

*μ TA[®] 2990
MicroThermal
Analyzer*



Operator's Manual

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(continued on next page)

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Notes, Cautions and Warnings

The following conventions are used throughout this manual to point out items of importance and safety as you read through the instructions.

A NOTE highlights important information about equipment or procedures.



A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.



A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Safety

Please read the safety information contained in this section carefully *BEFORE* using the instrument. The knowledge that you gain could save you from injury or equipment damage as you use the μ TA[®] 2990.

Laser Operation— AFM Scanning Head Laser



WARNING: NEVER LOOK DIRECTLY INTO THE LASER BEAM. In order to avoid the possibility of inadvertently looking into the laser, always use the software or hardware to switch the laser OFF *before* raising the head to eye level.




The diode laser in the μ TA 2990 scanning head complies with US 21CFR 1040.10 and is certified as a Class IIIa laser. The laser wavelength is 670 nm and the maximum power is 3 mW. In addition to the preceding warning, please follow laser safety control measures in American National Standards Institute Z136.1-1986.

High Voltage



WARNING: Wherever high voltage is present on the system, extreme care should always be taken to avoid direct contact while the instrument hardware is powered on. We do not recommend that you make any attempt to remove the panels on the instrument. Call TA Instruments Service for repairs. If you do attempt your own repairs, always turn the power off before attempting to remove any panels or PC boards and before touching any connectors by hand or with electrically conductive tools. High voltage is present at the

following locations on the system and where the  symbol is located:

- **μ TA™ 2990 head/Z scanner:** Z scanners should always be changed when the system is powered off or through the software/hardware procedure.
 - **ECU-Plus:** I/O boards and connectors (rear panel).
 - **Microscope Control Unit (MCU):** rear and front panel connectors.
 - **Power/connection cables:** μ TA 2990 head, ECU-Plus, computer, monitors.
-

CE Compliance

This equipment has been designed to comply with the following standards on safety:



- IEC 1010-1/1990 and A1/1992 and A2/1995
- IEC 1010-2-010/1992 and A1/1996
- EN 61010-1/1993 and A2/1995
- EN 61010-2-010/1994
- UL 3101-1, First Edition.

In order to comply with the Electromagnetic Compatibility standards of the European Council Directive 89/336/EEC (EMC Directive) and Directive 73/23/EEC on safety as amended by 93/68/EEC, the following specifications apply to the instrument:

- *Safety:*
EN 61010-1/1993 and A2/1995 Installation Category II
EN 61010-2-010/1994
- *Emissions:*
EN 55022: 1995, CISPR 22:1993
Class B (30-1000 MHz) Radiated
EN 55022: 1995, CISPR 22:1993
Class B (0.15-30 MHz) Conducted
- *Immunity:*
EN 50082-1: 1992, Electromagnetic
Compatibility—Generic immunity standard Part 1. Residential,
commercial, and light industry.

Instrument Symbols

The following labels are displayed on the μ TA[®] 2990 system for your protection:

Label	Explanation
	Indicates the presence of dangerous laser light. Eyes must be protected. Do not look directly into the laser beam. Follow all safety precautions listed in this manual to avoid injury.
	Indicates the presence of high voltage. Turn off the power to the instrument when directed. Follow all safety precautions listed in this manual to avoid injury.

Please heed these labels and take the necessary precautions when dealing with those parts of the instrument. This μ TA 2990 manual contains cautions and warnings that must be followed for your own safety.

Lifting the Instrument

The Electronic Control Unit (ECU-Plus), is heavy. In order to avoid injury, particularly to the back, please follow this advice:



WARNING: Use two people to lift and/or carry the ECU-Plus. Carrying handles are placed at the sides of the ECU-Plus for this purpose. The ECU-Plus is too heavy for one person to handle safely.

Part Numbers

If you need to order a replacement part for your μ TA 2990 instrument, refer to the following list of parts and then contact your service representative or the TA Instruments office closest to you.

Part Number	Description
899006.901	Thermal Probes (Pkg. of 5)
899007.901	AFM Probes—200 μ m (Pkg. of 10)
899008.901	Sample Mounting Stubs (Pkg. of 20)
899005.901	10- μ m Z Scanner (dry)
259508.100	Tweezers
259509.000	Spatula
899098.901	PET Film, Certified Reference Material
899096.901	Anisic Acid, Certified Reference Material
899097.901	Biphenyl, Certified Reference Material
271234.001	Tape, Double-Sided

Chapter 1

Introducing the μ TA[®] 2990

Introduction

The TA Instruments MicroThermal Analyzer (μ TA) 2990 is an analytical system that combines the capabilities of Atomic Force Microscopy (AFM) with thermal methods. AFM is a particular operating mode within the general field of Scanning Probe Microscopy (SPM). When you perform experiments with the μ TA 2990, you can image a sample and then characterize it using thermal analysis. This combination of techniques gives you visual and property-based information—but on a microscopic scale.

To run experiments, you mount the sample onto a substrate and then place it on the AFM sample stage. To obtain an image of the surface, a probe is rastered across the sample, while the Z-axis displacement required to maintain a constant force between probe and sample is measured. The thermal probe is used to obtain images not only on the basis of topography but also on the thermal properties of the sample. This is the first part of the experiment—the *visualization* of the sample.

You can then place the thermal probe at any specific location, using the images to identify domains, features, and phases. In this mode, the system acts like a MicroThermal Analyzer to obtain the *characterization* of the sample. As the sample is heated by the probe, temperature-dependent properties are measured, such as softening and melting. This process is called Localized Thermal Analysis.

In this manual, we use the term “ μ TA 2990” to refer to the TA Instruments’ analyzer and “MicroThermal Analysis” (or MicroTA) to refer to the technique.

The μ TA 2990 is used in conjunction with a TA Instruments controller (TA 5300 or higher) running μ TALab (MicroThermal Analysis Laboratory) software. The μ TALab software can be run at the same time as TA Instruments *Thermal Advantage* or *Thermal Solutions* Software. This means that a single controller can run both conventional and MicroThermal Analysis instruments simultaneously.

Components

Your μ TA[®] 2990 consists of a number of components, shown in Figure 1.1:

