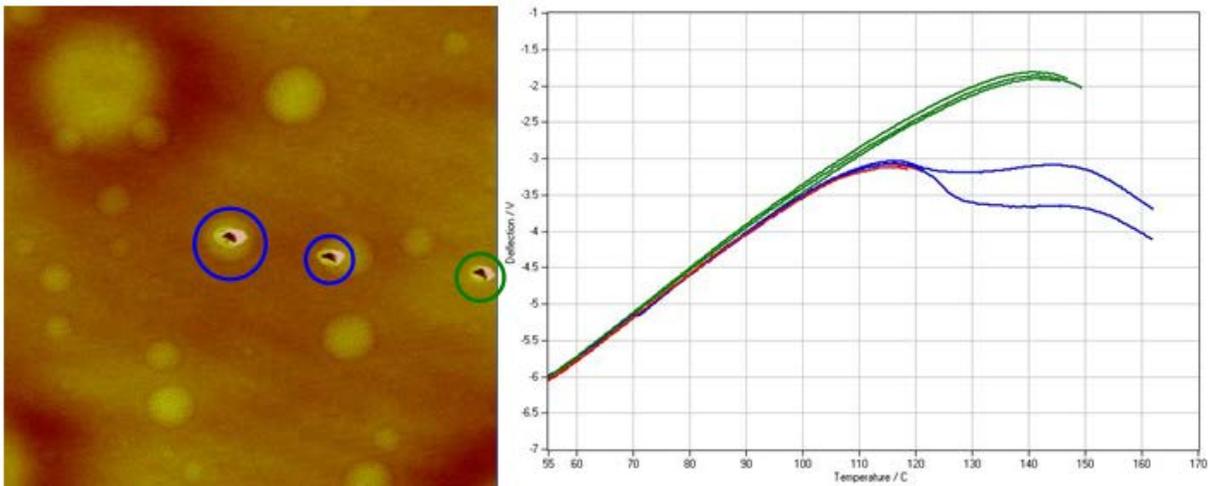


NanoTA

On afm+™ and nanoIR™ Systems



User's Manual

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Chapter 1

About NanoTA

1.1 Basic Concept

NanoTA (Nano Thermal Analysis) is a technique used to measure local thermal transitions of materials that can be imaged with Atomic Force Microscopy (AFM). A special thermal probe that can be controllably heated is used to do the AFM. Once the sample has been imaged, the probe is placed on the region of interest for analysis. Then the temperature of the probe is increased. This heats a small region of the sample around the probe's point of contact. The deflection of the probe is monitored during the thermal ramp. Below is a schematic showing the initial upward deflection of the probe due to thermal expansion of the sample, followed by the penetration of the probe due to softening or melting. This will typically happen at the glass or melt transition of the sample.

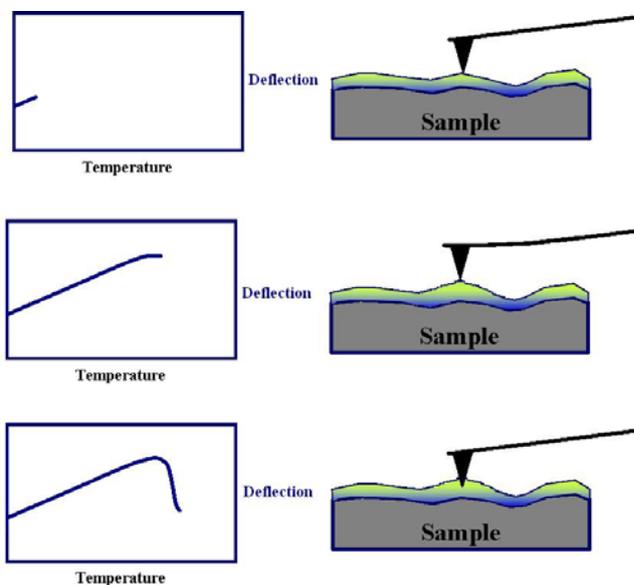


Figure 1-1: Schematic of the nanoTA process.

The heating of the probe is due to a high resistance region incorporated into a silicon probe that is otherwise much like a standard silicon probe. This allows rapid, controllable heating via the voltage applied to the probe. During the ramp, the operator can monitor up to four available signals including the deflection and resistance of the thermal probe, and the power applied to the probe.

1.2 ThermaLever Probes

Anasys Instruments has two styles of micro machined ThermaLever probes available. One has a cantilever length of ~300 μm and the other has a length of ~200 μm . The ThermaLever probes have tip heights of 3-5 μm and a large area to reflect the AFM laser. The 300 μm cantilevers can achieve a maximum temperature typically of 400° C and the 200 μm cantilevers achieve a maximum temperature of 350° C. Shown below are SEM images of the probes. Please contact Anasys Instruments to help determine the best probe for your application.

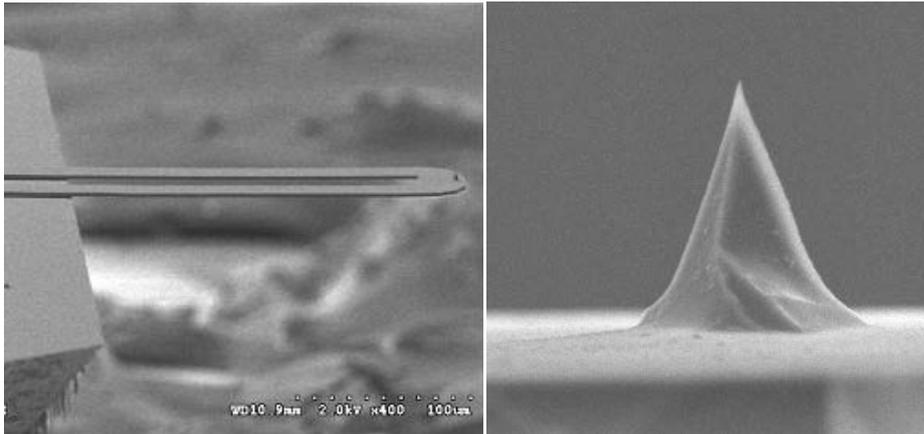


Figure 1-2: SEM images of a ThermoLever probe.

Both styles of probes are made from doped Silicon which means that the temperature change with resistance is nonlinear. This means that when calibrating the probe temperature, three calibration samples are required to accurately calibrate the probe. Also, the probe will have a maximum temperature as mentioned above. This maximum temperature is the highest temperature that can be achieved before the resistance turnaround point. Shown below is a plot of resistance versus voltage for a number of ThermoLever™ probes not in contact with a sample surface. As can be seen the resistance which correlates to the probe temperature is nonlinear and at a certain voltage the resistance begins to decrease. At this point, the correlation between the resistance and temperature ceases and the temperature of the probe begins to increase rapidly.



The voltage applied to the probe should be kept below the level of the resistance turnaround to prevent damage to the probe.

The resistance turnaround point can be easily measured by performing a thermal ramp with the probe not in contact with a sample surface while the system is monitoring the resistance of the probe.

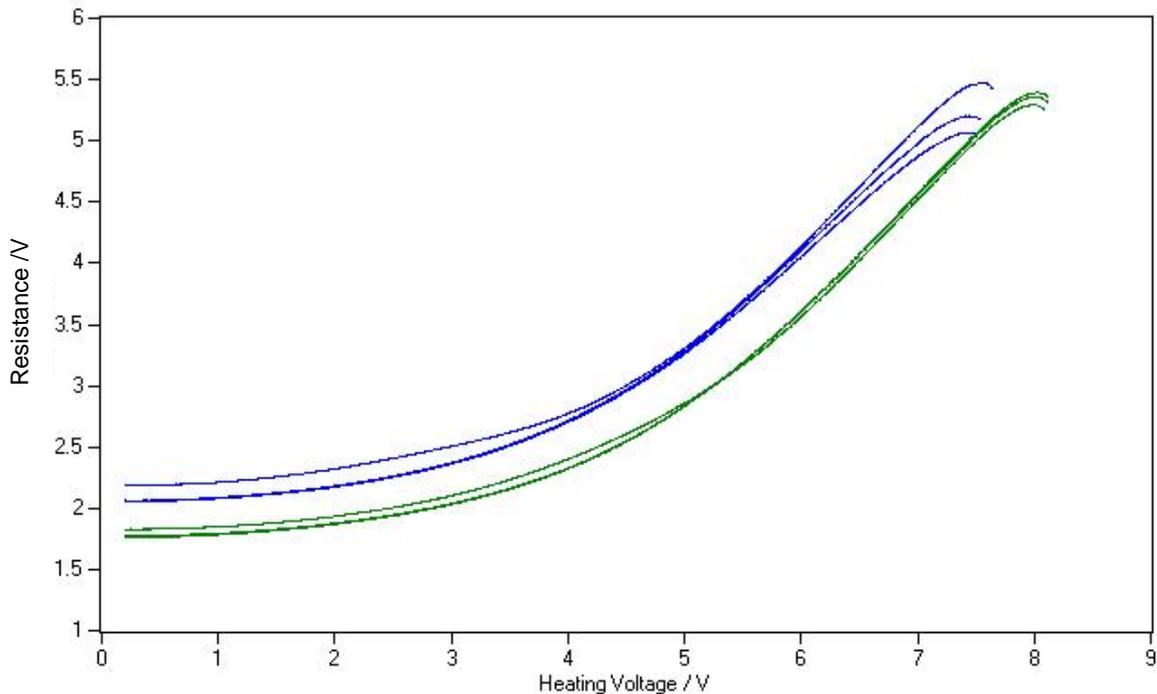


Figure 1-3: Resistance vs. Heating Voltage plots from ThermoLever probes that are off the sample surface demonstrating the resistance turn-around.

Because the ThermoLever probes require electrical connections to relatively small contacts on the cantilever substrate, the probes are glued to a larger half-disc structure and wire-bonded from probe substrate to the half-disc for the electrical connection. When exchanging a probe, the user will exchange this complete unit. Extreme care should be taken when handling the ThermoLever™ probes as the cantilever and wire-bonds are both very fragile and can easily be broken by any contact or rough treatment. **The ThermoLever must be disconnected before power to the system is turned on or off.**

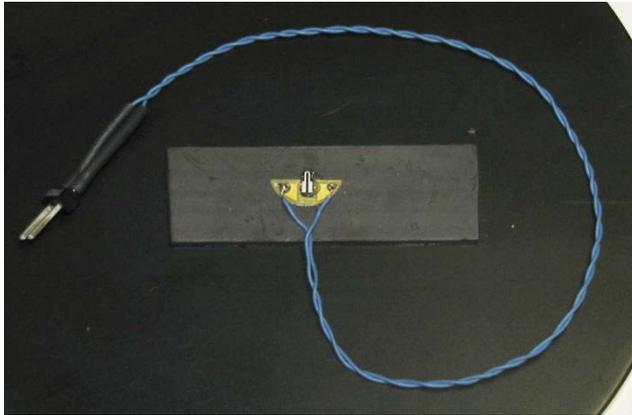


Figure 1-4: ThermoLever

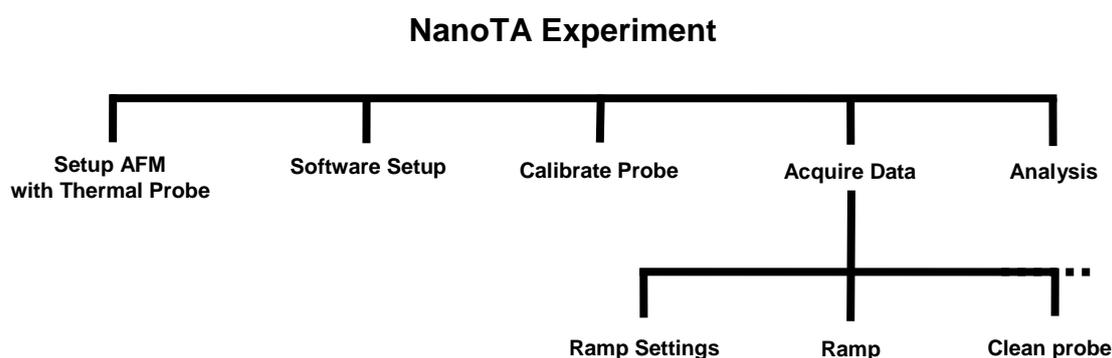
Before using a probe it is recommended that you check the resistance value to make sure the probe still has connections and is in a good resistance range. This can be done with the probe still in its box. Connect the probes from a Multimeter to the 2 pins of the black plug at the end of the blue wires. It is important that a non-autoranging Multimeter is used for this measurement and the range is set to 10 kOhm. This will minimize the current flowing through the probe and avoid voltage spikes caused by the autoranging function of the Multimeter that can easily destroy the resistive element on the probe. The expected resistance for the ThermoLever™ probes can range from 0.5 to 3 kOhm.

Chapter 2

Performing NanoTA

2.1 Standard Operating Procedure

The procedure below summarizes the basic steps in performing nanoTA experiments. The steps are covered in this and the following chapters. Once you have gained experience with the various steps involved in using the software, you may find this outline a useful checklist to refer to when setting up an experiment or scan.



Performing nanoTA begins with setting up the AFM with a ThermoLever probe. If needed, the software is configured for nanoTA. The probe is calibrated using the 3 standard calibration samples. Then the AFM is used to locate the area of interest on the sample. Temperature ramps are acquired and the data is analyzed. The chapters/sections associated with these operations are listed below.

1. AFM Setup and Operation (Section 2.2 and main product manual)
2. Software setup (Section 2.3)
3. Calibrating the Probe (Section 3.2)
4. Acquiring Data (Section 2.3)
5. Data Analysis (Software Manual)

2.2 AFM Setup

A nanoTA experiment begins with getting the AFM portion of the instrument running. This includes installing a probe and sample, aligning the AFM laser and detector, and engaging the probe onto the sample surface. The AFM setup is nearly the same as it is for general use. The two main differences are that a thermal probe is used and the AFM detector is aligned differently. Both items are covered in this section along with other AFM advice relevant for nanoTA. ***This manual assumes the user is familiar with operating the AFM, instructions for which are in the main product manual.***



Extreme care should be taken when handling the ThermoLever™ probes as the cantilever and wire-bonds are both very fragile and can easily be broken by any contact or rough treatment.



TURNING ON AND OFF THE CONTROLLER POWER SHOULD ONLY BE DONE WHEN THE PROBE IS DISCONNECTED!

Loading a ThermoLever Probe

1. Rotate the optics arm so that it is out of the way of the head.
2. Pivot the head up and back using the handle (rod sticking out of the head). Do not use the Detector Knob to flip the head. The back edge of the head should now rest on the black plate.
3. Using tweezers, remove a probe from the box in which they are shipped. Before doing this remove the tape that is holding the blue wires in place. The thermal probes are held in the box by a magnetic plate. The easiest way to remove a probe is to slide it to the edge of the magnet until it hangs slightly over the edge. Then use tweezers to grasp the blue connecting wires and lift the probe away from the plate.
3. Place the probe into the recessed semi-circular seat on the underside of the head. The seat contains magnets to hold the probe firmly in place. For accurate measurements, the probe must be seated firmly into the holder at the correct angle.
4. Connect the probe wires to the two socket connector on the cover behind the head.
5. It is best if the wires are positioned such that they extend out of the back of the head and do not interfere with the sample or optics arm. Also, the wires should be routed such that they are not taut. If they are, they may pull on the probe mount and shift the position of the probe.

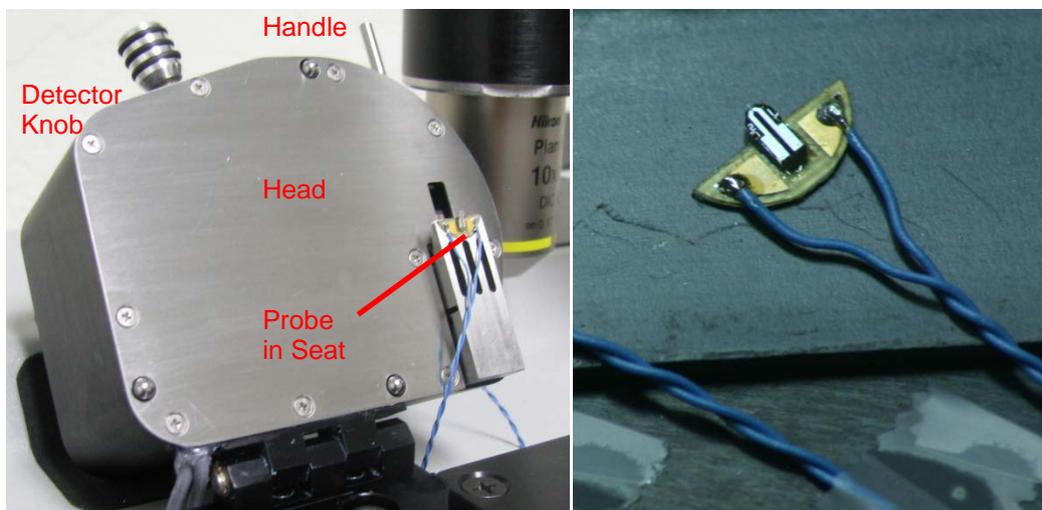


Figure 2-1: A ThermoLever™ probe both on and off the head.

The laser spot needs to be positioned such that it is near the end of the cantilever. ThermoLevers have two legs to the cantilever and one option to position the laser spot is at the end of the cantilever on the cross piece between the two legs. Remember that once the laser spot appears to be positioned in the video view, fine tune the position by watching the Laser Sum value. The Sum should be maximized while still keeping the laser spot close to the end of the cantilever in the video view. We have found for some applications that it is better to position the laser spot on one of the cantilevers legs slightly back from the end of the cantilever.

