

Force Measurement in an SEM Using the Zyvex Force Characterization Package

By Kanzan “KZ” Inoue, Applications & Development Scientist
and Adam Hartman, Production Mechanical Engineer

Introduction

The Zyvex Force Characterization Package (FCP) provides a simple, plug-in upgrade to any Zyvex Nanomanipulator, allowing nano-scale force measurement capability.

By integrating a piezoresistive cantilever with 5 nanometer resolution positioners, and high precision electrical measuring hardware and software, the Zyvex FCP provides researchers with a unique and powerful tool to investigate the forces ranging from nanonewtons to millinewtons on nano and micro structures.

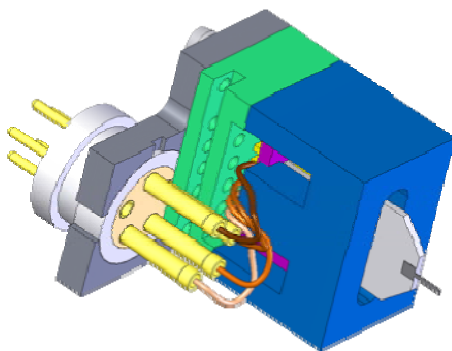


Figure 1. Drawing of the FCP End-Effector Assembly.

System Requirements

The FCP uses the following components to allow users to manipulate nanostructures and measure applied forces.

- Piezoresistive cantilevers.
- Zyvex FCP End-Effector Assembly, Part No. T10-C-2250-01.
- Zyvex Nanomanipulator, including the S100, sProber, dProber, and nProber product lines.
- High resolution scanning electron microscope (systems equipped with a turbo-molecular pump, TMP, are preferred).
- High precision voltage supply and characterization system, such as the Keithley Instruments 4200 Semiconductor Characterization System equipped with at least 4 Medium Power SMUs w/ Pre-Amps.

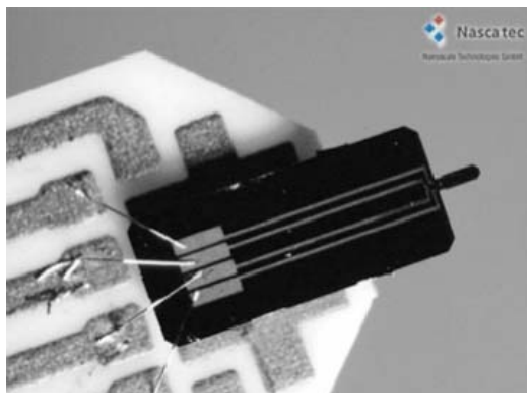


Figure 2. Close-up image of a piezoresistive cantilever.

Installing a Force Sensor Assembly

By integrating a commercially available piezoresistive cantilever assembly, the Zyvex FCP allows great flexibility in choosing the right force sensor for the desired measurement range. Custom cantilever dimensions and spring constants are also available, subject to pricing, availability, and lead time. The design of the FCP end-effector allows quick and easy exchange of force sensors without the need to mount, bond, or wirebond delicate cantilevers. When a new spring constant is desired, or when a cantilever is damaged, the old sensor assembly can be quickly unplugged from the End-Effector and a new one put in its place.

The steps to follow in order to install a force sensor assembly into the FCP end-effector are given below, with pictures of the procedure included after each step. Be sure to always wear clean, powder free, latex or nitrile gloves and to use clean, sturdy tweezers when handling any parts that will go into the SEM chamber. Placing a piece of clean, uncoated aluminum foil on a flat surface is recommended as a working surface.

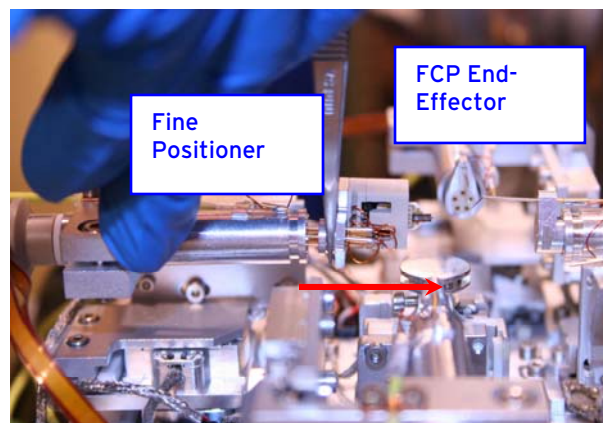
1. Remove the FCP End-Effector Assembly from the Nanomanipulator fine positioner.

The fine positioners are delicate and may be damaged by the forces involved in installing force sensors.

Remove the End-Effector using tweezers by placing the tips in the round channel at the back of the Assembly and pulling straight out, along the central axis of the fine positioner tube, while simultaneously holding the back of the positioner with the fingertips of your free hand.

Warning: Avoid rocking or twisting the End-Effector, as this may damage the fine positioner.

Set the End-Effector Assembly on a clean surface, taking care not to damage the cantilever of any previously installed force sensor.



- Step 1. Pull the FCP End-Effector Assembly straight out of the fine positioner.

2. If the End-Effector Assembly already has a force sensor assembly installed, it must be carefully removed in order to install a new sensor assembly. If no sensor is installed, skip to step 3.

Hold the End-Effector assembly with the fingertips of one hand and a clean pair of tweezers in the other hand. Approach the tips of the tweezers slowly toward the side of the force sensor ceramic printed circuit board (PCB), near the 45° cut corner and the mounted silicon chip.



- Step 2. Carefully remove the old force sensor.

Warning: Take caution to not touch the silicon chip or the wirebond wires with the tweezers.

Firmly grasp the ceramic PCB with the tweezers and pull the force sensor assembly straight out from the End-Effector Assembly.

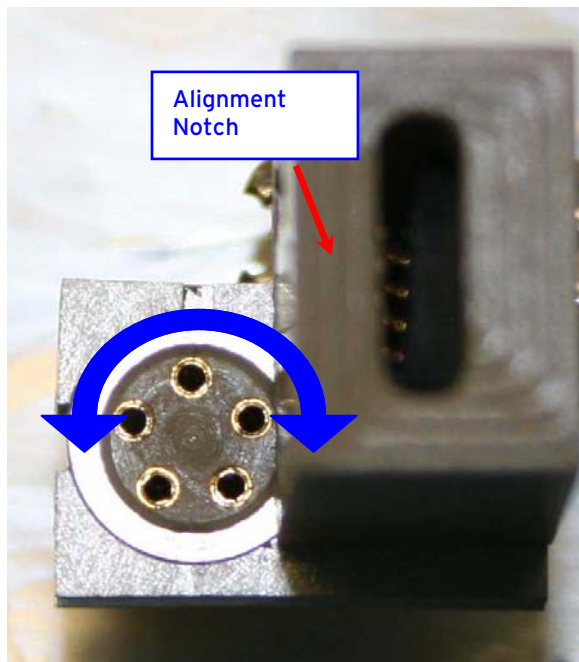
Carefully place the old sensor assembly on the adhesive strip inside the force sensor storage case, making sure to touch the back of the ceramic PCB to the adhesive before gently laying the rest of the PCB surface down flat.