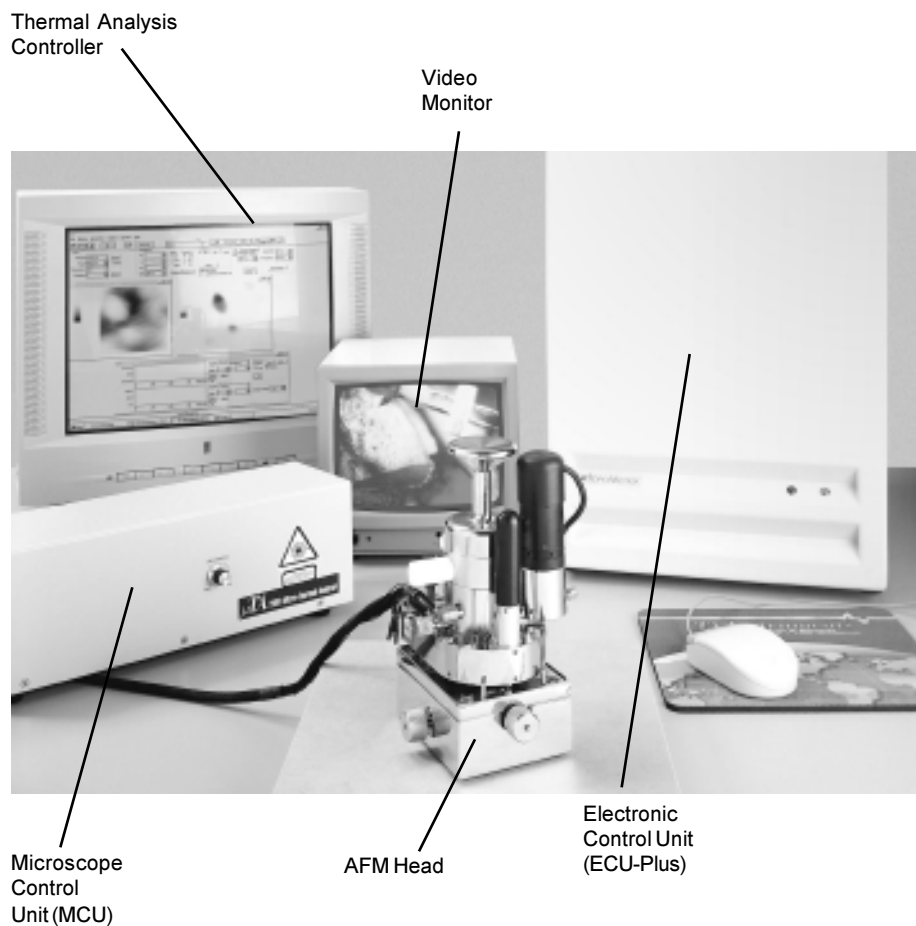


Figure 1.1
 μ TA 2990 System Components

The sections that follow describe each of these components in further detail.

μ TA[®] AFM Head

The μ TA AFM head is a modular stage microscope designed for stable, accurate scanning and easy sample mounting. The μ TA AFM head contains a TrueMetrix[™] scanner system incorporating a closed-loop scanner linearization technique that eliminates non-linearity and hysteresis effects. The system also incorporates Scanning Tip Technology[™] (STT) to raster the scanning tip over the sample. This design ensures accurate imaging as well as accurate thermal probe placement for Localized Thermal Analysis experiments

The μ TA AFM head uses an integral video camera for viewing the tip and sample at a 45° angle. With the standard μ TA scanner, the largest scan range is 100 μ m in the X and Y directions and 10 μ m in the Z direction.

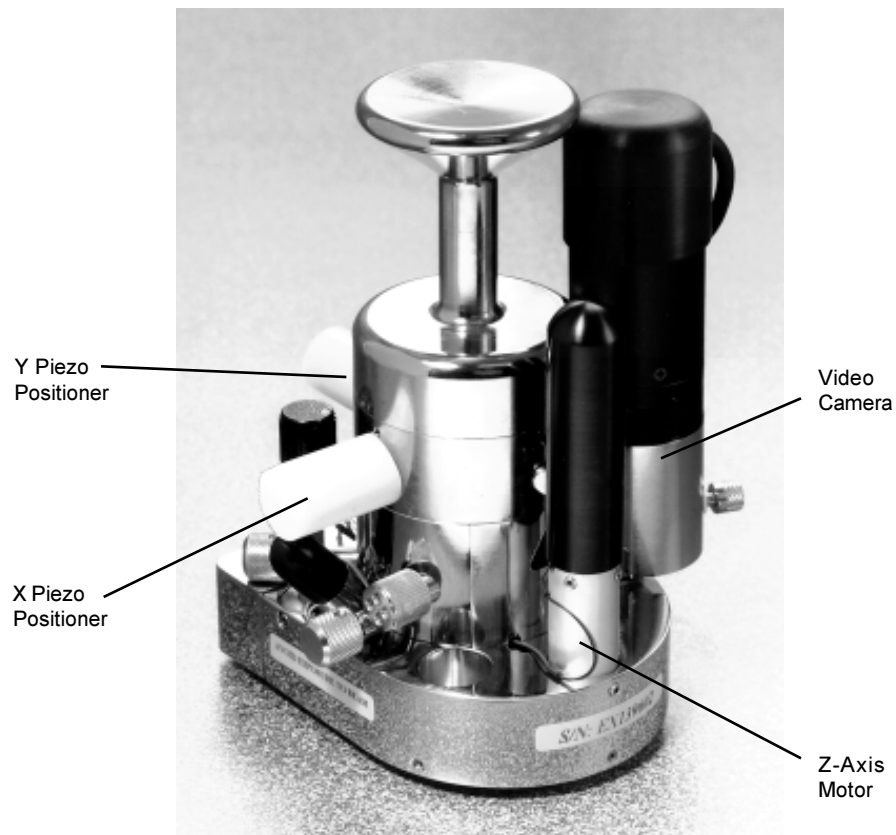


Figure 1.2
 μ TA[™] AFM Head

Electronic Control Unit: ECU-Plus

The ECU-Plus is an advanced electronic control unit that provides 16-bit scanner control in X, Y, and Z directions—at all scan ranges and at any scan offset. The ECU-Plus provides the electronic signals that control the scanner, positioning devices, and amplifiers in the microscope stage and chassis. It contains the mother board, which connects the ECU-Plus to the Digital Signal Processor (DSP) board in the computer, connectors, and four power supplies.



WARNING: High voltage is present at the I/O boards and connectors on the rear panel of the ECU-Plus. Extreme care should always be taken to avoid direct contact while the instrument hardware is powered on. Always turn off the power to the equipment before attempting to remove any panels or PC boards and before touching any connectors by hand or with electrically conductive tools.

Microscope Control Unit: MCU

The MCU is the interface between the ECU-Plus and the μ TA AFM head. It also contains the brightness control for the light that is built into the μ TA AFM head to improve illumination for the 100X Video Camera.

Microscope Video Monitor

The microscope video monitor offers an easy way to view the thermal probe and sample surface. The 100X Video Camera is located on the μ TA AFM at a 45-degree angle. The picture that is viewed on the monitor is used to align the laser onto the probe mirror as well as furnish a close-up view of the probe tip and surface during approach and scanning.

Thermal Analysis Controller

The TA Instruments Thermal Analysis controller is a computer that performs the following functions:

- Provides an interface between you and the μ TA 2990.
- Enables you to set up experiments to obtain images and Localized Thermal Analysis data.
- Stores data obtained from the μ TA 2990.
- Provides image analysis and data analysis programs.

Specifications

Tables 1.1 and 1.2 contain information about the μ TA[®] 2990 dimensions and product specifications.