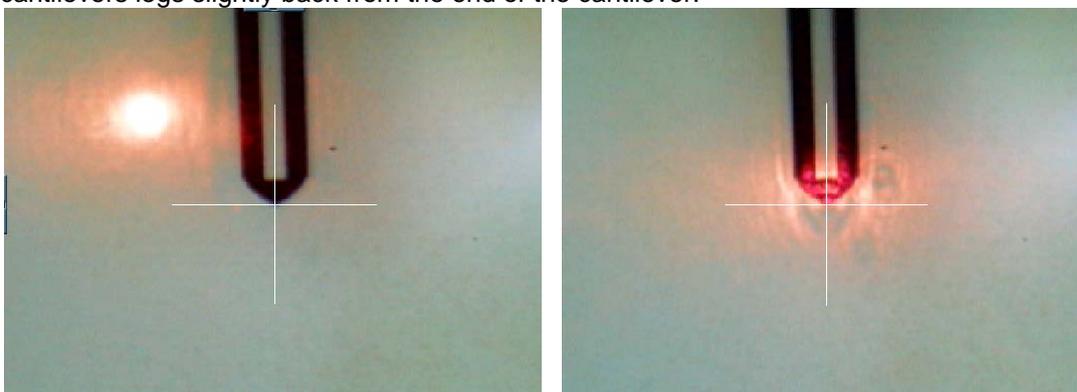


Figure 2-2: The Video view showing the laser spot to the side (left) and correctly centered on the end of the cantilever (right).

Tracking the thermal expansion of samples during nanoTA often utilizes a significant amount of the AFM detector range. More range is available if during setup, the detector is aligned away from its center. In the Setup/Engage Settings menu, set the “free air calibration target deflection” to -4 V. This sets the value for the green indicator light in the center of the Deflection light bar (in the AFM Probe panel). Now the alignment procedure is the same as usual. (Adjust the detector knob on the front of the head until the Deflection indicator is green, making sure the Sum remains above 1 V). If you find that the deflection is running out of range during your nanoTA experiments, start with a lower free-air deflection (down to -7 V typically works fine).

We recommend that both the engage and imaging forces be minimized when using ThermaLevers. The engage force is set via the Setup/Engage Settings menu. A conservative starting value for the Engage Force is 0.3 V. This value can be increased as needed if you find the probe will not engage consistently. The imaging force is determined by the Setpoint relative to the probe’s deflection just off the sample surface. The initial value of the Setpoint (in the AFM Scan panel) will be set automatically at the end of the engage process. To minimize the imaging force, follow the procedure given in the Acquiring Data/AFM Image Acquisition section of the nanoIR or afm+ manual (reproduced below) to find the minimum Setpoint.

Once the probe is engaged on the sample surface, the imaging parameters in the AFM Scan panel can be optimized. The first parameter to set is the Setpoint value. This parameter controls the force between the tip and sample. The lower the Setpoint, the smaller the force is. With a very low setting the probe may come off the sample surface because it cannot maintain stable contact with the surface. A higher setting (more force) can damage the sample more easily and dull the probe more quickly. The basic way to optimize this parameter is to reduce the Setpoint until the probe retracts from the sample surface. When the probe retracts, the light on the Z Piezo meter will move to the left end of the bar. It usually requires a Setpoint of -2 V or lower to retract the probe. Then check the Deflection value in the Volt Meter panel. Set the Setpoint to a slightly more positive value (~0.1 V) than that value. This should bring the probe back into contact with the sample surface (the light on the Z Piezo meter will move back toward the center of the bar). More force (larger Setpoint) may be needed for some samples and imaging conditions (larger or faster scans) but this establishes the baseline.

While the AFM is engaging, watch the deflection in the meter panel to see if it drifts downwards. If it does, this can be a sign of electrostatic attraction between the sample and probe, which can be due to static charge on the sample. If you see this, reduce the charge using a static elimination device.

AFM scan parameters with the ThermaLevers are similar to what is done for any etched Si contact mode cantilever. It is generally better to image such that the fast scan direction of the AFM image is perpendicular to the long direction of the cantilever.

2.3 Software Setup

Some setup in the software may be needed before running a nanoTA experiment. Most of the software settings do not need to be changed each time. The system remembers the settings from the last session.

On a nanoIR system, the bottom of the Control Window displays either the nanoIR panel or the nanoTA panel depending on the current mode. To change the mode, go to the Setup/Control panels menu and select the desired application. On an afm+, the nanoTA panel is typically available and this step is not needed.

In the nanoTA panel, click the Settings button on the toolbar.  Settings Shown below are the default settings. NanoTA should always be run in “Single” mode. The user can designate which signals to collect during nanoTA ramps. If the user does not want to collect all four channels, any channel can be set to Disabled.

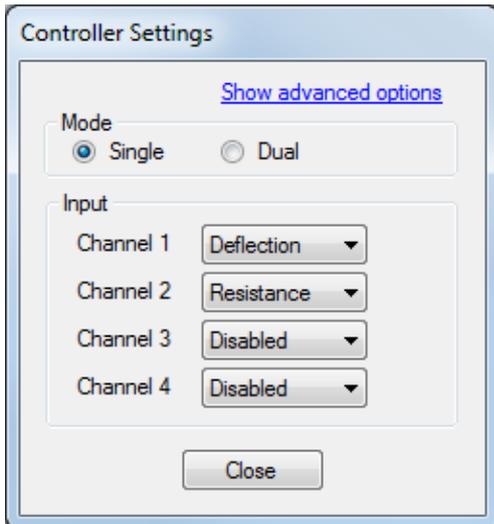


Figure 2-3: Default NanoTA Settings.

The basic method of nanoTA uses Power feedback during the temperature ramp. This is the default configuration and does not need to be changed unless the user is doing advanced modes of operation (or a previous experiment was run using thermal feedback - the settings persist, even after closing the software). The user may check the nanoTA feedback state by clicking on “Show advanced options”. For details on how to use the Power and Resistance feedback options, refer to the Advanced Procedures chapter.

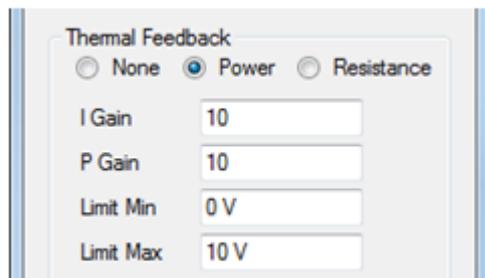


Figure 2-4: Default Thermal Feedback Settings.

In the Setup/nanoTA channels menu the default color of the nanoTA signals can be changed along with their display names, units, scaling, and offsets. See the Setup/nanoTA channels section of the Software Manual for more details.

2.4 Acquiring Data

When the Analysis Studio software is initialized and configured for nanoTA, the bottom of the Control Window will have the nanoTA panel and TA Meter. The initial (pre-calibrated) values shown in the nanoTA panel below are all in volts (V) because a calibration file is not loaded. Once a calibration file is loaded the values will appear with the units of degrees Celsius (C). You can check the calibration state by looking at the status bar at the bottom right corner of the Document Window. If a calibration file is loaded it will show the name of the file and if none is loaded it will not show a file name. Following are some reasonable starting values for the nanoTA settings. Different probes will require different voltages to achieve a certain temperature. Because of this we recommend using caution when operating with an uncalibrated probe. The user may need to abort early in the ramp if the temperature goes too high.

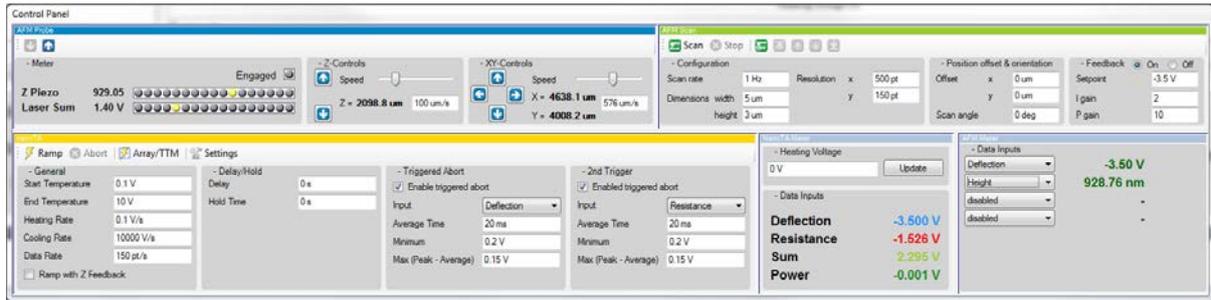


Figure 2-5: The Control Window configured for nanoTA.

Once the AFM is setup and engaged, image the sample to find the desired location for analysis. Create a new document if needed and capture an AFM image into the document. This will provide a reference for the subsequent nanoTA locations. Then stop the scanning and position the probe at the desired location. Click the Stop button  in an appropriate area or use the target  cursor to place the probe somewhere on the scanned image. Verify the ramp parameters in the nanoTA panel are set to appropriate values.

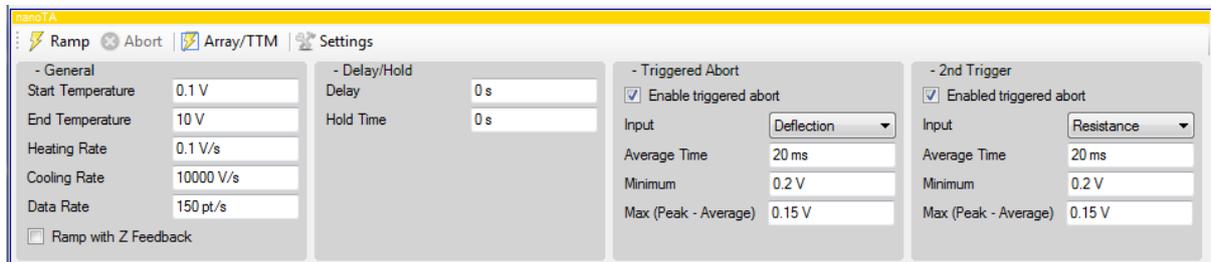


Figure 2-6: Reasonable ramp settings for uncalibrated mode. Parameters in [V]

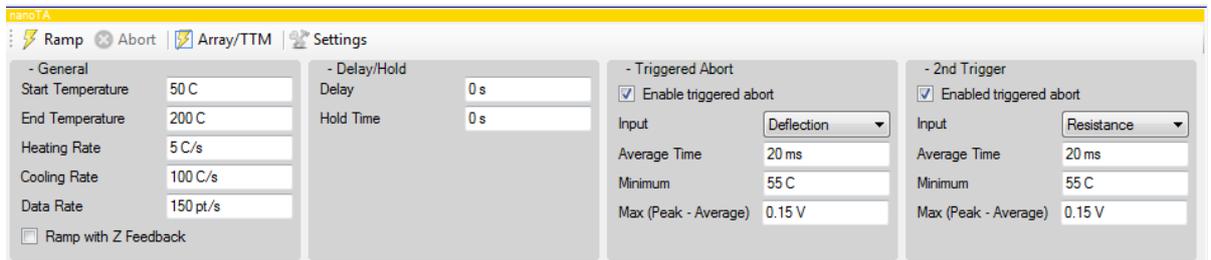


Figure 2-7: Reasonable ramp settings for calibrated mode. Parameters in [°C]

Start a thermal ramp by selecting the Ramp button in the toolbar at the top of the nanoTA panel. The system will perform a thermal ramp, which does the following (given the settings shown in above figure):

- The AFM's Z (height) feedback is turned off.
- The heating voltage to the probe is ramped.
- The ramp is stopped when any of the following conditions are met:
 - The heating voltage reaches the specified 'End Temperature' (ex. 10 V).
 - The user hits 'Abort'.
 - Trigger 1 is reached (above example: Deflection decreases by 0.15 V, nominally from a thermal transition in the sample).
 - Trigger 2 is reached (above example: Resistance decreases by 0.15 V, intended to prevent significantly passing the resistance turnaround point of the probe).
- The heating voltage holds its value while the probe is retracted away from the surface.
- Once the probe is retracted, the probe is cooled.
- The Z feedback is turned back on. The probe returns into contact with the sample and is ready for imaging or more nanoTA.

At any time, the user can abort the ramp with the 'Abort' button. This is useful if any unusual behavior in the deflection or other electrical signal is observed. A graph of Deflection versus Heating Voltage will appear in the Document Window. Shown below is a typical curve on PET with an uncalibrated probe.

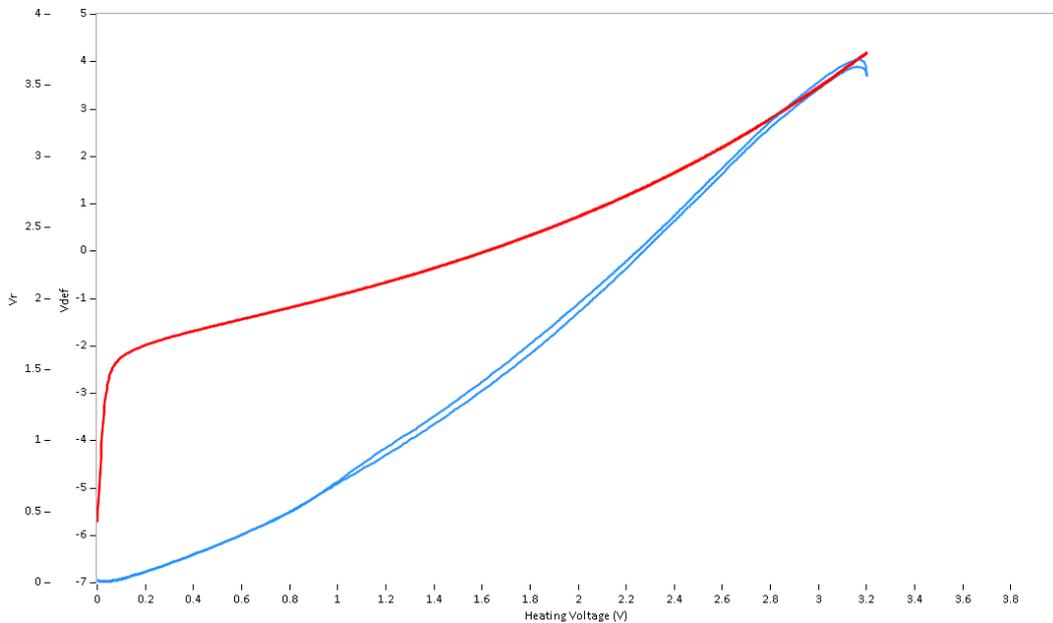


Figure 2-8: Example thermal ramps on the PET calibration sample.

When a thermal ramp is performed within the image map its location will be marked by a cursor on the map. The figure below shows an AFM image with cursors designating some of the nanoTA measurements.

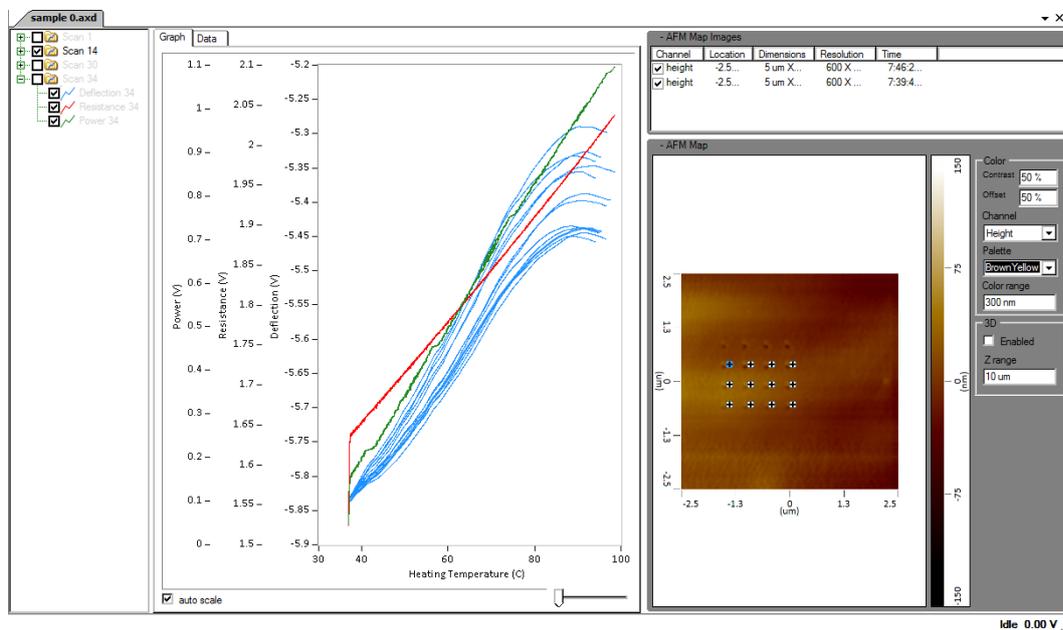


Fig 2-9: NanoTA document with nanoTA and AFM data.



CAUTION - The largest failure mechanism for the ThermoLever™ probes is from penetrating too far into the sample surface and either being irreversibly contaminated or becoming stuck in the sample surface such that they cannot be retracted.

If the probe is still in contact with the surface when it is cooled, it will stick to the surface. If a probe is stuck, the best way to remove it from the sample surface is to apply a heating voltage above the transition temperature of the sample and then withdraw the probe (using the withdraw up-arrow button in the AFM probe panel).

If you do not see an abrupt decrease in the deflection signal on the PET with the settings used above, there may be a problem with the probe or you may need to increase the voltage applied to the probe. Other possible problems can be that the probe may not be connected or may be electrically open, in which case the resistance signal will be at the maximum value. Alternatively the probe may be connected, but may not be on the sample surface, in which case, the deflection signal will not increase much and will not show a sharp melting transition on the PET. If this is the case, increase the setpoint value to make sure the tip is on the sample surface before starting the temperature ramp.