Set the End-Effector Assembly on a clean surface.

3. Check that the alignment notch on the End-Effector Assembly aluminum bracket is aligned with the top hole in the round End-Effector Plug.

If the alignment notch is not aligned with the top hole of the plug, grasp the back of the plug firmly with the fingertips of one hand and the aluminum bracket in the fingertips of the other hand.

Twist the two parts until they are aligned.

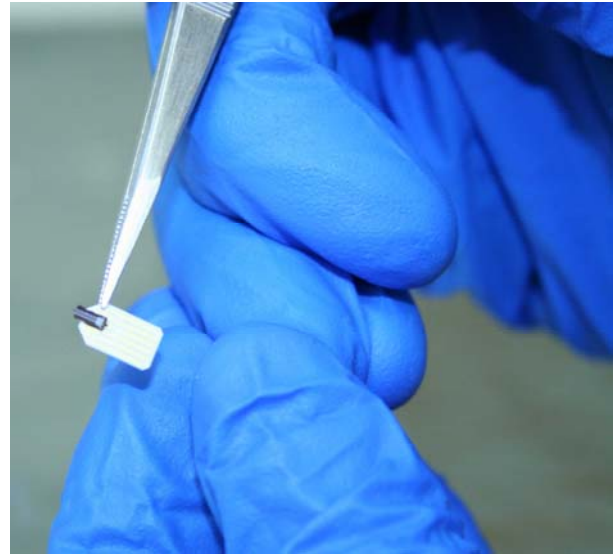


Step 3. Twist the plug and End-Effector assembly to align with notch.

4. Carefully remove a force sensor assembly from the adhesive strip in the storage case.

Approach the tips of the tweezers slowly toward the side of the force sensor ceramic PCB, near the 45° cut corner and the mounted silicon chip.

Warning: Take caution to not touch the silicon chip or the wirebond wires with the tweezers. The tweezers should be pointing perpendicular to the long axis of the force sensor assembly.



Step 4. Move the tweezers to hold the PCB near the front.

5. Pick up the End-Effector Assembly with the fingertips of your free hand so that the slot faces outward.

Gently insert the back end of the sensor PCB into the center of the slot. There should be a slight feeling of force as the spring fingers inside the slot are forced open by the ceramic PCB.

Warning: Take caution not to touch the bonded silicon chip or wirebond wires, as this may damage the force sensor. The PCB may twist slightly during insertion, and this is not a problem.



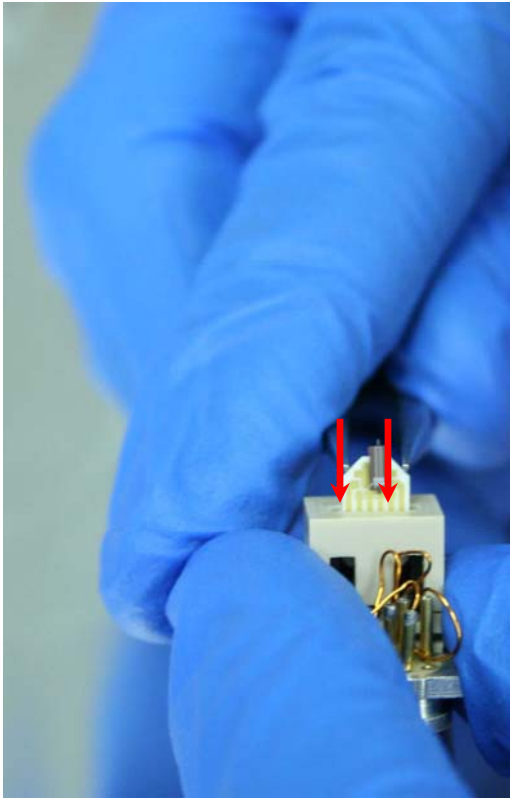
Step 5. Slowly insert the PCB into the End-Effector Assembly slot.

6. Change the position of the tweezer tips so that the tweezers are perpendicular to the ceramic PCB and the sides of the tips are pressed against the 45° cut corners.

Pressing against the corners, continue sliding the PCB all the way into the slot until it stops moving.

The PCB may appear slightly tilted forward or backward once inserted. This is not a problem.

Warning: Do not attempt to tilt the PCB in the slot, as this may damage the force sensor or the End-Effector spring contacts.



Step 6. Use the tweezer tips to push the ceramic PCB all the way into the slot.

7. Using the tweezers, carefully adjust the position of the sensor PCB in the slot to ensure that it is centered, side-to-side, in the opening.

Warning: Avoid contacting the bonded silicon chip or wirebond wires.



Step 7. Carefully adjust the position of the sensor to be centered in the slot.

8. Install any samples in the Nanomanipulator, making sure to secure the sample stub with the available set screw.

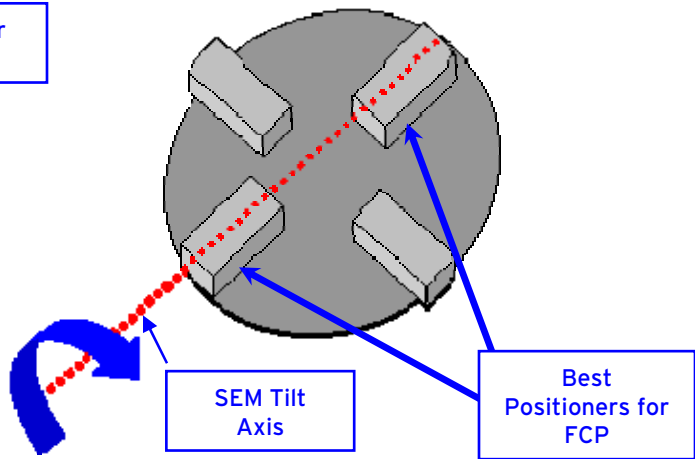
After installing the sample, the FCP can be installed. It is best to install the FCP on a positioner that is aligned with the tilt axis of the SEM stage, to allow any slight misalignment of the sensor assembly to be corrected through stage tilt.

