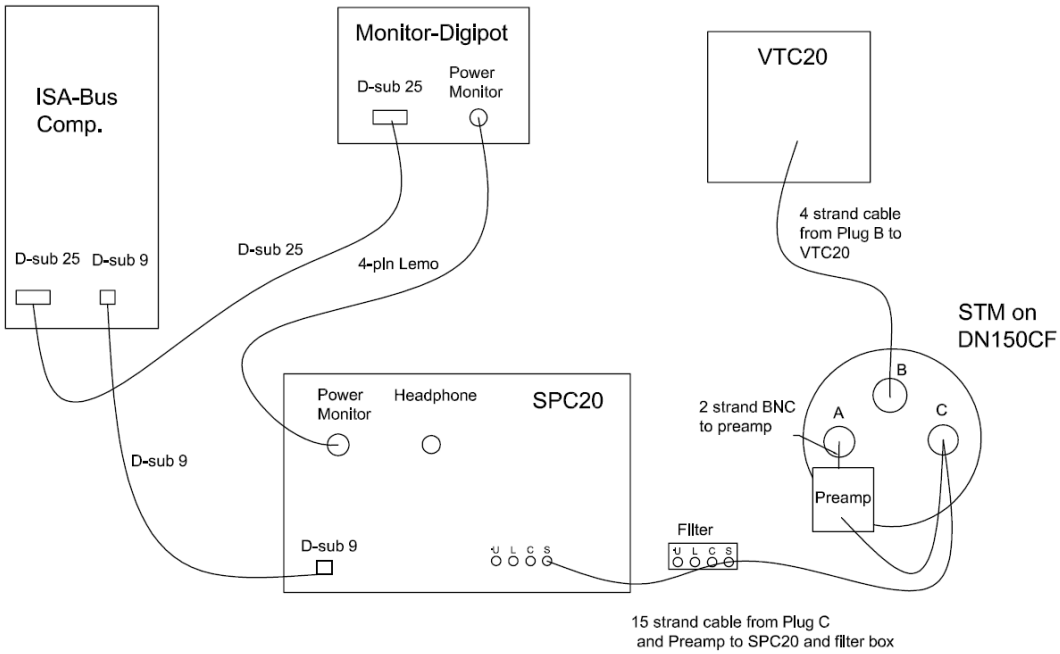



*Figure 6: Schematic Wiring Diagram of the STM 150 Aarhus*

### 1.3.3 Inserting a Tip

The STM is delivered with a sharp, etched tungsten tip still inserted in the tip holder. So for the first operation you can use this one. Nonetheless, because it is an important standard procedure to change the tip, read the following remarks and take a look at

the scanner and the tip-holder. If you want to change the tip follow the step-by-step explanation given below the remarks.

Pay attention to the cooling piston blocking the cradle. It protects the cradle from swinging and fixes it for tip exchange. In order to make the tip holder accessible, place the STM on the dedicated holder (see figure 1, page 2). Carefully remove the sample holder (metal plate acting as carrier) protecting the scanner. Now you can inspect the STM scanner and check the tip.

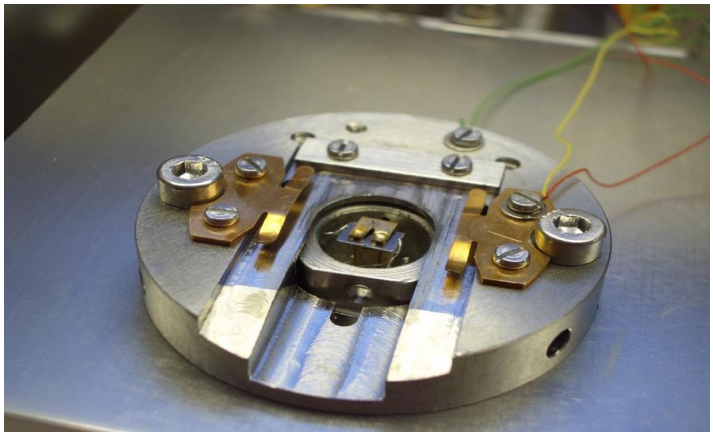


*Figure 7: The Sample Stage - scanner/tip holder is moved up*

In order to make the tip holder easily accessible, the approach motor needs to be moved up with the data acquisition program SpecsProbeUSB (see Chapter 3 “SpecsProbeUSB Software” on page 49). It is not possible to move the approach motor by hand[1]. It is important that no sample holder (metal plate acting as carrier) covers the scanner. Figure 7 shows the complete sample stage. The sample holder was removed and the scanner was moved up in order to get access to the tip.

When the approach motor has been moved up, the voltages have to stay switched on in order to tightly clamp the motor axis. Thus, potentially lethal voltages are present on

1 Because of the cradle potentially getting blocked and then the feedback loop of the STM wouldn't work. You will get the warning “tunneling current less than 40 mV”. As a consequence the motor will not stop if touching a metal sample holder.

the STM scanner and the approach motor. It is mandatory to wear latex gloves during tip exchange as a protection against high voltage. Even though there is no high voltage on the tip holder, accidentally touching the scanner could be dangerous. Zero the scanner position (no offset voltage on lateral scanning units) before changing the tip.

The tip is held in the tip holder by spring force. The tip has to slide into the holder by applying only little force. Move the tip back and forth if you experience any difficulty in pushing the tip plate completely in. Applying too much force on the tip holder can break the scanner.

**Always wear latex gloves during tip replacement. Move the scanner to the Zero position before doing the tip replacement. Watch the Monitor and make sure that -X, +X, -Y, +Y are all on zero volts (yellow LED)!**

**The approach motor could be destroyed if it is moved by hand. Applying too much force on the tip holder can break the scanner.**

Follow the step-by-step explanation to replace the tip:

1. Remove the STM from the vacuum chamber and park it in the dedicated holder.
2. Connect plug C of the STM and the electronics (the other cables can stay connected as well, but are currently not needed).
3. Start the data acquisition program SpecsProbeUSB. Refer to the software chapter for further references concerning the SpecsProbeUSB operation. In that chapter, tag numbers are used to explain the large variety of software features. In the following we refer to those features using those tags in quotation marks.
4. Because of the blocking of the cradle, the feedback loop of the STM cannot work. You will get the warning “... tunneling current less than 40 mV”. As a consequence the motor will not stop if touching a metal sample holder. Make sure that no sample holder is covering the scanner and ignore the warning.
5. Start scanning by pushing “1”.
6. Move the scanner to the Zero position by pushing “25”.
7. Stop scanning by pushing “1”.
8. Switch to the Motor Control window “53”.

9. Set the correct Motor Control parameters for your instrument. Refer to the specification sheets to look up the correct setpoint value “79” and the correct offset values “77” and “78” for your instrument at room temperature.
10. Move the scanner upwards by pushing “69”. You can choose a faster speed with "20" (green).
11. Stop the approach motor by pressing “68” when the correct height for the tip replacement is reached. Figure 8 shows the correct position of the motor. The tip plate needs to be accessible with tweezers.

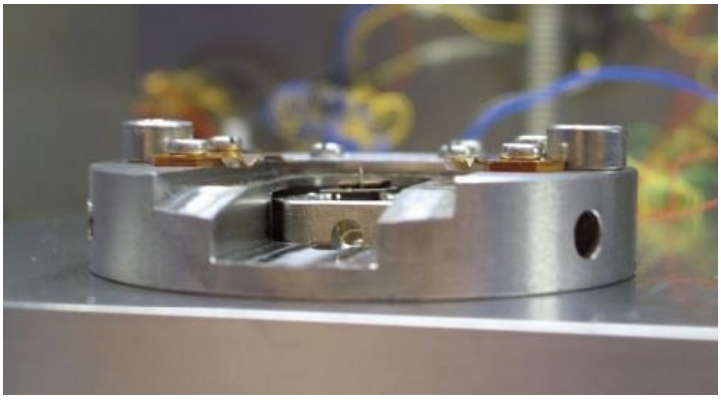


*Figure 8: Position of the Approach Motor for Tip Replacement*

The correct position of the approach motor for tip exchange is shown above.

12. Keep the high voltage switched on. This ensures that the motor holds its position while pushing in the tip.
13. Watch the monitor and make sure that +X, -X, +Y and -Y are all on zero Volts on Voltage Monitor, see section 3.2.1.10. Now no high voltage is present near the tip holder. Nevertheless, always wear latex gloves when inserting the tip!
14. Remove the old tip by carefully pushing it out with a sharp tweezers. Push successively at the tip wire and at the left and right side of the tip plate. The tip plate has notches to make it easier to move it.
15. Grab a new, sharp tip with appropriate tweezers as shown in figure 9. This figure shows the correct orientation for inserting a tip into the tip holder.

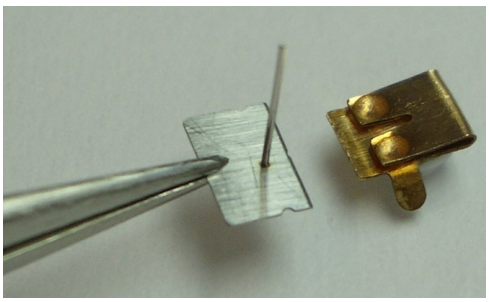


*Figure 9: Orientation for inserting a tip into the tip holder*

16. Carefully insert the tip into the holder until it is about 1 mm in. Then continue pushing it in with the tip of the tweezers, pushing on the left, on the right and on the tip wire successively. The tip is in the correct position when the tip wire sits at the end of the slot. Figure 10 shows the correctly mounted tip.

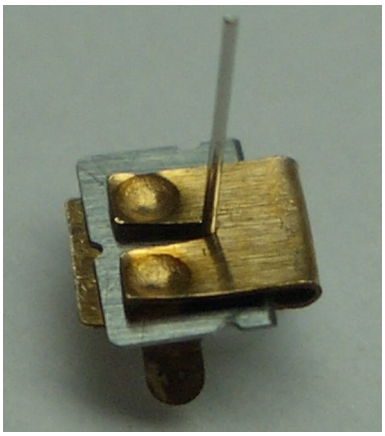


*Figure 10: The Tip Holder with a tip inserted*

17. Drive down the approach motor by pushing “67”. The motor will stop automatically having reached position -1, i.e. after retracting the tip 1mm. So you have to start retraction several times after zeroing the position indicator with "66" to move the motor several millimeters downwards.
18. Stop the approach motor by pressing “68” when the tip is far enough withdrawn to insert the sample. A safe position is reached when the tip is not visible anymore if one looks at the STM horizontally.
19. Quit the SpecsProbeUSB program and switch off the SPC 260 controller.

### 1.3.4 Choosing the Correct Damping

The STM 150 Aarhus can be equipped with different levels of damping. For damping, Viton bands can be inserted between the STM metal block (“cradle”) and the STM support frame. Figure 11 shows the STM on its DN150CF flange from below. In this figure, three Viton bands are installed for maximum damping. To reduce the damping, simply remove one, two, or all three Viton bands. The bands are held with set screws. If you take out the band, also remove the associated set screws and keep them for future installation of the damping. To observe the effects of changing the damping, the STM needs to be unlocked by retracting the cooling piston. The cradle needs to hang free in its spring mount (see section 1.4.5 “Working at Low Temperatures” on page 24 for the action of the cooling piston).

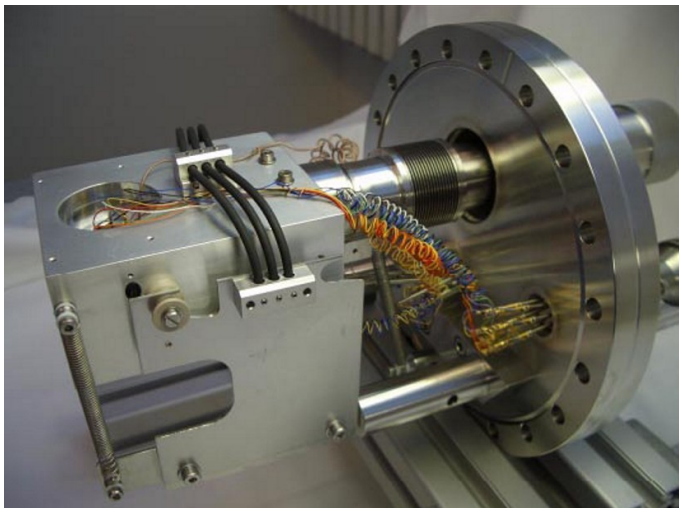


*Figure 11: The STM from below*

Three Viton bands are installed.

Choosing the correct damping means finding a good compromise: strong damping means that the STM is more closely coupled to the outside world and can pick up high frequency vibrations (5-200 Hz). Low or zero damping can lead to a resonant behaviour at low frequency (1-3 Hz, see below).

Experiments at the SPECS laboratory have shown that the strongest damping with four Viton bands gives excellent stability of below 5 pm at all frequencies (0-8 kHz). The SPECS laboratory is in the fifth floor of a very noisy building. Thus, SPECS recommends trying the STM first with all three dampers installed. The specification data is obtained with this setup.

If the STM is mounted in a low level floor or the basement, an even higher stability could be reached by removing one, two or all three Viton band dampers. The original Aarhus STM used in the university of Aarhus has no damping installed. This is also possible with the SPECS STM 150 Aarhus. However, if the damping is not enough, the STM metal block will constantly move with its resonance frequency of a few Hz. This movement is generated by low frequency building vibrations. With more damping, this resonance vibration can not significantly be excited anymore, and the metal block stays in

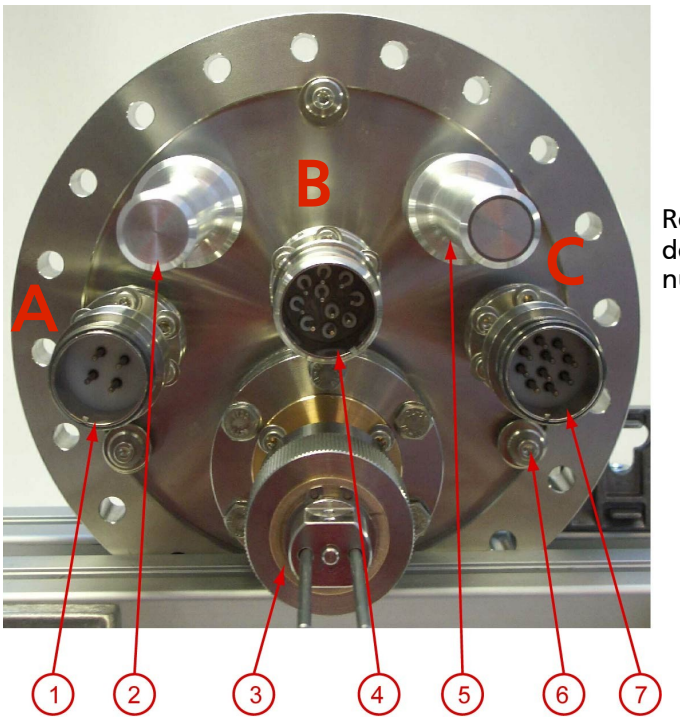
position. The block vibration can be observed by eye. If the block moves constantly, more damping needs to be installed.

We do not recommend to use air leg dampers. Typically the STM chamber on these legs has a resonance frequency of a few Hz. This frequency is very close to the STM metal block vibration frequency. As a consequence, a system of coupled pendulums can be created, which leads to even higher vibrations than without air leg dampers.

Now you can test the STM at ambient air pressure by using HOPG for example (see section 1.6.2 , "Preparation of Standard Samples" on page 33 and section 1.4 “Running the STM 150” on page 19) or mount it to a vacuum chamber as decribed in the following 3 subsections.

### 1.3.5 Mounting the STM 150 to the Vacuum Chamber

Before inserting the STM flange into the vacuum chamber, make sure that the metal block is locked with the cooling piston.
Insert the STM in the chamber: take care to not hit the chamber with the bottom of the “Cradle”. The viton dampers are held in a small aluminium block, which itself is fixed by glass balls. This part of the “Cradle” can easily break. Do not tighten the bolts yet. Figure 12 shows a back view of the STM flange.




Refer to the text below for descriptions of each tag number.

*Figure 12: The STM Flange from the back*

Level the STM with a level that can be put on the two aluminium handles (“2” and “5”). Undo the three fixing screws “6”. Rotate the flange until it is level. Now unlock the STM by turning the big screw “3” counterclockwise. The STM metal block has to be hanging free to be electrically and physically isolated from the frame. Look into the chamber and make sure that the metal block does not touch the STM frame anywhere. It has to make a few vibrations when the chamber is moved. If there is a short to ground, the metal block is still touching the STM frame somewhere and the feedback loop does not work. Check that there is no short to ground by measuring the resistance between the chamber and pin A on feedthrough A (“1” in figure 12). If you measure “no contact”, tighten the three fixing screws “6” and close the flange.

**Switch off the controller SPC 260 when pumping down the chamber or when venting the chamber. No voltages can be present when the Corona-region within 100 mbar and 1e$^{-4}$ mbar is crossed. The STM will be destroyed if this warning is neglected !**

The STM can be operated when a pressure of less than $5e^{-5}$ mbar is reached. It can certainly be operated at ambient pressure as well. Connect the preamplifier to feedthrough A and connect all powersupplies and the computer according to the wiring diagram shown in figure 6.

### 1.3.6 Baking the STM

To achieve good pressure in the STM chamber the whole system has to be baked. This, as always, should only be done when it is clear that there is no vacuum leakage present. Before the baking is started, the cradle (metal block) should be locked and all cables to the STM flange must be removed.