Hints for Optimal Performance:

1. **Do not ramp the temperature very fast**, you might go past the transition temperature and the tip will penetrate deeply into the sample surface. We recommend not using a heating rate higher than 1 V/sec until you are an expert on the system operation.
2. When ramping the temperature, the Deflection signal might reach the upper limit of the signal. This is more common with materials that have higher melting temperatures and high coefficients of thermal expansions. **To avoid Deflection railing**, set the initial vertical deflection of the AFM at some negative number like -4 or -5 V and then set the setpoint just slightly more positive. This will give you more range. Also, if you are ramping slowly increasing the heating rate will give less time for heat to diffuse into the sample and therefore the thermal expansion will be less.

Chapter 3

Controls

3.1 NanoTA Panel

The nanoTA panel contains the parameters that control how the thermal ramp is performed. This section gives descriptions for the parameters and buttons on this panel.

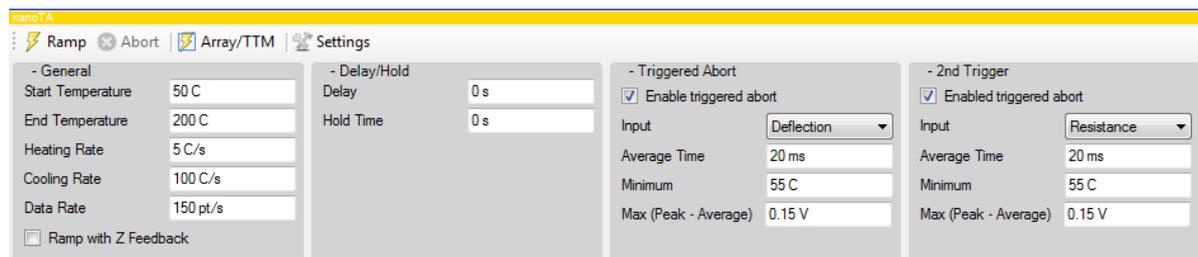


Figure 3-1: The nanoTA panel when the probe is calibrated. Parameters in [°C]

Start Temperature - The Start Temperature is the value of the heating voltage (uncalibrated) or the probe temperature (calibrated) at the beginning of the thermal ramp. The range is 0 to 10 volts. The corresponding temperature range is unique to each calibration file.

End Temperature - The End Temperature is the maximum allowed probe voltage/temperature of the thermal ramp, though the ramp is often stopped before reaching the end value by one of the triggers or a manual abort. The End Temperature must be greater than the Start Temperature and has a range of 0 to 10 V. Typically the End Temperature used will be less than 10 V to help prevent damage to the probe. Most sample materials have a transition at a lower value and the probe needs to be kept below a certain voltage to prevent damage to the probe. The maximum applied voltage for a given probe is defined by its resistance turn around as covered in the Thermal Probes Section 1.2. The ThermoLever™ probes are doped silicon and have a maximum power which can be applied before there is a chance of damage to the probe.



CAUTION – If too high of a voltage is applied to the probe it can be irreversibly damaged. Minimize the End Voltage/Temperature just so that the transitions of the material to be measured can be seen.

Heating Rate - The Heating Rate controls how quickly the temperature is increased. This needs to be set such that the transitions are clearly visible, but also so that the heating is limited to as small an area as possible. A typical value for this is 0.05 – 0.2 V/s in the uncalibrated mode or 1 – 10 C/s in the calibrated mode. Another factor in determining the heating rate is the Data Rate. If the Heating Rate is set too high in relation to the Data Rate the resolution of the scan will be poor. More information on this subject is included in appendix B.

Cooling Rate - The Cooling Rate is less important since the probe is cooled while it is off the sample surface and no data is taken during this time. It is typically set to a value 50 times higher than the Heating Rate.

Data Rate – The Data rate is an important parameter in controlling the data collection. The Data Rate will typically be set to a value where there are at least several data points per each degree. A typical rate of 100 points per second will work unless the Heating Rate is set significantly higher than 10 deg C/s. The maximum value is 20,000 pt/s, but this should only be used when the Heating Rate is set high enough so the total ramp time is less than a second, otherwise the experiment and saved data will be memory intensive.

Delay & Hold Time - The Delay and Hold Time are delays before and after the ramp respectively. The units are seconds and the default value is 0 S. These can be used if there is a need for a pause in the temperature at either the beginning or end of the scan.

Triggers - The Triggered Abort parameters allow the user to set parameters and enable two triggers which will cause the abort to happen automatically. This feature is enabled by checking either box labeled “*Enable triggered abort*”. The feature monitors an average value of one of the data channels. The average time is set by the *Average Time* parameter and is typically set to a value of 20 ms. It can be set to a lower value if the Heating Rate is set to a very high value. Increasing this value will reduce noise sensitivity and cause the temperature ramp to continue further past the transition. Each average value is compared against the previous value. If the sign of the *Max (Peak-Average)* value is positive and the new average value is higher than the previous average it replaces the previous average to become the new peak value. If it is lower it compares the difference to the *Max (Peak-Average)* to see if it is greater. If so, it aborts the scan. In this way the system will ramp the temperature until the selected input signal reaches a peak value and then decreases by the *Max (Peak-Average)* value. If the sign of the *Max (Peak-Average)* value is negative, then the input signal should initially decrease and then increase at the transition temperature. The Input parameter allows the Triggered Abort to operate on any of the channels. Typically, the Deflection channel is selected for the first trigger and the resistance channel is selected for the second trigger. The final parameter in the menu, the *Minimum Value*, prevents the trigger from occurring during the initial part of the temperature ramp. Often during the beginning of the temperature ramp there will be some change in slope that could cause an unwanted trigger to occur. This value needs to be set above where the changes in slope occur but below any potential transitions.

NanoTA Toolbar

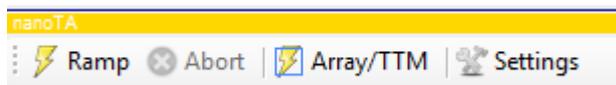


Figure 3-2: The nanoTA panel toolbar buttons.

Ramp - initiates a thermal ramp.

Abort - aborts a thermal ramp.

Array/TTM – accesses a new window which allows automatic acquisition of multiple ramps. Measurements are located within an AFM image and can be arbitrary points, a line array, or grid array. Selecting ‘Acquire’ initiates a measurement at each consecutive location. With a grid selected, there is an option to create a TTM (transition temperature microscopy) which is a false color map of the detectable transition temperatures across a sample surface (see Advances Techniques section).

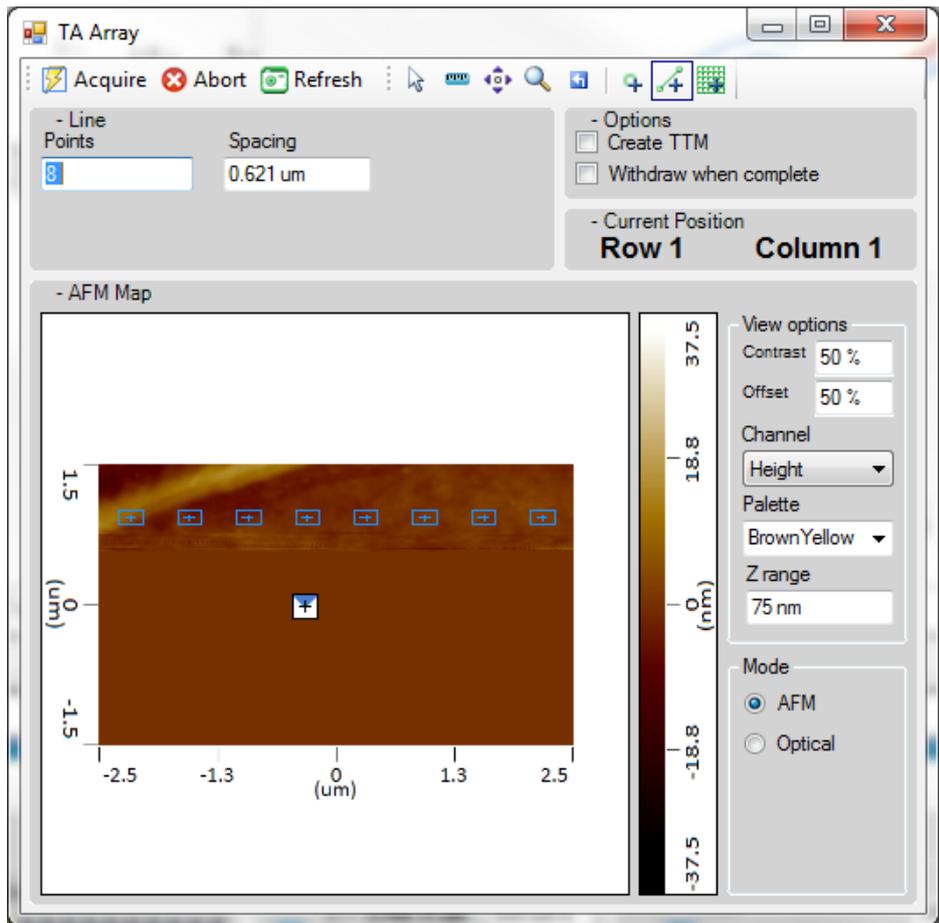


Figure 3-3: The TA Array Window.

Settings button – opens the nanoTA Settings menu shown below. Here, the user selects which signals to collect during nanoTA. This is also where Thermal or Z Feedbacks can be enabled, which are covered in the Advanced Procedures chapter.

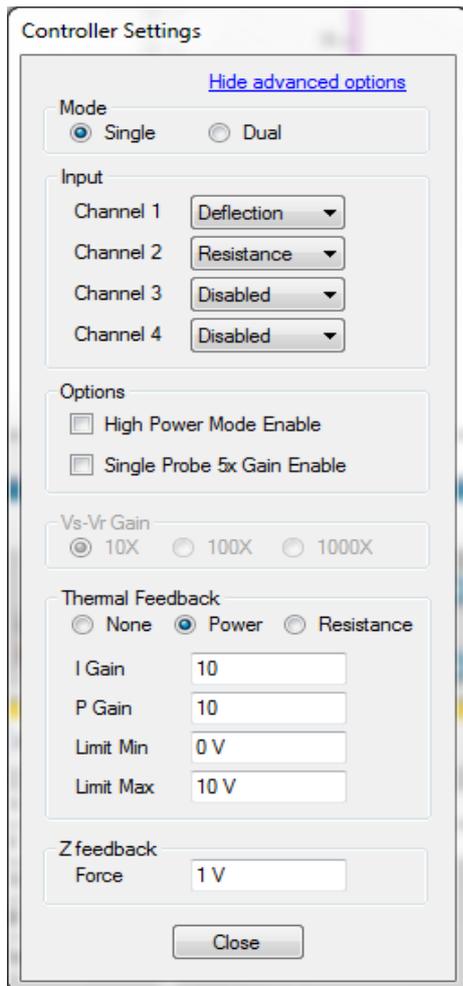


Figure 3-4: The nanoTA Settings menu.

NanoTA Signals

Following is a list of the nanoTA channels and their descriptions. It is helpful to refer to the nanoTA Circuitry figure below.

Heating Voltage (V_{heat}) – the voltage applied across the fixed resistor and probe.

Current (V_i) – a voltage proportional to the current through the probe (calculated by subtracting Probe Voltage from the Heating Voltage).

Probe Voltage (V_s) – the measured voltage across the probe.

Resistance (V_r) – a voltage proportional to the resistance of the probe (calculated by dividing the Probe Voltage by the Current).

Power (V_{pwr}) – a voltage proportional to the power through the probe (calculated by multiplying the Probe Voltage by the Current). Power can be used as the input to a feedback loop to ramp the probe's power instead of voltage.

Deflection (V_{def}) – the deflection of the probe.

Z Feedback Output ($FbOut2$) – the output of the Z feedback loop (same as the height channel for imaging) which is relevant during thermal ramps if the “Ramp with Z Feedback” option is selected in the nanoTA panel.

Sum – the sum from the AFM Photodetector.

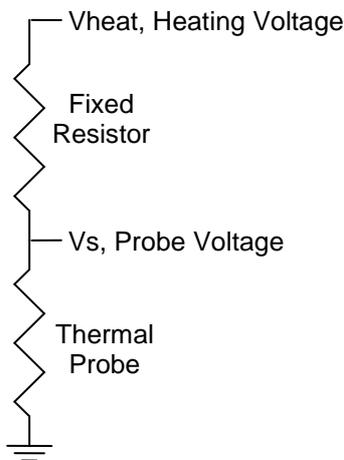


Figure 2-16: A diagram of the nanoTA circuitry.

3.2 NanoTA Meter

To the right of the nanoTA panel is the NanoTA Meter which allows the user to monitor the nanoTA input channels and also directly set the voltage/temperature on the probe. The probe voltage/temperature can be set by typing a value into the box labeled Heating Voltage and then hitting enter on the keyboard or clicking on the Update button on the meter. This function can be used to check that the probe has a resistance within the expected range. It can also be used to set the probe to a specific temperature for the Heated Tip AFM (HT-AFM) mode in which the system performs the normal AFM imaging while the probe is at an elevated temperature. The Channels displayed in the meter are those that are selected via the Settings button on the nanoTA panel (shown above).

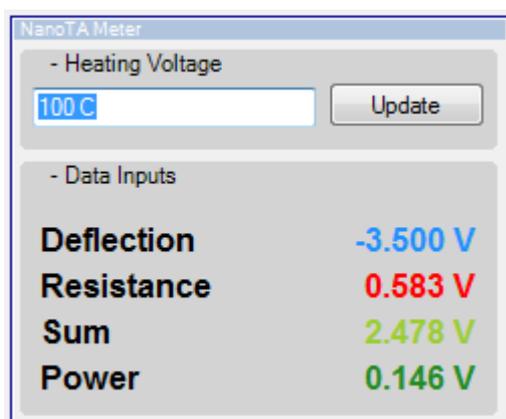


Figure 3-5: The NanoTA Meter (calibrated mode).

Chapter 4

Tools

4.1 NanoTA Calibration

When to Calibrate

The ThermoLever™ probes should be calibrated for temperature accuracy *once every day* or more often, if you suspect that there has been a change in the probe, such as damage or a significant change in its angle to the surface. This section provides the instructions needed to temperature calibrate the probe.

As with any analytical instrument, it is important that you calibrate the probe under the same conditions as those used for running your samples. The Analysis Studio software allows you to create, store, and recall multiple calibration files that you can use for different experimental conditions. If any of the experimental conditions change, or you change the sample, you should check your temperature calibration using one of the calibration samples provided by Anasys Instruments and recalibrate the probe as necessary.

The nanoTA Calibration Sample kit contains three polymeric calibration materials with sharp melting points. These are PCL ($T_m=55\text{ }^\circ\text{C}$); HDPE ($T_m=116\text{ }^\circ\text{C}$) and PET ($T_m=235\text{ }^\circ\text{C}$). All of these values are the onset of melting in these samples. Shown below is a picture of the nanoTA Calibration samples which should have been provided with your system.



Figure 4-1: Calibration Sample kit.

Additionally, nanoTA can be run in a number of different modes. The procedure detailed below covers calibration for the most basic nanoTA method. This will work well for a broad range samples that have higher transition temperatures as well as ones which are close to ambient. It is important when using this mode that the samples are relatively flat. If you have samples that have a significant amount of roughness, we suggest operating in the Resistance feedback mode. Using either feedback method requires its own calibration in that mode. There are more details on the operation of the feedback modes and the benefits of these modes in Chapter 6.4. The Power feedback mode is the default mode that we recommend currently.

Performing the Calibration

1. Load the supplied PET calibration sample.
2. Run a temperature ramp using the instructions from the Acquiring Data section, looking for the voltage corresponding to the melt.

3. We recommend that you run a minimum of 5 ramps to ensure repeatability. Each ramp should be separated laterally on the surface of the sample by at least 2 microns, so that the first ramp does not impact the next and so on.
4. Next run the HDPE sample and repeat exactly the same procedures as steps 2 and 3 above. The melt for HDPE will be seen at a much lower voltage than that of PET. We recommend that to prevent possible contamination of the probe, the tip is retracted from the sample surface and the scan aborted as soon as the transition temperature is reached. This is best done using the Triggered Abort functions.
5. Finally run the PCL sample using the same procedure as above. Again this sample will have a significantly lower voltage for the transition temperature in relation to the HDPE.
6. Go to the Tools menu and then click on the nanoTA calibration/New function. This will open the Calibration Window.
7. Select the cursor which is furthest to the right in the plot and position it at the average transition point for PET. The heating voltage corresponding to that cursor position should be displayed to the left of the 235 temperature in the Calibration fit box.
8. Next position the middle cursor on the peak for the HDPE and confirm that this voltage value is displayed next to the 116 temperature. Position the final cursor on the peak for PCL and confirm the correct voltage is displayed next to the 55 temperature value. These temperature values can be changed by double clicking on the value and inputting a different temperature if you are using alternative calibration samples.
9. Once you have the three values, confirm that the fit type is set to Quadratic. Verify that the Fit Plot is consistent with what is expected.
10. Select "Save" to save this calibration to a file and enter a file name and location as prompted. This calibration file will be automatically loaded.
11. The parameters will now be in units of degrees C and the temperature ramp will plot in degrees C. You will need to check the values in the nanoTA panel to make sure they are reasonable values before performing any thermal ramps with the calibration (refer to the Acquiring Data section)
12. If you have any samples available that have flat, smooth topography, are uniform across the sample surface and have a sharp, repeatable melting point these could be used as calibration samples instead of the ones provided.