Hold the back of the desired fine positioner with the finger tips of one hand and grip the back of the End-Effector Assembly with tweezers in the other hand.

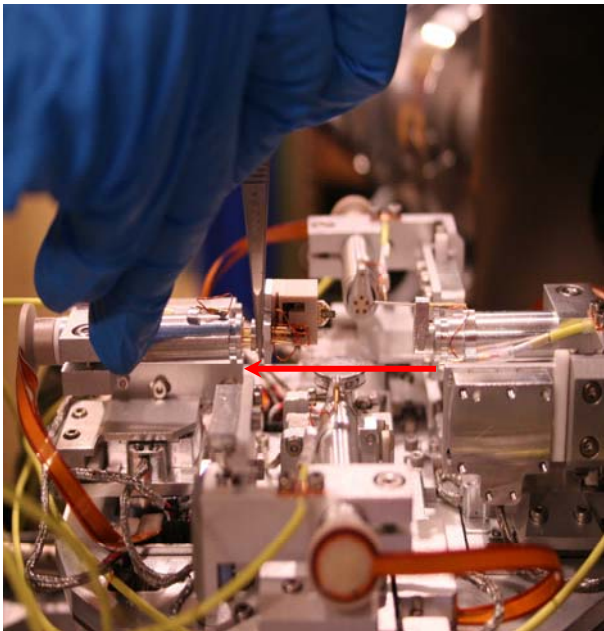
Carefully insert the pins at the back of the End-Effector Assembly straight into the sockets at the front of the fine positioner. Make sure that all five pins are lined up with the sockets in the positioner.

Warning: Use caution to avoid damaging the fragile cantilever on the front of the sensor, and do not use excessive force when inserting the pins into the positioner.

Generic 4-Positioner
Nanomanipulator



Step 8a. Install the FCP on a positioner aligned with the SEM stage tilt axis.



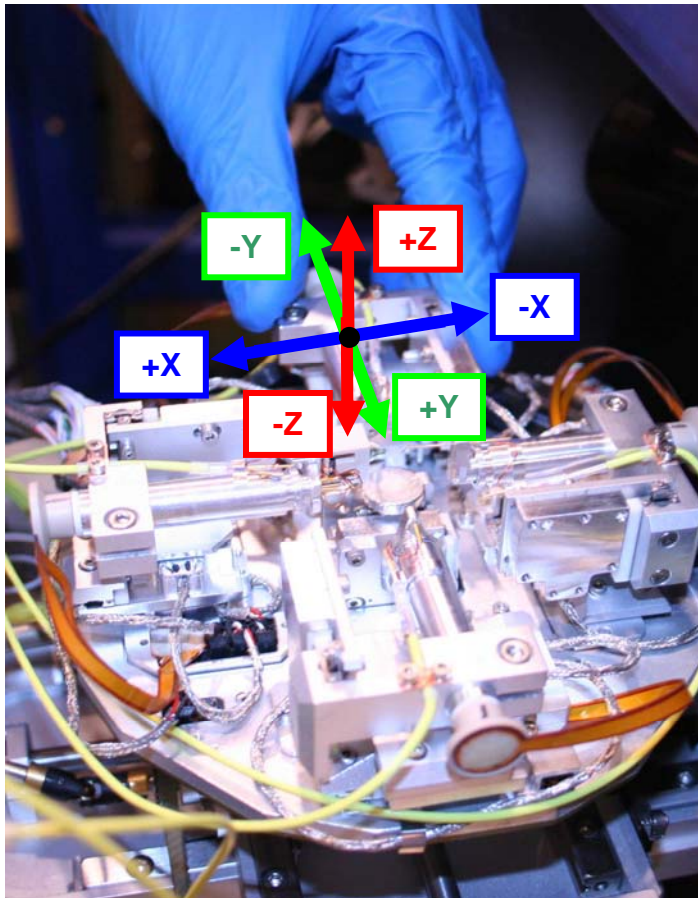
Step 8b. Gently insert the pins of the End-Effector Assembly into the fine positioner.

Once the End-Effector Assembly has been installed, move all positioners into the desired locations by gently holding the back of a positioner with the fingertips and adjusting its X, Y, and Z position.

Warning: Use caution to avoid crashing the sensor cantilever or the End-Effector Assembly into the sample, other probes, or other positioners, as this may damage the sensor or positioners.

Before closing the SEM chamber door, make sure that all positioners are low enough to clear the bottom of the column and any other SEM detectors or equipment.

Pump down the SEM as normal, running an Anti-Contamination Unit script if necessary.

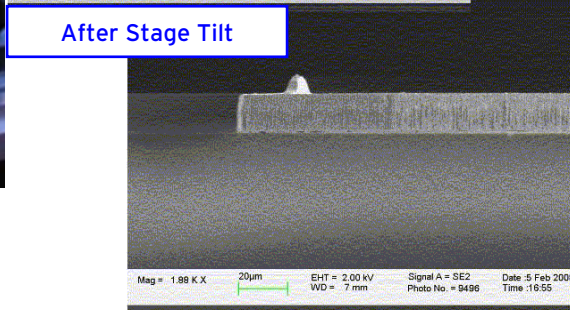
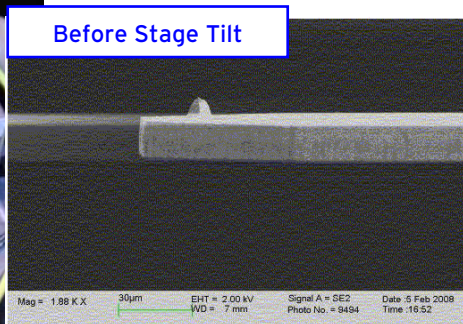


Step 9. Adjust the location of each positioner to place the FCP sensor and probes near the sample.

9. After the system has reached working pressure, turn on the beam and locate the sensor cantilever in the SEM image.

Check to see that the cantilever is exactly perpendicular to the beam axis, by making sure that only the edge of the sensor is visible. If any misalignment is seen, carefully tilt the SEM stage and re-check the SEM image until only the edge of the sensor can be observed.

WARNING: Be careful when tilting the stage to ensure nothing damages the SEM column.



Step 10. Tilt the SEM stage to correct any sensor misalignment.

Electrical Connections for the FCP

The Zyvex FCP uses a piezoresistive force sensor with an integrated Wheatstone bridge to convert mechanical forces on the sensor tip into a change in bridge resistance.