Table 1.1
 μ TA 2990 Dimensions

Component	Width	Depth	Height	Weight
μ TA AFM Head containing 100 μ m scanner	10.7 cm (4.2 in.)	10.2 cm (4.0 in.)	15.2 cm (6.0 in.)	1.4 kg (3.1 lb)
Electronic Control Unit (ECU-Plus)	33 cm (13 in.)	46 cm (18 in.)	53 cm (21 in.)	26.8 kg (59 lb)
Microscope Control Unit (MCU)	13 cm (5 in.)	36 cm (14 in.)	13 cm (5 in.)	4.5 kg (10 lb)
Monochrome-Microscope Video Monitor	23 cm (9 in.)	25 cm (10 in.)	25 cm (10 in.)	5.5 kg (12 lb)

Table 1.2
Product Specifications

Manual X-Y Translation Stage	Range 6 mm x 6 mm
Scanner X-Y Range	100 μm x 100 μm Linearized using TrueMetrix
Scanner Z Range	> 10 μm Linearized using TrueMetrix
Optical Microscope	Integral 100X, CCD with 9-inch monitor
Sample Size: —without base —with base	Unlimited 22 mm diameter, up to 5 mm high
Visualization Modes	Topography, Thermal Conductivity, and Thermal Diffusivity
Characterization Modes	$\mu\text{MDTA}^{\text{TM}}$ $\mu\text{TMA}^{\text{TM}}$
Temperature Range: —standard —with cooling stage	Ambient to 500°C -70°C to 300°C
Heating/Cooling Rates	0 to 1500°C/min
<i>Specifications are subject to change.</i>	

Output Values and Experiment Types

The following information may be obtained from your μ TA 2990.

Images (Visualization)

Five image modes are available to provide image contrast for your sample:

- Topography
- Sensor
- Thermal Conductivity
- Temperature Modulation (Phase)
- Temperature Modulation (Amplitude).

Images may be obtained in the forward, reverse, or both directions.

Localized Thermal Analysis (Characterization)

The following signals are stored, for each location, during a Localized Thermal Analysis experiment:

- Sensor Position (μm)
- Power (mW)
- Programmed Temperature ($^{\circ}\text{C}$)
- Time (min)

The following AC signals are included if AC measurements are made:

- AC Amplitude (V)
- AC Phase (rad)

Chapter 2

Installing the μ TA[®] 2990

Overview

Before shipment, the μ TA 2990 is tested and calibrated. Installation involves the following procedures, covered in this chapter:

- Inspecting the system for damage or missing parts
- Choosing an appropriate location for the μ TA 2990
- Unpacking the ECU-Plus, Microscope Control Unit (MCU), and Monochrome Monitor
- Unpacking the μ TA 2990 head and 10 μ m Z-axis scanner
- Installing the 10- μ m Z-axis scanner into the μ TA 2990 head
- Connecting the cables and power cords
- Installing the μ TALab Software on your Thermal Analyst Controller
- Installing and aligning the 100X Video Camera onto the μ TA 2990 head
- Installing AFM or thermal probes

If you wish to have your instrument installed by a TA Instruments Service Representative, contact your local representative (see list in frontmatter of manual) for an installation appointment when you receive your instrument.



CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

Inspecting the System

When you receive your μ TA 2990, look over the instrument and shipping container carefully for any signs of damage. Please check the parts received against the enclosed packing list.

- If the instrument is damaged, notify the carrier and contact your local TA Instruments office immediately.
- If the instrument is intact but parts are missing, contact your local TA Instruments office.

A list of TA Instruments offices can be found in the frontmatter of this manual.

Choosing a Location

Before you place your new μ TA 2990 in its final location, make sure that the place you have chosen meets the following requirements.

Power Requirements

The ECU-Plus is either 110/220 or 110/240 Vac ($\pm 10\%$), depending on country, and requires 15 or 20 amp, 50 to 60 Hz, 1 phase, 1200 W max. (In 110 Vac countries, the instrument is shipped with a surge protector power strip.)



CAUTION: If the line power in your laboratory is extremely noisy, an additional power conditioner may be required.

The equipment is designed for safe operation within the defined environmental conditions. For optimal performance, however, more stringent temperature and humidity conditions are recommended.

Table 2.1
Operating Conditions

Safe Operating Temperature	5 to 40°C
Optimum Operating Temperature	15 to 25°C Stable to $\pm 2^\circ\text{C}/\text{hour}$
Safe Operating Relative Humidity	$\leq 80\%$, Non-condensing, at 31°C decreasing linearity to $\leq 50\%$, Non-condensing at 40°C
Optimum Operating Humidity	$< 60\%$, Non-condensing

Temperature

Changes in temperature can cause the metal parts in the microscope stage to expand and contract. AFM images will have drift when the temperature of stage components changes. Maintain the microscope in a constant-temperature environment (range of 15 to 25°C \pm 2°C/hour) to avoid drift problems. Do not locate your microscope near heating vents, heat sources, or windows (where varying sunlight could cause frequent temperature changes).

Humidity

The optimum humidity level for the use of the μ TA 2990 is less than 60% (see previous page). We recommend that when you choose the location for your instrument, you try to follow these guidelines. See the following CAUTION for more information.



CAUTION: Your microscope is engineered for use in a standard laboratory environment. The manufacturer's specifications indicate that failure is possible if the piezoelectric positioner is operated while wet or when humidity is greater than 70%. This fact should be considered in your choice of location.

Vibration

The μ TA 2990 should be located on a vibration-free surface. It is recommended that you place the μ TA 2990 head on a table that is separate from the other components of your system (*e.g.*, Thermal Analyst Controller, ECU-Plus).

Vibration is the greatest single source of image noise in an atomic force microscope and can cause a substantial decrease in resolution. Vibrational noise is generated because vibrations cause excessive motions of the probe tip relative to the sample. When the thermal probe is used, an environment equivalent to that used for an analytical balance is recommended. For the highest-resolution AFM images, the microscope must be maintained in a vibration-free environment, such as a ground-floor laboratory. For optimal AFM image performance, the μ TA 2990 should be operated on a vibration isolation platform. The major sources of vibrational noise are:

- **Sound**—Acoustic waves can excite vibration-induced noise in the AFM cantilever and should be minimized.
- **Table**—A table with a poor support structure can transmit vibration frequencies that reduce microscope resolution. Mechanical components (such as a fan sitting on the microscope stage table) can cause excessive vibration. Excessive vibrations are also generated when the mouse button is clicked, for example. It is advisable to place the microscope on a separate table from any other components in the instrument configuration.
- **Floor**—Vibrations can be transmitted through the floor of your building. Natural low-frequency vibrations in the floor occur because of “bowing” motions of the building. Mechanically generated vibrations from elevators and air conditioners can also be transmitted through the floor. A coarse test of the vibrational characteristics of the surface that supports your microscope is to take a glass of water and place it on the table near the stage. Any stray vibrations can be seen on the surface, especially when a light source is reflected off the water. Note that the presence of ripples definitely does indicate vibrations that may affect imaging but that the absence of ripples does not prove that no vibrations are present.

Unpacking System Components

Follow these instructions to unpack the system components. As you remove each piece, place it in a safe location (an area where it will not be knocked to the floor, etc.).

1. Locate and unpack the ECU-Plus. The front cover of the ECU-Plus is packed separately.
2. Connect the cables from the front cover to the main cabinet of the ECU-Plus. The front cover is held on by push-fit snap connectors.
3. Locate and unpack the Microscope Control Unit (MCU) and monochrome monitor.
4. Unpack the μ TA 2990 head from the larger of the two wooden boxes. Turn the μ TA 2990 head upside down and place it on a flat surface.
5. Remove the 100X Video Camera from the larger wooden box.
6. Remove the 10- μ m Z-axis scanner from the smaller wooden box. The next section provides instructions on how to install the scanner on the μ TA head.