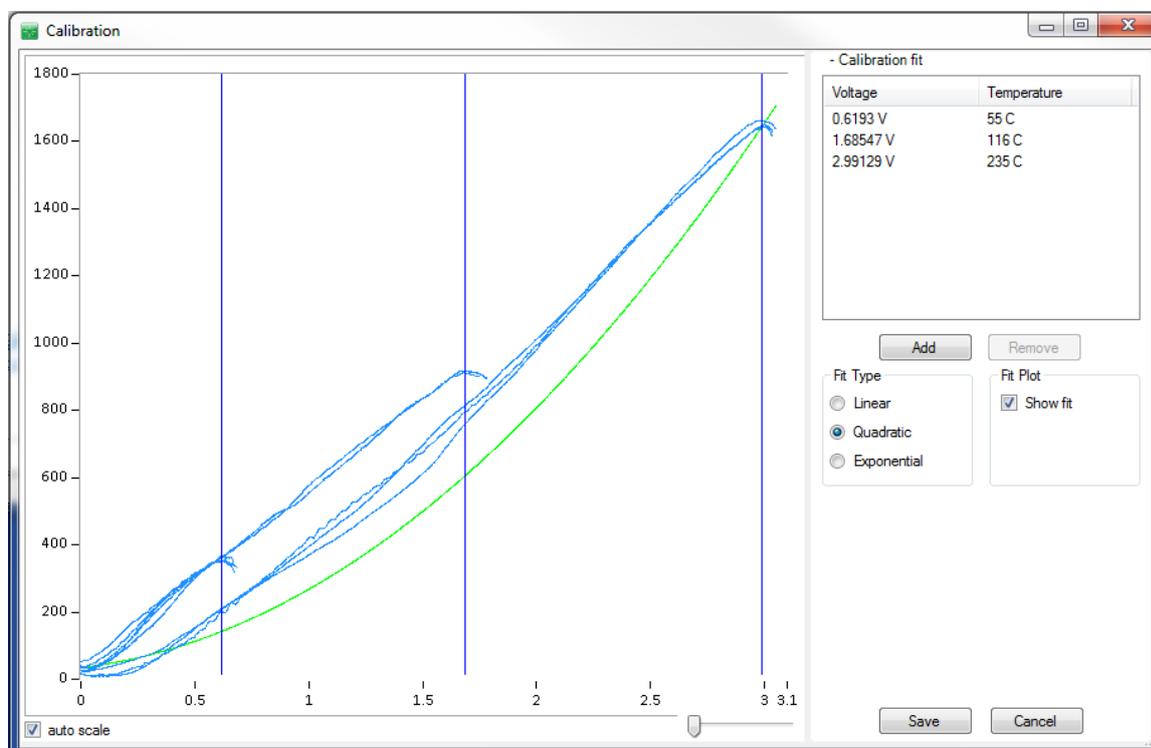


Figure 4-2: The Calibration window and data from all three calibration samples.

After the probe is calibrated, you can check the calibration to see if it is correct by running a temperature ramp on one of the calibration samples or another known sample. Shown below is a temperature ramp on the PET sample with a calibrated probe. Once the probe is calibrated the resistance and deflection signals should be relatively linear except for an initial start-up variation.

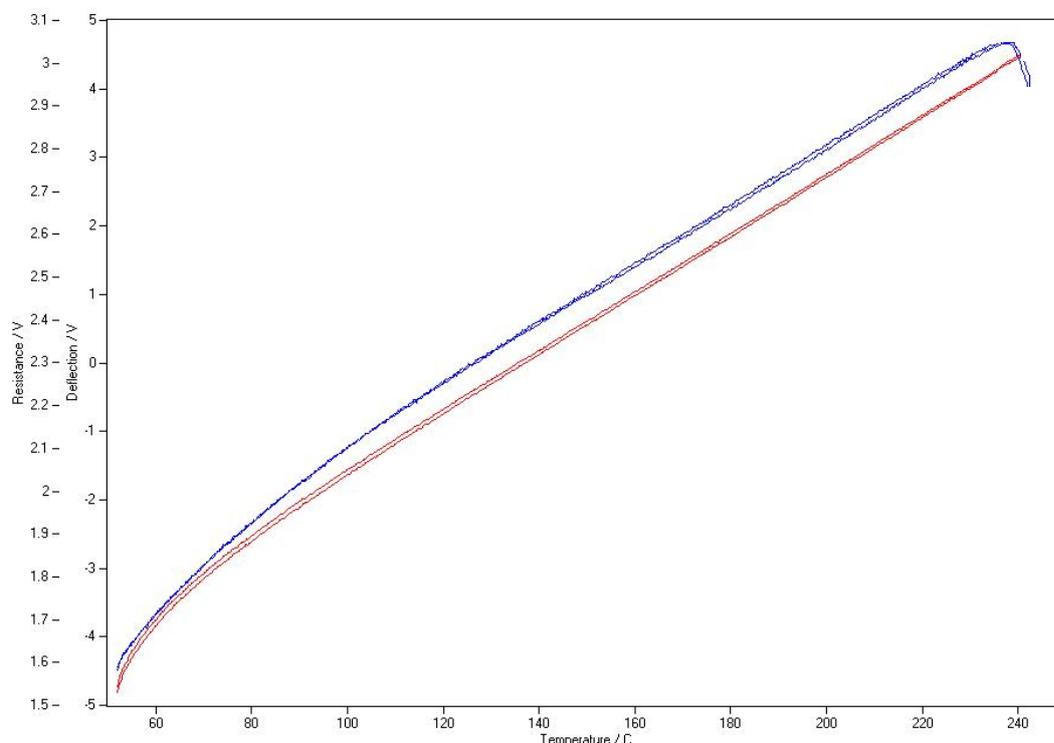


Figure 4-3: A temperature ramp on the PET calibration sample after the calibration has been performed.

Calibration Menu Functions

The following functions appear in the Tools/nanoTA calibration menu:

New – Accesses the Calibration window which allows the user to position cursors on the data shown in the current document to create a new calibration file.

Load – This allows the user to load a previously saved calibration file which will convert the voltage applied to the probe to degrees C. Once a calibration file is loaded, its name will be displayed on the status bar at the bottom of the Document window.

Clear – Selecting this function clears any calibration that is currently loaded which will convert the units of the software back to volts. This should be done whenever a new probe is first used so that it can be calibrated or if the calibration of the probe shifts. When the clear calibration function is selected the status bar should reflect that there is no active calibration by not displaying a calibration file on the status bar.

4.2 Clean Probe

In the event that the probe becomes contaminated, typically the AFM imaging resolution will become worse. The user can then make use of the Clean Probe function to try to remove the contamination from the end of the probe. When you press the Clean button in the Clean Probe menu, the voltage/temperature of the probe is raised to the selected value for the number of seconds set in order to burn off any contaminants that may have adhered to the probe. The Clean Probe function should only be used as a last resort to clean the probe. Quite frequently the probe can be cleaned by

performing another temperature ramp on the sample or changing to one of the calibration samples and performing a temperature ramp on that sample. Because the probes are silicon and a semiconductor they have a different response to increasing voltage on the probe. As the voltage increases the resistance increases, but at some voltage the resistance will start to decrease. This is due to the fact that there exists a critical temperature at which the thermally-generated intrinsic carriers exceed the doped carriers. Below this temperature, the main factor to affect the resistivity is the mobility of the doped carriers. Since the mobility decreases with temperature, the resistivity of doped silicon increases with temperature. Above the critical temperature, however, increasing carrier concentration overcomes decreasing mobility, resulting in the decrease of the resistivity. If the voltage continues to be increased at this point a significant amount of current can flow in the probe causing significant heating. This is OK if this voltage is held for a very limited time and the current limiting resistor in the bridge box has a higher value than the actual probe. But if the voltage is held for any significant time, the probe can reach very high temperatures which can change the resistance or even seriously damage the probe. Because of this it is recommended that initially the voltage/temperature is set very low and gradually increased. Also the time should be kept to a few seconds or less. After finishing the probe cleaning, it is also best to recalibrate the probe as typically the calibration will change with cleaning.

On some types of samples the probe becomes more easily contaminated and may become contaminated enough such that the calibration shifts significantly after only a few hundred temperature ramps. In this event we recommend that the Auto Clean function is enabled to prompt the user after a preset number of temperature ramps to clean the probe. We find that as long as the voltage applied is kept below the resistance turnaround value the change in calibration is minimal.

Clean Probe Functions

The Clean Probe window is accessed through the Tools menu.

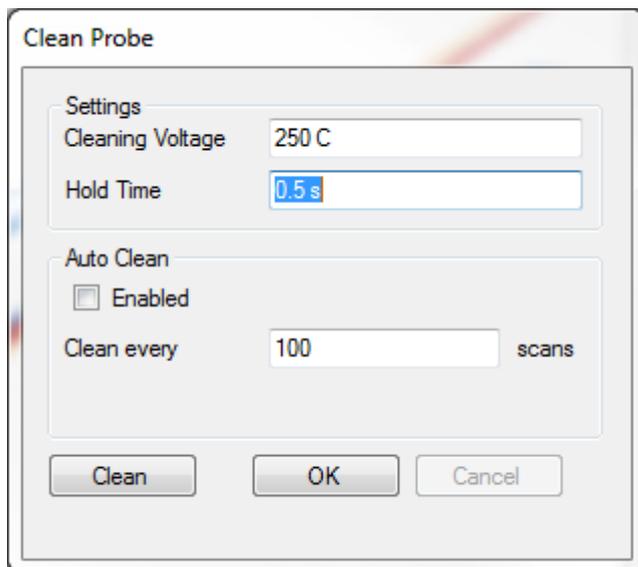


Figure 4-4: The Clean Probe window

Cleaning Voltage – Voltage or temperature of the probe during cleaning, depending on whether the probe is calibrated or not.

Hold Time - is the length of time the probe is held at that voltage/temperature. Typically set to a value of less than 1 s.

Auto Clean - If the Auto Clean Enabled checkbox is checked, the software will track the number of temperature ramps performed and when this exceeds the number of scans input will prompt the user to perform a clean probe function. During an array, the probe will be automatically retracted, the probe will be cleaned and the probe brought back into contact with the sample surface. On some samples this can be helpful in maintaining the calibration accuracy as the probe picking up material will typically shift the calibration of the probe more than performing a clean probe function. Care

should be taken when using this function to not select a voltage or temperature which is significantly above the resistance turnaround of the probe.

Clean button – Starts the cleaning process

Cancel button – Aborts the cleaning process

OK button – Closes the window without initiating the cleaning process.

Chapter 5

Advanced Procedures

5.1 Z Feedback

The standard method of nanoTA turns off the afm+ Z feedback during a temperature ramp. This method works well to prevent the tip from penetrating too far into the sample surface. The tip will typically only penetrate until the deflection in the cantilever caused by the thermal expansion of the sample and the original deflection caused by the setpoint value are gone. One disadvantage to this technique is that the force will increase as the temperature increases due to the increased deflection of the probe. Another problem that can arise is if the sample has a large coefficient of thermal expansion, the deflection signal can reach the limit of its range before the transition temperature is reached. Shown below is a temperature ramp in which the deflection signal is out of range due to the large thermal expansion of the sample and the high transition temperature of the sample. If this occurs, the best technique is to first decrease the deflection of the cantilever when it is off the sample surface to a value that is as low as possible. This will give more range for the deflection signal to increase.

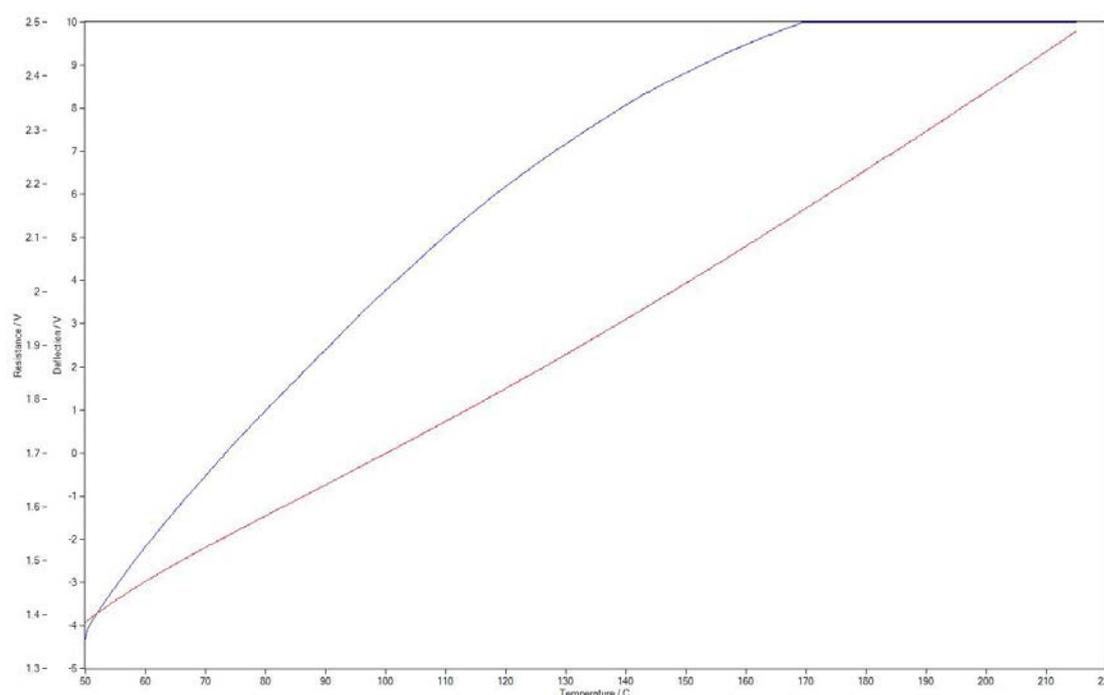


Figure 5-1: A temperature ramp in which the deflection signal is out of range.

If the deflection signal is still out of range, the user can try the alternative method of keeping the Z feedback turned on. This is done by selecting the 'Ramp with Z Feedback' option accessible from the Settings button in the nanoTA panel. The disadvantage to this technique is that if the transition is sharp the probe can penetrate more into the sample unless a Triggered Abort is used. When using the triggered abort with the Z feedback the parameters for the trigger will need to be changed. Default values are shown below. The Average Time and Max (Peak – Average) parameters may need to be optimized for the specific probe and sample. The negative value for the Max (Peak – Average) parameter is because the Z Feedback Output signal typically decreases with increasing expansion of the sample, so the curve will start going downwards and then go up at the penetration of the probe into the sample.

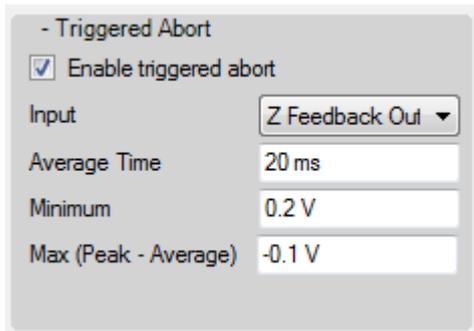


Figure 5-2: The Triggered Abort parameters when operating in the Z Feedback mode.

There is one additional parameter to set when using the Z Feedback mode. This parameter is in the Settings menu and is called Z feedback Force. This parameter is used to reset the Setpoint during a temperature ramp when the “Ramp with Z Feedback” checkbox is checked. When ramping with the Z feedback, the probe can pop off the sample surface if the thermally induced bending causes the deflection of the probe to be higher than the Setpoint. A positive Z Feedback Force can prevent this from occurring. The exact value of this parameter will depend on the sample transition and the probe so it is best to start with a value of 1 V and then increase the value as needed until the thermal transition is seen before the tip pops off the sample surface.

5.2 Thermal Feedback

There are two options for using thermal feedback during nanoTA: Power and Resistance feedback. These operate a feedback loop with an input that is either the power applied to the probe or the resistance of the probe. This section of the manual will describe the benefits to each mode.

In nanoTA operation with no feedback and without a calibration loaded, the heating voltage to the probe is linearly ramped. The temperature of the probe is roughly proportional to the square of the voltage. The calibration procedure uses a quadratic fit to establish the relationship between the heating voltage and temperature. The calibration determines the shape of the voltage ramp used during subsequent data acquisition (calibrated mode), with the intent to ramp the temperature linearly with time. No feedback is involved in this method.

Power Feedback

When taking near ambient temperature measurements (65 °C or below) it is best to use the power feedback mode. Power feedback gives more accurate temperatures and heating rates in the low end of the system’s temperature range. In power feedback, the power (rather than voltage) to the probe is ramped. This is done using a feedback loop (on the DSP in the Controller). The input to the feedback loop is the power, the set point is the heating voltage, and the output (Feedback Output in the software) is applied to the fixed resistor and probe combination. In this way, the power to the probe is ramped along with the heating voltage. As before, the calibration procedure establishes the relationship between the heating voltage and temperature. The calibration curve is different than in standard operation because the temperature is much more linear with the power.

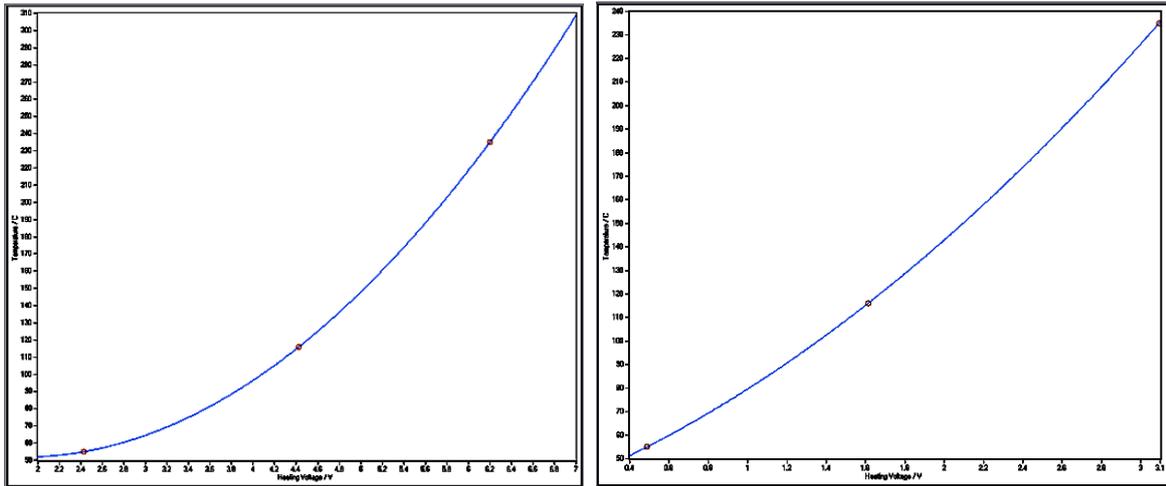


Figure 5-2: Calibration fits - temperature vs. heating voltage, standard operation (left) and power feedback (right)

Figure 2 shows the calibration fits from calibrating the same probe in standard operation and in power feedback mode. For standard operation, the 55 °C transition of the PCL sample occurs near 2.4 V with this probe. The curve is clearly not valid as a calibration at voltages much below that point because the parabola reaches its minimum and would turn upward again at lower voltages. The minimum is at 1.8 V corresponding to 51.8 °C which is the minimum temperature allowed in the ramp parameters for this particular calibration. A transition occurring below this minimum accessible temperature will not be observed or will be distorted. Typically it looks like an apparent transition immediately at the start of the data or the deflection is already going downward when the data begins (since the probe is already pushing down into the softened material).