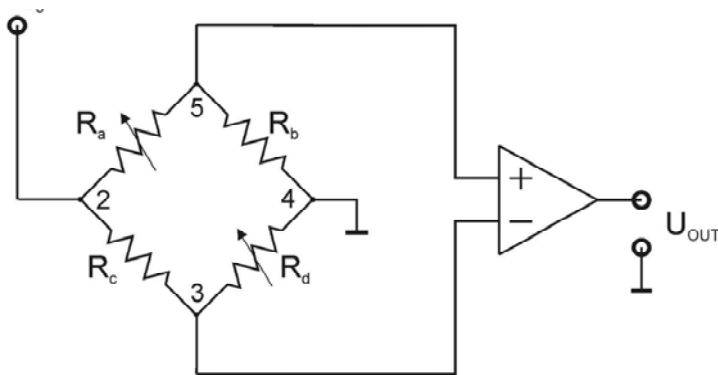


Figure 3. Schematic of the Wheatstone bridge built-into the force sensor.

A method to supply and measure small voltages and currents, such as a Keithley Instruments 4200 Semiconductor Characterization System (Keithley 4200), is required to apply the voltage to the bridge and measure its output.

In this example, 4 Keithley Source Measurement Units (SMUs) are connected to the channels of one positioner. Once installed on a chosen positioner, the channels 1, 3, 4, and 5 for that positioner (on the front panel of the rack) need to be connected to the back of the Keithley 4200 as shown below. Connect the outputs at the back of each SMU to the front panel connections for the chosen positioner on the Zyvex equipment rack as indicated in Table 1.

Note: If your Keithley 4200 unit has only 2 SMUs, you may use another external voltage source-measure unit (ex. Keithley 2410). If the external unit has 2 channels capable of supplying 2 different voltages simultaneously, one channel should be set at 1.75V and the other at -1.75V. The unit common must then be connected to the 4200's common line. If the external unit has only 1 channel, then the voltage should be set to 3.5V, with the common similarly connected to the 4200's common. Finally, in the case of a differential external voltage unit such as the Keithley 2410, its "High" terminal should be set at 3.5V and the "Low" terminal must be connected to the 4200's common (see the Figure 4 on page 7). Notice that this external voltage unit does not have a common connection.



Figure 4. Keithley 2410 Voltage Source Meter.



Figure 5. Electrical connections on the front panel for FCP measurements.

SMU # (Back)	Front Panel Channel	Name
1	1	V in 1
2	3	V out 1
3	4	V in 2
4	5	V out 2

Table 1. Electrical connections for the FCP.

For the case of a set-up made up of 2 Keithley 4200 SMUs plus an external voltage unit, the following connection configuration must be used.

SMU # (Back)	Front Panel Channel	Name
1	1	V in 1
2	3	V out 2
External Unit “High” or +V Terminal	4	V in 1
External Unit “Low” or -V or Common Terminal	5	V in 2

Table 2. Electrical connections for the FCP.

Note that V in 1 should be connected to the 2410’s “High” output line, and V in 2 needs to be connected to the 4200’s common, hence, it also connects to the 2410’s “Low” output connection.

Force Measurements with the FCP

When using the Keithley 4200, the FCP is equipped with DC Measurements software that automates sensor calibration and force measuring. Measurements are performed in real-time to allow the user to apply varying mechanical loads to the sample as they watch the SEM video, and are collected as both a graph and as raw data, allowing further data analysis in any software package desired. All of the software and hardware settings used for the test are also included in the data files, for comparing different tests to each other. Instructions for setting and using the FCP software are given below.

Software Configuration

Before force measurements can be made, it is necessary to properly configure the Zyvex Nanomanipulator software for force measurement. If you do not have the DC Measurement Package from Zyvex then refer to the other vendor’s documentation. The steps for configuration are given below:

1. Make sure that all electrical connections are made and that a force sensor is correctly installed in the Nanomanipulator.

Ensure that the Keithley 4200 is turned on and that the Keithley External Control Interface (KXCI) has automatically started.

Also make sure that the Nanomanipulator software is running and the DC Measurement window is active. The DC Measurement window can be activated by clicking Tools, then selecting DC Measurement in the drop down menu of the Nanomanipulator software.

2. In the DC Measurement window, a list of measurement sequences is shown. Near the top of the list is a sequence called Force Sensor.

This sequence contains the tests which will perform calibration and measurement.

Double click the sequence Force Sensor and verify that the sequence is configured as shown in Table 3:

Channel	Configuration	Name
1	Med. Power SMU w/ preamp	V in 1
2	Med. Power SMU w/ preamp	V out 1
3	Med. Power SMU w/ preamp	V in 2
4	Med. Power SMU w/ preamp	V out 2

Table 3. Configurations for the Force Sensor sequence.

For the case of a set-up made up of 2 Keithley 4200 SMUs plus an external voltage unit, a special DC Measurement sequence has been included near the top of the list in the DC Measurement window, and is called “Force Sensor (2 SMUs + external V source)”.

This test sequence works in the same as the case of 4 SMU set-up, except with the added requirement of manually triggering the external voltage source. For this sequence, the operator manually starts the external voltage sourcing prior to performing the measurements.

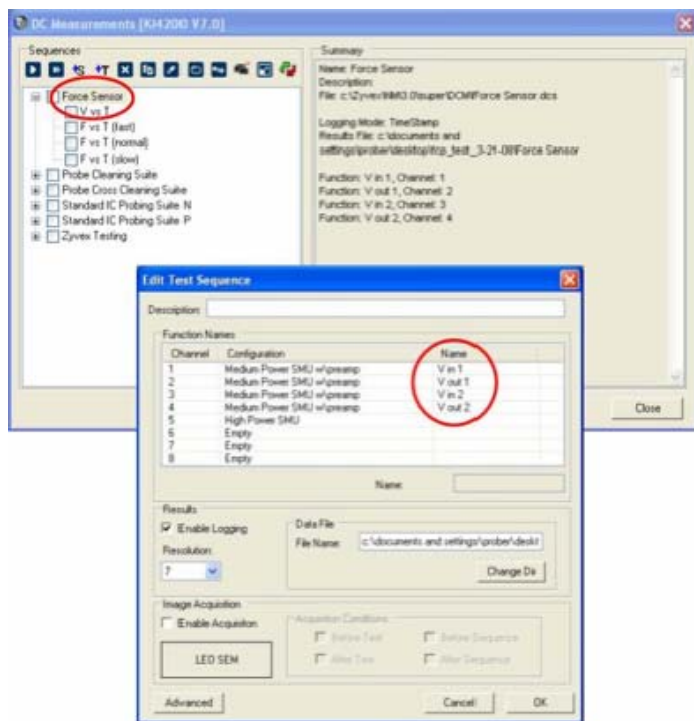


Figure 6. Double click to configure the Force Sensor sequence.