**! The STM 150 Aarhus can be baked at a temperature of maximum 150°C. Place a thermocouple near the STM flange and carefully monitor the temperature! Due to the construction of the STM the user must not exceed this temperature. For malfunctions of the STM due to overheating SPECS can not bear responsibility !**

Assuming sufficient pumping facilities, baking periods of maximum 12 hours should be enough to establish good pressure in the chamber. After the baking has finished it takes about 12 hours until the STM and the sample stage have reached room temperature (RT) again (don't forget to lock the cradle). We recommend to wait until room temperature has been reached before the STM is operated again. Nevertheless the STM can be operated at elevated temperatures, as described in "Working at Elevated Temperatures" on page 23.

### 1.3.7 Checking Motor Performance after Bakeout

When room temperature in the STM and the metal block housing of the STM has again been reached, (connect the VTC 20 via the four wire cable to plug B and check temperatures) connect the cables via plug C and the preamp via plug A. Start the electronics SPC 260 and the scanning program SpecsProbeUSBUSB. Go to the Motor Control menu (see section 3.2.4 "Motor Control Window" on page 60), insert the default values for "Offset Upper", "Offset Lower" and "Setpoint" used before the last bakeout. Without a sample holder inserted into the STM check by driving up and down a few millimeters that the motor is running in a way it did before the bakeout.

In rare cases the motor does not move after bakeout. You might increase the "Setpoint" to more negative values, by steps of 10 to 20 Volts, and try again.

As soon as the motor has started to move, reset "Setpoint" voltage to the before used value. Typically the motor should move with those values, again.

### 1.3.8 Adjusting Asymmetry of Coarse Approach Motor

The approach-motor enables a very fast approach and retraction speed of the scanner head over distances of several millimeters. Its linear motion is performed stepwise and results to a "wandering"-movement of the axis holding the scanhead. The width of each step and the repeat-frequency can be chosen by the user. The approach-sequence is controlled by the software which avoids tip crashes very dependably. When having reached tunneling contact, the motor does not perform fast, "step-like" motions, but holds/finetunes the position of the scanhead. So the imaging performance is not influenced by the Motor Control.

Because of the stepwise motion over large distances, it is only possible to count the number of "steps" instead of measuring exact distances. In practice this is of minor importance, because you can always retract the tip to a save distance (about 3mm), change the sample safely and get into tunneling contact again. The tunneling contact should always be used to "zero" the motor-position, i.e. it should be taken as a reference point.

The exact actual speed of the motor depends on temperature. It also depends on the direction of the movement of the motor (asymmetry). Both variations are in a range of few percent. To correct this, the temperature dependence was tested during certification. This testing resulted in a motor parameter-set as a function of temperature of the motor. Those sets can be found in the certification document of your STM. Please check the appropriate set and enter it to the software.

From time to time one should recheck the performance of the coarse approach motor (especially after bakeout). This is a kind of safety procedure: in practice it is not very important to reach a perfect symmetry in motor movement. A deviation of 5 percent does

usually not cause problems. But you should not apply a lot of up and downward movement to the motor at large distances without referring to the physical benchmark “tunneling contact”. This is because the position-offset caused by a slight asymmetry could sum up to rather large values.

The details of the test procedure are described in the following:

Since the STM has no direct physical reading of the overall position of the motor in the z-direction, it is important to be able to control and adjust the speed in both directions (to and from the sample). To do this, one can use the tunneling contact as a physical benchmark:

(1) Approach into tunneling contact and reset the position to zero (button “66”, figure 23, “Motor Control window (approach motor control),” page 60) . Choose the "slow" speed in you are working with a good tip. If you are practicing in air, you can choose medium speed with "20" (green).

(2) Drive down the motor by a certain nominal distance (e.g. -1 mm, as the software automatically stops then). Driving up the motor will end in tunneling contact again. This new tunneling contact will give a new reading for position. If the reading is 0.00 mm, the speed in both directions is identical. Still the overall speed and hence the exact distance of retraction of the tip is unknown, because the step size of the inchworm cannot be measured but only the number of cycles and the voltages applied to the motor.

(3) If the speed upwards is larger than downwards (negative reading for position after regaining tunneling contact), then typically the clamp force on the upper clamp is typically smaller than the clamp force of the lower clamp, and vice versa. One now can adjust this difference (to a certain extent) by increasing the clamp force on the upper clamp (more positive voltage via button “77”, figure 23, “Motor Control window (approach motor control),” page 60, and loosen the clamp force on the lower clamp by decreasing “Offset Lower” voltage button “78”, same figure, in a symmetrical fashion with change in “Offset Upper” voltage. Do this in steps of 10 to 20 Volts.

(4) Repeat the procedure by starting to zero the position with “66”, drive down and up to tunneling contact. With this procedure you should be able to decrease asymmetry close to zero. If asymmetry can not be adjusted by offsetting upper and lower clamps (see (3), above), one can try it by also offsetting “Setpoint” voltage in the negative direction. This is the appropriate choice if the motor axis is too loose.

The “Setpoint” voltage, button “79” in the same figure is the common reference to all the involved electrodes of the approach motor (upper, center and lower). So, by setting “Setpoint” to a negative voltage value, one increases the applied voltage to each electrode with respect to this common reference. This results in a tighter clamp for both relevant clamps and vice versa. Also offset “Setpoint” voltage by 10 to 20 volts each run.

(5) If you have parameters that result in an offset of zero, you should use those parameters as the new default values for driving the motor. It is good to have a screenshot of those parameters on the desktop of your computer for each user to set those values as default before driving the motor.

## 1.4 Running the STM 150

For getting started quickly, please refer also to the section "Quick Start" in the software chapter.

### 1.4.1 Inserting a Sample

Before inserting a sample, make sure that the tip is withdrawn far enough. If the tip is too high, the sample might touch the tip. As a consequence, the tip will break and in the worst case the scanner can break off.

**! Make sure that the tip is withdrawn far enough before a sample is inserted. Make use of the motor to withdraw the scanner and the tip a few millimeters !**

Lock the STM with the cooling piston by turning the big screw, (“3” in figure 12) until a distinct resistance is felt.

Grab a sample from the storage garage or from the manipulator with the sample transfer tool. To do so, slide the tool over the handle on the sample holder (see figure 13, page 28) and turn it 90° clockwise to lock it to the transfer tool. Turn carefully, do not apply too much force. Do not turn further than 90°. Pull the sample out and push it carefully into the STM until the stop. Now turn the transfer tool 90° counter-clockwise and pull away the transfer tool.

**Remark**: For the first time it may be difficult to release the sample from the transfer tool without applying too much force on the STM sample holder mechanism. You should train it by putting sample holders out of and into the storage garage.

Now unlock now the STM by turning the big screw “3” counter clockwise. Do not attempt to turn the screw beyond the mechanical stop. You can obtain about 1.5 centimeters retraction of the cooling piston.

Before approaching the tip, make sure that the STM hangs free. Keep in mind: if the cooling piston is still touching the metal block, the tunnelling voltage will be shorted and the tip will crash!

### 1.4.2 Approaching the Tip

Refer to the Quick Start section in the software manual for approaching the tip. This chapter gives an additional short description of how to approach the tip.

Go to the approach-motor window. Set the correct setpoints for your instrument (those are given in the certification report).

Make sure that the STM hangs free. Make sure that all cables are connected, especially the preamplifier. Start SpecsProbeUSB. By default the tunnelling current is set to 0.001 nA and the tunnelling voltage to approx. +1.2 V. You should not alter these values for the approach (to achieve negative voltages you can switch polarity after having reached tunneling contact).

Start the approach sequence by clicking the up arrow button “69” in the approach motor window, and choose "slow" speed with "20" (green). When the tip is close to tun-

neling contact the approach sequence is terminated by the electronics. The software then does some fine adjustment of the tip position. This fine adjustment is finished as soon as the $I_t$ value, which is “60” in the approach-motor window, shows actual values again. After this, click the up arrow  button once again . A fine tuning of the motor position is done once again.

Go back to the Scan Control window. Set the tunnelling current and voltage to the desired values. Start a scan. Adjust gain and speed.

In order to change the polarity to negative voltages, first wait until a scan is finished, then reduce the current to zero and switch voltage.

Before going back to the approach-motor menu, e.g. for withdrawing the tip, set the tunnelling current to minimum (0.001 nA)!

### 1.4.3 Taking STM Images- basics

Measuring good STM images takes some practice. It is important to set the correct values for the tunnelling voltage, the current, the feedback loop gain and the tip speed. Refer to the Quick Start section in the software chapter to see where to change these parameters and how to save images. Most important is the quality of the tip and the sample. We present some tips for measurement practice in section 1.6 , "How to Get Good Images" on page 32.

In the current chapter we want to focus on basic considerations concerning the interplay between motor for stepwise approach and the scanner itself with its limited Z-excursion of about 125nm in both directions. For highly corrugated samples it is important to use the entire z-range of the scanner. One has to operate the scanner such that its “zero” Zposition equals the overall (average) height of the sample of the scanned region.

The motor position is always fixed during scanning of a sample. So it could be that the Z-value of the scanning voltages comes close to the limit of +200 or -200 V. Then the tip can either lose contact (values larger than +200V) or it can crash (values smaller than

-200 V). The Z-voltage is displayed in “24” of the Scan control window, see figure 21, page 54.

**! If the $V_z$ is close to -200V the tip will crash. Stop scanning, reduce tunneling current to zero and move tip with motor using button “10” in the scan control window (figure 21, page 54) away from the sample !**

In any case you can simply move the approach motor with button “10” in the scan control window (figure 21, page 54). This will first only change the voltage on the center electrode C of the motor. C is displayed beside the motor position in “9”. In addition, it can be monitored on the Monitor-Digipot. If C comes close to +200 V or -200V (upper and lower red LED on the Monitor-Digipot), the motor will make a coarse step. Before the coarse step occurs, the tip has to be withdrawn by turning the tunnelling current down to 0.001 nA. If a coarse step is performed during tunnelling with currents higher than that, the tip could crash.

**!Reduction of the tunneling current to zero leads to a retraction of the tip, thereby avoiding tip crashes!**

For normal operation there is an elegant software feature: AutoVz (see section 3.2.3 , ‘Scan Control Window II” on page 57). This AutoVz- function “37” adjusts the voltage Z (on the scanner) and the voltage C on the motor such that Z approaches 0 Volts. In other words, the software fine tunes the mentioned voltages such that Z always has maximum range to and away from the sample surface. This feature is very useful and you should make extensive use of it. Nevertheless when C is close to +200 or -200V AutoVz can not work anymore, a coarse step as described above should be performed. So again reduce tunneling current to minimum beforehand! Alternatively you can use this fine-tuning of the motor position manually by the shortcut “Ctrl-Home”, also see the list in section 3.4 , on page 81).

There is another reason why it is important to find the optimum zero-position for the scanner. The Z-voltage is converted by an A/D converter to digital data with a finite res-

olution. To obtain best digital data (with no disturbing artificial steps in the grayscale) it has a programmable range. I.e. out of the +/-200 V that are available for Z, only a certain portion is converted to digital data. This converted range can be selected to be +/-5 V (appropriate for very flat images on a small scale), +/-10 V, +/-20 V, +/-50 V, +/-70V, +/-140 V, or +/-200V (for large area scans on highly corrugated surfaces). This converted z-range can be selected with “14” in Figure 21, “Scan Control Window I”, page 54 ). If the current Zvoltage is outside this range, the image will either be saturated dark or bright. Nevertheless, the tip will not yet crash until Z reaches -200 V. To keep the Z-voltage value within the A/D converter range, the approach motor can be used to center the Z-voltage near Zero by using AutoVz.

### 1.4.4 Working at Elevated Temperatures

The temperature controller VTC 20 allows one to control the temperatures of the sample stage and the STM and to regulate them (almost) independently in the specified range.

For stability in STM measurements, elevated temperatures (above RT) are disadvantageous: one has to expect higher noise levels and higher drift rates (especially if the STM and sample are not at the same temperature). For temperatures higher than room temperature it is possible to heat the STM to the same temperature as the metal block and the sample. This minimizes relative drift of sample and STM.

The Aarhus STM can be operated at elevated temperatures (up to 400K). But extensive use at 400K can cause depolarisation of the piezo ceramics used in the scanner. Firstly, calibration is lost and pictures get distorted. In the worst case piezo ceramics do not respond to applied voltages anymore. To avoid this, **high scan voltages with negative polarity should not be used if the STM is hot**. So do scanning in a region in the vicinity of zero or at positive offset values in X and Y (stay in the center or in the upper right quadrant of the scan window “48”, figure 22, “Scan Control Window II”, page 57. Do not operate the scanner permanently with highly negative Z-excursions. First use AutoVz as discribed above to zero the scanner z-position, then subsequently move away the tip with the motor causing the scanner to elongate. This should lead to a positive voltage of about 50V at the z-scanner.

Repolarisation of the piezoceramics is a standard procedure at the University of Aarhus after high temperature mesurements. Please contact SPECS for details if you think you partly depolarized your scanner. Repolarization is an easy task and can even be done routinely.

Nonetheless baking the STM at maximum temperature 423K does not cause depolarisation, because no voltages are present in the piezo ceramics. For precise measurements however you have to recalibrate the instrument after baking (typical change in excursion 1 to 2 percent after each baking, see also section 1.6.5 , "Calibration of the STM", on page 40.

### 1.4.5 Working at Low Temperatures

Working at low temperatures on the other hand improves the stability of STM measurements, as long as the increased drift due to different temperatures of sample and STM can be compensated. The samples in the Aarhus STM get cooled by pumping LN2 (liquid nitrogen) through the tubes of the cooling piston “3”, see figure 12, while the cradle that is holding the sample, is locked (turn the big screw clockwise until distinct resistance). Because of the thermal contraction of the cradle and the cooling piston, it is recomended to retighten the big screw one or two times during cooling.

**! Use appropriate handgloves if you touch the ultra-cold cooling piston - danger of severe injury (frostbite). Wear protection-glasses when you use liquid nitrogen!**

After the desired temperature of the sample is established (temperature reading T2 at the VTC 20) the cradle is unlocked and the STM (T1) is heated up to RT. The reason for this is that the piezo-constants keep their known values (no need for recalibration of measured data). Therefore, typically, sample and STM are not at same temperature, which is a source for drift. But this drift can be compensated for by the methods described in software part, submenu: Drift Compensation, see section 3.2.6.5 , on page 68. During tunneling the metal block hangs free and therefore the sample will warm up, starting from 90K, at an initial rate of approx. 10K/hour. The rate halves every hour, i.e.

the temperature curve is exponential. The warming up curve can be found in the certification report of your instrument (included in the shipment from SPECS).

### 1.4.6 Removing the Sample

**The sample must only be removed from the STM stage when the tip has been retracted sufficiently such that no damage to the tip and the scanner can occur (for this point see section 1.4.10 )!**

Additionally, the STM stage must be locked (clockwise turn of the big screw of the cooling piston until distinct resistance) and the high voltages HV should be turned off (the easiest way to do this is to terminate the SpecsProbeUSB program). Recheck the +-205V LEDs on the Monitor-Digipot, they have to be switched off. If this situation is established the sample plate can be removed just the opposite way it was introduced into the STM, see above section 1.4.1.

### 1.4.7 STM Tips

With the STM you have a selection of (pre)-prepared STM tips. These include 5 Pt/Ir-tips, 5 W-tips, and three etched W-tips. Also in the delivery you will find 15 bare tip-plates that are to be used for fixing tips. Fixing tips on those plates is done by spot-welding. See figure 8, page 10 for details. All tips should be finished to a length of 2-3mm. The Pt/Ir tips have to be cut with a sharp edge cutter. The tungsten tips are to be sharpened via tip etching to the same length (for details see "Tip Preparation (ex situ)" on page 32 or contact SPECS).

### 1.4.8 In-situ Tip Preparation

Even if no sputter gun has been ordered and delivered for tip etching there are various ways to prepare the installed tip. They all use the temporary application of voltages between tip and a surface (typically the sample to be measured). For details see the SpecsProbeUSB part of this manual, submenu Z(V), I(V) and the shortcut list to SpecsProbeUSB. Aside these "soft" methods there also exists the possibility to perform field emission between tip and "sample". For details please contact SPECS.

### 1.4.9 Tip Sputtering

Sputtering with noble gas ions is another way to prepare the tip. The sputter gun should face normal to the sample plate region (or equivalently, in an axis parallel to the tip). The sputter gun above the STM should be positioned such that the ion-flux focus can be adjusted directly onto the tip. This can be achieved by a port aligner (e.g. SPECS IQE 11/35 plus port aligner) or via a sputtergun with deflection units (e.g. SPECS IQE 12/38). In any case one has to be aware that sputtering into a STM is a delicate task: sputtering ions and secondary ions could hit the piezos of the STM scanner or the approach motor. Those ions could electrically charge up the piezos, destroy the piezo material, or, by depositing metal, bridge the necessary insulation between the various piezo elements.

Therefore the requirements for tip etching are:

Obtain a small sputtered area, therefore a short distance between sputtergun and tip or a sputter gun with adjustable focus.

Use a shield to protect the STM that reduces the sputtered area to the very vicinity around the tip. Customers that have purchased a SPECS sputter gun for tip etching have a shield for this purpose in form of a sample holder with a tiny bore hole of approx. 1mm in the center right above the tip.

Strictly avoid high pressures, especially **do not accidentally cross the Corona-region within 100 mbar and 1e$^{-4}$ mbar**, with voltages on the STM switched on. Do not use high ion currents during sputtering.