This limitation of the calibration at near ambient temperatures also affects the initial heating rate of the probe. Starting a ramp at the minimum temperature, the probe voltage will jump immediately from 0 to 1.8 V and the heating from ambient to 51.8 °C will not be done at a controlled rate (the rate will be determined by the thermal conductivity and contact of the probe and sample). Thus the probe and sample may not be in thermal equilibrium for some period at the beginning of the ramp, adding to the error in the calibration at low temperature.

The power feedback mode does not have these issues. The minimum of the calibration curve occurs well below 0 V (Figure above) so there is no artificial minimum temperature. The curve crosses 0 V at 36.5 °C, which is the minimum temperature available in the ramp parameters; corresponding to the ambient temperature of this specific probe and environment (the AFM laser heats the probe above general room temperature).

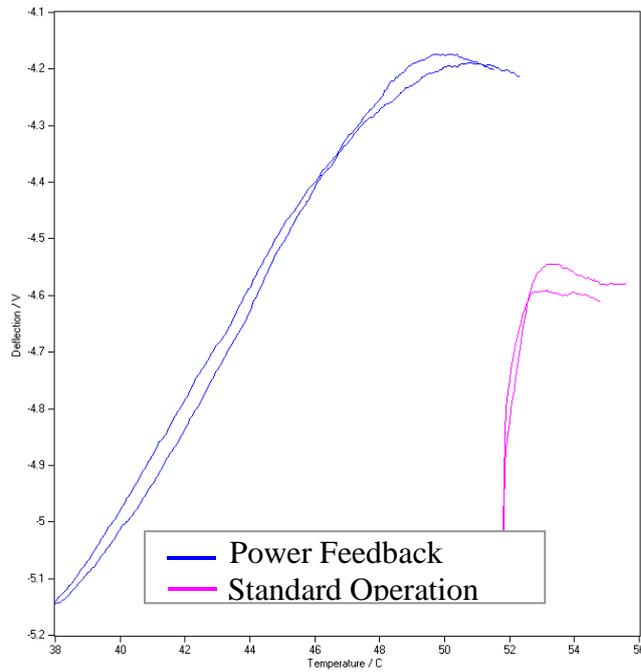


Figure 5-3: Nano-TA data on a material with a low temperature transition (1 °C /s)

The figure above shows nanoTA data taken on a material with a low temperature transition using the same probe for both modes. From the power feedback data (blue) we can see the transition occurs at 50 °C, which is below the minimum temperature accessible with standard operation. The peak at 53 °C in the standard operation data (pink) is misleading. It takes some time for the probe to heat up at the beginning of the ramp, which shifts the peak to a higher apparent temperature.

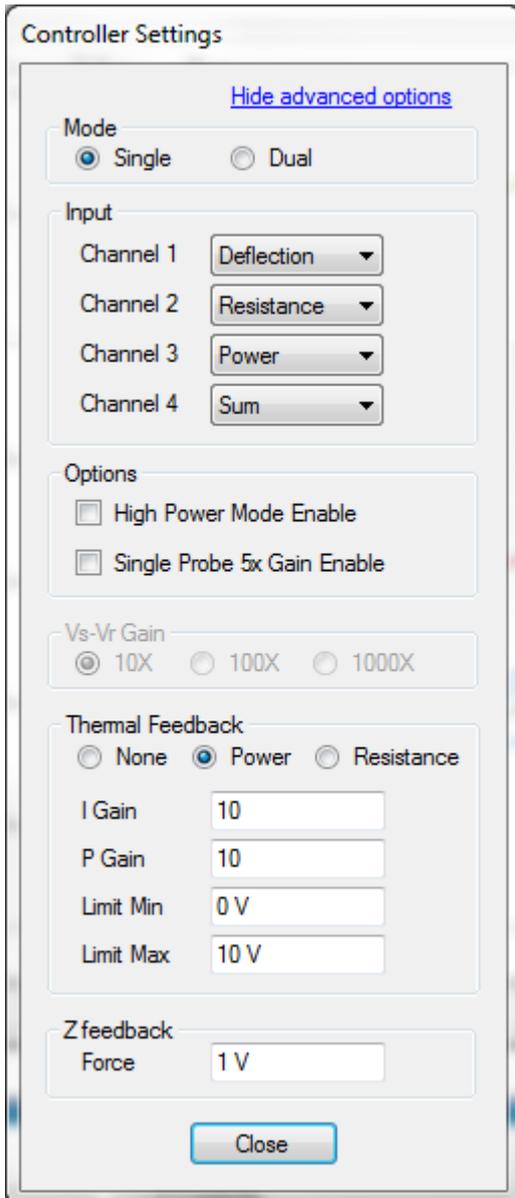


Figure 5-4: NanoTA Settings for Power Feedback

To use power feedback, make sure there is no voltage on the probe and clear any existing calibration. Select the Settings button in the nanoTA panel toolbar. The Mode remains set to Single just as in standard nanoTA. Set the Thermal Feedback to Power and set the I and P Gains both to 10.

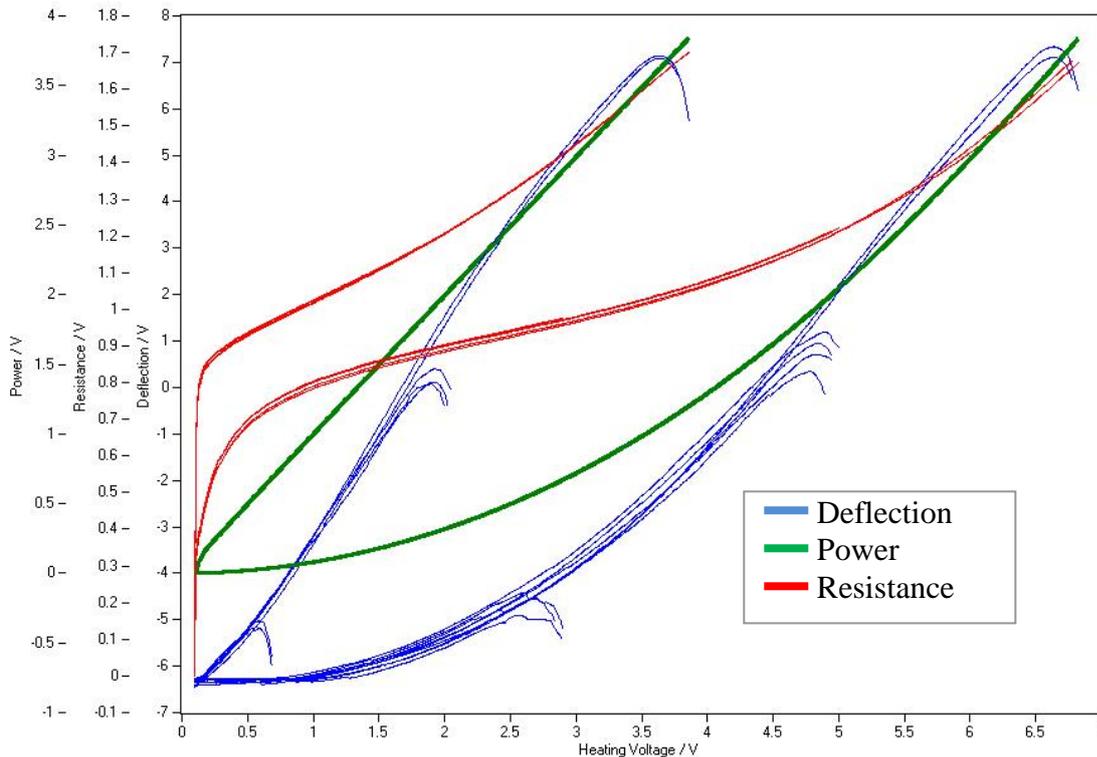


Figure 5-5: Nano-TA data on the three calibration samples. The right set of curves the feedback is set to None; the left set is Power feedback.

Calibrate the probe using these settings. Though the temperature is much more linear with power, it is not enough so that a linear fit can be used. A quadratic fit must still be done using all three calibration samples. Note that the gains can affect the calibration, so in the unlikely event that the gains need adjustment; a new calibration will be needed. If you are planning to use faster heating rates (>30 °C/s), you should set the feedback gains to higher values typically up to a maximum value of 15 so that the feedback can track the more rapid changes in the heating voltage. The process for data acquisition is the same as normal, just using the above settings and the appropriate calibration file. We recommend using slower heating rates, such as 1 °C/s, when measuring near ambient temperature features.

Resistance Feedback

In the resistance feedback mode, the resistance of the probe is used as the input to the feedback loop with the heating voltage used as the setpoint, then the output of the feedback loop is applied to the probe. This allows the user to ramp the resistance of the probe. Since the resistance of the probe is roughly equivalent to the temperature of the probe it is expected that this mode would produce the most reliable temperature measurements. This is true if the samples to be measured have significant roughness. In this case, the heat flow between the sample and the cantilever can be variable which can affect the temperature of the probe and therefore the temperature of the tip apex in either the No feedback or power feedback modes. In this case operating in the resistance feedback mode produces a more reliable measure of the transition temperature. We find that if the surface is relatively flat (<1 μm) the variation in heat flow does not significantly affect the calibration and either the resistance or power feedback mode can be utilized.

One challenge to the resistance feedback mode is that the resistance does not always increase with continued increases in the heating voltage, at the resistance turnaround point the resistance decreases with higher applied voltages. So above the resistance turnaround point, the feedback loop would drive the applied voltage to the maximum value when it should be decreasing the voltage. In order to prevent this we have added some limits to the heating voltage as well as a second trigger. When using the resistance feedback it is important to make sure to enable the second trigger with the correct values and to set the limits on the heating voltage. The following procedure describes how this can be accomplished.

Procedure for running with Resistance Feedback

1. Set up the system to run with no feedback with the tip far from the sample surface (>100 μm away).
2. Set one of the data channels to resistance and ramp up to the resistance turnaround point. Abort the ramp *immediately* after the resistance turnaround and cool the probe quickly to avoid damage to the probe. Using a Triggered Abort on the Resistance channel is highly recommended. The data should look similar to that shown below.

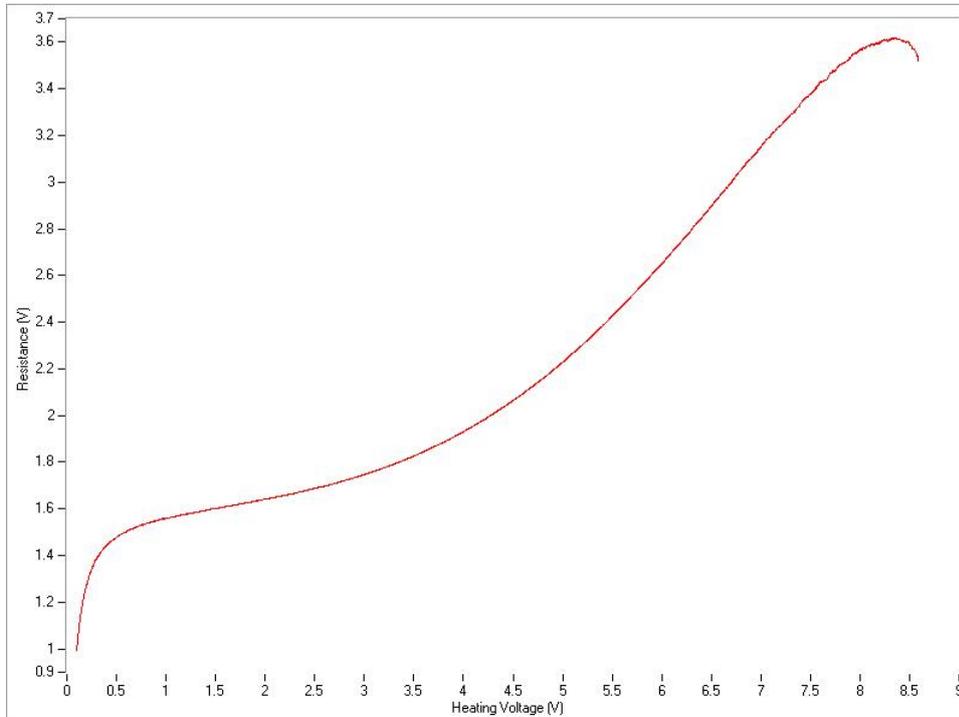


Figure 5-6: The Resistance data from a ThermoLever probe in the no feedback mode showing the resistance turnaround point at ~ 8 V.

3. Determine the Heating Voltage value where the resistance turnaround occurs. In the above data this is ~ 8 V. Also determine the Resistance value at the resistance turnaround and at a Heating Voltage of 0.2 V
4. Set the Start Temperature in the nanoTA panel to the value of the Resistance at a Heating Voltage of 0.2 V. In the above example this is ~ 1 V.
5. Set the End Temperature to a value slightly lower than the value of the Resistance at the Resistance turnaround. In the above example this is ~ 3.5 V.
6. Set the Heating Rate to a lower value than is normally used when operating with no feedback by approximately a factor of two. This is because the voltage required to reach a certain temperature is lower in the resistance feedback mode than in the standard mode. Then set the parameters for the second trigger value to the typical values shown below.
7. Select the Settings button on the toolbar of the nanoTA panel and set the Limit Min in the Feedback to 0.2 V and the Limit Max to the value of the Heating Voltage just before the Resistance turnaround. Confirm that the I gain and P gain are set to 10 and then turn on the Resistance feedback. The parameters should be like those shown below for this example.
8. Now start a temperature ramp. The Resistance data channel as shown in the data set below should have a linear increase. The grey line in the plot below is the Feedback Output showing the actual voltage out to the probe. As can be seen it ramps up to 8 V and then stops increasing. This is due to the Limit Max parameter and prevents the feedback from applying too much voltage so that the probe goes past the resistance turnaround point. This is one safeguard. The second safeguard is the 2nd trigger which is on the Resistance data channel. This is a less optimal safeguard because the trigger will not occur until the probe is past the resistance turnaround.

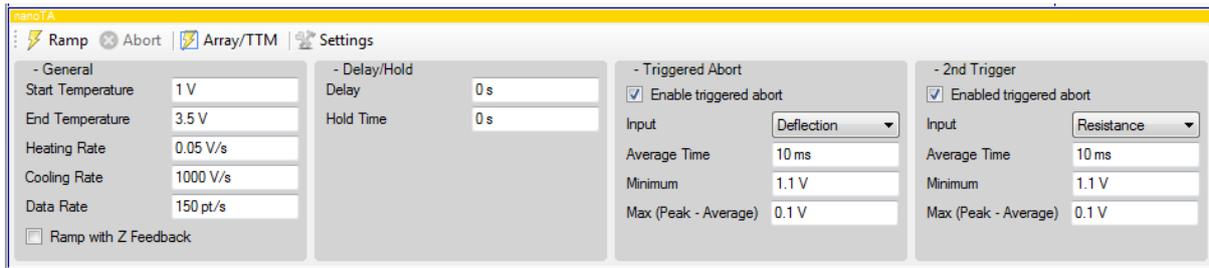


Figure 5-7: The nanoTA panel showing the settings for Resistance feedback mode.

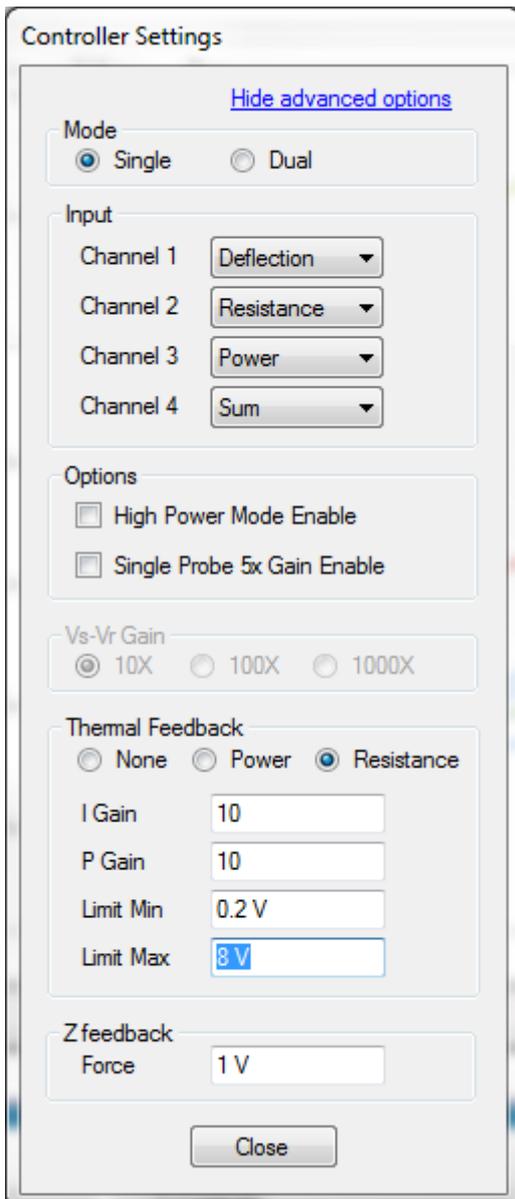


Figure 5-8: The nanoTA Settings menu for Resistance feedback mode.