Initial Sensor Calibration

After configuring software, initial sensor calibration is needed. The FCP software automatically calibrates at the beginning of each measurement using input parameters determined during initial calibration. The purpose of this calibration is to find a correlation between the output voltage of the Wheatstone bridge and the physical deflection of the sensor cantilever, in the form of

$$x = f(V_{out})$$

where “x” is the displacement and $f(V_{out})$ is some function of output voltage. Once the displacement is characterized, it can be combined with the cantilever’s given spring constant to yield force, as given in the equation

$$F = k \cdot x$$

where “F” is the measured force and “k” is the mechanical spring constant of the cantilever.

This initial sensor calibration is performed by using a probe installed in a Nanomanipulator fine positioner to deflect the cantilever by a known distance, in even steps, and measuring the bridge output voltages. The SEM measurement tool is used to measure displacement steps and the Keithley 4200 records output voltages.

The direction of sensor calibration is important, and depends on the expected deflections to be measured during the desired experiment. For example, if the sample to be measured is a thin and wire-like shape, a tensile (pulling) experiment may be desired. The direction of deflection caused by a tensile test should be the same as the direction in which initial sensor calibration was performed. It may be necessary to push on the backside of the cantilever to calibrate in the proper direction. Deflection in this way will yield effectively the same results as pulling from the front of the cantilever.

Figure 7 shows a method for deflecting the cantilever for initial sensor calibration. Cantilever displacement is strictly measured as the deflection of the cantilever tip before and after applying a force with the probe. In this way, any probe bending does not affect calibration data. If a compression (pushing) experiment is desired, it is oppositely necessary to push the cantilever from the front side of the lever. However, do not push on the tip of the sharp cantilever tip, which may cause to damage the tip. The tip is so sharp that a single walled carbon nanotube can be mounted; on the other hand, it is not designed to handle a great force as exerted during the calibration.

The measurement used for the initial calibration is structured as a voltage versus time test (V vs T). While watching a live image on the SEM, the user presses on the cantilever with the probe tip until the cantilever has deflected a known distance as measured with the SEM measurement tool. The probe motion is stopped and several data points are collected at the given displacement.

The following plot shows an example wherein a total displacement of 30 microns was broken into 6 individual 5 micron steps. The cantilever in this example had a k value of 142 N/m. The actual calibration size may depend on the k value as well as the dimensions of the cantilever, but it also depends on the deflection range you desire for your experiments.

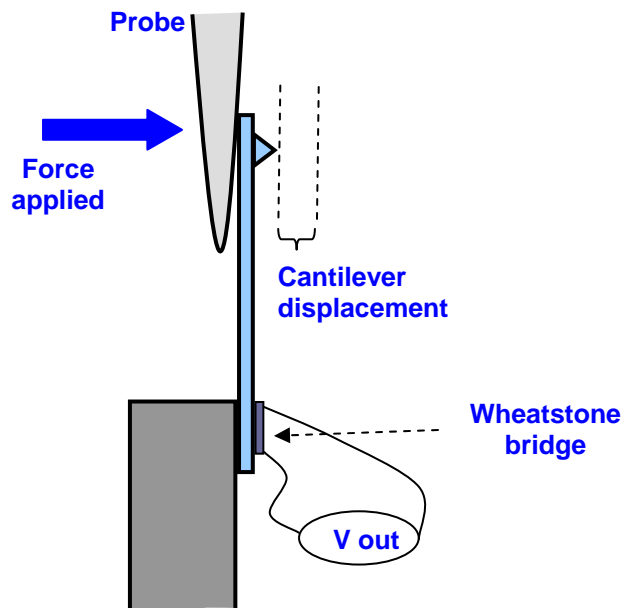


Figure 7. Example of initial sensor calibration.

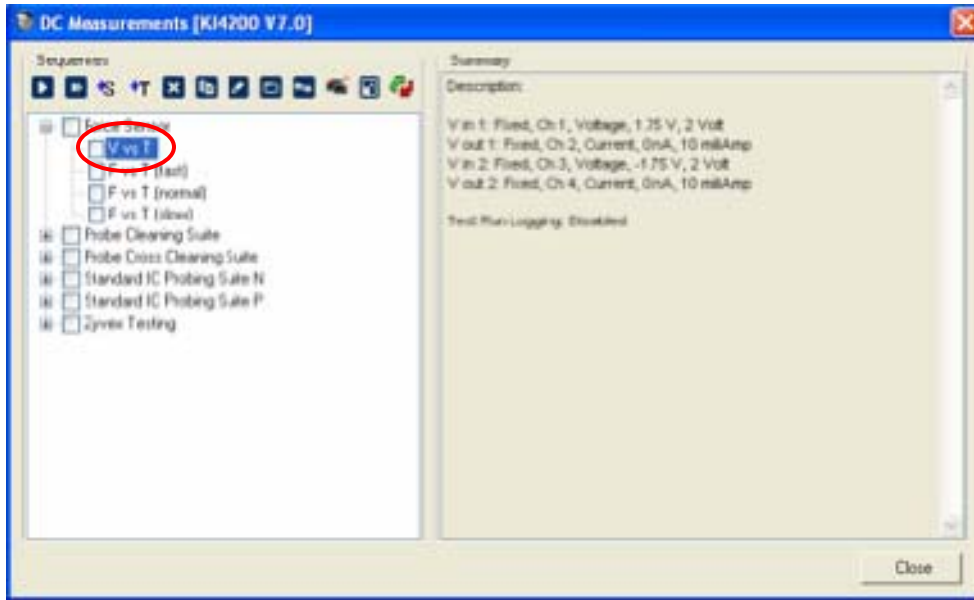


Figure 8. Voltage versus Time test.



Figure 9. Graph of an initial calibration test.

After successfully obtaining an initial calibration test with flat, well defined voltage steps for each displacement, click “List” (shown above) to access the raw data.

Take note of the “Delta Vout” for each displacement step, as shown below. These values must be recorded manually, and are based on the user’s best judgment of the stable voltage value for the given displacement.