Follow the step by step instruction:

1. Insert the sputter sample holder with the hole for the tip.
2. The tip is moved towards the sample, as if one wanted to do tunneling on that bare metal, see “Approaching the Tip” on page 20.
3. If the tip is facing straight up and is well positioned in the tip holder, the tip eventually will drive through that tiny bore hole without touching the shield plate and thus

not getting into tunneling contact. Tunneling contact and therefore automatic stop of the approach is achieved as soon as the tip base "touches" the sputter shield.

4. Having reached this point, drive the tip down approx. 0.002 mm and stop again (for this choose a low speed of the approach-motor). Now the scanner is physically shielded from the ions.
5. Lock the cradle, disconnect all cables and measure with a multimeter resistance between PlugA-PinA (sample bias) and PlugA-PinC (tunnel current: tip) - there has to be no contact.
6. SPECS recommends offsetting all the coordinates of scanner and inchworm by applying +200V with respect to ground. If you have purchased the sputter gun with port aligner and shield plate you can use the included green 10-pin plug on Plug C during sputtering for that purpose. This green Plug C with its Lemo connector can be applied to output channel S of the SPC 260 during sputtering. Channel S is not needed for the motor during sputtering and you can use it as a HV supply. It allows to offset all coordinates by maximum 200 V via the Motor Control menu (Setpoint voltage). By this means the electrodes are biased positively against ground to establish a repeller voltage for the sputtering ions.
7. Now monitor the current between PlugA-PinC (tunnel current: tip) and system ground, i.e. the sputtering current on the tip. As sputter parameters we recommend: Arions of about 2-4 keV, currents about 5 µA and sputtering time of approx. 15 min.

**WARNING**
**Make sure to put the Setpoint value back to its nominal value before moving the motor!**

8. After this, establish the original situation, exchange cables on Plug C and SPC 260, attach preamp again, retract tip (completely!), replace sample, and start tunnelling.

### 1.4.10 Sample Holders

The STM sample holders are based on the standard SPECS sample holders (see figure 13 left). These sample holders can be modified by the customer to special needs. But there are restrictions due to the set up of the STM that have to be met. These restrictions are depicted in the same figure on the right side: **the distance between the lower plane of the sample holder and STM body is only about 1mm! This limits the depth of the**

**sample surface facing towards the STM. If these restrictions are not met, introducing a sample into the STM could destroy the STM!** In the same way on the backside of the sample plate the two springs (not shown in figure 13), that hold the sample plate in the STM have a distance of 12mm from each other. **So the mounted sample should not have a diameter that exceeds 10mm, otherwise it can not be inserted into the sample stage on the cradle!**

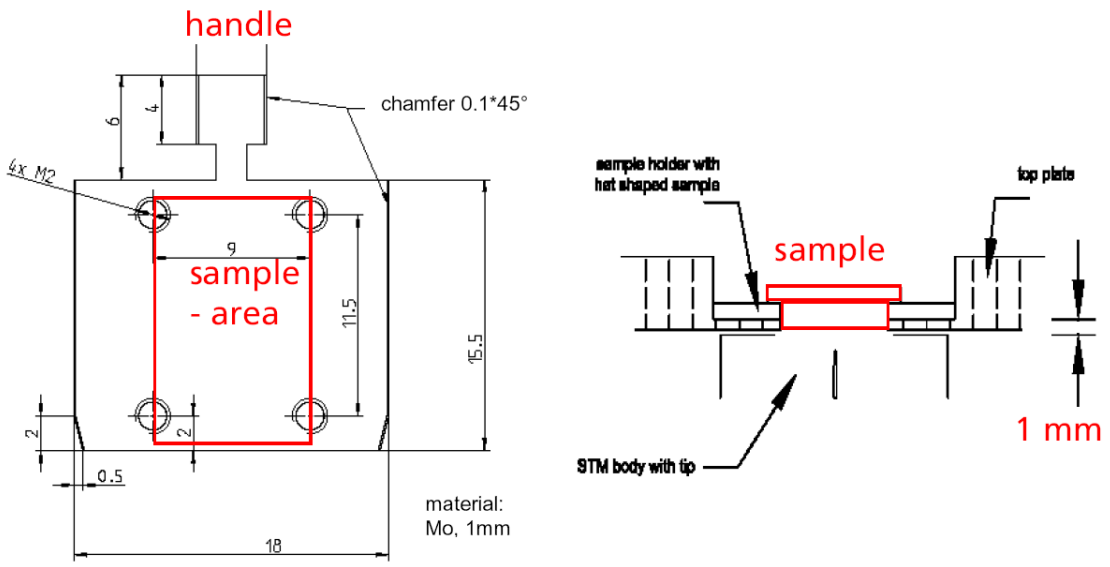
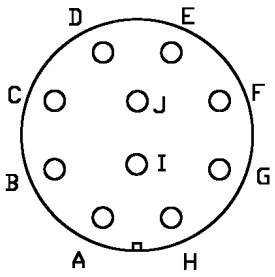



*Figure 13: Standard SPECS Sample Holder (l), Schematic Drawing of the Sample (r)*

Figure 13 shows the standard SPECS sample holder, that can be modified to special needs. Right of figure 13: Schematic drawing of the sample in the STM. Please note the small distance between sample plate and STM body of about 1mm!

### 1.4.11 Pin Assignment




(view onto feedthrough
from air side.
Note: Plug A is only 4-pin)

*Figure 14: Schematic Drawing of the Feedthroughs (STM Flange)*

The table below describes the pin assignments from the diagram above:

| PIN | Plug A (Current) 4-pin | Plug B (Thermo) 10-pin | Plug C (Current) 10-pin |
|---|---|---|---|
| A | Sample Bias, $U_{bias}$ | STM Zener Diode return | Z Electrode |
| B | | STM Zener Diode | |
| C | Tunnel Current, $I_t$ | STM thermo_A[2] | Y Electrode Neg. |
| D | Tunnel Current Braid | STM thermo_C | X Electrode Pos. |
| E | | Zener diode 1&2 return | Y Electrode Pos. |
| F | | | X Electrode Neg. |
| G | | Sample thermo_A | Piezo Motor bottom:= L |
| H | | Sample thermo_C | Piezo Motor centre:= C |
| I | | Zener Diode 1 | Piezo Motor reference:= S |
| J | | Zener Diode 2 | Piezo Motor top:= U |

*Table 3: Pin Assignment of the STM 150 Aarhus*

2 Pin Assignment on the DN150CF base flange. _C: Chromel, _A: Alumel. The colour codes of these cables are in vacuum/air: _C: Yellow/White, _A: Red/Green.

## 1.5 Tuning

### 1.5.1 Tuning the Preamplifier

The preamplifier (see figure 15) converts the tunneling current, which is in the nA-range to a voltage in a range of several volts. The zero point of current and voltage can be adjusted and the frequency response can be tuned. Because the currents measured are extremely small, the capacitive coupling to the tunneling junction has to be compensated, too. SPECS tunes the preamplifier before delivery, but this procedure should be repeated from time to time, especially if highly accurate spectroscopic data using specific tip shapes is required.

*Figure 15: Wiring of the Preamplifier*

Figure 15 shows a schematic diagram of the preamplifier wiring, which is interconnected between the tunneling junction (STM) and the controller SPC 260.

**Inputs of the preamplifier:**
The preamp input is connected to the tunneling junction. It sets the exact and stabilized tunneling voltage via “Ugap’” and measures the tunneling current via “It in”. The tunneling voltage is preset with the controller SPC 260 via “Ugap”.

**Outputs of the preamplifier:**
The tunneling voltage is connected with the controller by “Ugap, return”. In the preamp the tunneling current is converted to a voltage in a range of volts (it’s “ampli-

fication" is about 1V per nA) and send to the controller by "It out". There is a monitor point for the current also called "It out", using the LEMO connector type instead of BNC.

**Adjustments:**

The descriptions of adjustments presented below always start from normal operation conditions: all plugs connected, power supply via SPC 260 switched on.

Allow the preamp to warm up for at least 1 hour. The preamplifier should be in the 10nA range as set by the scanning program.

For compensation of the capacitive coupling between tip and sample you need a representative sample and tip, i.e. you should be ready for measurements.

**Required extra equipment: Voltmeter (mV-range), cable with BNC connectors, oscilloscope, wave generator (square wave), attenuator (-20dB), 2 pin to BNC adapter.**

**1. Adjusting zero point of tunnel current**

Disconnect "It in" (leave it "open") to achieve zero tunneling current. Disconnect "It out" (BNC-connector) and measure the DC output voltage (!) by a voltmeter in the mV range. Now adjust the voltage to zero using a screwdriver on the "It offset" screw.

**2. Adjusting zero point of tunnel voltage**

Unplug "$U_{gap}$", $U_{gap}$' and "It in" and connect $U_{gap}$' and "It in" in order to shorten the STM circuit, use a BNC cable for this (now the gap voltage equals zero). Disconnect "Ugap, return" and measure this DC output voltage by a voltmeter in the mV-range. Now adjust this voltage to zero using a screwdriver on "Ugap, offset".

**3. Adjusting the amplifier frequency response**

Unplug "It in". Apply a 1kHz square wave signal (from a wave generator) **using a -20dB attenuator (!)**: take an amplitude of 100mV (peak to peak) through the attenuator

being connected to “It in”. Disconnect “It out” and measure the AC output wave form by an oscilloscope. Now adjust to best square wave response using a screwdriver to adjust “comp1”.

**4. Adjustment to cancel the tip-sample capacitive coupling**

For this the STM needs to be connected and a reference sample and tip is recommended. Approach the tip until tunneling contact (see preceding section) occurs. Retract the tip to a distance of 0.1mm from tunneling contact.

Unplug “Ugap”. Apply a 10kHz sine wave signal to “Ugap”: use an amplitude of 600mV (peak to peak). For this you need an adapter BNC to LEMO. Disconnect “It out” and measure the AC output by an oscilloscope. Now adjust to minimum output amplitude using a screwdriver to adjust “comp2”.

## 1.6 How to Get Good Images

### 1.6.1 Tip Preparation (ex situ)

Pt tips are cutted with a sharp wire cutter- though they are not really sharp on atomic scale you can attain atomic resolution on any surface which is not corrugated to much. Etched tungsten tips are much sharper on a mesoscopic scale. They are the appropriate choice for rather rough surfaces. For etching recipes contact SPECS.

In vacuum sputtering of the tip is best to clean and sharpen the tip. For this refer to section 1.4.9.

## 1.6.2 Preparation of Standard Samples

### 1.6.2.1 Highly Oriented Pyrolytic Graphite (HOPG)

Highly Oriented Pyrolytic Graphite (HOPG) is polycrystalline graphite. The domain size of small graphite single crystals it consists of can be influenced in a certain range during fabrication (at least several hundred nanometers). Typically the cleavage axis of all graphite micro-crystals are oriented in one direction, but the micro-crystals are rotated in an arbitrary way along this axis. Hence its diffraction pattern is not spots, but concentric rings.

Preparation at ambient air pressure is very easy: a clean, almost step-free surface can be created by taking a piece of adhesive foil (simply take high quality sticky tape), putting it on the surface and peeling away the top layers of the HOPG (this is “cleaving”). There is no further preparation needed for UHV applications except annealing at 400K to get rid of adsorbed water.

The surface is rather inert (small reactive strength) and not much site-specific (low corrugation). A variety of large adsorbates create a carpet-like crystalline overlayer on this surface with no or few structural reference to the graphite micro-crystals. Typical STM parameters are +1V, 0.5nA. If the tip has previously crashed it is often very difficult to regain resolution. If the “in situ” tip sharpening procedures described in section 1.6.4 do not often work, then sputtering or cutting of the tip is recommended. For some reason it is easier to get atomic resolution under ambient air pressure than under UHV conditions.

### 1.6.2.2 Silicon

Silicon wafers are polished slides of large single cristal rods of silicon. Because of the requirements of the chip industry they are very precisely oriented and cut. Due to fabrication and storage at ambient air pressure they have got a thick oxide overlayer, which is not ordered and does not allow STM measurements.

To get rid of the oxide overlayer you can heat the sample by conducting electric current through the sample. The conductivity of a semiconductor drops with temperature, so you need to control the current at the power supply (not the voltage)! Pure silicon has got a very high resistance at room temperature, you either need a high voltage to send a current through it or you heat it by radiation to start the current. Doped Si has got a much lower resistance. SPECS uses wafer pieces of 0.5mm thickness and 2 mm breadth. We start the heating at 60volts ending up with 11 to 12 amperes. First heat the sample by a current of 0.6A overnight in order to degas the sample holder. The sample should glow red if turning off the light in the lab and in the chamber. To get rid of all the oxide “flashing” at a base pressure better than $5x10^{-10}$ mbar is recommended. For flashing, increase the current to about 11A for 1 second. The sample should glow uniformly in almost white color. After cooling down you should get a clean Si surface. In case of using Si(111) the famous 7x7 reconstruction is formed. SPECS uses such samples to obtain certification data.

#### 1.6.2.3 Noble Metals

Noble metals like silver or gold are available as single crystal rods. They are cut with appropriate methods in the desired direction to get for example (111), (110), (100) or even high indexed vicinal (stepped) surfaces. Subsequently they are polished to get a flat surface and etched to get rid of the polishing agent. All this is done at ambient air pressure.

After this the surface is sputter-cleaned and annealed several times in UHV. Ag crystals are sometimes contaminated with sulfur. Annealing always enriches such bulk contaminants at the surface. Sometimes this makes it impossible to obtain a clean surface. In general the chemical reactivity of noble metals is medium and slightly depends on the surface orientation. “Open” surfaces like (110) are most reactive, regularly stepped surfaces (vicinals) offer maximum corrugation for adsorbates. There is a variety of large adsorbates known (organic dyes for example) creating ordered overlayers matching to the atomic structure of the metal single crystal. This is possible because they often do not stick to the surface, but diffuse around at elevated temperatures. Clean gold or silver itself is very mobile even slightly above room temperature. As a consequence high temperature STM measurements on clean Au do not make much sense. Adsorbates trap the

gold atoms and reduce mobility. Some adsorbates can influence the terrace width distribution by trapping and bunching steps, i.e. they can create their desired surface to a certain extent.

Noble single crystals can be spoiled permanently even under UHV conditions. If one deposits a thick overlayer of organic molecules and subsequently performs some sputtering to clean the surface carbon microparticles are created. These particles cause step bunching and lead to rough surfaces. You never get rid of those particles even by excessive sputtering. Instead of sputtering you should remove thick overlayers by evaporation first if possible. Subsequent sputtering of the remaining monolayer of adsorbate is less harmful.

Typical tunneling parameters are 1-2V, 1nA for Ag and Au. The clean surface can be altered by the tip, i.e. the tip can move surface atoms. Hence the study of diffusion processes by STM is difficult.

### 1.6.3 Checking Quality of Sample Preparation

STM is a local probe of the electronic sample surface structure. It is not possible to check a smaller area in the order of some percent of the overall surface because of the extremely small scanning field. Hence a well defined uniform surface pre-preparation is needed. This has to be controlled prior to STM measurements using supplementary information from other techniques. Typically crystal surfaces are used. The sample surface is cleaned, and its chemical cleanliness and degree of ordering is checked by a appropriate “integrating” methods. Subsequently the desired surface is created by preparation and again checked by integrating methods. Now STM measurements taking hours or even days are performed to get local information. After this the same integrating methods can be used to recheck the surface properties. By this any perturbing influence of the residual gas in the chamber can be seen.

Often single crystal surfaces are used for surface science applications. A standard method of checking the degree of ordering is LEED (Low Energy Electron Defraction). With this method you easily get the diffraction pattern of the pattern of atoms on the surface. Sharp spots in this pattern indicate a well ordered crystal on a comparatively large length scale on the surface. Good LEED instruments like the SPECS ErLEED 1000A can

show pattern uniformity on length scales of about 20nm. I.e. if you have got patterned islands on your surface you can distinguish with this LEED between average islands sizes of say 5nm and 20nm but larger islands do not lead to sharper spots. With LEED, one can very precisely measure the unitary cell of a surface pattern, but in most cases you get little to no information about the surface structure within this smallest unit. Because this method is surface sensitive one only gets diffraction spots if the crystal is not covered by one or several unordered contamination layers. If only a part of the surface is patterned you will get the diffraction pattern of the ordered parts of the surface. The unordered parts simply increase background “glow” of LEED measurements. So in practice a LEED pattern does not tell much about the well ordered area fraction, but mostly about the average size and unit cell of the ordered parts. The chemical composition can be investigated with XPS (X-ray Photoelectron- Spectroscopy) (for example the high-end SPECS-XPS using the PHOIBOS analyser). This method is also surface sensitive and can detect for example unwanted contaminations on surfaces covering only a few percent of the surface. It allows also monitoring of the amount of adsorbate deposited. The results are very precise in the submonolayer region and in a covering-range of up to a few layers of adsorbate. Chemical reactions on surfaces can be monitored by measuring the so called chemical shift of the participating elements as compared to the unshifted spectra of the pure elements. Both methods help to get reliable information from STM data, because they complement this method very well.

### 1.6.4 Measuring with the Aarhus-STM: Useful Advice

#### How to avoid tip crashes

Always reduce the tunneling current to zero (in fact slightly above zero), when approaching or withdrawing the tip with the approach motor. This ensures that the tip is at a comparatively large distance from the surface. As a consequence, retraction of the tip can always be performed automatically by the feedback loop fast enough to prevent a crash.

Always keep an eye on the voltage of the scanner-head Vz: see “18” or “24” in figure 21, “Variables for tunnelling, scan control window I,” page 54. Negative voltages mean

a retraction, positive voltages a elongation of the scanner-head. The tip crashes if the minimum voltage of -200V is reached. You can prevent this by moving the whole scanner away from the sample with the approach-motor. This can be done manually with the keyboard shortcut <Ctrl-Home> (at any time, for example while scanning) or with the software feature AutoVz (see “37” in figure 22, “Scan control window II,” page 57).