Installing the μ TA[®] 2990

The μ TA 2990 system is installed in several stages: (1) the 10 μ m Z-axis scanner is installed on the μ TA head, (2) the power cords and cables are connected, (3) the software is installed, (4) the camera is installed, and (5) the probes are installed.

Installing the Z-Axis Scanner

Follow these instructions to install the 10 μ m Z-axis scanner on the μ TA head. Refer to Figure 2.1 as needed.

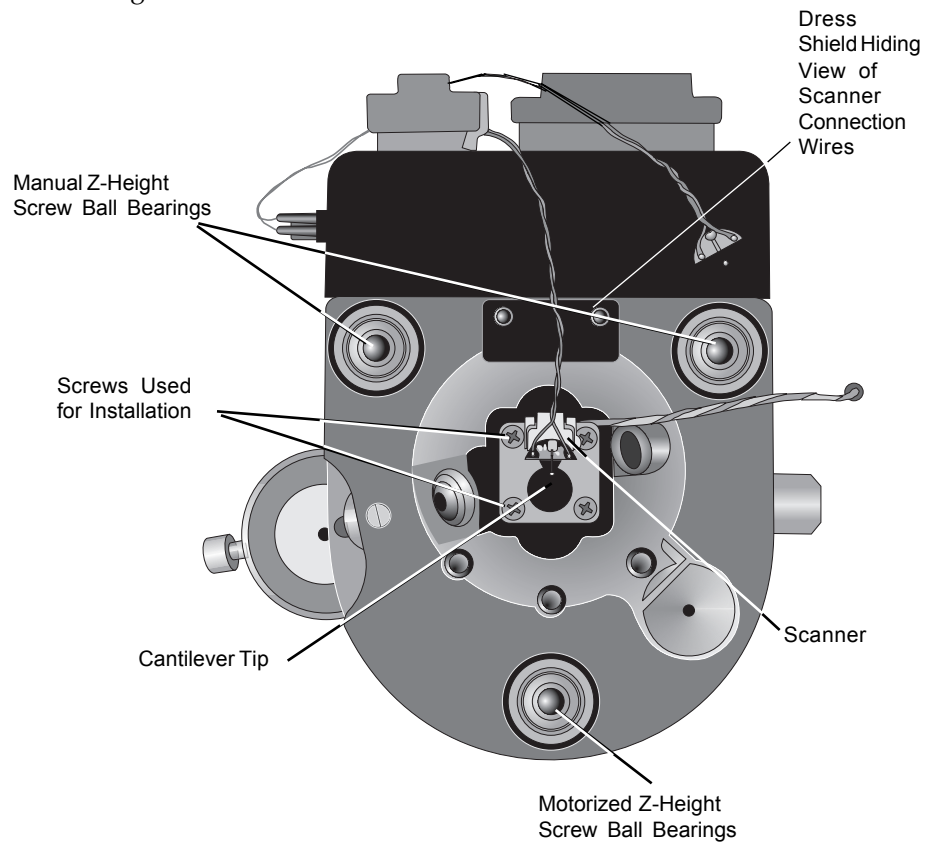


Figure 2.1
Underside of the μ TA Head

1. Install the 10 μm Z-axis scanner, using the four captive screws, onto the μTA 2990 head.
2. Using a small hex-wrench, remove the dress shield located between the two manual Z-height adjusters to expose the female scanner connection wires.
3. Using tweezers, connect the male scanner connection wires to the female scanner connection wires, making sure to observe the color coding (red to red, orange to orange).
4. Replace the assembly between the two manual Z-height adjusters, taking care not to crimp or cut the scanner connection wires.
5. Connect the additional scanner wiring harness to the connector on the top of the μTA 2990 head; see Figure 2.2.

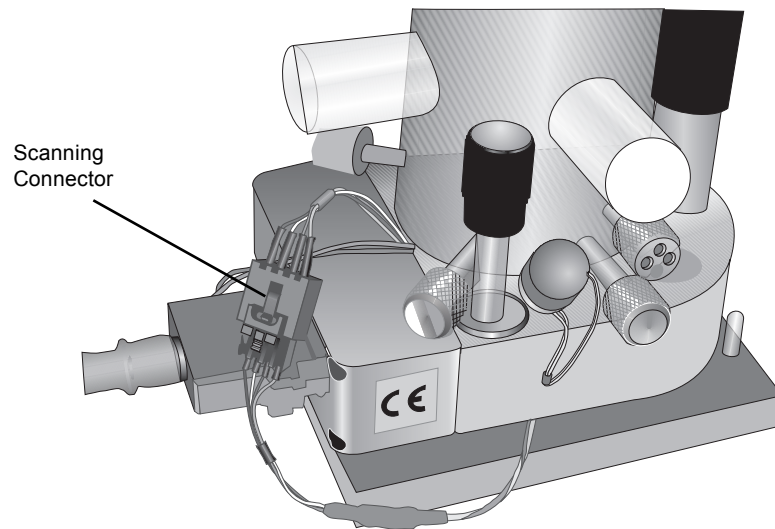


Figure 2.2
 μTA Head



CAUTION: The 10 μm Z-axis scanner can be damaged or destroyed if it hits either the sample surface or the surface that the μTA 2990 head rests on.

6. Adjust the manual Z-height screws so that the scanner is kept well clear of the laboratory bench before turning the μ TA 2990 head over and placing it right side up.
7. If you need to remove the 10 μ m Z-axis scanner, reverse steps 6 through 1.

NOTE: Because the X and Y tripod piezo positioners on the μ TA 2990 are permanently mounted in the head/tower assembly, only the Z-axis scanner (piezo positioner) is replaced when a scanner is changed.

Connecting Cables and Power Cords

The majority of connecting cables will each fit into only one socket. Where it is possible to insert a cable into more than one socket, a label on the cable connector identifies the correct socket.

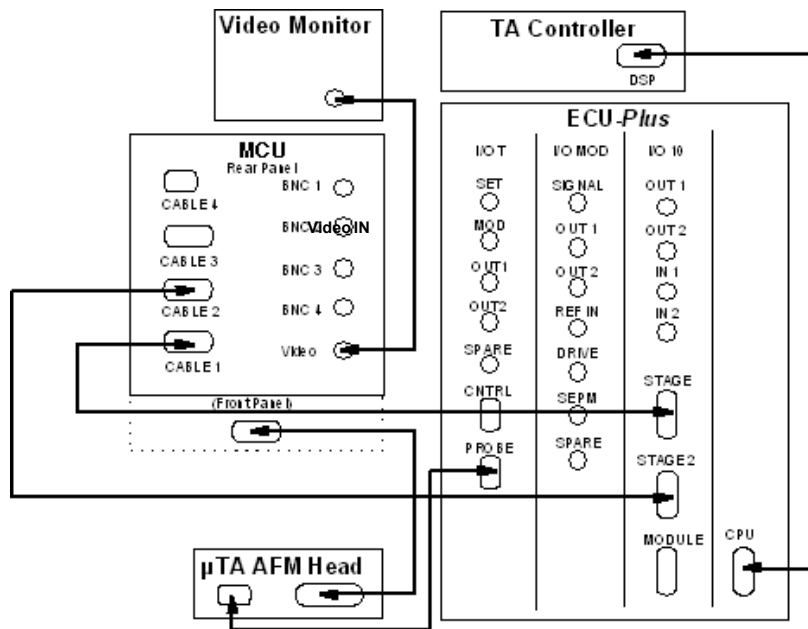


Figure 2.3
Cable Connections



WARNING: High voltage is present at the I/O boards and connectors on the ECU-Plus, μ TA™ 2990 head, and MCU. Always take extreme care to avoid direct contact while the instrument hardware is powered on. Always turn off the power to the equipment before attempting to remove any panels or PC boards, and before touching any connectors by hand or with electrically conductive tools.