	Time [A]	V in 1 Current [B]	V in 1 Voltage [C]	V in 2 Current [D]	V in 2 Voltage [E]	V out 1 Current [F]	V out 1 Voltage [G]	V out 2 Current [H]	V out 2 Voltage [I]	Delta Vout [V] [J]
1	859.375	0.000501559	3.499915	-4.519230E-0	-2.125702E-0	-3.686279E-1	0.01525803	-3.996207E-1	0.02155033	6.292E-03
2	11875	0.000501564	3.499913	-4.519190E-0	-2.092792E-0	-4.150276E-1	0.01525937	-4.172881E-1	0.02155061	6.291E-03
3	19546.875	0.000501562	3.499912	-4.519127E-0	-2.093602E-0	-4.523939E-1	0.01525681	-4.450494E-1	0.02155032	6.294E-03
4	27203.125	0.000501566	3.499917	-4.519076E-0	-2.095048E-0	-4.912217E-1	0.01525963	-4.627159E-1	0.02155063	6.292E-03
5	34829.125	0.000501566	3.499916	-4.519093E-0	-2.096389E-0	-4.959249E-1	0.01525766	-4.268762E-1	0.02154965	6.293E-03
6	42500	0.000501567	3.499911	-4.518968E-0	-2.094342E-0	-4.529422E-1	0.01525831	-4.647362E-1	0.02154829	6.29E-03
7	50125	0.000501563	3.499911	-4.518957E-0	-2.095562E-0	-4.912238E-1	0.01525898	-4.52527E-1	0.0215494	6.29E-03
8	57745.625	0.000501568	3.499912	-4.518932E-0	-2.094177E-0	-4.967702E-1	0.01525648	-4.521181E-1	0.02154909	6.29E-03

Figure 10. Raw data of an initial calibration test.

After the data has been recorded, external software may be used to plot the Displacement values versus the “Delta Vout” values.

Note that you make sure and plot the displacement values vs the delta voltage, but not the other way around because we would like to obtain the calibration coefficients for the displace as a function of output voltage.

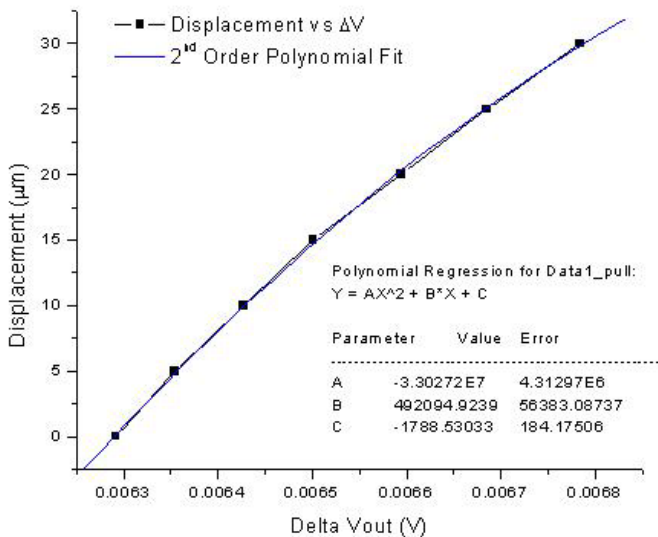


Figure 11. Graph of the data relating Displacement to Delta Vout.

In addition, a 2nd order polynomial fit should be added to determine coefficients to be entered for subsequent measurements. The equation found in this example is

$$x(\Delta V_{out}) = -3.30 \cdot 10^7 \Delta V_{out}^2 + 4.92 \cdot 10^5 \Delta V_{out} - 1.79 \cdot 10^3$$

Furthermore, to maximize the accuracy of the measurements, the force measurement test is designed to measure the delta voltage offset at the beginning of each test and feed the value back to the equation as a preset value (zero value).

We are only interested in the shape of the polynomial curve; hence, we subtract the “Delta Voltage” value at zero displacement (0.00629V) from every “Delta Voltage” that was obtained in the initial calibration earlier and re-plot the values once again.

	DeltaVoltage[X]	Displacement[Y]		DeltaVoltage[X]	Displacement[Y]
1	0.00629		→	0	0
2	0.00635	5		6.2E-5	5
3	0.00643	10		1.35E-4	10
4	0.0065	15		2.09E-4	15
5	0.00659	20		3.02E-4	20
6	0.00669	25		3.93E-4	25
7	0.00678	30		4.92E-4	30

Figure 12. Removing the beginning offset value from the data.

Therefore, the new and final expression will have a polynomial expression to match the shapes of the curves, while the resetting the off-set value is left to the software’s auto-calibration for each measurement. Note

that the force measurements software (F vs T normal) takes roughly a quarter of a minute to start taking measurements because of this automated calibration, and this is normal.

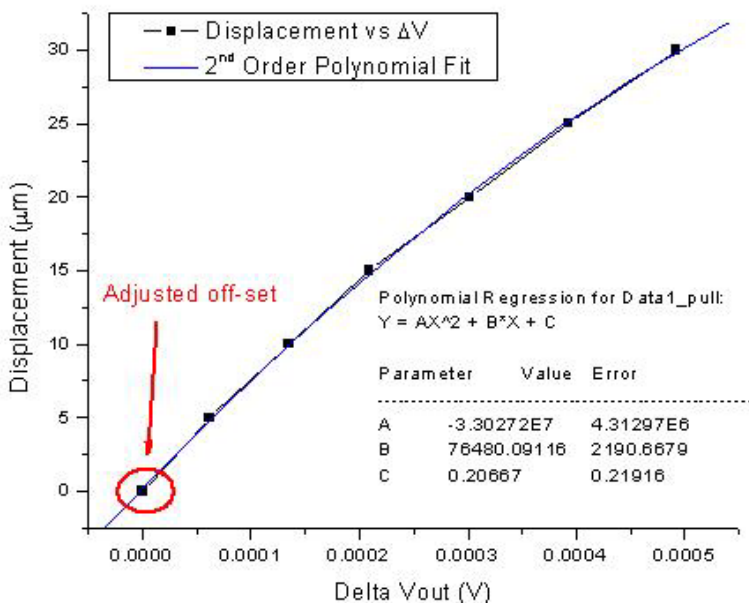


Figure 13. Graph of the data relating Displacement to Delta Vout with offset correction.

Hence, the new correlation is

$$x = -3.30 \cdot 10^7 V^2 + 7.65 \cdot 10^4 \cdot V$$

Also, notice that there is no constant coefficient is included in this final expression. This is because of the auto-zero function of the software which again reset the initial offset of the output voltage. Once the correlation coefficients are found, their values should be entered in

the “Force Measurement” data sheet along with the spring constant (k) of the cantilever. The next several screen-shots expressively show how to get to the value input page.