Take at look at the excursion off the center electrode of the motor C at the Monitor Digipot (see figure 38, “The displays of the Monitor-Digipot,” page 74), if you intend to re-adjust the tip zero-position by AutoVz (see “37” in figure 22, “Scan control window II,” page 57). In case of the C-voltage is either maximum or minimum (red LED glowing), moving of the motor will cause a coarse step of the motor. Although the design of the motor should prevent a tip crash, it is safer to reduce the tunneling current to zero before this action. Sometimes it is better not to use AutoVz, which makes the motor move after each image, but to do the same action manually from time to time using the keyboard shortcut <Ctrl-Home>.

Ensure that the feedback-loop gain (see figure 19, “Scan control window I,” page 50) is high enough to enable the tip to follow the surface corrugation. In practice start with the highest gain and reduce it while monitoring the tunneling current for slight tip crashes (spikes in the signal). Using a very low gain results in a constant height operation of the STM.

### Tip sharpening procedures while scanning (in situ)

The Z(V) spectroscopy function (see section 3.2.6.2 in the software chapter) is very useful for tip preparation. Right-click a few lines ahead of the currently scanned line in the current image. When the tip passes this point, a Z(V) scan will be performed using the parameters set in the Z(V) tab. Keep increasing the voltage range until the tip has changed and has become “sharp”. After this, move the tip away from this region, because the sharpening procedures may have caused some damage to the surface structure.

For the same purpose and with similar results you can also "abuse" the I(V) spectroscopy function to alter the tip (see section 3.2.6.2 in the software chapter) in the same fashion.

Tunneling at low voltages and high current is a common method to alter the tip shape. However provoking tip crashes does not work with the Aarhus STM like this so easily, since its stability is a crucial quality SPECS is proud to offer to the customer.

If you intend to induce tip crashes you can significantly reduce the feedback gain of the feedback loop (see "11" in figure 21, "Variables for tunnelling, Scan Control window I," page 54) and increase the current while scanning. SPECS does not recommend this method.

### The "Sound of Atoms"

We strongly recommend the use of headphones to monitor the tunnelling current. They provide a much better user-instrument-feedback than an oscilloscope. After some practice it is possible to judge from the sound whether the tip is sharp and whether the surface is flat. Terraces generate a sequence of clicks in the headphones. An area with large terraces has less clicks. Atoms can be heard as a beeping sound. If one zooms in, the sound frequency becomes lower (each atom makes a small click) until the atoms appear as a sequence of fast clicks. Even if the atoms are not visible in the image yet (because the scan range is too large or the number of pixels too small), they can be heard in the headphones.

### Are the pictures real?

With or without supplemental knowledge of the surface structure (by LEED for example), one is sometimes unsure if the topography measured is real or contains artefacts. First of all the topography has to be reproducible by serveral subsequent scans. It has to be independent of the scan speed, and it should be possible to zoom in and out. Variation of the scan direction has to lead to rotated pictures. By matching those re-

quirements you can be almost sure that it is the real topograpy you are observing. As a strict rule however, the only exact proof is to rotate the sample and hence to observe a rotated picture.

In rare cases sharp protrusions (sharper than the STM tip) on the surface can act as tips themselves. This results in imaging of the tip itself rather than an imaging of the surface. If so, vary the scan range - on larger scales you may see several large protrusions all looking the same. Those may be images of your tip. Hence it doesn't make sense to zoom in. Try to sharpen your tip with the in situ methods described in this chapter.

If the surface is extremely rough (matte finish) and the tip is not sharp enough, the electron tunneling may occur at different points of the tip, depending on the topograpy of the surface. As a consequence you get more than one image projected into one displayed image. In practice this data is worthless. In such cases it is easier to obtain very small scale images - they may be free from artefacts. To get a sharp tip (on a mesoskopic scale) you can use electrochemical tip etch procedures or buy etched tips.

For regular but false structures (stripes for example) in the images, keep reading.

### Coupling of noise to the STM

Sometimes the STM works as a kind of oscilloscope detecting unwanted relative periodic movements of tip and sample during a scan. This results in stripes on your data. To find the source of that, you can use the FFT feature of the software as descibed in section 3.2.5 . It helps you to find resonance frequencies.

Because of the extreme sensivity of this measurement, you can see even the slightest swing of the cradle cause relative movements of the scanner and the cradle in a range of only a few picometers at a few Hz (see also specification data taken in the 125Hz range). If this contribution is very prominent you may have problems with low frequency building movements coupling to the STM. The problem is severe if subsequent lines of the images appear to be on different height (it typically takes half a second to do a line scan). Try out more damping of the cradle as described in section 1.3.4.

The two clamps holding the sample holder can be bended during insertion of sample holders and therefore loose its strength. Sometimes they act as a pickup for noise at a few hundred Hz. In this case you can hear this sound with the headphones (or see it in the FFT) if knocking at the vacuum chamber. Try to improve the clamping strength or contact SPECS for new clamps.

**Moving scanned region on the sample for large distance**

The scanner of the Aarhus STM has a very large scanning field of 1500nm. Within this region you can choose any center of the image. Furthermore, once left, it is possible to return to the same place. If you want to move the tip on a large scale, withdraw the tip by the motor and approach it again. In general you will center the scanning region outside the scanning area used before. If you do not insert the sample to the sample holder till the mechanical stop, you can choose a region, which is one or two mm away from the center of your sample holder.

### 1.6.5 Calibration of the STM

Piezo ceramics respond very quickly with an elongation being almost exactly proportional to the applied voltage. Due to its design the Aarhus STM shows this linear behavior in the whole scan region. The size of the scanned region depends on the voltage applied, the design of the scanner and material property, namely the piezoelectric effect. This property underlies ageing effects (depolarisation), because it is not a state of thermal equilibium. Depolarisation by parts is caused by appliance of both high temperature and high voltage (with negative polarity, in our case) at the same time. In practice baking the STM at 423K with no voltages applied is not harmful, but slightly changes the response behavior of the piezo ceramics. So calibration is inevitable, at least when operating the system for the first time. Before doing so, it is important to know about different sources of distortion, which can be avoided:

If you offset the scanning region, i.e. center the scanning region to a new place by doing a fast and large step, the tip does not stay exactly at this new place. It “creeps”

along the direction of the applied step for some seconds or even minutes, depending on the stepsize. This causes a potentially large distortion of the image. It is not possible to correct this creep in a precise way, so avoid taking data under such conditions.

Thermal drift is an other main source of distortion. If the STM and the cradle are at the same temperature, the thermal drift is very small (less than 5 Angstrom per minute). However at elevated temperatures this is the main source for image distortion especially when taking small pictures. In this case you can make use of the drift compensation feature of the SpecsProbeUSB data aquisition software. For taking image size-calibration-data use an STM stabilized for hours at room temperature (take care of pressure gauges and other heat sources).

Now you can start to calibrate the instrument. Use a standard sample as described above (HOPG is easiest), find out the cristallographic data of the surface from literature and take pictures of different size with atomic resolution. Store the pictures and level it with the software to get rid of distortion caused by an inclined sample surface. Furthermore this increases contrast of pictures. Now you can take those pictures as a measure for distances and angles for each scan size and scan direction. For practical reasons this procedure of counting atoms (on HOPG for example) works only for small-scale pictures (up to 15nm) and atomic resolution. Because the Si(111) 7x7 superstructure is comparatively large, you can use this in a range of up to 100nm for calibration. For even larger image sizes you can use artificial calibration samples like periodically etched silicon wafers.

If maximum accuracy in measuring distances is required, diffraction methods like LEED are superior to STM-measurements. However, they only work for strictly periodic patterns.

# Chapter 2 Temperature Controller

The VTC 20 temperature controller is used to heat the sample and the STM chassis. The sample is in direct thermal and electrical contact with the metal block serving as a heat reservoir. This metal block can be heated by passing a current through two Zener diodes mounted on the block. A current of up to 100 mA can be passed at a voltage of 70 V through each diode. The STM body can be heated with a separate Zener diode with a current of up to 40 mA at 70 V.

For measurements at room temperature neither the block nor the STM body is heated. The VTC 20 need not be installed or can stay switched off in this case. For measurements at elevated temperatures, the metal block (and thus the sample) is heated to the desired temperature while the STM body is not heated. To reduce possible drift, sample and STM could be heated to the same temperature.

**! Please check that channel 1 is for STM and channel 2 is for the cradle (metal block), pay attention not to mix up heater cable and thermocouple of different channels. Keep in mind: max. temperature for the STM is 400K! Extensive use at 400K can lead to depolarisation of the scanner. Please refer to section 1.4.4 for more information.**

For measurements below room temperature, the metal body is pre-cooled with the cooling piston and then released. The STM body is heated such that it has a temperature near room temperature to assure functionality of the approach motor.

The exact procedure for operating the STM instrument at different temperatures is described in section 1.4.4 . This section only explains the operation of the heating power supply VTC 20.

The VTC 20 provides two independently operating 70 V current sources. The first circuit (left side, T1) heats the STM body diode, the second circuit (right side, T2) heats the two “cradle” diodes (metal block housing the scanner). The operation of both circuits is the same. The controller allows you to choose a setpoint temperature that can be controlled automatically. Alternatively, the current can be adjusted manually. The temperature is displayed in mV output of the thermovoltage amplifier. This amplifier does a zero-point correction automatically, but it does not linearize the type K thermo voltage. The output in mV corresponds roughly to the temperature in Celsius x 10. For an exact temperature reading, the displayed thermovoltage has to be converted into a temperature using the diagram shown in figure 18 or with table 4, middle column.

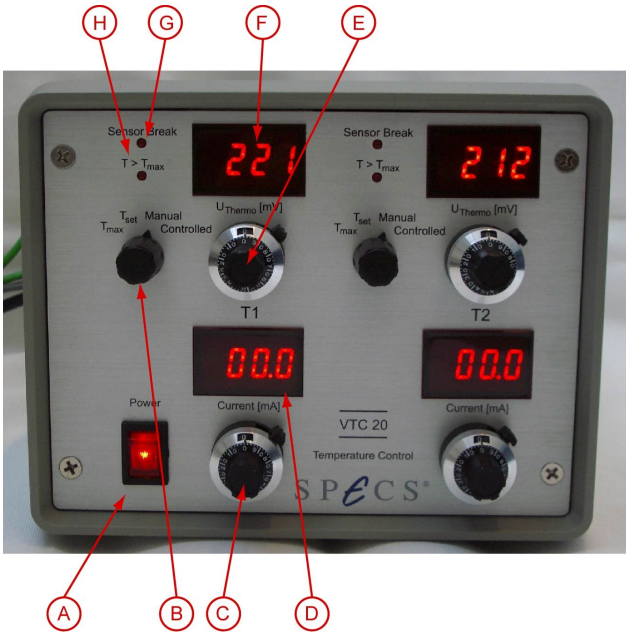



*Figure 16: Temperature Controller VTC 20*

The temperature controller VTC 20 has two independent channels. The tags are explained in the following text.

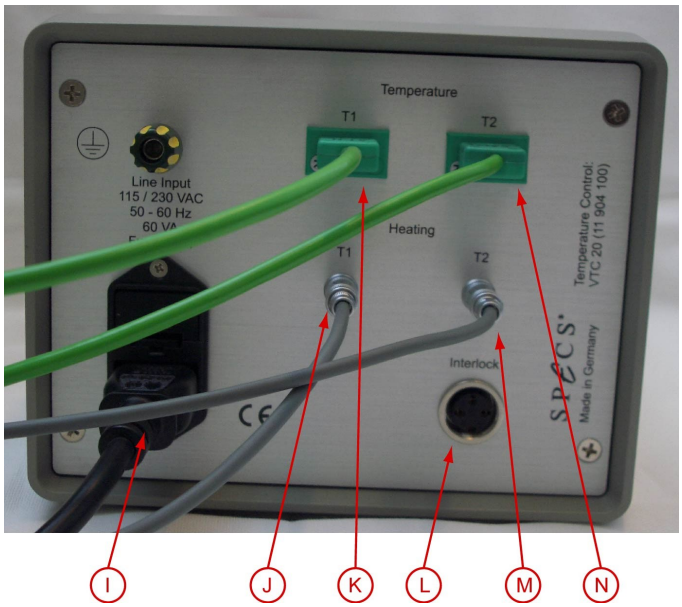



*Figure 17: Back View of the VTC 20*

The following list describes the front panel and back panel functions of the VTC 20:

A. Mains switch.

B. Four position selector:

   a. *Display Tmax*: the alarm thermovoltage is displayed in Figure 16, F. The alarm temperature is set internally. If one of the heating circuits reaches the alarm temperature, both circuits are shut off and the interlock at the back panel is set. To recover from an alarm, the mains has to be switched off.

   b. *Adjust Tset*: adjust the setpoint for the automatic temperature regulation. The setpoint thermovoltage is displayed in Figure 16, F. Change the setpoint with the potentiometer E. See d) below for regulated operation.

   c. *Manual operation:* The heating current can be adjusted with the potentiometer C. It is displayed in Figure 16, D.

   d. *Controlled operation:* The temperature controller regulates the temperature to the setpoint defined in b) with the potentiometer E. After heating to the desired temperature, the controller will hold this value precisely.

C. Ten-turn potentiometer for adjusting the heating current during manual operation. The STM body heater has a maximum current of 40 mA, the metal block heater gives a maximum of 2 x 100 mA.
D. Heating current display. For the T2 circuit the current through one diode only is displayed.
E. Ten-turn potentiometer for adjusting the setpoint thermo voltage in controlled operation.
F. Thermovoltage display. The displayed value in mV corresponds roughly to the temperature in Celsius multiplied by a factor of ten. To get a correct temperature value, the thermovoltage has to be converted to Kelvin using the diagram in figure 18 or table 4.
G. Sensor break alarm. If a thermocouple breaks, both heaters will switch off and the interlock at the back panel will be set. To recover from a sensor break, the controller has to be switched off.
H. Overtemperature alarm. If one of the heating circuits reaches the alarm temperature, both circuits are shut off and the interlock at the back panel is set. To recover from an alarm, the mains has to be switched off. The alarm temperature is set internally to 150°C.
I. Mains line, 110-230 V AC.
J. STM body heater ("STM").
K. STM body thermocouple ("STM").
L. Interlock output. Activates upon overtemperature or sensor break.
M. Metal body heater ("Cradle").
N. Metal body thermocouple ("Cradle").

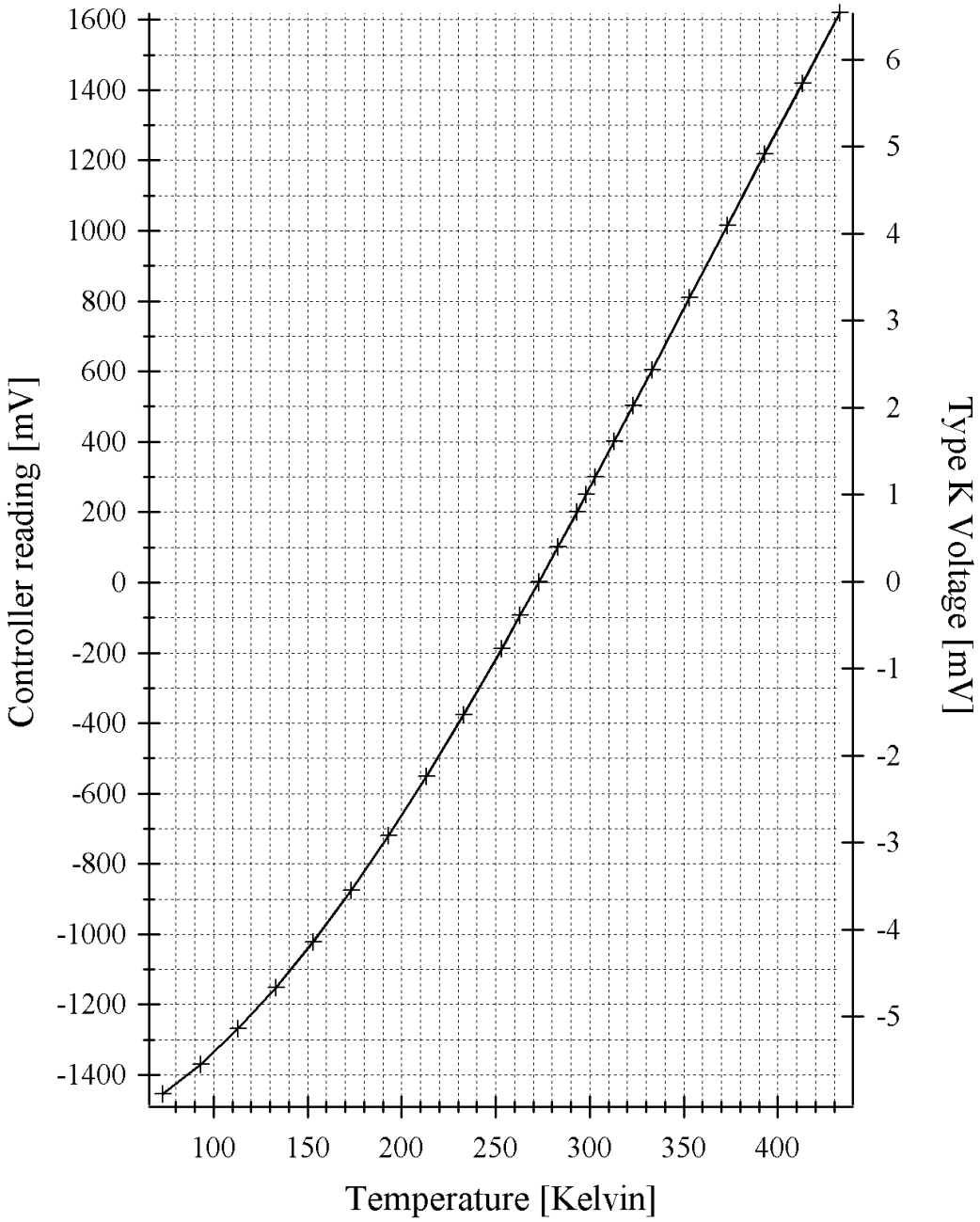



*Figure 18: VTC 20 Controller output as a function of temperature*

Additionally the thermovoltages for a Type K thermocouple are displayed (with reference junction at 0°C).