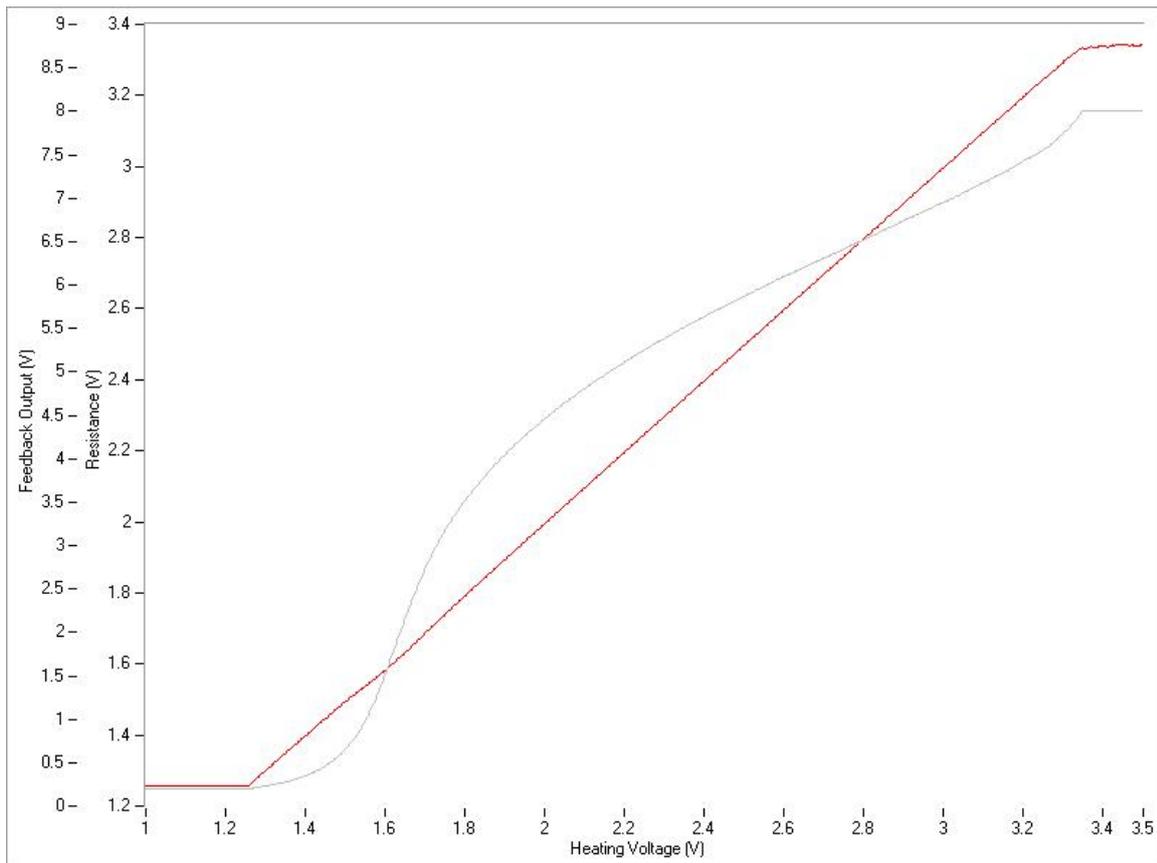


Figure 5-9: A temperature ramp showing the Resistance (red) and Feedback Output (grey) plots when using the Resistance feedback mode.

5.3 Fast Heating Rates

The system is capable of much higher heating rates than is described for standard operation. When doing fast heating rates you may need to set the data rate higher (its maximum value is 20,000 Hz). If you perform slower scan rates with the data rate set this high you can get curves in which the amount of data is very large. This will slow down any manipulation of the curve. Due to this it is recommended that you keep the number of data points in any one curve below 20,000 by setting the heating rate, heating range, and data rate to appropriate values.

At fast heating rates, it will be difficult to stop the heating of the probe at the transition temperature. We recommend that you either use the triggered abort function or set the end temperature such that the temperature of the probe does not go too high.

At very fast heating rates, it may be difficult to see the transition in the deflection signal. This will especially be true for softening transitions which may not be visible at all at higher scan rates. It will be easier if the transition is a melt transition. Due to the limitations of heat transfer from the probe to the sample when measuring with heating rates exceeding 100 °C/s, you will need to calibrate the probe at those rates. This will require estimating the calibration of the probe and setting the heating rate in the uncalibrated mode to a value that is somewhat equivalent to the ultimate calibrated heating rate desired.

5.4 TTM: Transition Temperature Microscopy

Arbitrary nanoTA measurement locations can be chosen using the TTM/array feature accessible in the NanoTA Control panel. The figure below shows a blank TA array. The user may select a multitude of individual points, a linear array, or a 2D array. If a 2D grid is selected the user will have the option to render the data as a TTM map or Transition Temperature Microscopy. When using the automated features it is very important to set appropriate end temperature and automatic triggered abort settings in order to capture a transition and to appropriately withdraw without damage to the thermal probe.

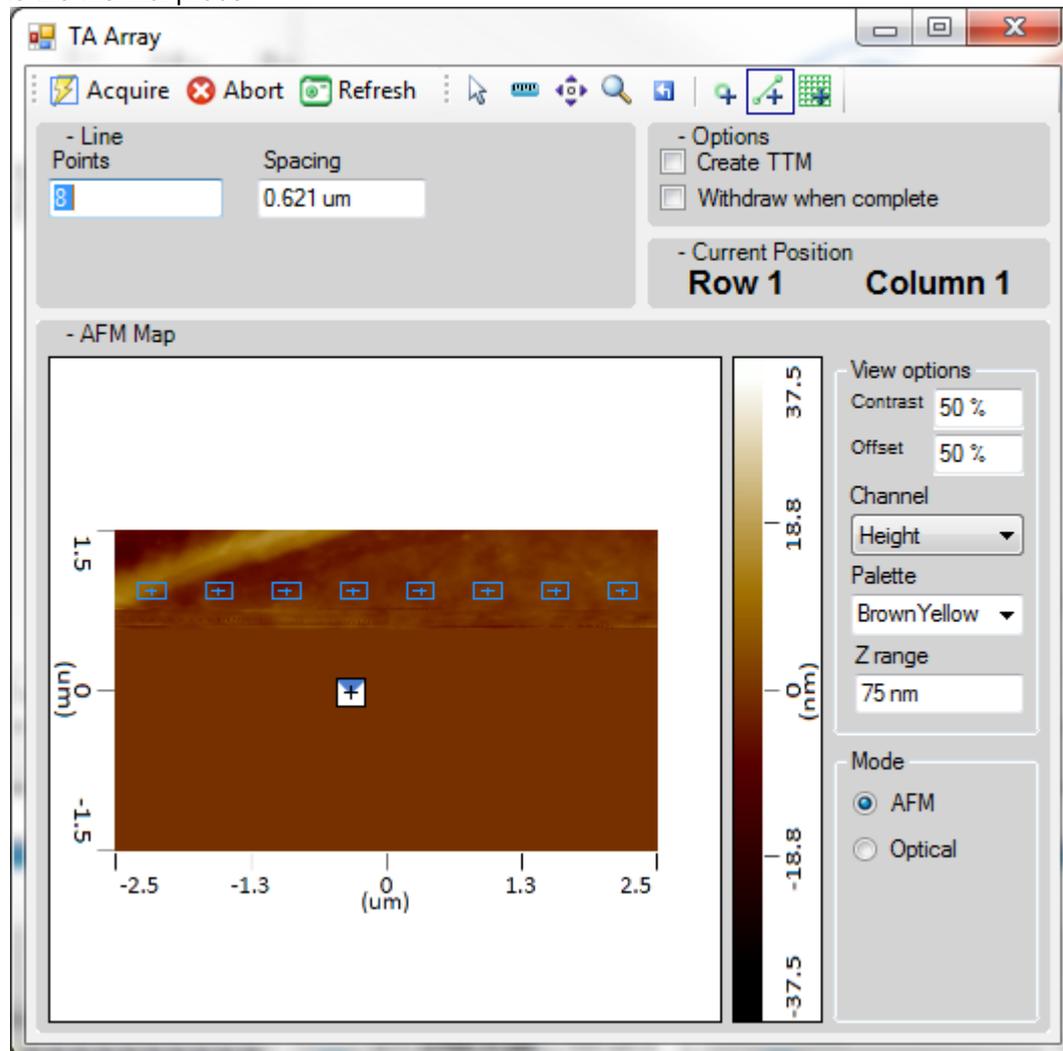


Fig 5-10: TA Array window.

Transition Temperature Microscopy (TTM) is a method that utilizes advanced automation and rapid measurement speeds to acquire numerous localized thermal measurements within a user defined region on a sample surface. The software automatically analyzes each thermal measurement to determine a transition temperature at each unique spatial location. The detected thermal transition is then color coded and placed in the TTM map which is a spatially resolved visualization of the transition temperatures on the sample surface. In a TTM map, the X and Y axis depict locations on the sample surface and the Z axis corresponds with a color bar which correlates a thermal transition temperature to a color.

To initiate a TTM map first select the *Array/TTM button* in the NanoTA Control panel toolbar. In the TA Array window, select the 'Create TTM' option. After each measurement the software will identify a peak in the nanoTA curve and store it at a pixelated location corresponding to a point on the map starting from the top left of the image and ending in the bottom right. The TTM measurements will begin when the user selects the Acquire button. To stop the TTM at anytime select any of the Abort icons located in Ansys Studio. If aborted, the user will only have access to the measurements acquired prior to aborting the TTM imaging. The remaining TTM points that were not completed will

remain empty. Before initiating a TTM image it is very important that the user review all settings defined in the control panel paying close attention to the triggered aborts.

After a TTM is acquired, the user will have the opportunity to view and manipulate various characteristics of the TTM map. First, the channel containing the thermal transition is selected. Next a user can choose between the different color palettes offered for the TTM map. The selected palette will define the color gradient that will be applied to the transition temperatures in the Z axis. The Z scale can be altered by defining a minimum and maximum value. The color palette can be offset within the minimum and maximum range by dragging the blue arrow up and down. The color contrast can be altered by dragging the green arrow relative to the blue offset arrow.

Because the TTM is composed of numerous thermal measurements the user may review the data for any point or points by selecting those locations on the map. The individual data curves corresponding to the selected locations will be displayed in the plot below the TTM map. Locations may either be selected by individually clicking on the TTM locations or by dragging a box around the desired region on the map. The 'Ctrl' key can be used to add additional measurements to an existing selection. The plot below the TTM map contains additional parameters used in the detection of thermal transitions. Using the range parameter, the user may choose to only search for thermal transitions above or below a specified temperature. The peak fit parameter determines the intervals within which the software will search for a turn-around or thermal peak. Sharper transition temperatures will require a smaller interval whereas longer gradual thermal peaks may require a larger peak fit parameter. The peak fit has an additional option for determining peaks that are highest or lowest in X or Y in the data plot. This is useful in filtering data curves that may contain multiple peaks such as in the case of a layered film. Lastly Peak displays either the temperature at which a thermal peak is detected or an average with standard deviation in the case that multiple TTM locations are selected. In order to calculate the average thermal transition temperature with standard deviation for the complete set of all thermal measurements present in the TTM map, create a selection box with the mouse selecting all the measurement locations in the TTM map. The data from all TTM locations will be displayed in the data plotter below the map with the calculated average for the thermal transition temperature over the entire region. Lastly, the TTM map can be exported to a graphic by going to File->Export->Image. An example of a TTM map is shown below.

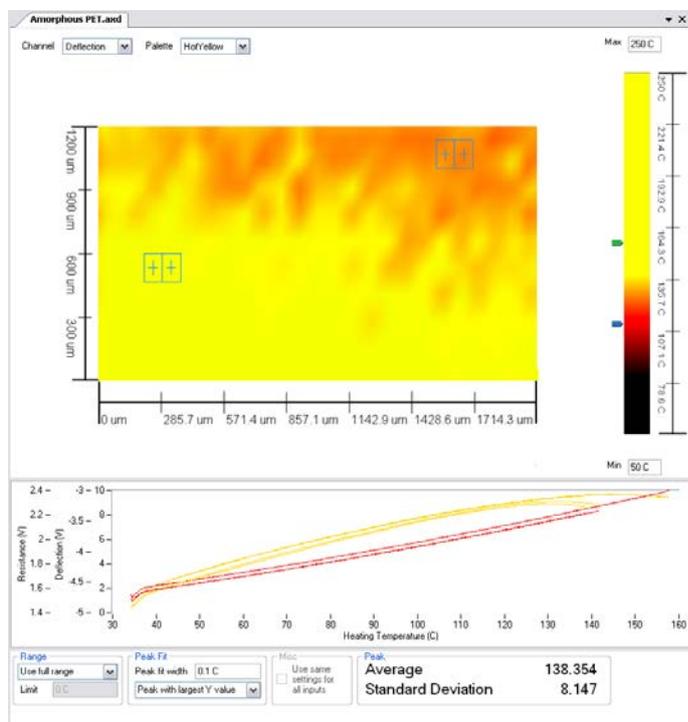


Figure 5-11: An example TTM map showing local variation along a sample surface. The plot below the map is displaying the measurement results of the four selected points on the map. The peak average and standard deviation is calculated for the selection in the bottom right hand side.

Chapter 6

SThM Mode

6.1 Introduction

The SThM mode is a mode where a special AFM tip is scanned across the sample surface in the contact mode. This tip has a resistive element near the apex of the tip as shown in the below SEM image.

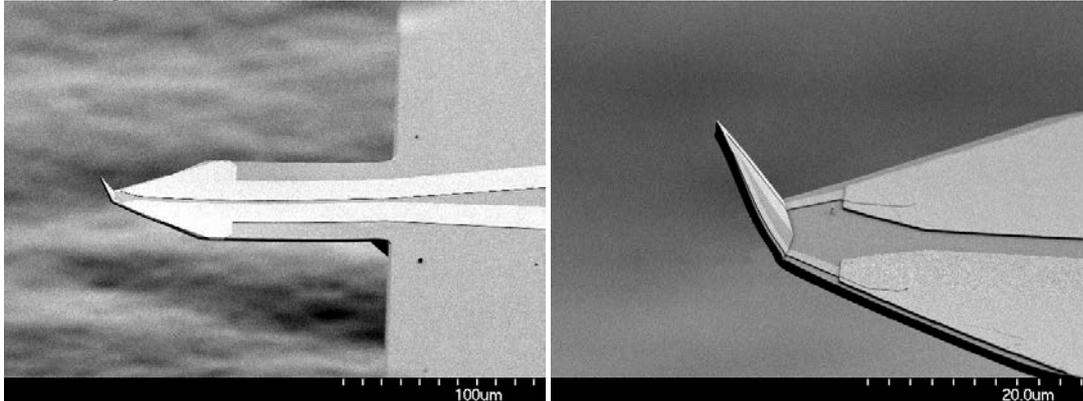


Figure 6-1: The SEM image on the left is the GLA style probe and the one on the right is a zoom of the end of the probe.

As the tip changes temperature due to variations in the thermal conductivity or temperature of the sample in contact with the tip, the resistance of this element changes. This resistance change is monitored by the hardware using a Wheatstone bridge circuit and can be output to the controller to generate an image. To run experiments, you mount the sample onto a substrate, place it on the sample stage, and obtain an image of the surface in the contact mode, following the normal AFM procedure but using a resistive thermal probe. Then the user applies a voltage to the resistive thermal probe, balances the Wheatstone bridge and measures a voltage which is characteristic of the resistance of the probe. This voltage displayed along with the other images in the Analysis Studio software. As example image taken on the SThM Test sample which is a carbon fiber/epoxy composite sample is shown below.

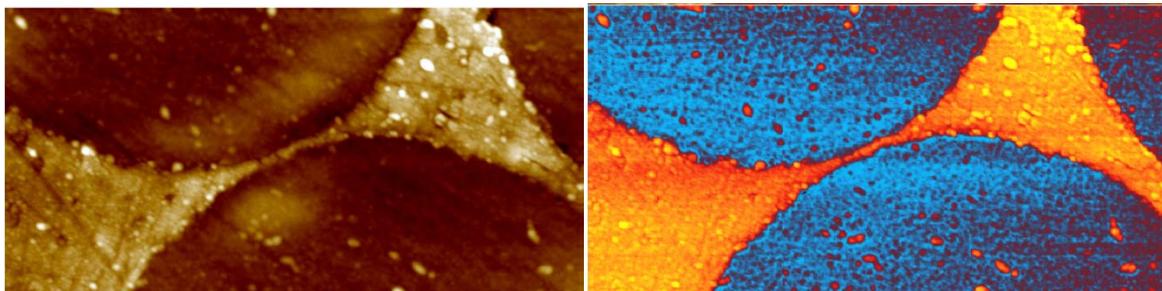


Figure 6-2: A $4\mu\text{m} \times 8\mu\text{m}$ SThM image of a carbon fiber – epoxy composite sample. The height image (left) shows a number of carbon fibers, while the SThM image (right) shows the change in probe temperature on the two materials due to their differences in thermal conductivity.

6.2 SThM Setup

AFM start up, Sample preparation, Laser alignment and Probe approach

Follow the procedure given in the afm+ or nanoIR manual for these steps. The laser alignment and parameter settings should be very similar to what is done for the ThermaLever or any etched Si contact mode cantilever. The only difference is we recommend that for both the engage and the imaging the force is minimized as much as possible. The metal film coating the apex of the tip used to measure the temperature is soft and can easily be damaged with repeated scanning with higher forces. This may mean only setting a very small difference between the vertical deflection signal and the setpoint.