1. Connect a power cord to the ECU-Plus and TA Controller.
2. Connect all power cords to main outlets (or, if supplied, through the surge protector power strip). The monochrome monitor has an integral power cord.
3. Turn on the power as directed in the next section.

Starting the μ TA[®] 2990

The components of the μ TA 2990 system need to be powered up in the following order:

1. Check all connections between the various components of your μ TA 2990 system.



CAUTION: Raise the probe (thermal or AFM) *well above* any sample or surface. If the probe tip is too close to the surface at startup, it could be broken during the software/hardware initialization sequence. See Chapter 3 for more information on moving the probe.

2. Turn on the following components in this order:

- Monochrome Monitor
- ECU-Plus
- Thermal Analyst Controller.

NOTE: The μ TA 2990 system needs 1 to 2 hours to fully warm up and reach thermal equilibrium with its environment. Please wait for sufficient warm-up time before you calibrate the instrument or begin making measurements.

3. Load the Windows[®] NT operating system. For information on how to load this software, follow the instructions provided by Microsoft Corporation.
4. Install the μ TALab software as directed in the next section.

Installing μ TALab[®] Software

μ TALab (Micro-Thermal Analysis Laboratory) Software is a software program designed to operate the μ TA 2990 MicroThermal Analyzer. After you install it on your TA Instruments Controller (TA 5300 or higher), you can run μ TALab Software at the same time as *Thermal Solutions* Software. This means that you can run conventional and MicroThermal Analysis instruments simultaneously.

You should use μ TALab Software to set up and perform μ TA experiments and analyze image data. You should use the Windows Universal Analysis Software to analyze and print data from Localized Thermal Analysis experiments.

The μ TALab software is used to:

- Control the μ TA 2990
- Acquire data (both images and Localized Thermal Analysis)
- Enhance and measure images
- Provide a means to optimize and format your data prior to printing.

Installation Procedure

Diskettes containing the μ TALab[™] Software are shipped to you along with the μ TA[™] 2990 MicroThermal Analyzer. You need to install the software on your Thermal Analysis controller in order to use the instrument.

To install the software on your TA controller, follow these steps:

1. Put Disk 1 of the software in the floppy disk drive of the controller.
2. Click on **Start** from the taskbar, and select **Run**.
3. To enter the correct file in the "Open" field, either use the keyboard to enter "A:\setup.exe" or click the **Browse** button and find this filename by browsing. Click OK to start the installation.
4. When prompted, remove Disk 1 and put Disk 2 of the software into the floppy drive.

3. When prompted, remove Disk 2 and insert the Scanner Calibration Data disk.
4. When the software files have been loaded, you will see a message advising to reboot/restart your computer. Follow these instructions.

Starting μ TALab Software

To start up the μ TALab Software, double-click on its icon on the controller screen. The μ TALab **Main** window appears, as shown in Figure 2.4.

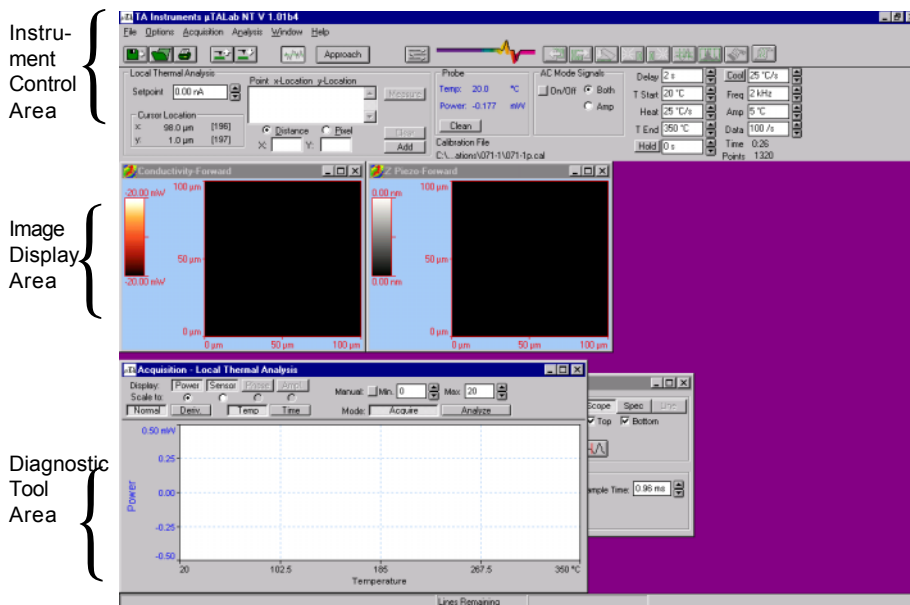


Figure 2.4
Areas of the μ TALab Main Window

At first this window may seem very complicated because it contains many different pieces and windows. However, it can be divided into three main areas:

- **Instrument control:** The *top* of the **Main** window consists of menus, buttons, and panels used to collect and analyze images and data.

- Image display: The *middle* of the **Main** window displays the images being obtained during scanning as well as real-time plots.
- Diagnostic Tool Area: The *bottom* of the **Main** window contains displays for and gives access to diagnostic tools, such as meters and the oscilloscope.

The top of the **Main** window is discussed in detail in Chapter 3. The other areas of the window are explained elsewhere in the manual.

Installing the Camera

Use the following procedures to install or remount the video camera on the head of the μ TA 2990:

1. Adjust the camera's Y-adjust/fine focus thumbwheel, located on the μ TA 2990 base (see the figure below), to allow a small space between the camera mounting base block and the bottom of the recessed ledge. This allows room for later adjustments.

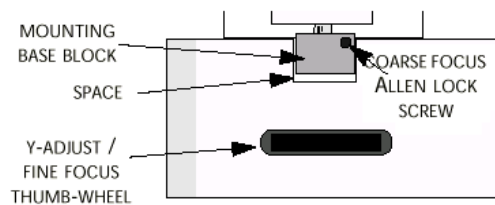


Figure 2.5
Adjusting the Space

2. Make sure that you have sufficiently loosened the two mounting Allen screws at the camera's base. Then, mount the camera on the optical shaft, sliding it down on the shaft until the two mounting screws can be secured in the rounded openings in the slotted guides on either side of the shaft.