| Controller Reading | Type K Thermovoltage [mV] | Temperature [K] |
|---|---|---|
| -1454 | -5.891 | 73 |
| -1370 | -5.55 | 93 |
| -1269 | -5.141 | 113 |
| -1152 | -4.669 | 133 |
| -1021 | -4.138 | 153 |
| -876 | -3.553 | 173 |
| -719 | -2.92 | 193 |
| -552 | -2.243 | 213 |
| -375 | -1.527 | 233 |
| -189 | -0.777 | 253 |
| -94 | -0.392 | 263 |
| 2.7 | 0 | 273 |
| 101 | 0.397 | 283 |
| 200 | 0.789 | 293 |
| 250 | 1 | 298 |
| 300 | 1.203 | 303 |
| 401 | 1.611 | 313 |
| 503 | 2.022 | 323 |
| 605 | 2.436 | 333 |
| 810 | 3.266 | 353 |
| 1015 | 4.095 | 373 |
| 1219 | 4.919 | 393 |
| 1420 | 5.733 | 413 |
| 1620 | 6.539 | 433 |

b. VTC Controller output and corresponding type K thermovoltage for temperatures in Kelvin

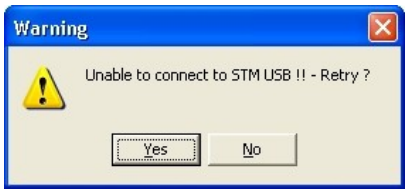
*Table 4: VTC 20 Controller Output Table*

# Chapter 3 SpecsProbeUSB Software

This chapter is intended to help you using the SpecsProbeUSB software as soon as possible. The first part gives an overview over the functions of the program. It uses screenshots to guide you through all the features of the program. The next part gives you a Quick Start introduction to using the microscope. If you follow the step by step description you will get your first images quickly. We recommend that you flip through the function overview with the screenshots first, in order to get a rough impression of what to expect when using the instrument. Then you will be ready to start taking images using the Quick Start introduction. After some experience with using the software, you will find the Function Overview useful as a reference.

## 3.1 Installation of SpecsProbeUSB

The SpecsProbeUSB software can be installed by simply running the file "SpecsProbeUSBinstall.bat", and can be uninstalled by running the file "SpecsProbeUSBuninstall.bat". Please refer as well to the "readme.txt" files on the software CD. After restarting the computer, the program and the USB interface are ready to use. An icon in the status bar of the Windows desktop will show if the SPC 260 electronics are available (connected via a USB cable and switched on). The program can be started in demo mode by answering "No" in the Retry prompt window.



## 3.2 Overview of Functionality

This section gives a quick overview over the software and its display and control functions. The first section contains some important functions that are not immediately accessible or obvious from the user interface. The other sections show screenshots of the SpecsProbeUSB software with tag numbers on the key control and display elements. The accompanying lists describe the functions of the numbered items in a short sentence. A more detailed description of the functions is given in the subsections of section 3.2.6, "Spectroscopy Tabs".

### 3.2.1 General Functions and Features

Program windows: SpecsProbeUSB consists of three main windows accessible by tabs: Scan Control window, Motor Control window and FFT Control window. The Scan Control window contains a section which is again divided into tabs. These tabs mainly contain the spectroscopy functions and some additional settings.

Figure 19 shows the Scan Control window as it typically appears during data acquisition.

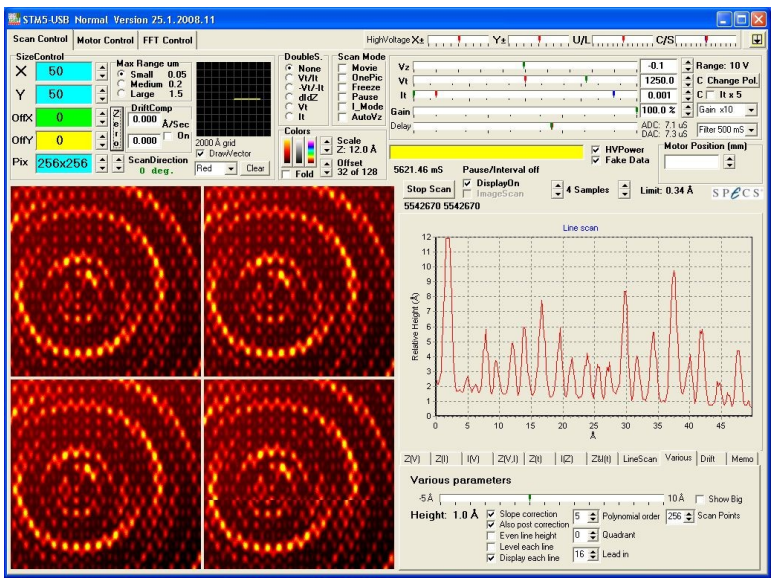



*Figure 19: Scan Control Window I*

#### 3.2.1.1 Data Buffer Concept

The images are acquired into data buffers. The buffers can hold 16 images of 64x64 pixels size or 8 images of 128x128 pixels size or 4 images of 256x256 pixels size or one image of 512x512 pixels size. The data will be acquired into the buffers sequentially and will overwrite the data that resides already in the buffers (special overwrite rules can be defined using “40” or “41”, see below).

Saving data: the image data can be saved in two different ways: the function key F5 saves the current image after the scan has been finished. A file name will be asked for when saving the first image. SHIFT F5 closes the current file and saves the images into a new file. Alternatively, an image can be saved by a SHIFT-left-click (same as F5) or a shift-right-click (same as Shift-F5) into a buffer. This will save the buffer contents after the current image has finished. This way, images that are in other buffers than the current one can also be saved.

#### 3.2.1.2 Centering the Scan on a Feature

When the cursor is a cross-hair, a left-click will immediately centre the scan area onto the x-y position where the click occurred. The left-click can be done in any data buffer, not only in the current image. A "Ctrl-click" in a buffer will change the crosshair icon into a hand icon. Dragging the image will reposition the scan area accordingly. Another Ctrl-click will make the crosshair icon reappear.

#### 3.2.1.3 Controlling the Scan Speed

Every data point takes a certain aquisition time. The maximum scan-speed results from the number of data points (image size) and the step-width during scanning as set by “7”. “8”is the number of samples per data point. Increasing the number of samples improves image quality and slows down the scan speed.

The scan speed is also affected by the analog-to-digital and digital-to-analog conversion speed. A slider (item 1, figure 21) enables you to set this conversion speed. By changing the slider position, the scan speed can be varied in a rather wide range.

#### 3.2.1.4 Spectroscopies

A right-click in any buffer will start a spectroscopy scan when the tip passes the point (no matter which buffer is active) where the click has occurred. Which kind of spectroscopy to be applied is determined by the active tab in the spectroscopy part of the Scan Control window. E.g. a Z(V) (height versus tunnelling voltage) scan will be performed if the foremost tab is the Z(V) tab.

#### 3.2.1.5 In situ Tip Preparation

The Z(V) spectroscopy function is useful for tip preparation. Perform a right-click a few lines ahead of the currently scanned line in the current image. When the tip passes this point, a Z(V) scan will be performed using the parameters set in the Z(V) tab. Keep increasing the voltage range until the tip has changed and is “sharp”. Similarly, you can use I(V) curves for preparing the tip.

#### 3.2.1.6 Controlling SpecsProbeUSB using the Keyboard

Most of the functions (and some additional ones) of SpecsProbeUSB are also available via the keyboard. Advanced users typically run the microscope using the keyboard. A list of keyboard functions is given in Section 3.4.

#### 3.2.1.7 Atomic Resolution Images

Atomically resolved data is best measured using the "Small" mode for the x-y amplifiers (see “52”) and an AD conversion range of less than 50 V (see “14”).

#### 3.2.1.8 Headphones

We strongly recommend the use of headphones to monitor the tunnelling current. They provide a much better user-instrument-feedback than an oscilloscope. After some practice it is possible to judge from the sound whether the tip is sharp and whether the surface is flat. Terraces generate a sequence of clicks in the headphones. An area with large terraces has less clicks. Atoms can be heard as a beeping sound. If one zooms in,

the sound frequency becomes lower (each atom makes a small click) until the atoms appear as a sequence of fast clicks. Even if the atoms are not visible in the image yet (because the scan range is too large or the number of pixels too small), they can be heard in the headphones.

#### 3.2.1.9 Moving the Approach Motor

The approach motor can be moved during tunnelling with the control field “10”. This is useful to keep the Z-Voltage Vz within the Z-AD converter range (see “14” and “24”). It is safe to move the motor as long as the center piezo voltage of the motor C is within -180 and +180 V. This voltage is displayed beside the approach-motor position as well as on the voltage monitor. The motor will make a coarse step when C gets bigger than 195 V or smaller than -195 V; this is when the monitor LEDs for C are red. Before the coarse step occurs, the tip has to be withdrawn from the sample by turning the tunnelling current to 0.001 nA. Then the tip will not suffer if the motor makes a coarse step. After the coarse step occurred, the current can be increased again.

#### 3.2.1.10 Voltage Monitor

The top display in the sofware displays the high voltages of the STM with red and blue indicators. The center position is zero volts; left and right are minimum and maximum voltage.

*Figure 20: Voltage Monitor*

#### 3.2.1.11 Saving a Configuration

Upon exiting SpecsProbeUSB, some settings are stored in an .ini file. This file can be edited if necessary.

### 3.2.2 Scan Control Window I

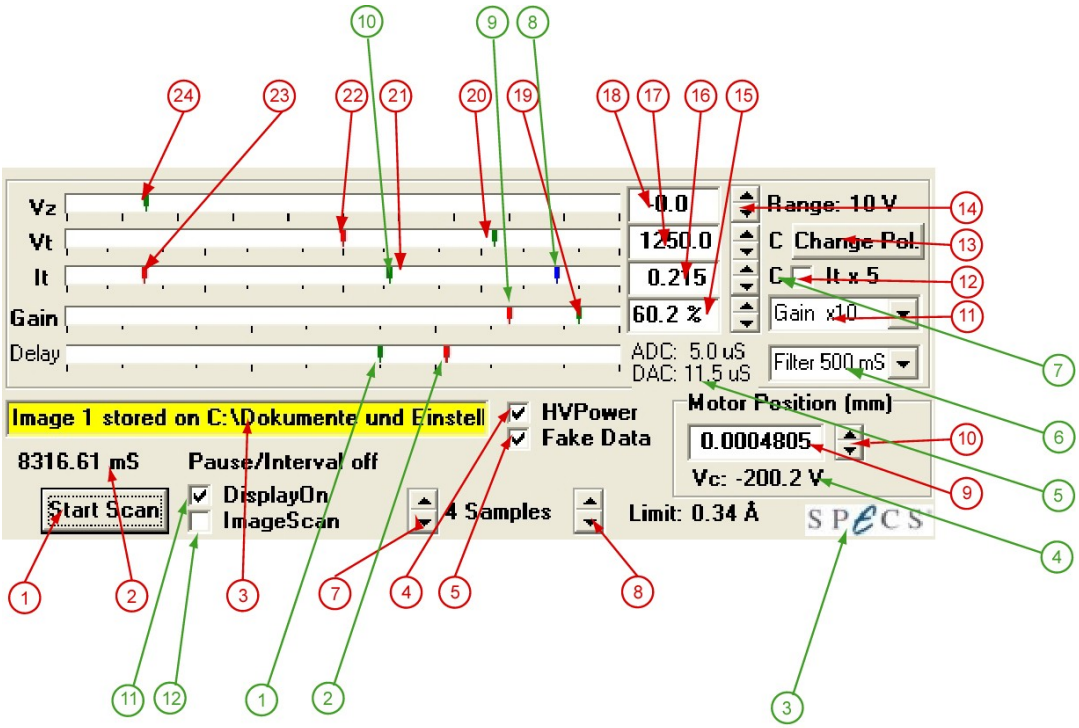



*Figure 21: Variables for Tunnelling, Scan Control Window I*

The screenshot above shows the most important variables for tunnelling. Coloured tag numbers refer to the text below. Please take note of the color codes when reading the descriptions. Codes in red represent functionality that has existed in legacy versions of SpecsProbe, and the green tags indicate new features in the software.

**Red** Tags

1. Start or Stop scanning.
2. Acquisition time for last image.
3. Message display area (see 93).
4. Switch high voltage on/off.
5. Display fake data.
6. Use fast AD conversion (useful only on slow PCs).
7. Number of samples per data point.

8. Largest allowed step in x-direction.
9. Approach motor position in mm.
10. Move tip towards/away from sample using approach motor (upper arrow moves tip closer to sample).
11. Set feedback loop amplifier to 100 x amplification.
12. Set tunnel current preamplifier to 50nA mode.
13. Change polarity of tunnelling voltage (changes upon start of next image).
14. Change range of AD converter for Z-voltage Vz. Available ranges are 5, 10, 20, 50, and 135 V. Z-voltages higher or lower than the conversion range will saturate the converter.
15. Display of feedback loop gain in percent of maximum gain (0-100%). See “11” for switching maximum gain by a factor of 100.
16. Display of tunnelling current (0.001-10 nA or 0.001-50 nA). See “12” for switching maximum current by a factor of 5.
17. Display of tunnelling voltage (1-10000 mV).
18. Display of actual Z-voltage on scanner. Needs to be within AD conversion range (see “14”). Is only updated during scanning.
19. Slider for adjusting feedback loop gain for positive tunneling voltage. Use **9** for negative voltage. Drag with mouse or use up/down arrows. Changes will become effective immediately.
20. Slider for tunnelling voltage (for "green" parameter set, see “46”, “161-164”). Drag with mouse or use up/down arrows. Changes will become effective immediately.
21. Slider for tunnelling current (for "green" parameter set, see “46”, “161-164”). Drag with mouse or use up/down arrows. Changes will become effective immediately.
22. Slider for tunnelling voltage for "red" parameter set, see “46”, “161-164”; used in double scans and line scans. Drag with right mouse button.
23. Slider for tunnelling current for "red" parameter set, see “46”, “161-164”; used in double scans and line scans. Drag with right mouse button.
24. Slider displays Z-voltage value, same as “18”.

Green Tags

1. Slider for adjusting the ADC (Analog-to-Digital Converter) delay. Drag with the left mouse button (slider 2 is moved the same amount). Affects the scan speed[3].
2. Slider for adjusting the DAC (Digital-to-Analog Converter) delay. Drag with the right mouse button (slider 1 stays fixed). Affects scan speed[3].
3. Click on "SPECS" to enter expert mode. Password is "SPeCS". (Case sensitive!)
4. Display of centre voltage C. When negative, the tip motor is retracted and when positive, the motor is extended.
5. Display of ADC and DAC delays. Change with 1 and 2.
6. Set delay filter of the feedback loop: Use smaller values for faster scanning. Small values can lead to loop oscillations. Turn current to 0.001nA before switching.
7. Click on letter to change the stepsize of the arrows beside the letter. C = coarse, M = medium, F = fine, S = superfine. This way $I_t$ and $V_t$ can be set with higher precision than by dragging the sliders.
8. Indicator that displays the tunnelling current.
9. Slider for adjusting feedback loop gain for negative tunneling voltage. Use 19 for positive voltage. Drag with mouse or use up/down arrows. Changes will become effective immediately.
10. Is the same as 21.
11. If checked, then every line is displayed after it has been measured. If unchecked, the display is only updated when a full image has been completed. Affects the ultimate scan speed. Uncheck for maximum speed.
12. Enable "Image Scan" feature. See section 3.2.7. In Image Scan mode, "11" (green) has to be active.

3 Typically, ADC and DAC should have the same delay.

### 3.2.3 Scan Control Window II

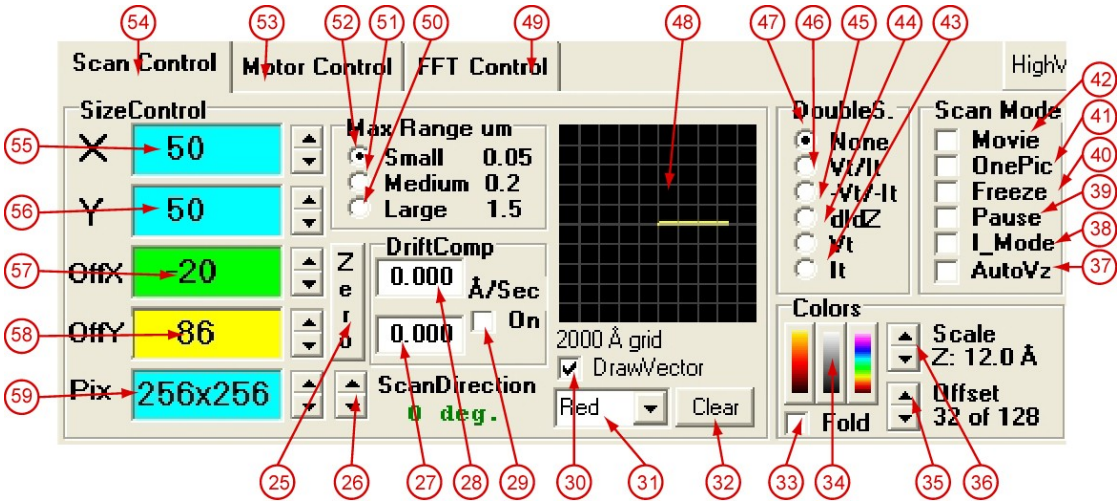



*Figure 22: Scan Control Window II*

This selection of the Scan control window displays various parameters like the scanned area, scan display and scan mode. Tag numbers refer to the text below.