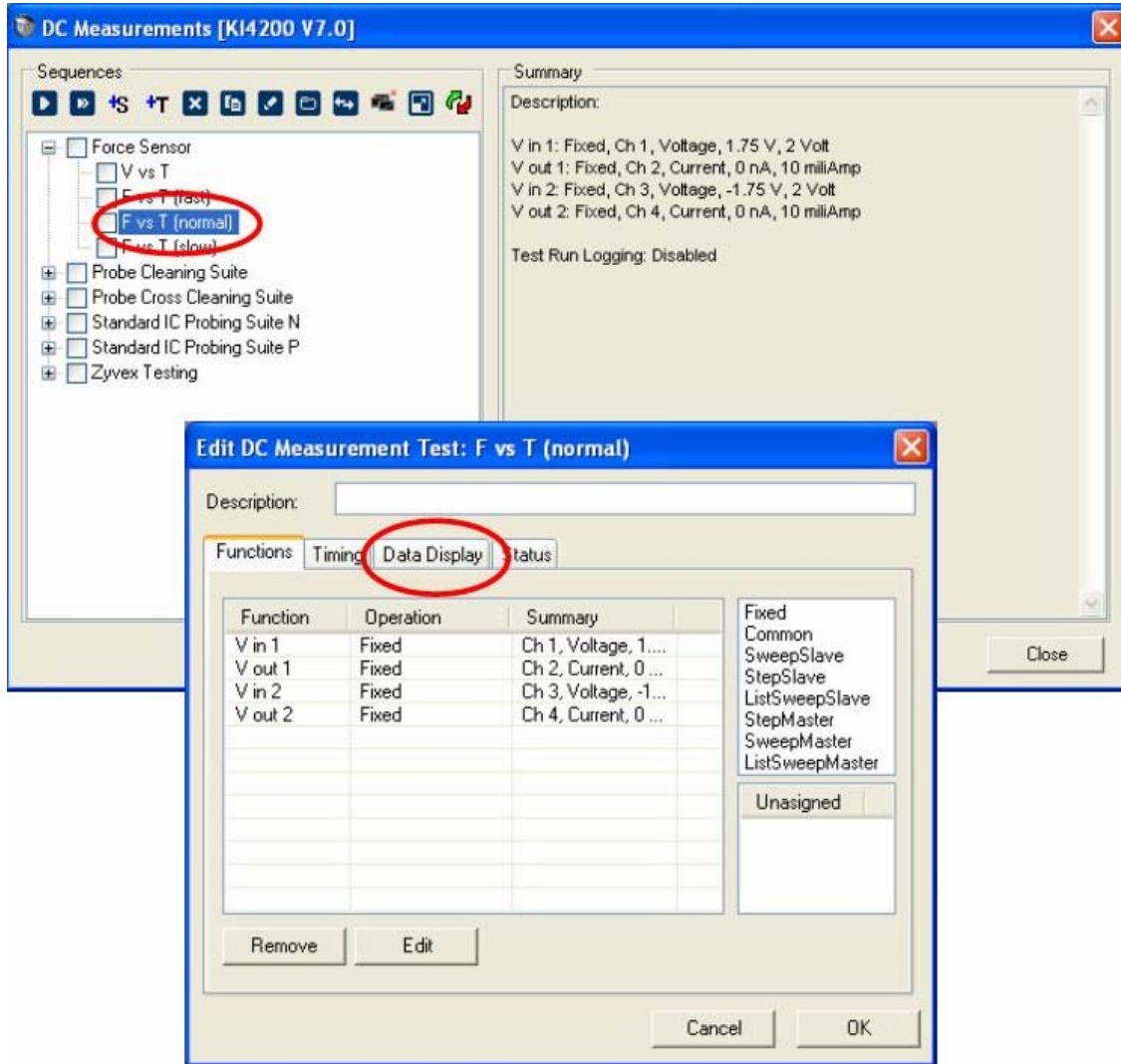


Figure 14. Navigating to the data sheet to enter coefficients.

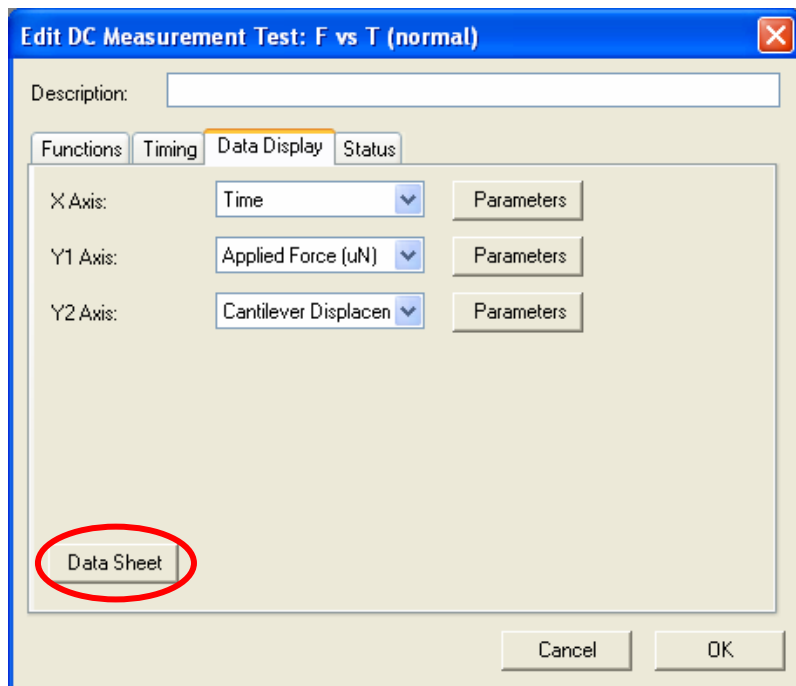
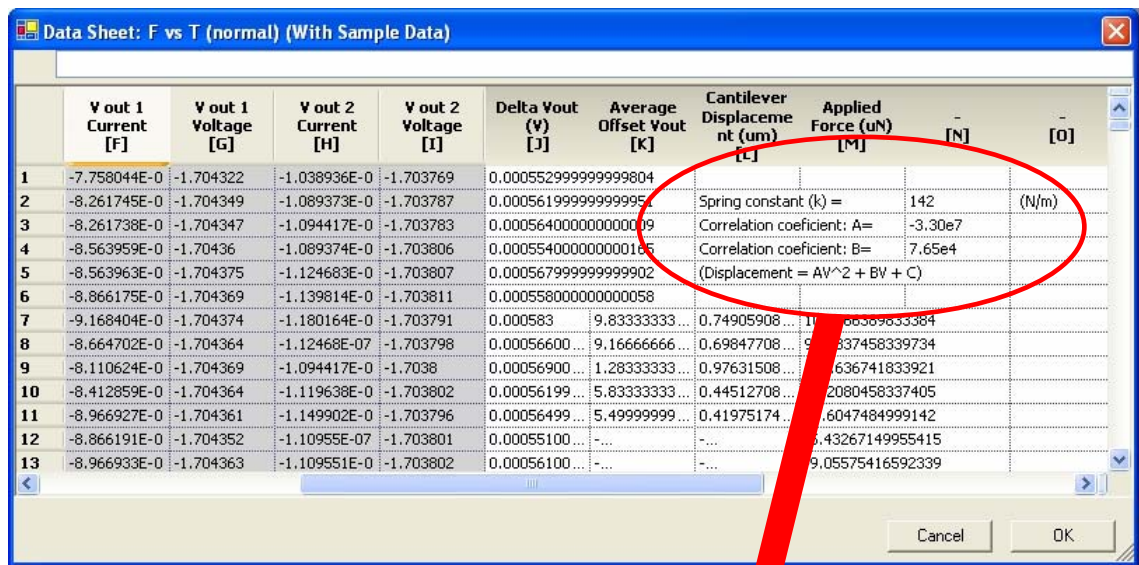