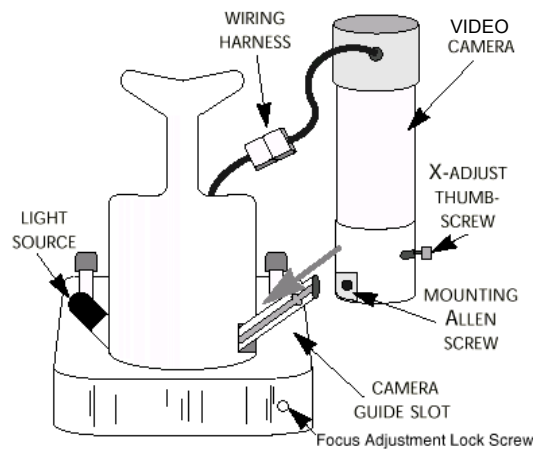


Figure 2.6
Mounting the Camera

3. Tighten both screws. Tighten the fine focus adjustment locking screw.
4. Connect the camera's wiring harness to the appropriate socket on the μ TA 2990.
5. Turn on the monochrome monitor.

Go on to the next section to adjust the light source. Instructions on how to adjust and focus the camera can be found at the end of this chapter.

Light Source Adjustment

To adjust the light source:

1. Loosen the adjustment screw for the nylon illuminator light bulb on the light source housing (see the figure below).
2. Rotate the light until the sample surface is satisfactorily illuminated, compensating for the surface reflectiveness or tilts.
3. Retighten the screw.

To replace/remove the bulb, loosen the bulb-removal Allen screw.

You may need to raise or lower the optical microscope's bulb brightness level (with the control on the MCU) to adequately see the bulb's field of illumination.

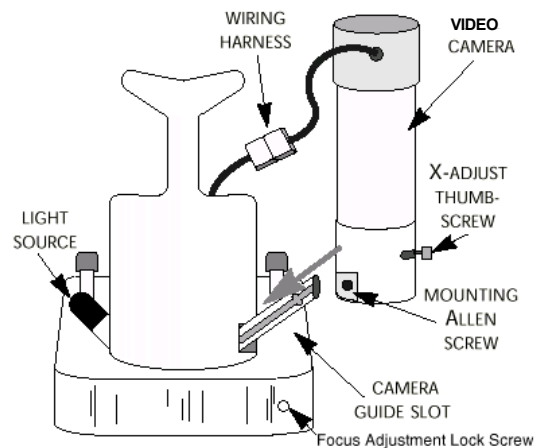


Figure 2.7
Light Source Housing

Video Image Polarity

The standard black and white video monitor comes with a NORMAL/ REVERSE image polarity switch at the back of the unit. This switch reverses the video display, creating a "mirror" image. For most scanning applications, the setting is a matter of personal preference.

Installing Probes

Two types of probes can be installed on the μ TA 2990 head—the AFM probe and the thermal probes. Before installing either of the two probes, simultaneously rotate the (two) Z-height adjusters clockwise and adjust the Z-axis motor using the software so that the cantilever is elevated well above the surface. (See the figure below for the location of the thumbscrews needed for the Z-height adjustment.) This will ensure that the probe does not collide with the surface after being mounted into the Z-axis scanner.

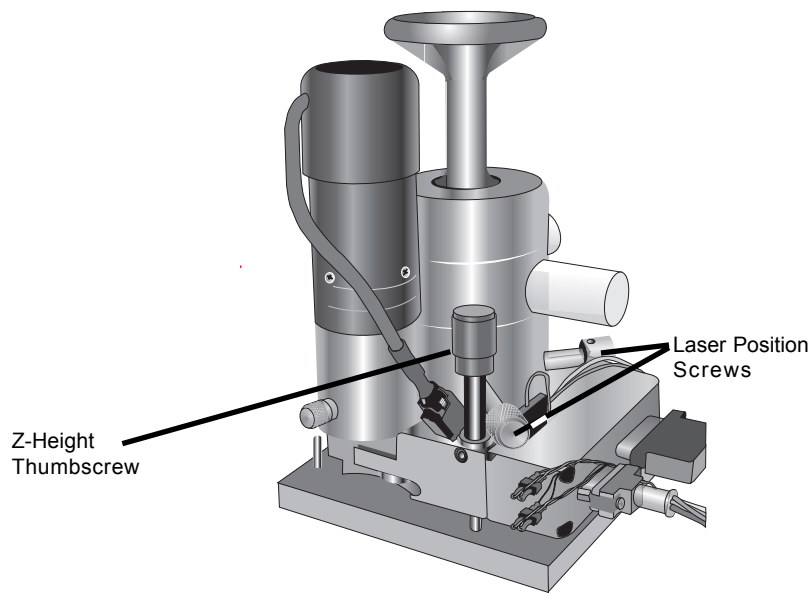


Figure 2.8
Z-Height Thumbscrews

WARNING: Never look directly into the laser beam.

Installing an AFM Probe

A box of contact topography AFM probes is supplied with your μ TA 2990 system. These probes are used to check the calibration of the scanner and to ensure that the μ TA 2990 performs in specification as an AFM. (Contact your TA representative, listed in the front matter of this manual, for scanner calibration instructions.) The contact topography probe consists of two V-type Silicon Nitride (Si_3N_4) cantilevers.

1. Turn the μ TA 2990 head upside down and place it on a flat surface.

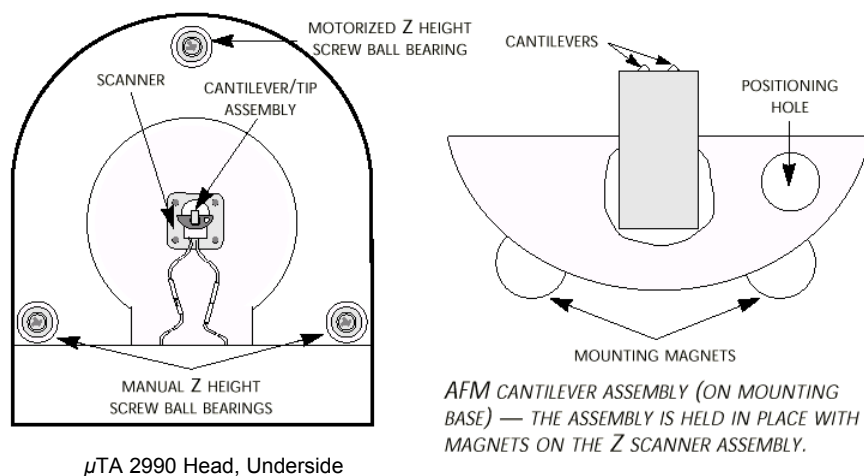


Figure 2.9
Mounting a Probe

2. Use tweezers to remove a contact topography probe from the box. The probes are held in the box by magnetic force. The easiest way to remove a probe is to slide it to the edge of the plate until it hangs slightly over the edge.

Then use the tweezers to grasp the edge that overhangs and lift the probe away from the plate.

3. Place the contact topography probe into the recessed seat on the Z-axis scanner. This seat contains a magnet to hold the probe firmly in place. For accurate measurements, the probe must be seated firmly into the holder.
4. Return the head to the upright position

5. Simultaneously rotate the (two) Z height thumbscrews counterclockwise to slowly lower the head while observing the tip position, being careful not to allow the tip to collide with anything.



CAUTION: Do not allow the tip and Z scanner to collide with the mounting or sample surface—these parts can be damaged if they come into contact.