25. Set X and Y offsets to Zero (use with care! Tip will immediately move to the centre of the scan area (X=Y=0 Angstroms)).
26. Change scan direction (azimuthally) in 1 degree steps. See “30” for using the mouse as alternative. Scan speed will be maximum at 0°, 90°, 180°, 270°. Intermediate orientations require some CPU time and will slow down scanning.
27. Drift compensation vector Y-value (Angstrom/sec). See “Drift Compensation” on page 68 for setting drift compensation parameters.
28. Drift compensation vector X-value (Angstrom/sec). See “Drift Compensation” on page 68 for setting drift compensation parameters.
29. Switch drift compensation on/off.
30. Display X-scan direction vector in scan history window (see “48”). The displayed vector can be dragged with the mouse to change the scan direction. See also “26”.
31. Set colour for tip trace in scan history window.
32. Clear the scan history window.

33. Use rolling colour table for image colour. Values higher than the maximum will be displayed after subtracting the scale maximum.
34. Choose colour table (red hot, grey or rainbow).
35. Use arrows to adjust colour scale offset.
36. Use arrows to adjust the colour scale range. The display shows either the z-voltage range that can be displayed with the colour scale or the current range that can be displayed.
37. Activate automatic Z-voltage centering. The approach motor will be set after each image such that the average Z-voltage is Zero.
38. Check for using constant height mode from next image on. The images will represent the tunnelling current. Uncheck to return to constant current mode.
39. Immediately pause scanning. Uncheck to resume.
40. Freeze current image. The current data buffer will not be overwritten by new data. Uncheck to make this data buffer available for data acquisition again.
41. Check for using only one data buffer. All images will be acquired into the current data buffer and will continuously overwrite the old data. Uncheck for using all data buffers.
42. Start movie acquisition. A file name will be asked for. Uncheck for stopping the movie acquisition.
43. Double image mode; line by line acquisition. Second image is a constant height image. Scaling of the image is done by F4/SF4.
44. Double image mode; line by line acquisition. Second image is an image of the tunnelling voltage (-25 mV < V < +25 mV). This voltage is the true gap voltage, corrected for the amplifier input impedance. It is measured in the preamplifier and read back by the electronics. This method is typically referred to as thermovoltage imaging. Scaling of the image is done by F4/SF4.
45. Double image mode; point by point acquisition. The second image shows the work function. It shows the change in tunnelling current when changing the tip height (open feedback loop). Height change is set in “154,” "Various". Scaling of the image is done by F4/SF4.
46. **a.** Double image mode; line by line acquisition. The first image uses the “green” parameter set. The second image uses the same current, but opposite polarity (see “20-23”). **b.** Double image mode; line by line acquisition. The first image uses the

"green" parameter set. The second image is acquired using the "red" parameter set for tunnelling voltage and current (see "20-23").

47. Single image mode. Only the "green" parameter set is active (see "20-23").
48. Scan history window. The trace of the tip is shown as a coloured line. Choose colour with "31". Clear display with "32". Area corresponds to a 2 micrometer x 2 micrometer square. Subdivisions are 2000 Å x 2000 Å squares. If "30" is checked, a vector will show the scan direction. The vector can be dragged to change scan direction.
49. Switch to the FFT Control window.
50. Switch X-Y amplifiers to "Large" mode. Maximum scan area is 1.5 x 1.5 micrometers in this mode.
51. Switch X-Y amplifiers to "Medium" mode. Maximum scan area is 0.2 x 0.2 micrometers in this mode.
52. Switch X-Y amplifiers to "Small" mode. Maximum scan area is 0.05 x 0.05 micrometers in this mode.
53. Switch to approach-motor window.
54. Switch to Scan Control window (this is the current window).
55. Change X-scan size using the arrows. Changes also apply for Y-scan size (see "56"). Value is displayed in Angstroms. Changes will become effective in the next image. Display is yellow if new range is not yet active, blue if the displayed range is the actual range in the current image.
56. Like "55" but for Y-scan size. Can be set independently from X-scan size.
57. X-offset in Angstroms. Use arrows or the offset knobs on the Monitor-Digipot to change the offset value. Changes will become effective immediately.
58. Like "57", but for Y-offset.
59. Image size in pixels. Change with arrows. Changes will become effective in the next image.

### 3.2.4 Motor Control Window

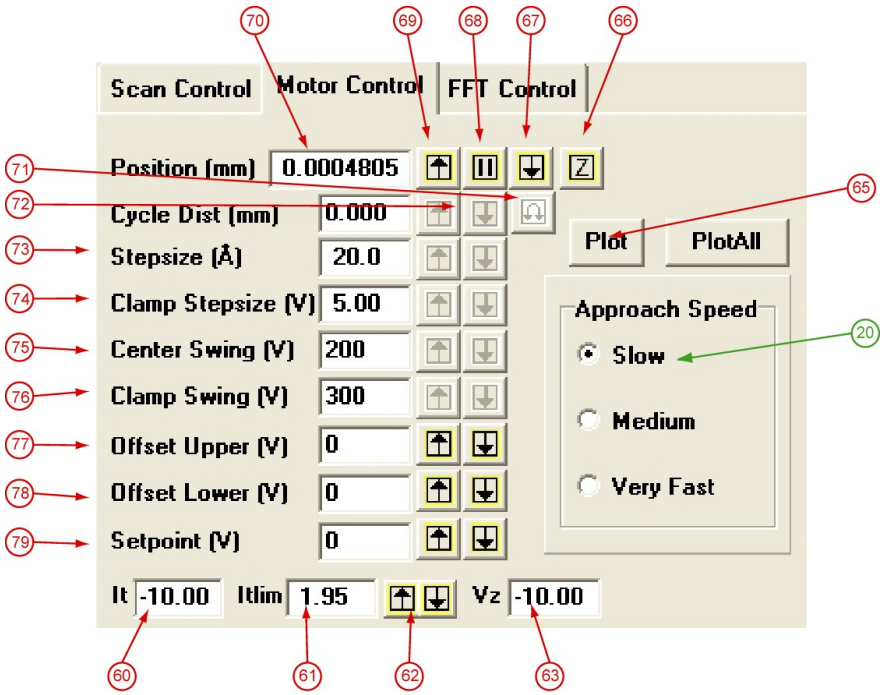



*Figure 23: Motor Control Window (Approach Motor Control)*

The window above shows the relevant parameters for the approach motor control. Tag numbers refer to the text below.

60. Actual tunnelling current.
61. Safety switch-off value for the tunnelling current. Approach will shut off immediately if the current is higher than this limit. This is only a safety feature. The approach algorithm uses a very small current value to sense the presence of the sample.
62. Change Itlim with these arrows.
63. Actual Z-voltage value.
64. Check for safe approach feature. This slows down the approach speed. Not necessary in new instruments.
65. Plots the voltages on the approach motor as well as the tunnelling current in the right window.

66. Define the current approach motor position to be the Zero position.
67. Withdraw the tip from the sample. Turn down the tunnelling current to 0.001nA in the Scan Control Window before withdrawing the tip.
68. Stop approach-motor.
69. Approach tip to the sample. The approach algorithm will stop when the tip is within tunnelling range. Make sure that all connectors are connected and that the cradle hangs free! Turn down the tunnelling current to 0.001 nA in the Scan Control window before approaching the tip.
70. Display of approach motor position. The displayed value is a relative value. The Zero point can be set with “66”. The actual motor travel can differ from the displayed value. Check the actual travel of the motor from time to time and estimate the deviation from the displayed travel. Refer to the STM 150 Aarhus hardware manual for fine tuning motor parameters.
71. Start cycling the motor. Only used at the factory.
72. Define cycle distance using the arrows. Only used at the factory and disabled in user mode. Change to Expert Mode (3) to enable.
73. Stepsize. Define the step size of the approach motor. This affects the approach speed. SPECS recommends a value of about 20 Angstroms. Larger values could lead to tip changes upon approach. Disabled in user mode. Change to Expert Mode (3) to enable.
74. Clamp Stepsize. Clamp electrode step size. Affects approach speed. SPECS recommends a value of about 5 V. Disabled in user mode. Change to Expert Mode (3) to enable.
75. Center Swing. Motor movement between clamp change sequences. Only used at the factory. Should be 200V. Disabled in user mode. Change to Expert Mode (3) to enable.
76. Clamp Swing. Gripping tightness of motor. Only used at the factory or when tuning the motor. Standard is 300V. Disabled in user mode. Change to Expert Mode (3) to enable.
77. Offset Upper. Adjusts motor up/down speed asymmetry by changing the tightness of upper bearing. Only used at the factory or when tuning the motor.
78. Offset Lower. Adjusts motor up/down speed asymmetry by changing the tightness of lower bearing. Only used at the factory or when tuning the motor.

79. Setpoint. Adjusts average gripping tightness of the approach motor. Needs to be changed for different motor temperatures. Refer to the specification data sheet delivered with your instrument for the factory preset value.

Green Tag

20. Select motor speed. Do standard approach with "slow". Approach a sputter shield with "medium" or "slow". User "Very Fast" only when there is no sample inserted.

### 3.2.5 FFT Control Window

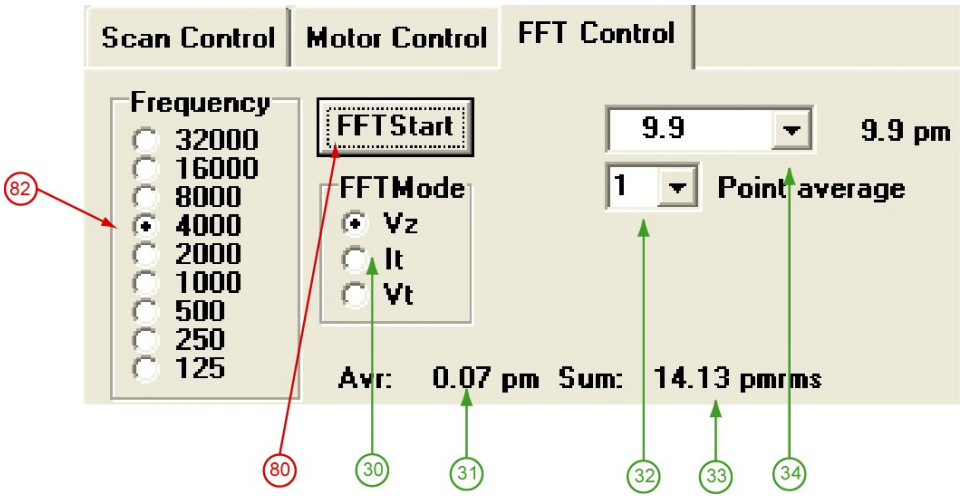



*Figure 24: The FFT Window*

80. Start Fast Fourier Transformation (FFT) of tunnelling current or tip height (according to the last scan mode, defined with “38”). The frequency spectrum is displayed in the window below.
82. Select the frequency range of the spectrum (e.g. 4000 will display a spectrum from 0-4000 Hz). Lower frequency ranges need longer data acquisition time.

Green Tags:

30. Selects which signal is measured, transformed, and displayed: Vz, It, or Vt.
31. Average measured value.
32. Number of averaged FFT-transforms before display.

33. Sum of all values in the displayed frequency range.
34. Select the full scale Z-voltage or tunnelling current for the frequency spectrum display.

### 3.2.6 Spectroscopy Tabs

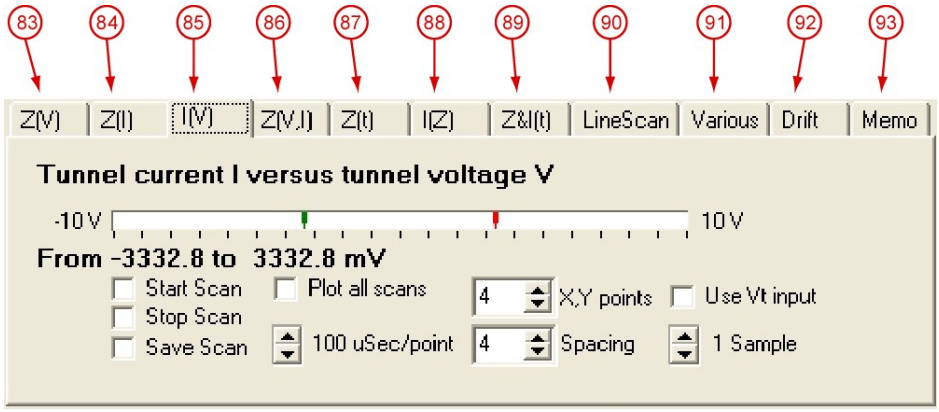



*Figure 25: Spectroscopy Tabs in the Scan Control Window*

83. Z(V) scan. Tip height versus tunnelling voltage. Tunnelling current from current image is used.
84. Z(I) scan. Tip height versus tunnelling current. Tunnelling voltage from current image is used.
85. I(V) scan. Tunnelling current versus tunnelling voltage with open feedback loop.
86. Z(I,V) scan. A new image is built up with I on the X-axis and V on the Y-axis. Z is displayed as colour value.
87. Z(t) scan. Tip height over time.
88. I(Z) scan. Tunnelling current over tip height with open feedback loop.
89. Z & I(t) scan. Tip height and tunnelling current over time
90. Line scan. Tip moves along a user defined line back and forth, once or twice, with different tunnelling parameters.
91. Various parameters. Contains parameters that affect the data display as well as some imaging modes.
92. Drift compensation. Contains parameters for the drift compensation feature.

93. Memo. Displays important system messages.

### 3.2.6.1 I(V) Scans

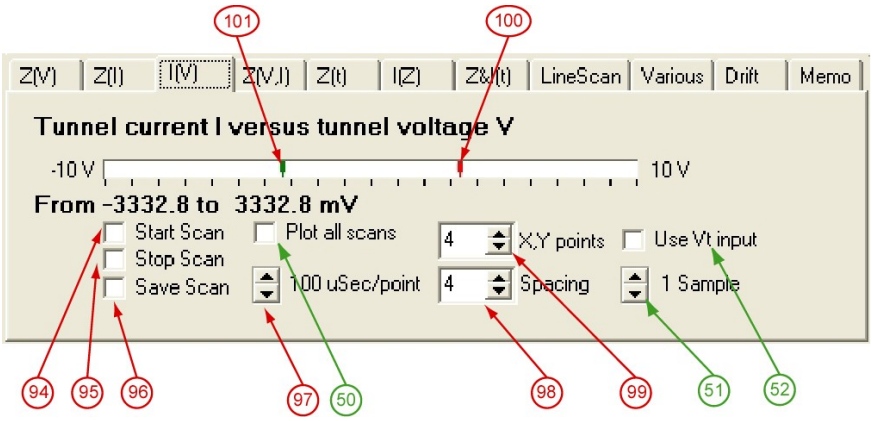



*Figure 26: I(V) Scans*

94. Start matrix of N x N I(V) scans in next image. Spectra will be acquired on a N x N grid defined in "98" and "99". One single scan can be started by right-clicking into any image buffer. The single I(V) scan will be done where the right-click occurred.
95. Abort scan.
96. Save scan; filename will be asked for.
97. Idle time between data points. This ensures that Vt (=:Ugap) has settled.
98. Spacing in pixels between points on the N x N grid (see "94"). Grid is centred on the image.
99. Number N of points on the N x N matrix (see "94").
100. End value for the voltage in the I(V) scan. End can be smaller than start.
101. Start value for the voltage in the I(V) scan. Start can be larger than end.

Green Tags:

50. Plots all scans if a matrix of I(V) curves is measured.
51. Number of samples per measured point.
52. Instead of the tunnelling current I(V), the voltage Vt input (=Ugap return) is used. This feature is only used when a lock-in amplifier is used for I(V) spectroscopy. SPECS recommends using the built-in I(V) spectroscopy. A lock-in is not necessary.

### 3.2.6.2 Z(V) Scans

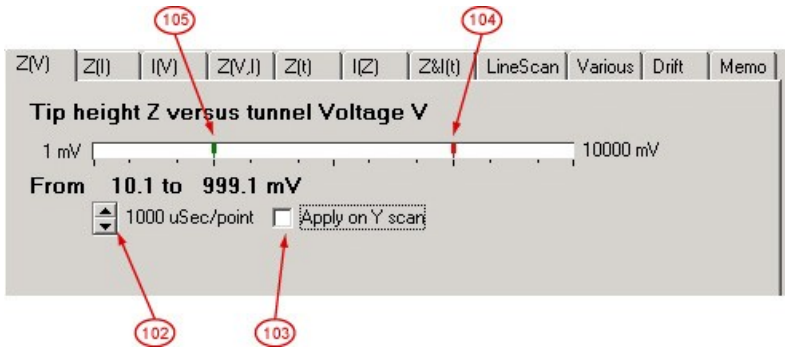



*Figure 27: Z(V) Scans*

102. Idle time between data points. This ensures that the tunnelling voltage Vt has settled.

103. Normally a Z(V) scan is started by right-clicking into any image buffer. The Z(V) scan will be performed where the right-click occurred. Alternatively, the tunnelling voltage V can be swept in parallel to the Y sweep in the image. This feature is activated with this checkbox. The next image is an image with non-constant tunnelling voltage. Vt will change from first to last line from Vstart to Vend.