Figure 15. Navigating to the data sheet to enter coefficients.



Delta Vout (V) [J]	Average Offset Vout [K]	Cantilever Displacement (um) [L]	Applied Force (uN) [M]	[N]	[O]
0.000552999999999804					
0.000561999999999951		Spring constant (k) =	142		(N/m)
0.000564000000000009		Correlation coefficient: A=	-3.30e7		
0.000554000000000165		Correlation coefficient: B=	7.65e4		
0.000567999999999902		(Displacement = AV^2 + BV + C)			
0.0005580000000000058					
0.000583	9.83333333...	0.74905908...	106.366389633384		

Figure 16. Enter the coefficients here.

Taking a Force Measurement

To take force measurements, F vs T programs may be used. There are three types of programs are provided to choose from. For most measurements, “F vs T (normal)” may be used, while in case of needing fast

response thus also higher noise level, the “F vs T (fast)” can be used. Oppositely, when slow steady-state measurements are required, the “F vs T (slow)” may be chosen.

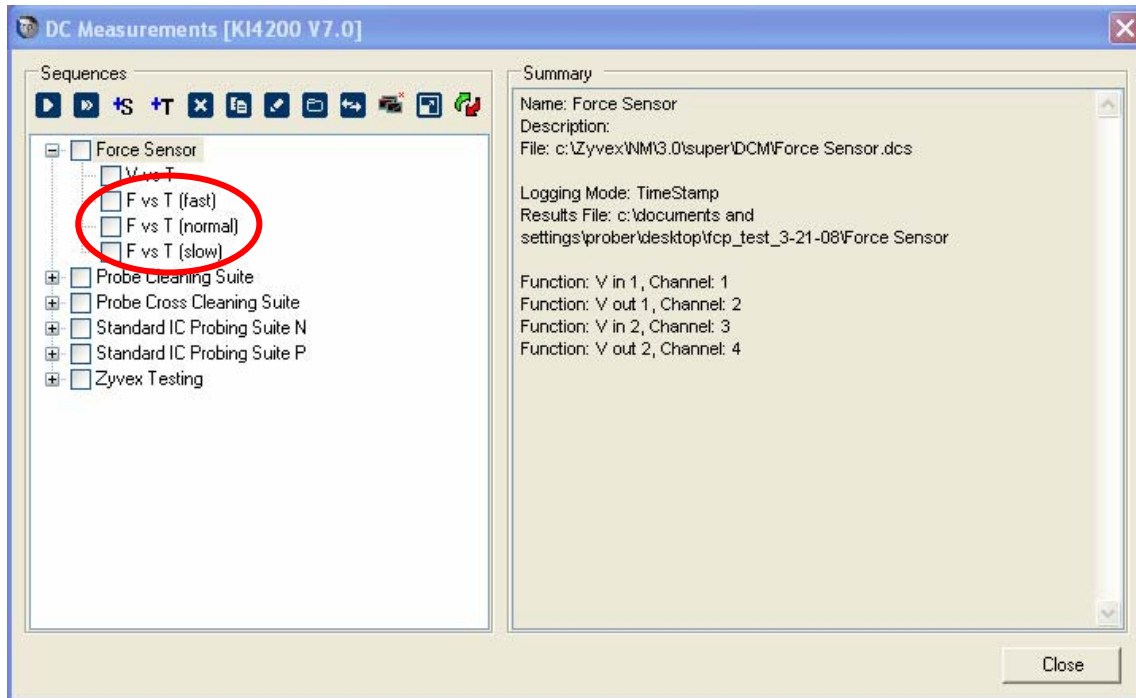


Figure 17. Navigating to the data sheet to enter coefficients.

The sample on which the experiment will be performed should be mounted between a probe and the cantilever, if a tensile experiment is desired. One way of attaching a material to a desired surface is to use electron-beam induced deposition (EBID). The intrinsic Wheatstone bridge voltage-out needs to be measured. Ideally a Wheatstone bridge is balanced, so that voltage-out is zero when no force is applied. However, in reality, these photo-lithographed Wheatstone bridges are not balanced at exactly zero.

Hence, there is a slight built-in offset, which needs to be determined and set as a preset initial value by the Zyxex software. When the measurement test (F vs T) is executed, this calibration process is automatically done. The time lag in the plot is because of this process. The probe, with attached sample, should be moved away from the tip of the cantilever slowly until the slack of the material disappears. This can be checked by monitoring the voltage-out change from the preset value, while pulling the material gradually.

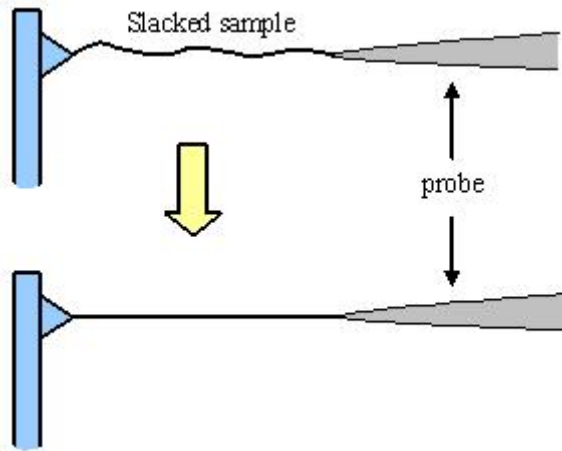


Figure 18. Taking out the slack in a tensile test.

To measure the strain of the sample, it will also be necessary to measure the displacement of the probe tip at the point where the sample is attached. The

difference between the probe displacement and the cantilever displacement will be the overall strain of the sample.