CAUTION: Do not manually lower the tip close to the sample surface until you have started μ TALab software.

Installing Thermal Probes

Two thermal probes are required; one to measure the sample, the other to function as a reference probe.

Reference Probe

To install the reference probe on the μ TA 2990, follow these instructions:

1. Use tweezers to remove a thermal probe from the box. The thermal probes are held in the box by magnets. The easiest way to remove a probe is to slide it to the edge of the plate until it hangs slightly over the edge. Then use the tweezers to grasp the connecting wires and lift the probe away from the plate.
2. Place the thermal probe (tip upwards) on the bottom of the μ TA 2990 head (see Figure 2.1), using double-sided tape to secure it.
3. Connect the reference probe into the upper thermal probe socket, marked "REF." The thermal probe sockets are located on the right side of the μ TA 2990 head when it is held upright with the Z-axis motor facing you.

Sample Probe

To install the sample probe on the μ TA 2990, follow these instructions:

1. Turn the μ TA 2990 upside down and place it on a flat surface.
2. Using tweezers, remove a thermal probe from the box. The thermal probes are held in the box by magnets. The easiest way to remove a probe is to slide it to the edge of the plate until it hangs slightly over the edge. Then use tweezers to grasp the connecting wires and lift the probe away from the plate.
3. Place the thermal probe (with tip upwards) into the recessed seat on the Z-axis scanner. The seat of the Z-scanner contains a magnet to hold the probe firmly in place. For accurate measurements, the probe must be seated firmly into the holder.

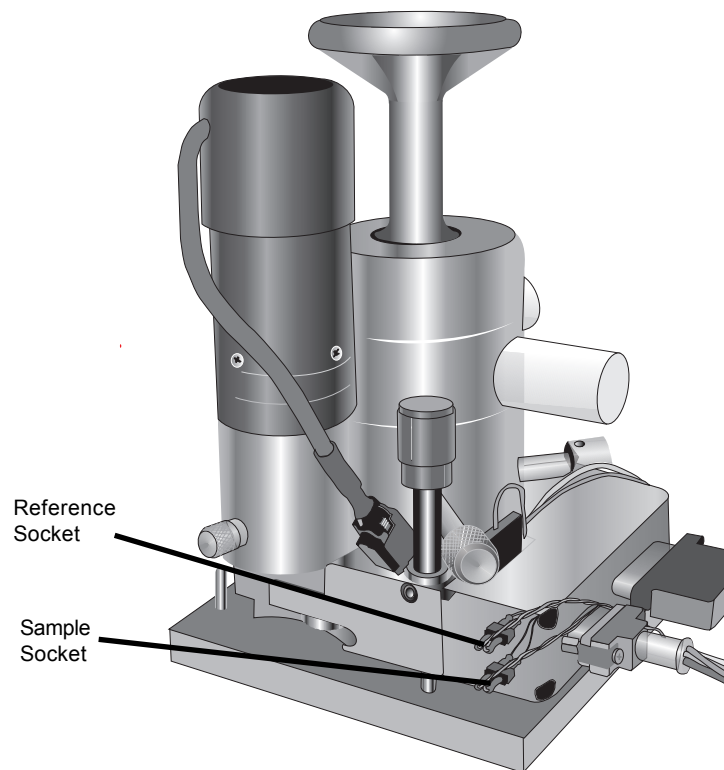


Figure 2.9
Thermal Probe Sockets

4. Connect the sample probe into the lower thermal probe socket, marked "Sample." The thermal probe sockets are located on the right side of the μ TA 2990 head when it is held upright with the Z-axis motor facing you.
5. Simultaneously rotate the (two) Z-height thumbscrews counterclockwise to slowly lower the head while observing the probe position, being careful not to allow the tip to collide with anything.

Once you have the probes installed, you can adjust and focus the camera as instructed in the next section

Adjusting and Focusing the Camera

After the camera and probes have been installed, follow these steps to adjust and focus the image:

1. Ensure that the X-adjust thumbscrew is centered in its slot or adjust its position so that the mounted cantilevers are centered in the video image.
2. For coarse focus:
 - a. Use an Allen wrench to loosen the screw at the mounting base block.
 - b. Slide the camera up and down until you achieve the best coarse focus.
 - c. Retighten the coarse focus Allen lock screw at the mounting base block.
3. Use the Y-adjust/fine focus thumbwheel to achieve the best fine focus. To adjust the location of the video field of view, use the X and Y adjustments to locate the desired feature. Tighten the fine focus adjustment locking screw.

NOTE: When the μ TALab software establishes communication with the μ TA 2990 head (via the ECU-Plus electronics), the scanner performs a quick "self-test" by moving in a square. This movement can be observed in the monochrome monitor. If this self-test fails to occur, please check all cables and reload the μ TALab software.

Chapter 3

Using the μ TA[®] 2990

Standard Operating Procedure (SOP) for Using the μ TA[®] 2990 MicroThermal Analyzer

This procedure summarizes the basic steps in making good MicroThermal Analysis experiments. The chapters in which specific information is given for each are listed in parentheses.

Once you have gained experience with the various steps involved in using the μ TA 2990 and the μ TALab software, you may find this outline a useful checklist to refer to when setting up an experiment or scan.

Start Up

1. If necessary, turn on ECU-plus and video monitor, and wait 1–2 hours to allow the instrument to reach thermal equilibrium.
2. Start the μ TALab software. Ensure that “DSP:Oh” is not visible in title bar.
3. Watch the video monitor to see whether the thermal probe performs initialization as software loads (Chapter 4).
4. Adjust I/O-T offsets (Chapter 4).
5. Check the date of probe calibration; if required, check thermal probe temperature calibration, and recalibrate (Chapter 4).

Sample Preparation and Alignment

(See also: Appendix A)

1. Mount the test sample onto a metal mount (stub).
2. Mount the metal stub onto the ambient XY translation stage.
3. Enter sample and operator information.
4. Raise legs of the μ TA 2990 so that thermal probe will not collide with the sample when the head is placed on the XY translation stage.
5. Carefully lower the head onto the translation stage. Watch on the video monitor to ensure that the probe does not collide with sample.
6. Lower the legs of the head so that the probe shadow is visible on the sample surface.
7. Translate the sample so that the thermal probe is approximately over area of interest.
8. Turn on the laser and align the laser beam on the probe mirror (Chapter 3).
9. Adjust T-B and L-R signals (Chapter 3).
10. Adjust the set point (Chapter 3; Appendix B).

Image Acquisition

1. Select the images you wish to acquire.
2. Set image size, speed, and PID settings (Chapter 5).
3. Turn on the probe and set temperature and modulation parameters (Chapter 5).
4. Lower the probe close to the surface (observe the probe shadow).
5. Press **Approach**.

6. Select **Line Scan**. Check the roughness of the sample surface, tilt, and thermal signals. Adjust scan and probe parameters accordingly (Chapter 5).
7. Collect and save the images (analyze them using μ TALab Software; Chapter 6).
8. Raise the probe tip.