104. End value for the voltage in the Z(V) scan. End can be smaller than start.

105. Start value for the voltage in the Z(V) scan. Start can be larger than end.

### 3.2.6.3 Various

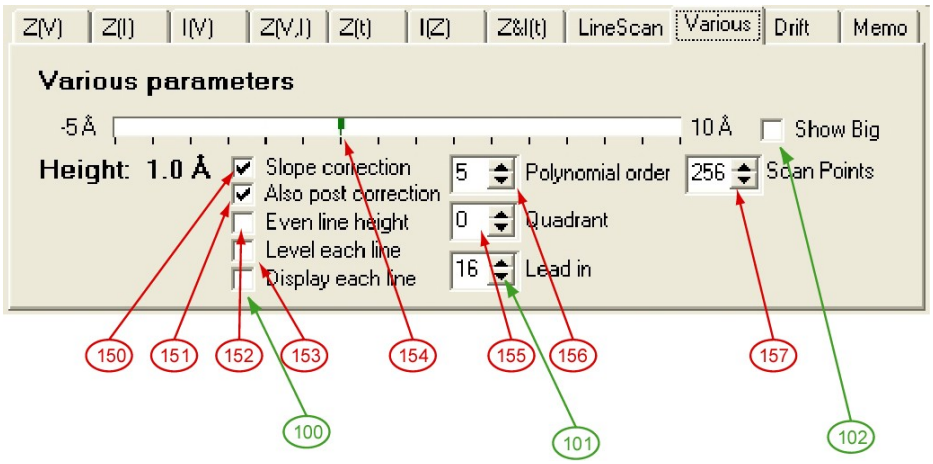



*Figure 28: The Various Tab*

150. Applies a slope correction to each measured image. Correction parameters are taken from the last acquired image. After image completion, correction parameters are re-calculated and applied to the next measured image. If “151” is checked, the recalculated parameters are applied to the current image as well. Slope correction is done with a 2 dimensional first order polynomial. Basis for the interpolation is a user defined image section defined in “155”. “150” has to be unchecked if “153” is checked. The correction only affects the display and not the original data.
151. Applies the recalculated slope correction to the image after the image acquisition has finished. See “150”. The correction only affects the display and not the original data.
152. After image acquisition has finished, each line gets an offset such that the average height of all lines in the image is the same. The correction only affects the display and not the original data.
153. After a line has finished, the line is slope corrected with a linear fit and an offset. The correction only affects the display and not the original data.
154. Slider sets the height variation for work function images. See “45”.
155. Selects the image quadrant from which the slope correction “150” and “151” is calculated. 0: full image; 1: upper right quarter; 2: upper left quarter; 3: lower left quarter; 4: lower right quarter; 5: centre quarter.
156. Sets the polynomial order for the fit to measured I(V) scans. Both this fit and it's derivative are displayed in the I(V) data window.
157. Defines the number of data points used in all 1 dimensional scans. This can be used to decrease the effects due to noise in these scans: increase the number of points and data averaging is done by the software afterwards.

Green Tags:

100. Displays each scan line. This will slow down scanning speed a bit. Uncheck for very fast scanning.
101. Defines the number of extra pixels measured at the bottom of the scan window. These points will not be saved and not be displayed. The reason for being a lead in is to give the scanner some time for its "creep" to reach a constant state before the actual imaging starts.
102. Shows an extra window with an enlarged view of the currently measured image.

### 3.2.6.4 Line Scan

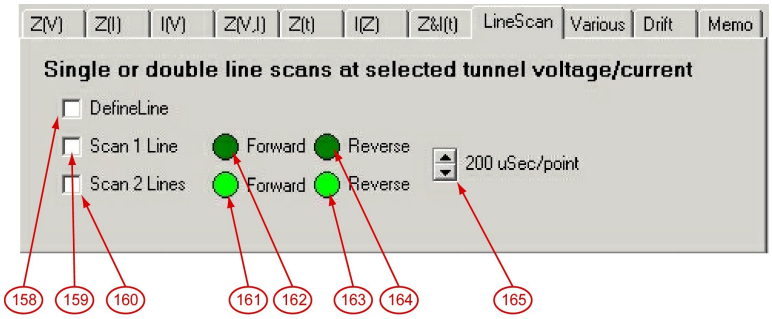



*Figure 29: The LineScan Tab*

158.Define a line by left-clicking in an image buffer for the start point of the line. Click the left button again to define a stop point.

159.Scan one line, forward and reverse. The scan is started immediately upon checking this box.

160.Scan two lines, forward and reverse, forward and reverse. The scans are started immediately upon checking this box.

161.Choose "green" or "red" set of tunnelling parameters for the line scan (see “20-23”). Indicator changes colour when you click on it. “161” chooses the red/green parameter set for the second line, forward direction.

162.As “161”, but for first line forward direction.

163.As “161”, but for second line reverse direction.

164.As “161”, but for first line reverse direction.

165.Time delay between points in the line scan.

### 3.2.6.5 Drift Compensation

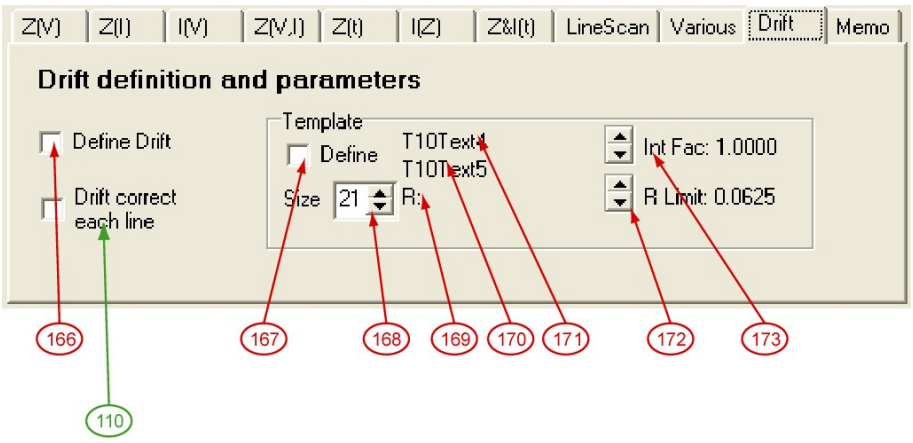



*Figure 30: The Drift Tab*

Drift compensation can be done in two ways. Either a prominent feature is defined in one image and in a later image. This defines a constant drift compensation vector that is applied as an image offset to compensate the drift. Alternatively, the drift compensation can be done automatically by template matching. This means that a certain feature can be defined as a "template". SpecsProbeUSB tries to find this feature in the next images with an image-recognition algorithm and tries to keep this feature at a constant position in the images. This requires a regulating-algorithm that can be tweaked with the integration factor (see “173”).

166. Initiate the drift definition. After this, left-click in an image buffer at a prominent feature. Wait for 10-100 seconds and left-click the feature again. The difference in position and time gives an X and Y correction written in the DriftComp fields “27” and “28” in the ScanControl window.
167. Define the position of a template by left-clicking in an image buffer.
168. Size of the template image area in pixels (always odd).
169. Quality factor of the template match (always < 1).
170. Current X and Y co-ordinate of the drift algorithm.
171. X and Y co-ordinate of the drift algorithm at the time of definition.

172.Limit of the quality factor R. If R becomes less than this value the template matching is stopped.

173.Integration factor of the drift correction feedback loop for template matching. 1 is normally a good value. At higher values the correction may start to oscillate.

Green tags:

110. Applies the drift correction algorithm after each line and not only after each frame.

### 3.2.6.6 Memo Function

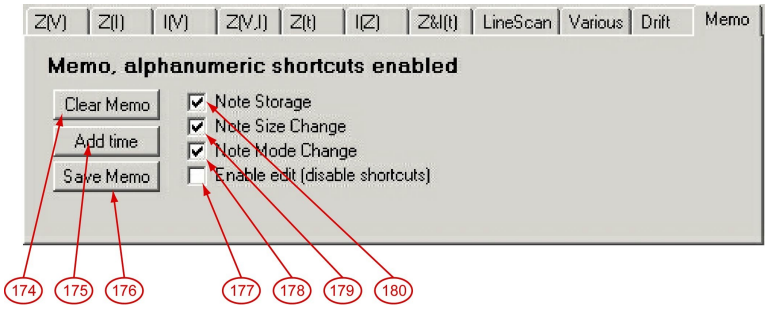



*Figure 31: The Memo Tab*

174.Clears the memo field.

175.Adds a time stamp to the memo field

176.Saves the memos to a file.

177.Enables the manual editing of the memo area. All program shortcuts are disabled.

178.All imaging mode changes are noted.

179.All size changes are noted.

180.Storing of images is noted with the full path name.

### 3.2.6.7 Z(I) Scans

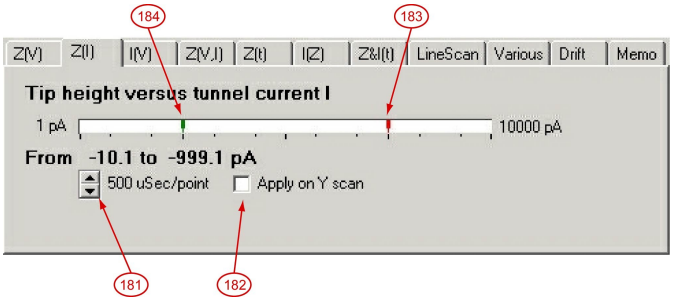



*Figure 32: The Z(I) Tab*

181. Idle time between data points. This ensures that the tunnelling current I has settled.
182. Normally a Z(I) scan is started by right-clicking into any image buffer. The Z(I) scan will be done where the right-click occurred. Alternatively, the tunnelling current I can be swept in parallel to the Y sweep in the image. This feature is activated with this checkbox. The next image is an image with non-constant tunnelling current. I will change from first to last line from $I_{start}$ to $I_{end}$.
183. End value for the current in the Z(I) scan. End can be smaller than start.
184. Start value for the current in the Z(I) scan. Start can be larger than end.

### 3.2.6.8 I(Z) Indentation Scans

This feature is used to measure the tunnel current versus tip height Z. It also can be used for tip indentations and may give information on work function and quantized conductance. It is armed by right-clicking on an image. The measurement is performed next time the tip passes this point. First the feedback loop is opened and the tip is moved to the start height at high speed. After a delay of 500µsec the scan is started and the tip is moved to the stop height in half the number of scan points (as defined in Various) with the default delay per point, “189”, of 100µsec. In this stop position the tip remains for 500µsec, as set by “188”. The number of points may be altered in the Various menu.

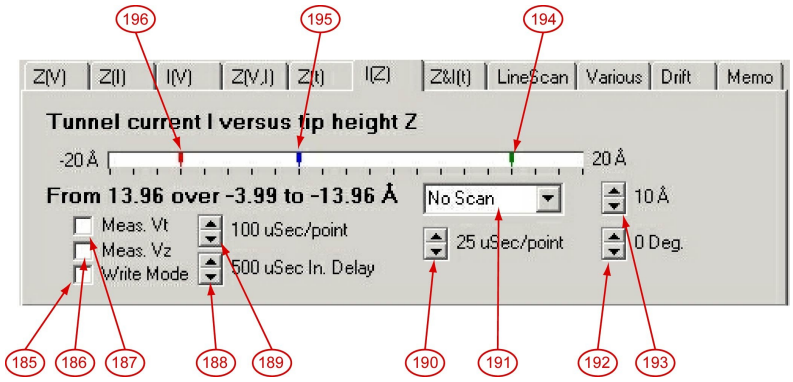



*Figure 33: The I(Z) Tab*

185.Write mode for I(Z)- scans, not yet fully implemented.

186.Records the actual Z-voltage.

187.Records the tunnel voltage to obtain tunnel conductivity.

188.Rest time in stop position.

189.Delay per point for moving the tip from Start to Stop value of the scan.

190.Delay per point for time, longitudinal and transversal scans selected in “191”.

191.Measure tunnel current at any tip height on the way out either versus time or longitudinal or transversal position.

192.Orientation of the transversal scans selected in “191”.

193.Length of longitudinal and transversal scans selected in 2191”.

194.Start value of the scan, selected by left mouse button.

195.Scan height, selected by Shift+ left mouse button.

196.Stop value of the scan, selected by right mouse button.

### 3.2.6.9 Z(V,I) Scans

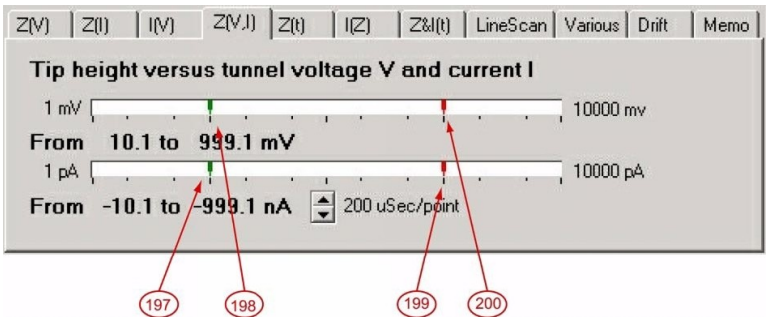



*Figure 34: The Z(V,I) Tab*

This is a 2 dimensional scan mode where the tip height is measured as a function of the tunnelling voltage and the tunnelling current. The tip stays at the same position during the measurement. The data is displayed in an image buffer and can be saved like an image.

197.Start value for the tunnelling current in the Z(V,I) scan. Start can be larger than end.

198.Start value for the tunnelling voltage in the Z(V,I) scan. Start can be larger than end.

199.End value for the tunnelling current in the Z(V,I) scan. End can be smaller than start.

200.End value for the tunnelling voltage in the Z(V,I) scan. End can be smaller than start.

### 3.2.6.10 Z(t) Scans

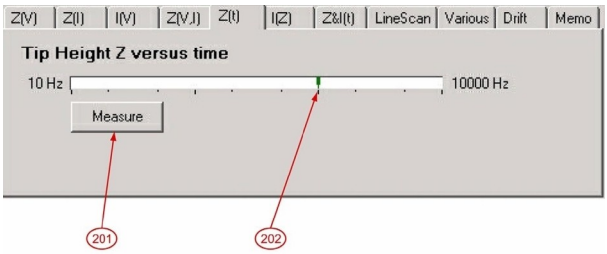



*Figure 35: The Z(t) Tab*

201.Start the measurement of Z versus time.

202.Define the highest frequency that can be identified in the measured data. This simply affects the span for taking the Z(t) data.

#### 3.2.6.11 Z & I(t) Scans

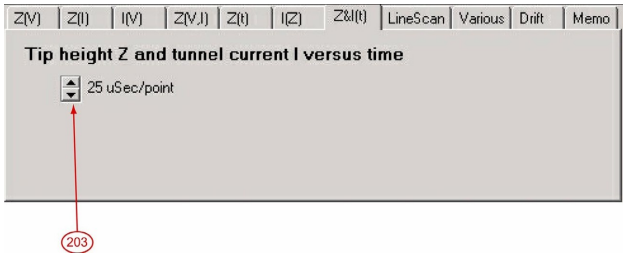



*Figure 36: The Z & I(t) Tab*

203. Sets the idle time between measured points in the Z & I(t) data.

### 3.2.7 Image Scan

The Image Scan tickbox (see (12) on page 54) activates/deactivates the Image Scan feature. Image Scan allows especially fast scanning. When image scan is unchecked, the scanning is done by the SpecsProbeUSB program. Each digital step in each ramp is sent to the SPC 260 via USB. When image scan is checked, the STM program requests a full image from the SPC 260 in an FPGA program, making extremely fast scans possible.

During each scan, the program will not respond until one scan is finished. The program will provide a warning that responsiveness might be slow if the scan speed is slow.

During Image Scan, spectroscopies are deactivated. Most other program funtions are still active however. The spectroscopy tab changes according to Figure 37.

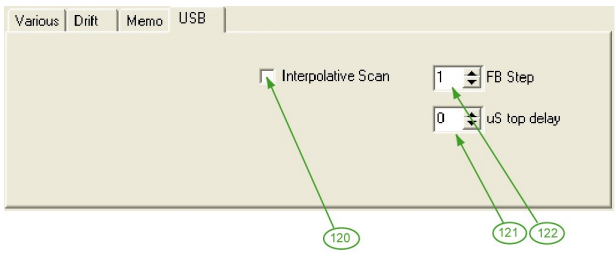



*Figure 37: The USB Tab in Image Scan Mode*

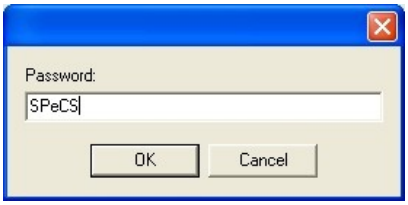
The new tab "USB" allows you to set some details of the Image Scan algorithm:

Green Tags:

120. When activated, only each even line is measured while each odd line is simply a duplicate of the preceding even line. This results in a doubled scan speed because the number of measured lines has just been halved.
121. ### not yet written
122. ### not yet written

### 3.2.8 Expert Mode

When clicking on (3) in the main scan window (see page 54), a dialog will ask for a password:




Typing in "SPeCS" will enable the Expert Mode of the software. To disable Expert Mode, click on (3) again and simply click OK (no password).