The SThM probe will need to be plugged into the afm+ or nanoIR system using the same connector which is used for the nanoTA probes.



CAUTION – The SThM probes are shipped in an ESD box with a shorting connector between the two pins on the connector to the probe. Before the shorting connector to the probe is removed and the probe is handled, the user needs to be wearing a ground strap. The SThM probes are highly ESD sensitive and can be easily damaged. In addition any samples which will be imaged should be grounded or the static dissipated before the probe is engaged to the sample surface.



CAUTION – If too high of a voltage is applied to the probe it can be irreversibly damaged. The voltage applied to the probe should always be set to zero before plugging in or unplugging the probe.

System Configuration

Before running a SThM experiment, the Settings in the software need to be set so that the system is in the correct configuration. Shown below is the Settings menu. For SThM, the mode needs to be changed to Dual mode and the Thermal Feedback needs to be set to None. In addition, the Vs-Vr gain should initially be set to 10X.

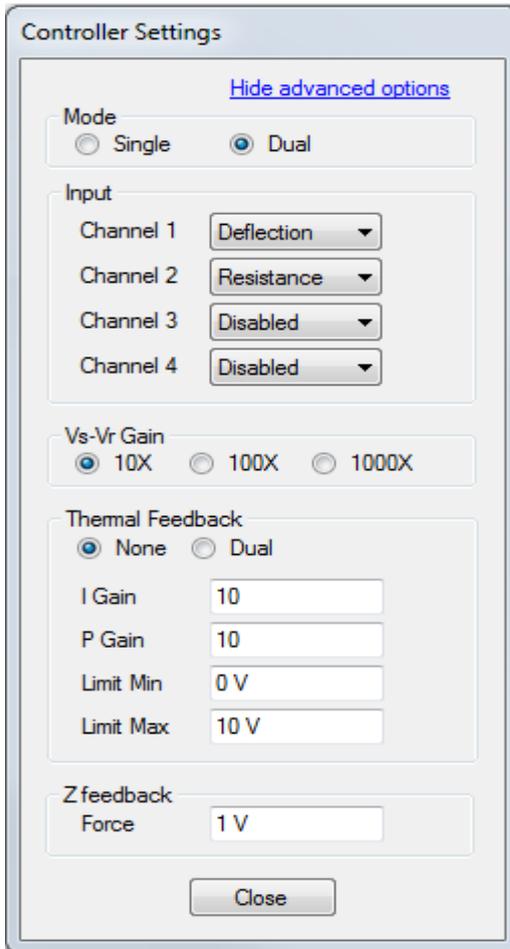


Figure 6-3: The Settings menu when running the SThM mode.

The Dual mode changes the electronics so that the system is using a full Wheatstone bridge like is shown in the following diagram.

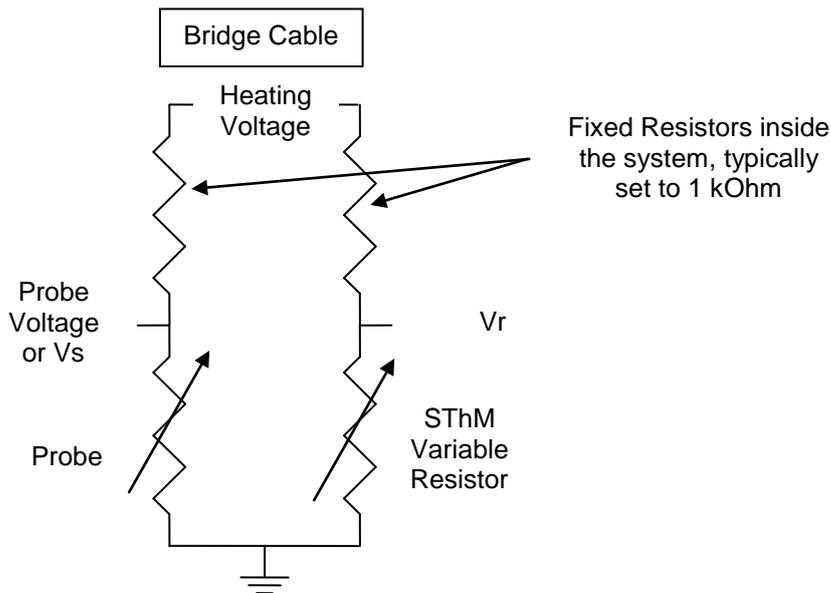


Figure 6-4: A diagram showing the signal names and configuration of the bridge circuitry.

Applying Voltage and Balancing the Bridge

In order to make measurements of the resistance of the probe the Wheatstone bridge needs to have some voltage applied and the variable resistor needs to be adjusted to the same resistance as the probe. When initially starting the software, the Heating voltage to the probe will be set to zero volts. This voltage is the voltage applied to both legs of the Wheatstone bridge as shown in the figure above. It is important that not too much voltage is applied to the bridge as this will cause too much current to flow in the SThM probe and this will damage the probe.



CAUTION - As mentioned previously, the SThM probe is made of a thin metal film on a cantilever which will act much like a fuse and break open if too much voltage is applied. The current applied to the probe should always be kept below 2 mA and below 1 mA for typical operation.

The fixed resistor in the Wheatstone bridge inside the system is 1 kOhm and the probe resistance is typically 0.4 kOhm, this means limiting the applied voltage to <1.4 V for typical operation. The higher the applied voltage the better the sensitivity on the resistance change will be, but also the higher the self heating will be. Self heating occurs in the probe due to the current flowing through the high resistance region at the end of the cantilever. This can cause the probe to heat up to significant temperatures. We recommend that the temperature of the probe is kept below ~160°C to minimize damage to the probe.

To start, set the output voltage to 0.2 V with the Vs-Vr gain set to 10X. In the Analysis Studio software, the Vs-Vr gain is set in the Settings menu as shown in figure 6-3. The Heating voltage can be set in the Analysis Studio software by going to the NanoTA Meter.

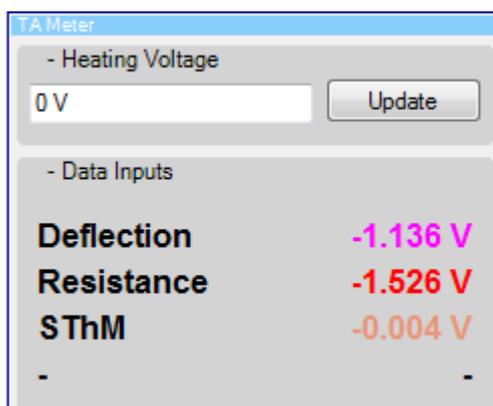


Figure 6-4: The Meter view allowing setting of the heating voltage

Next the SThM signal in the NanoTA Meter should be zeroed by adjusting the variable resistor. The maximum value that can be measured with the SThM signal is +/-4.240 V. Once the Input signal is zeroed, the voltage that will be used during the measurement can be set and the system rezeroed. There is typically some offset in the value depending on voltage. After the final voltage has been set, the Vs-Vr gain can be increased up to 1000X which is what is typically used for measurements where there are small changes in temperature of the probe. In the Analysis Studio software this is done by changing the Vs-Vr gain in the Controller settings menu. It is not required that the voltage is exactly at zero, but just within the range of the system. Typically as the probe approaches the sample surface the temperature will change and may require more adjustment of the variable resistor to balance the bridge.

A good test to confirm that the probe can measure temperature variations is to move the laser which detects the deflection of the cantilever on and off the cantilever. This should only be done when the probe is retracted from the sample surface. There should be a noticeable change in the SThM signal when the laser is on or off the cantilever. The difference will depend on the exact position of the laser on the cantilever but it is expected that the difference will be close to if not more than a volt.

6.3 Acquiring SThM Data



It is important before bringing the probe into contact with the sample surface that the sample does not have any applied voltage, static charge or is at a temperature that is too high for the probe.

After confirming this, bring the probe into contact with the sample surface and minimize the force between the probe and sample. Next optimize the feedback loop by adjusting the feedback gains to the maximum stable value which does not cause feedback oscillation. Once the probe is on the surface there may need to be some readjustment of the variable resistor due to coupling of the temperature of the probe to the sample. Adjust the variable resistor so that the V_s - V_r signal is within range and as close to zero as is possible. With a 1000X gain the signal will be very sensitive and even very small changes in the variable resistor will cause large changes in the V_s - V_r signal.

In order to improve the temperature resolution, it is recommended that a relatively slow scan rate is used (0.5 lines per second or slower). Also it is recommended that the probe is kept on the surface of the sample and with voltage applied for the minimum amount of time required to get the measurement. This will optimize the lifetime of the probe. We recommend during scanning that the probe is scanned perpendicular to the long axis of the cantilever which means that the Scan Angle should be set to 0 degrees. The cantilever has a very high aspect ratio at the end due to the probe essentially being made out of the cantilever angled down. If the end of the cantilever is pushed into a steep edge, the cantilever will bend as opposed to moving up and over the step. This bending can cause the metal film on the cantilever that forms the resistor to break. Finally, select the SThM channel as one of the Input channels in the Channel view.

The SThM measurement is amplifying a small voltage change. As such, it is possible to get noise in the image due to radiation sources or ground loops. This will typically appear at the fundamental frequency of the outlet power (50 or 60 Hz) or a higher harmonic of these frequencies. If this is seen in the image, try to minimize the sources of radiation such as monitors and other electronics. The image that is generated will display hotter areas as light in the image because the hotter the probe, the higher the resistance and the higher the output voltage. Care should be taken when trying to make these measurements as changes in the room temperature or AFM temperature can affect the baseline value of the V_s - V_r signal.

The sensitivity of the SThM system is determined by the gain and the output voltage to the probe. If you are trying to measure an active sample which may have significant temperature variations across the sample surface you can use a lower gain and or output voltage. Lowering the output voltage will decrease the amount of self heating of the probe due to current flow in the probe which will lessen the sensitivity of the probe to thermal conductivity variations on the sample.

When stopping measurements or turning the system off you should perform the following steps:

- Raise up the probe a reasonable distance above the sample surface.
- Verify there is no voltage applied to the probe.
- Disconnect the thermal probe from the connector and reapply the shorting jumper.

6.4 Effects of topography on SThM image contrast

The SThM images obtained are affected by the topography of the sample. As the thermal probe travels over a sample, its temperature will change due to the proximity of the cantilever to the sample surface. When the probe is on a higher spot on the sample, it is surrounded by less sample and more air than when it is on a lower part of the sample. Air has a lower thermal conductivity than the sample; therefore, the apparent thermal conductivity (k) measured by the probe is lower. If measuring a heated sample in which the probe is cooler than the sample, when the probe is on a lower part of the sample it can cause the appearance of a local hot spot. These observed changes in the SThM signal (and, therefore, in the images) are due entirely to topographical effects—not to changes in the thermal properties of the sample. Consequently, the flatter your sample, the less effect the topographical features of your sample will have on the SThM images. When possible, use sample preparation techniques that produce flat surfaces in order to make image interpretation easier.

Also, as the probe moves over the sample surface if the contact area between the tip and sample changes this can cause a change in the temperature of the probe which can be interpreted as a change in the thermal conductivity or the local surface temperature. So even on smooth samples if the probe is on the top of a small peak versus in a valley it will have a different contact area which will cause more or less heat to flow out of the tip into the sample. Also if the probe moves from a "hard" to a "soft" material you can get contrast due to the contact area difference caused by the tip indenting further into the softer material.

As a result of the topographical effects, the SThM images obtained with any AFM based scanning thermal technique are not quantitative. The functions of the SThM images are to provide image contrast based on the thermal properties of the phases or components in your sample.

Appendix A

Sample Preparation

Overview

One of the most important things that you need to do when running nanoTA experiments is to store and handle samples in such a way that the surface is not degraded or damaged in any way. NanoTA is essentially a surface technique. Therefore, any changes that occur to the surface because of poor storage or handling conditions will affect the quality of the results. It is a good idea to get into the habit of using tweezers and/or wearing gloves when handling unknown samples. Moisture and oils from the skin can be easily transferred to your samples. If you do use your hands to pick up a sample, make sure that your fingers never touch the area on which you plan to image or analyze. Samples can be stored in plastic bags, on microscope slides, in Petri dishes, or in any way that will ensure that the surface is not damaged prior to measurement.

Preparing samples

Although you can use a large range of specific sample preparation techniques in AFM imaging, all of these techniques must satisfy a few basic requirements. The sample must be held securely, so that:

- The sample does not move.
- The sample position relative to the probe can be controlled during scanning.
- Both high and low points are within the range of the Z scanner.

You must consider three basic questions for all samples:

- What substrate do you need to use?
- How can you treat the substrate so that the sample can be held securely?
- How can you attach the sample to the substrate, so that it will be held securely, but its surface will be maintained intact for imaging?

For many samples, one or more of these questions are trivial. For others, all three questions require serious thought and experimentation. The examples that follow address the issues that are relevant to each sample. Not all procedures are suitable for all samples. If you believe that the procedure described may affect the sample surface in a manner that could produce erroneous data, you need to seek a different sample preparation procedure.

Sample tilt

First, you need to remove the effects of sample tilt by ensuring that the sample is as level as possible with the scanner. Samples such as thin films will typically be very level but pellets and sectioned samples may not. It is easiest if the sample is made level by cutting parallel faces, one to image and the other to use to mount to the sample mount. If that is not possible you can use a piece of modeling clay to hold the sample to the sample mount (stub). Because the clay is malleable, you can move the sample so that it is parallel to the sample mount. Unfortunately this can also cause drift in the sample both laterally and vertically because of the clay. If using this mounting technique it is recommended that the sample is mounted and then set aside to stabilize for some period of time of at least an hour.

In nearly all cases, you should level the image using the image analysis software. Even if no data is lost due to limited Z-range, the amount of tilt is often significant compared with the size of sample surface features. When this occurs, the surface features will not be clearly seen until the overall image tilt is removed by software.

Static on the sample surface

Static charge can build up on the surface of some samples. This can interfere with imaging because the static charge creates a force on the thermal probe. Follow one or more of the methods described here to reduce or eliminate this problem:

- Ground the sample, if the sample is a conductor or semiconductor
- Use other static elimination devices to minimize or eliminate the static. These include alpha sources or ionization bars. We recommend use of a Staticmaster static elimination brush. This brush includes an alpha source which can be detached from the brush and placed close to the sample surface. This brush can be purchased from a number of sources by searching on the web. This device can be dangerous if not used and stored properly. Anasys Instruments strongly recommends following the instructions included with the Staticmaster brush and all local laws regarding use of this material. Anasys Instruments assumes no obligation or liability for usage of alpha sources.
- Wipe the sample surface with water, using a cotton swab. The water will dissipate the charge. Allow time for the sample to dry before imaging or you may obtain false feedback.

CAUTION: This technique may alter the sample surface or damage the sample.

Debris on the sample surface

If there is loose material on the sample surface, it is likely to interfere with imaging. If you image a dirty surface, small particles may adhere to the thermal probe, making it larger and more unstable. Later, the small particles may fall off. This results in streaks across the image as particles adhere to and fall off the probe. To reduce the effect of debris on samples, follow one or more of the following guidelines:

- Use a micro-duster air gun (available from most laboratory supply houses) to remove very loose debris.
- If the sample will not be damaged by contact with alcohol, put some alcohol on a tissue and drag it across the sample. Do not push—simply let the tissue drag across. The sample could even be dipped into alcohol if doing so would not cause damage.
- Let the system scan several times at a large scan range. This will tend to push loose particles to the edge of the scan range. Then zoom into the area of interest. The risks of this procedure are that the tip (1) may be damaged by repeated scanning or (2) may become contaminated with debris. If the probe is contaminated and cannot be cleaned by continued scanning, a nanoTA ramp can be performed which can clean the probe or the Clean Probe function can be used.
- If possible, scan with a higher probe temperature. This can decompose the small dirt particles. Then reduce the probe temperature and scan the sample again.

Soft samples

Soft samples are easily damaged or distorted by very small forces. The most important problems in imaging soft samples are:

- Securely holding the sample without deforming its surface
- Avoiding surface deformation due to tip-sample forces or mechanical interaction

In order to minimize damage to the sample surface, the feedback gains and setpoint should always be optimized so that minimal force is applied between the tip and sample.

Some guidelines to keep in mind when measuring soft samples are as follows:

- Be careful to avoid formation of air bubbles under the sample.
- Optical glue* can be used to hold the sample.
- Wicking glues (such as Superglue** or cyanoacrylic) are often not compatible with these samples—they may wick through membrane samples or may permeate the sample. Furthermore, wicking glues may chemically react with the sample.
- Soft samples that are hydroscopic, such as a photoresist, may be difficult to image with low forces. This is because the capillary action of the surface moisture tends to pull the probe into

the sample. You can minimize this problem by imaging in a dry environment, which is accomplished by purging the system with dry nitrogen. Also hydroscopic samples may give unstable results with nanoTA. As the temperature of the probe is increased, the water in the sample can be driven off which will cause the sample to shrink. This will look like a transition in the nanoTA data but will typically be at a significantly different value than the correct transition temperature and will have more variability.

Polymer films

Use either double-sided tape, “Spot-o-glue” tabs, or glue to hold the film on sample mounts. Simply cut a small piece of film and place it on the adhesive. Use a flat object or a lint free cloth to push the sample down onto the adhesive. Gently push down on the sample in order to firmly attach it, without causing roughness to be added to the sample.

Polymer pellets

You can use double-sided tape, glue, or modeling clay to easily mount polymer pellets onto sample mounts. When imaging full pellets, be careful to increase the clearance of the probe with respect to the sample surface so the probe is not crashed into the sample surface during the approach. Another option is to cut a slice off the pellet which will create a thinner sample. Use a sharp scalpel or fresh single-edged razor blade to cut the polymer pellet, creating a slice. The thin slice can then be treated as a “polymer sheet,” described in the previous section.

NOTE: Take care when using a sharp scalpel to prepare samples. Obtain assistance if needed.