In Expert Mode, the following new features are available: A fully enabled Motor Control window, a USB details Window, and a Lithography window.

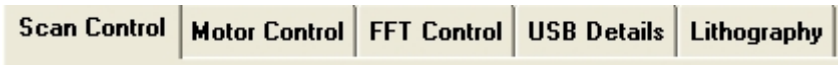



*Figure 38: Available Tabs in Expert Mode*

#### 3.2.8.1 Motor Control Window in Expert Mode

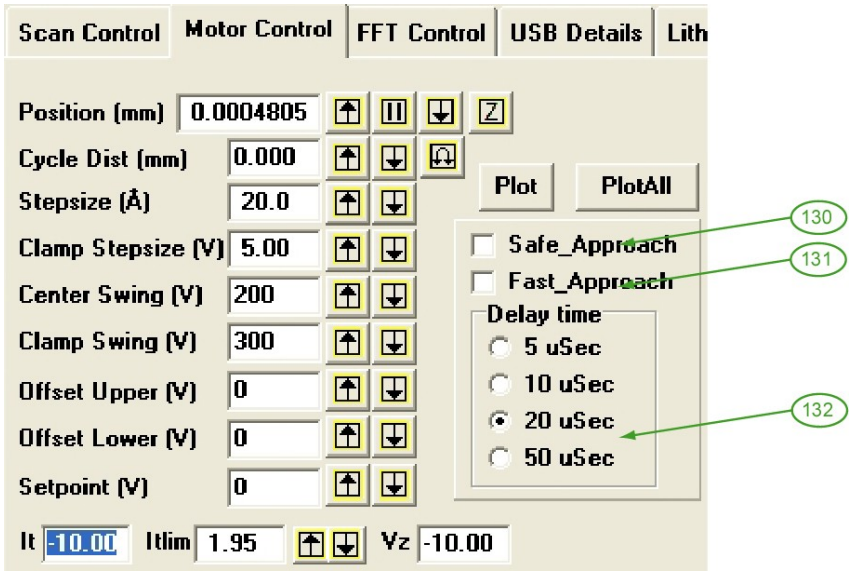



*Figure 39: Motor Control Window in Expert Mode*

In Expert Mode, the Motor Control Window allows access to all motor parameters:

Green tags:

130. Safe_Approach withdraws the centre Motor voltage a few % before a clamp change starts. Typically Safe_Approach does not have to be activated.
131. Fast_Approach delegates the important parts of the approach algorithm from the program into the FPGA of the SPC 260. This enables faster approach speeds.
132. The delay here influences the wait times in the approach algorithm. The typical value is 10µs. Note that smaller values can lead to tip crashes.

#### 3.2.8.2 USB Details Window

This window enables direct access to all functions of the SPC 260 electronics and the SpecsProbeUSB program. This panel should only be used with the support and guidance of SPECS Support Personnel.

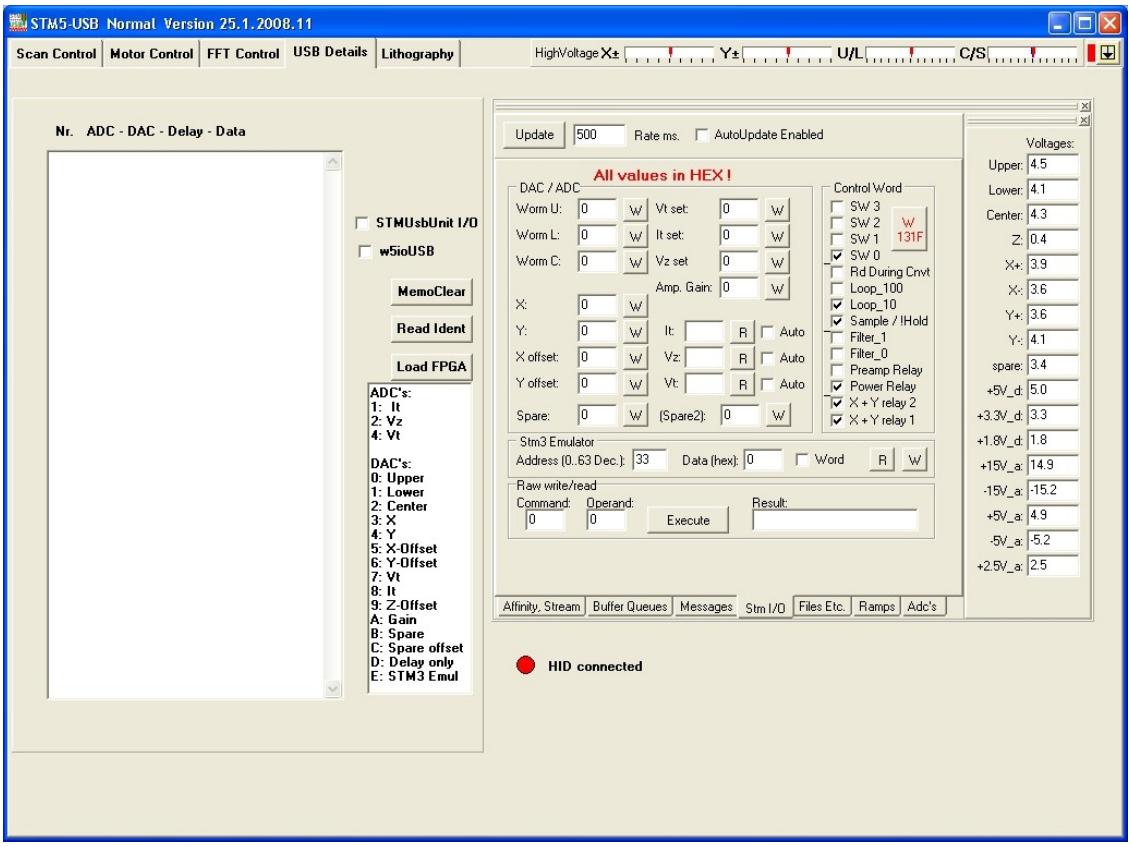



*Figure 40: The USB Details Window. Use only with the help of SPECS Personnel!*

### 3.2.8.3 Lithography Window

This window controls the lithography features of the software. Contact SPECS for further details (support@specs.de).

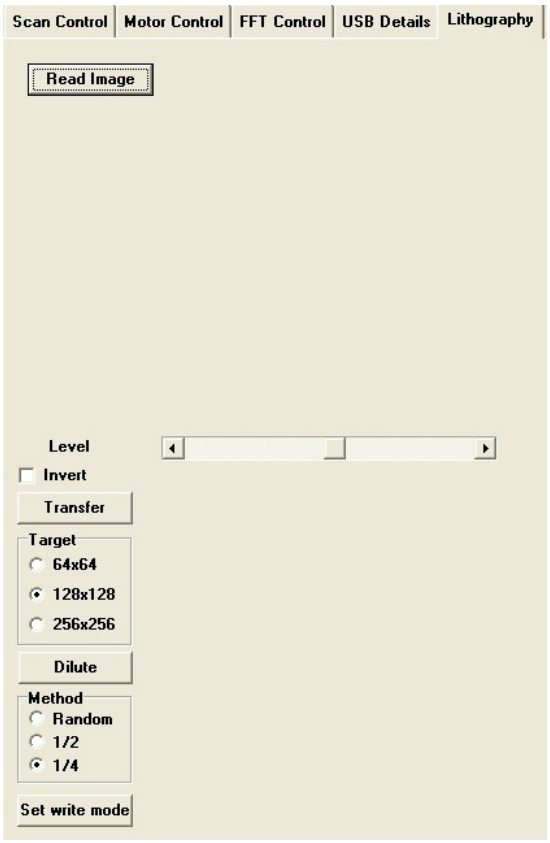



*Figure 41: Lithography Window*

## 3.3 Quick Start

This section serves as a guide to taking the first images with the STM 150 Aarhus and the SpecsProbeUSB software.

1. Insert a sharp tip in the tip holder. Make sure that the approach motor is completely withdrawn before installation of the instrument. Refer to sections 1.3 “Installation of the STM 150” and 1.4 “Running the STM 150” for inserting the tip and withdrawing the Motor Control.
2. Mount the STM and make sure that it is installed horizontally. Use a level to check this.
3. Do not connect any cables to the STM yet, or make sure that the power supply is switched off.
4. Pump down the chamber to a pressure better than $5e^{-5}$ mbar. When the pressure is within the so-called Corona-region (about 100 mbar to $1e^{-3}$ mbar), it is mandatory that no voltages are present on the STM! The instrument will be otherwise destroyed.
5. Alternatively, the STM can be used in air. To do so, mount the STM in its transport case and put it on a stable table.
6. Connect all cables to the STM and the power supplies SPC 260 and VTC 20.
7. Switch on the power supplies and start the SpecsProbeUSB software.
8. The program will display the system number of the electronics:

*Figure 42: The System Number*

9. Confirm that you are ready for switching on the high voltage:

*Figure 43: Ready for High Voltages. Is the Pressure OK?*

10. Enter the type of your STM. "1" is the standard (typical) value for the standard length scanner. Enter "2" if you have purchased the custom, short-length scanner.

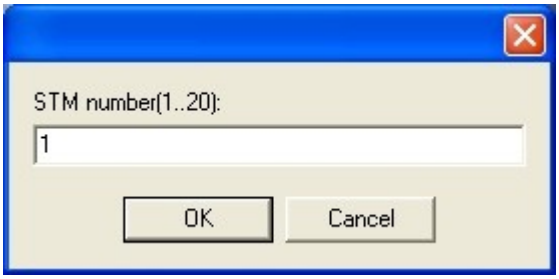



*Figure 44: STM Number*

11. SpecsProbeUSB will then display a number that characterizes the computer speed:

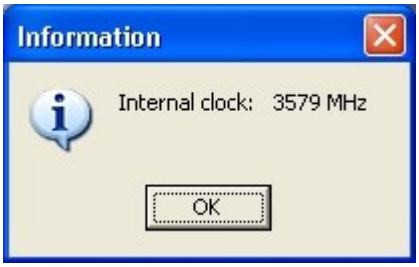



*Figure 45: The Internal Clock Speed*

12. Make sure that the tip is completely withdrawn. Insert a sample into the STM.
13. Unlock the STM by withdrawing the cooling piston completely. Now the STM should swing freely in its springs. If it touches the support frame, you need to readjust it by rotating the STM flange.
14. Go to the InchWorm Control window (see “53”).

15. Enter a setpoint value in the field “79”. You find the correct setpoint values for your instrument in the certification report (included in the shipment from SPECS). The setpoint effects the speed of the approach motor.
16. Double check that the STM hangs free. If it is locked by the cooling piston, the tunnelling voltage is shorted and the tip would crash into the sample upon approach.
17. Start the approach by hitting the button “69”. The approach steps are audible in the headphones (muted clicking) and are visible as spikes in the tunnelling current on the oscilloscope (if connected). The approach will stop automatically if the tip is close to the sample and tunnelling starts.
18. If the approach has stopped, click “69” again. The approach motor will not make another step, but the tip height will be readjusted, which takes about 20 seconds.
19. When the approach is finished, go back to the Scan Control window by clicking on the according tab.
20. Slowly increase the tunnelling current using the slider “21”.
21. Adjust the tunnelling voltage to an appropriate value (typically, 1200 mV is a good value) using the slider “20”.
22. Start the image acquisition with button “1”.
23. Change the image size with “55”. The number displayed are Angstroms.
24. Choose an appropriate size range with “50-52”.
25. Choose an appropriate gain by sliding “19” or switching to gain "x100" with “11”.
26. Change the Z-AD converter range with “14”. The actual Z-voltage displayed in “24” must be within the chosen voltage range or else the image will be saturated.
27. Adjust the image colour scaling with “36”.
28. Switch on line-by-line slope correction with “153”.
29. Save the current image with F5. A file name will be asked for when saving the first image. The next saved images will be stored in the same file in the mul format (multi image format). Save any image buffer with Shift-left-click into the buffer.
30. Change the number of image pixels with “59”.
31. Change the tip speed with “7” and/or “8”, or sliding "1".
32. Stop the image acquisition with “1”.
33. Reduce the tunnelling current to 0.001 nA. This is mandatory before withdrawing the tip or else a tip crash can occur during withdrawal of the approach motor.
34. Change to the InchWorm Control window by hitting “54”.

35. Click on "67" to withdraw the approach motor. Pull the tip back at least 2 mm before removing the sample from the STM. Stop the movement of the motor with "68". The sample must always be the highest point of the sample holder. If the sample has an odd shape, then remove the tip further than 2 mm.
36. Go back to the Scan Control page and quit the program.

## 3.4 Shortcut List to SpecsProbeUSB

Many features and functions in the SpecsProbeUSB software can be controlled via shortcuts. This makes working with the software very comfortable and easy. The functions are listed below.

### Keyboard Shortcuts to Functions in the Program SpecsProbeUSB

| | |
|---|---|
| F1 | Increase the number of pixels in X and Y by a factor of 2 |
| Shift-F1 | Decrease the number of pixels in X and Y by a factorof2 |
| F2 | Increase the size of the scanned area in X and Y. |
| Shift-F2 | Decrease the size of the scanned area in X and Y. |
| F3 | Increase the scanned size in Y only. |
| Shift-F3 | Decrease the scanned size in Y only. |
| F5<br>Shift-LeftClick | Quick save of present picture. As Shift-F5 but title is not asked for |
| Shift F5,<br>Shift-RightClick | Save present picture on present multifile. If no multifile has been specified yet, you will be prompted for a filename. The extension .MUL will be added to the filename. |
| Ctrl-click | Switch cursor from crosshairs to hand tool. With the hand icon, image offset can be controlled by clicking and dragging any image. |
| F7 | Take one Piezo Motor step up. |
| Shift-F7 | Take one Piezo Motor step down. |
| Alt-F7 | Start automatic Z adjustment. |
| Ctrl-F7 | Stop automatic Z adjustment. |
| F8 | Increase tunnel current by a factor of 1.2 |
| Shift-F8 | Decrease tunnel current by a factor of 1.2 |
| F9 | Increase tunnel voltage by a factor of 1.2, max is 10 V. |
| Shift-F9 | Decrease tunnel voltage by a factor of 1.2, min is 0.2 mV. |

| | |
|---|---|
| Alt-F9 | Change polarity after this scan. |
| Ctrl-F9 | Perform tip clean by applying a tunnel voltage of 5 V for 0.1 Sec. |
| F10 | Exit the program. |
| Right arrow | Move scan 1/6 of the width to the right. |
| Left arrow | Move scan 1/6 of the width to the left. |
| Up arrow | Move scan 1/6 of the height up. |
| Down arrow | Move scan 1/6 of the height down. |
| END | Halt the scan, the mouse is still active. Restart if halted. |
| ESC | Restart present scan. |
| PgUp | Start drift compensation. |
| PgDn | Stop drift compensation. |
| Shift-j | Increase the max. current to 50 nA. |
| j | Decrease the max. current to 10 nA. See J. |

### Function buttons in the approach motor menu.

| | |
|---|---|
| F1 / Shift-F1 | Run Up / Down. |
| F2 / Shift-F2 | Increase/Decrease the step size. |
| F3 / Shift-F3 | Increase/Decrease the clamp step size |
| F4 / Shift-F4 | Step Up/Down |
| F5 | Stop Motor. |
| Shift-F5 | Start Cycling . |
| F6 / Shift-F6 | Cycle distance Up/Down |
| F7 | Clear position. |
| F9 | Show histogram. |
| F10 | Exit from the Piezo Motor Menu. |
| F11 / Shift-F11 | Increase/Decrease the current limit when searching tunneling. |

*Table 5: Keyboard Shortcuts to Common Functions*

# Chapter 4 SpecsFFT Analysis Software

## 4.1 Installation of SpecsFFT

The SpecsFFT software consists of one executable file: **"SpecsFFT"**

This data analysis software for .mul files software is simply installed by copying the file from the CD on the hard disk. After this, it can be started.

### 4.1.1 Functions of SpecsFFT

Please refer to the online help in SpecsFFT for a description of the available data analysis functions.

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SPECS®

# Health and Safety Declaration for used Vacuum Equipment and Components

The repair and/or service of vacuum equipment/components can only be carried out if a correctly completed declaration has been submitted for every component.

**1._____Description of components**

Type: _____Serial No:_____

2. **Reasons for return**_____

**3. Equipment condition**

**Has the equipment ever come into contact with the following (e.g. gases, liquids, evaporation products, sputtering products ...)**

| | Yes | No |
|---|---|---|
| • toxic substances? | ❑ | ❑ |
| • corrosive substances? | ❑ | ❑ |
| • microbiological substances (incl. sample material)? | ❑ | ❑ |
| • radioactive substances (incl. sample material)? | ❑ | ❑ |
| • ionising particles/radiation (α, β, γ, neutrons, ...)? | ❑ | ❑ |

| | Yes | No |
|---|---|---|
| **Is the equipment free from potentially harmful and hazardous substances?** | ❑ | ❑ |

**4. Decontamination Procedure**

Please list **all harmful substances, gases and by-products** which have come into contact with the vacuum equipment/component during the decontamination methode used.

| SUBSTANCE | DECONTAMINATION METHODE |
|---|---|
| | |
| | |

**5. Legally Binding Declaration**

Organisation: _____

Address: _____

Phone/Fax: _____

Name/Position: _____

I hereby declare that the information supplied on this form is complete and accurate.

**Date:_____Signature:_____Company stamp**