Hard surfaces

Hard surfaces (such as crystal surfaces, thin rigid films, polymer moldings, or semiconductors) are generally the simplest samples to image. Cut a portion of sample and mount it using glue, double sided tape or “Spot-o-glues” onto a sample disk. During testing of hard-surface samples, the following potential problems can occur:

- The sample is not held securely.
- The plane of the sample is not kept level with the scan plane.
- Dirt is found on the sample surface.
- Static buildup occurs on the sample surface.

Refer to the guidelines listed previously in this chapter for solutions to these problems.

Fibers

Fibers—both *natural* (e.g., hair) and *manufactured* (e.g., nylon)—can be readily analyzed using nanoTA. You can image fiber surfaces as well as their cross-sections. The most important challenge when imaging fiber samples is holding them securely for imaging. The fibers must not vibrate or move when the probe passes over them during scanning. Use one of the following preparation methods to fix the fiber samples to a sample mount. After the fibers are securely fixed to the mount, they can be imaged.

Optical Glue* Method

1. Place a very thin layer of the adhesive on a sample mount, and cure until tacky.
2. Hold each end of the fiber with tweezers, and place it on the mount.
3. Push the fiber slightly into the adhesive, so that at least 1/2 of the fiber diameter is above the surface of the adhesive. The fiber may bow slightly when attached. To eliminate bowing, press gently with a flat surface.

NOTE: This method can alter the sample surface.

Carbon Paint* Method

If the previous method is not sufficient to hold the sample securely, use the following carbon paint method:

1. Cover a sample mount surface with carbon paint and allow to dry.
2. Lay fibers on the stub, and tack the ends with carbon paint.

3. You may also need to tack the center of the fiber to reduce vibration of the fiber during scanning.

Tips for fiber sample

Some guidelines to keep in mind when using fiber samples are:

- Place 2 to 3 fibers on the sample mount in case one comes loose.
- Clean certain samples to remove oils before imaging—particularly carbon fibers, which can be cleaned with acetone.
- *Do not* clean hair samples. Hair samples are generally imaged in order to observe surface treatments, and cleaning would change the effect.
- Only the top 1/6 to 1/10 of the sample can be imaged, because the probe geometry will generally dominate the image beyond this depth. Also, owing to the effect of fiber curvature, you may need to reduce the image size in order to keep the Z-piezo scanner displacement to less than 5 μm .
- Adhesives that wick, such as Superglue**, are not appropriate for imaging fiber samples or cross-section imaging of fibers, because the adhesive often wicks over the top of the fiber.
- The optical glue* method can also be used for fibers. The fibers will sink into the optical glue to about half their diameter.

You can image stiff fiber cross-sections using the techniques described for hard surfaces. The key is to obtain a flat cleave. The fiber can then be held in place by putty, clay, or epoxy. This method is useful, however, only for stiff fibers that will not bend.

For soft fiber samples, cross-section images can be obtained only by using a microtome, because they will not be held firmly enough if the ends of the fibers extend beyond the adhesive.

Powders

You can image the surface of powder particles using an AFM, provided that the powder particles are firmly secured to a sample substrate. The most significant problem you will face when imaging powders is adequately securing the sample to the substrate. If the powder is loose, particles will adhere to the probe or will be moved around on the substrate during imaging. Use one of the following two methods to fix powder samples to substrates for AFM imaging:

TEMPFIX*** Method

1. Place the sample substrate on a hot plate, and warm the substrate so that it is slightly warm to the touch.
2. Move the waxy optical glue stick over the stub to lay down a layer of adhesive. Be careful not to make the temperature too hot.
3. Remove the sample mount from the hot plate, and turn the hot plate temperature to its lowest setting. The optical glue on the mount will spread to an even coating.
4. Allow the mount to cool completely.
5. Sprinkle the powder sample onto the optical glue. When the hot plate has cooled so that it is slightly warm to the touch, place the mount back on the hot plate.
6. Monitor the status of the optical glue with a match stick or other thin object by touching near the edge of the mount. When the glue is tacky, remove the sample from the hot plate and allow it to cool. The powder particles will sink down into optical glue and adhere. The particles will sink only partially into the adhesive, leaving the top portion uncoated for imaging.

Solution Deposition Method

1. Cut a piece of silicon wafer to approximately 1 cm x 1 cm.
2. Attach the wafer to a sample disk using tape, epoxy or carbon paint.
3. Put a small amount of the powder sample into a test tube with water or other solvent.
4. Sonicate or stir to make a slurry.
5. Use a pipette to take some of the sample material from the top of the slurry, avoiding the large particles that may be at the bottom of the tube.
6. Place the slurry sample on the wafer, covering the spot in the center.
7. Allow to dry. Capillary surface adhesion will hold the powder particles for scanning.

This technique works well for smaller particles which are less than a few microns in diameter and ideally sub-micron.

Tips for powder samples

Mica or glass slides can be used in place of silicon wafers for the solution deposition method. Mica has steps that can hold very small powder particles. This can help in some applications by preventing powder particles from moving. Both glass and mica are good substrates also because they have lower thermal conductivity than silicon. The lower thermal conductivity will cause less error in the calibration when measuring small particles on a substrate.

Obtaining materials

The materials cited in this appendix can be obtained from the following sources:

*Carbon paint and optical glue are available from SPI Supplies, <https://secure.2spi.com/>

** Superglue is a product of the Loctite Company.

*** TEMPFIX is a product of the Gerhard Neubauer Company.

Appendix B

Optimizing NanoTA

Overview

If you have followed the instructions contained in the initial chapters of this manual, you should be familiar with the basic procedure for conducting nanoTA experiments. This section will give you recommendations for obtaining the best results possible. The system can produce two types of measurement: Atomic Force Microscopy (AFM) images and nanoscale thermal analysis data. These two types of information, when obtained and used together, constitute the technique of nanoTA. You do not need to be an expert in AFM or thermal analysis to get good results. However, a reasonable understanding of both disciplines is desirable in order to get the most out of your instrument.

Imaging

To help you produce high-quality AFM images, we have provided guidelines in this section that specifically target the parameters associated with imaging of your samples.

ThermaLever probes in general have a different value of spring constant to that of a typical contact AFM probe. This means you need to be careful in optimizing the force between the tip and sample. Also, it may be better to image such that the fast scan direction of the AFM image is perpendicular to the long direction of the cantilever.

Rough Samples

CAUTION: You may damage the probe if you image a sample whose roughness exceeds the Z scanners range.

Typically, *rough samples* are defined as those samples with a Z displacement exceeding 5 μm over the 100 μm by 100 μm image area. For these rough samples, you will need to reduce the image size until the roughness of the sample is under the limit imposed by the range of your Z scanner. If you are using an unknown sample, you will need to check the roughness before you get a complete image, by using a line scan. Also, an additional limit on the roughness of the surface is due to the length of the tip on the ThermaLever™ probes. The AN style probes have a tip which is only 3-6 microns tall. Because of this, the roughness of the sample needs to be less than ~2 μm p-p and also the tilt of the sample needs to be very low. If not, there is a good probability that some other part of the cantilever will touch the sample surface before the tip. The flatter the sample is the better the nano-TA results will be, so it is preferable that the surface roughness of the sample is less than 200 nm RMS.

Characterization

To help you produce high-quality nanoTA results, we have provided guidelines in this section that specifically target the parameters associated with nanoscale thermal analysis of your samples. The procedure for optimizing thermal parameters in nanoTA experiments is similar to those for traditional thermal analysis experiments.

Heating Rate

When you want to obtain standard nanoTA data, follow the recommendations given here to set up the heating rate for your experiment:

- Using faster heating rates means that less overall damage will be done, because the heating will be more localized than slower heating rates. However, it should be remembered that the penetration measured by the nano-TA technique is a viscoelastic phenomenon; it is possible to heat and cool so fast that there is little time for indentation to occur. Therefore, slowing the heating rate can make some transitions, typically glass transitions (T_g), clearer.
- Consequently, a compromise is required between fast heating rates for primary transitions (such as melting) and slower heating rates for secondary transitions (such as the T_g). Heating rates in the range of 1 to 20°C/sec usually yield the best overall results.
- Also when measuring glass transitions and attempting to compare the results to traditional thermal analysis equipment, the user should be aware that the heating rate will affect the measured transition temperature. This heating rate dependence is logarithmic and so the measured transition temperatures will change the most at slower heating rates. Since traditional thermal analysis systems typically have heating rates of 5-10°C/minute versus the 2-10°C/second of nano-TA, the measured transition will be much higher in the nano-TA.

Reproducibility

Variation in the shape of the deflection during temperature ramps or measured transition temperatures, obtained at multiple locations using the same sample, can be due to either the sample itself or the instrument. The best way to assess the reproducibility possible with your sample is to first use a similar type of sample that you know is flat and homogeneous. Measure multiple locations on this known sample before measuring unknown samples. A 10-mg sample of your material, analyzed in a traditional DSC, will produce a curve that has a “specimen-averaged” result. NanoTA data are obtained from a cubic sample about 1 μm or less in size. Local variations across the surface of your sample can be measured by nanoTA. These variations are not seen using the traditional thermal techniques, so you must be careful when comparing values from the two techniques. Clearly, the relative positions of events, with respect to temperature, should not change when moving from conventional to nano Thermal Analysis. The lower temperature transition in a two-phase system always shows the lowest transition temperature, regardless of whether it is measured in your DSC instrument or your NanoTA2 system. You will need to assess the reproducibility for your own types of samples before drawing conclusions about the similarity or difference between two samples.

Also, you can see variations in the Deflection signal due to artifacts in the AFM system. These can include mechanical/acoustic noise, laser noise, thermal drift and piezoelectric creep. The Deflection signal is a high resolution measurement over a relatively long time of the relative positions of the tip and sample. As you have probably experienced with your AFM, loud noises, vibration, strong air currents or other effects can disturb your images. In the AFM images, these are typically minimized by filtering the data using a flatten or planefit filter. This is not possible with nanoTA as this information is what we are attempting to measure, so the system needs to be very well isolated from acoustic noise, mechanical vibration, air currents and temperature variations. Both mechanical noise and laser noise will typically add periodic higher frequency noise to the measurement. The mechanical noise can be minimized by adding a vibration isolation system. The laser noise can be minimized by allowing the system sufficient time to come to thermal equilibrium after it is turned on. The thermal drift and piezoelectric creep in the Z direction will typically cause slow changes in the position of the tip relative to the sample which can cause a change in slope or even a reversal of the slope when initially starting the temperature ramps. The thermal drift can be minimized by allowing the system to reach thermal equilibrium. The piezoelectric creep of the Z scanner is more difficult to eliminate. Minimize as much as possible any large Z motion immediately before performing a temperature ramp. This can be done by allowing the system to scan for a small amount of time after it is engaged or after the tip has been put back on the surface after the tip has been cooled before performing any temperature scans.

Appendix C

Troubleshooting

All forms of analysis have the potential to create data which includes artifacts. It is important to understand what data is due to the sample and what is due to the system. Shown below are a number of examples of data which includes artifacts. Hopefully this will decrease misinterpretation of data and improve results.

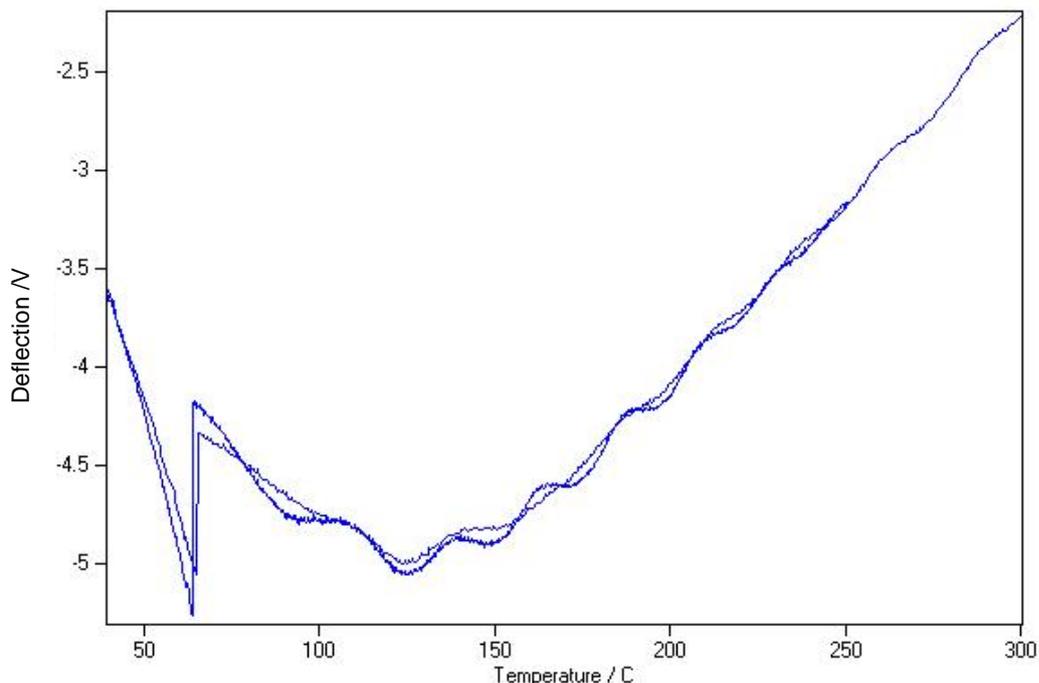


Figure C-1: Probe retracting from the sample during a nanoTA ramp

The above temperature ramp shows what happens if the sample initially moves away from the probe during the temperature ramp. This can occur due to thermal drift or piezo creep causing the probe and sample to separate or the bending of the cantilever due to thermal stress being greater than the thermal expansion of the sample. The initial linear portion of the curve from 40-65° C is when the probe is on the sample surface. The sharp step up is due to the probe popping off the sample surface. The rest of the curve occurs with the probe not touching the sample and is a combination of optical interference and thermal stress induced bending of the cantilever.

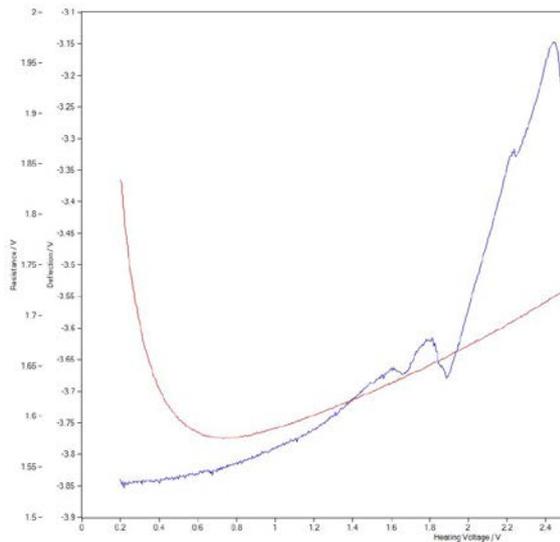


Figure C-2: Probe initially not on the sample

If the probe is in the attractive region of the forces, the probe may not initially be in contact with the sample surface during the temperature ramp. In Figure C-2, the probe is not in contact with the sample surface from a heating voltage of 0.2 to ~1.8 V. At this point the probe comes into contact with the sample surface due to electrostatic forces between the probe and sample, the thermal induced bending, or the thermal expansion of the sample. After this, the temperature ramp will display the characteristic behavior of the sample such as seen on the PCL sample above. If the probe does not come down into contact with the sample for a significant portion of the temperature ramp, adjust the setpoint to a larger value before starting the thermal ramp.

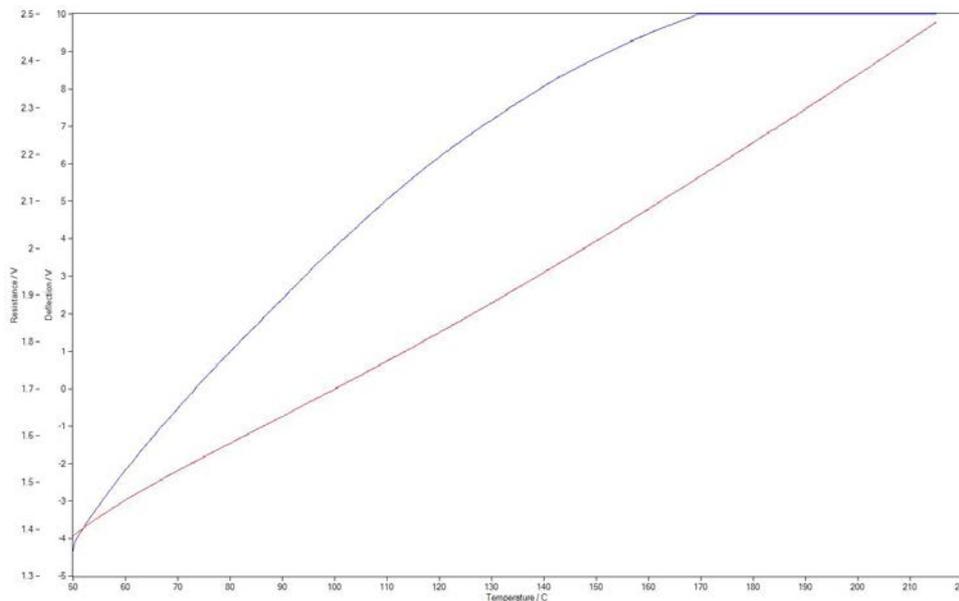


Figure C-3: Thermal expansion causing deflection signal to be out of range

If the thermal expansion of the sample is large and causes the Deflection signal to be out of range, try a lower “free air calibration target deflection” when aligning the AFM detector. This is discussed in the AFM Setup, Section 2.2. If there is still not sufficient range, try running nanoTA with Z feedback on, as described in the Advanced Procedures section. Another option is to decrease the amount of sample expansion by either increasing the heating rate (less heat transfer to the sample) or to decreasing the sample thickness if possible.