Localized Thermal Analysis

1. Select **Local Thermal Analysis**.
2. Select up to 9 points on any of your acquired images for LTA measurements (Chapter 5).
3. Adjust, if required, the LTA temperature program and set point (Chapter 5).
4. Select baseline and probe cleaning options on the **Preferences** window (Chapter 5).
5. Measure LTA curves (Chapter 5).
6. Save LTA curves. Process the data using Universal Analysis Software.

Shutdown

1. Raise the back legs of the AFM head so that thermal probe is well clear of the sample.
2. Remove the sample from the XY translation stage.
3. Shut down the software.

Top of the μ TALab Main Window

The top, or instrument control part, of the μ TALab Main window is shown in Figure 3.1.

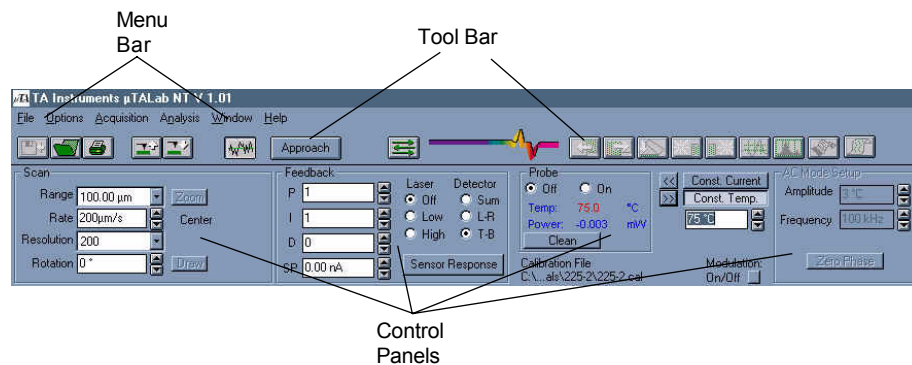


Figure 3.1
Instrument Control Part of the μ TALab Main Window

This window contains a tool bar, a menu bar, and some control panels that are used in different stages of a μ TALab experiment. As you select and use the various modes and functions, the control panels (situated below the tool bar) change to give you access to relevant control parameters.

NOTE: If a menu item or tool bar button is "grayed out" (disabled), the command or function it represents is not available either within the current operating mode or with the window currently selected. For example, the image analysis tool bar buttons are grayed out when the active window (the window that is currently selected) is not an image window.

NOTE: Because the screen colors and image palettes are user selectable within the μ TALab software, the colors used in the figures in this manual may not exactly match your own system colors.

The tool bar and its buttons are described in the section "Using the Tool Bar Buttons."

The control panels on the **Main** window, which are used to set up and control experiments, are explained in Chapter 5.

The six menus in the menu bar are explained in the section "Using the Menu Bar."

Using the Menus

Throughout the program, drop-down menus are available, such as the one shown in Figure 3.2.

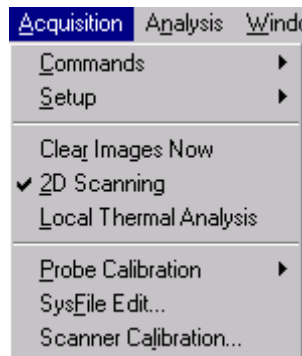


Figure 3.2
The Acquisition Menu,
a Drop-Down Menu on
Menu Bar

Drop-down menus often appear when you click on a menu or submenu item. To choose an item on a menu, drag the mouse to highlight your choice and release the mouse button.

Using the Menu Bar

The menu bar on the Main window of the μ TALab Software (see Figure 3.1) contains the following menus:

- **File** menu
- **Options** menu
- **Acquisition** menu
- **Analysis** menu
- **Window** menu
- **Help** menu

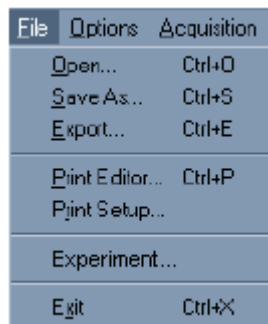
The **Window** menu contains the standard items available for any Microsoft Windows[®] NT* program—**Next**, **Previous**, **Tile**, and **Cascade**. Please refer to your Windows[®] NT documentation for explanations. The other menus are explained in varying detail here.

NOTE: This documentation assumes that the user is familiar with the operation of Windows software. For more information, refer to the manuals specific to Windows that were supplied with your PC.

File Menu

The **File** menu (shown in Figure 3.3) contains items that allow you to open and close files, save data in different formats, and arrange the data for printing.




Table 3.1, on the next page, explains the items available on the **File** menu. If an item can also be accessed with a tool bar or other button, the button is shown below the item name.



File	Options	Acquisition
Open...		Ctrl+O
Save As...		Ctrl+S
Export...		Ctrl+E
Print Editor...		Ctrl+P
Print Setup...		
Experiment...		
Exit		Ctrl+X

Figure 3.3
File Menu

Table 3.1
Items on the File Menu

Menu Item	Explanation
Open 	Opens the File Manager window, which displays all files with image (e.g., ".tfr") or LTA file format. Standard file management tools are available from this window, such as Delete, Copy, and Rename (see Chapter 7).
Save As... 	Opens the Save Data As... window, which allows you to save the currently selected data, either images or LTA data (see Chapter 7).
Export...	Opens the Export Data window, which allows you to save data in the ASCII text format ("*.txt"), so that it may be used by other applications. This feature allows you to export graphs, images, and oscilloscope displays (see Chapter 7).
Print Editor... 	Opens the Print Editor so that you may optimize and format the position of your data prior to printing (see Chapter 7).
Print Setup	Displays the standard Windows Print Setup window, which enables you to select or change the printer, paper orientation, and paper size prior to printing (see Chapter 7).
Experiment...	Opens the Sample and Experiment window, which enables you to enter the operator and sample information either before or after running a sample. The operator and sample information is stored in the LTA data files.
Exit	Closes down the μ TALab Software after prompting you to save any unsaved data.

Options Menu

The **Options** menu (shown in Figure 3.4) contains functions that allow you to customize the acquisition parameters, changes default preferences in the software, and change the color and other display parameters of your images.



Figure 3.4
Options Menu

Table 3.2 contains explanations of the **Options** menu items. If an item can also be accessed with a tool bar or other button, the button is shown below the item name.

Table 3.2
Items on the Options Menu

Menu Item	Explanation
Acquire	Opens the Acquisition Setup window, which allows you to select images and set parameters for acquisition (see Chapter 5).
Preferences	Opens the Preferences window, with which you can change default settings for some of the activities and displays (see Chapter 5).
Screen Colors	Opens the Color Settings window, which allows you to change some colors used in μ TALab™ displays. All changes made in the Color Settings window are saved when you exit μ TALab software.
Contrast & Brightness	Opens the Contrast & Brightness window, with which you can change the contrast and brightness of the screen display (see Chapter 6).
Palettes	Opens the Palettes window, which enables you to change the color palette used to display an image (see Chapter 6).

Acquisition Menu

The **Acquisition** menu, shown in Figure 3.5, gives you access to the controls you use to set up and acquire scanning and LTA data. Some of the items (Commands, Setup, and Probe Calibration) are actually submenus giving access to two or more items.

Table 3.3 contains explanations of the **Acquisition** menu items. If an item can also be accessed with a tool bar or other button, the button is shown below the item name.

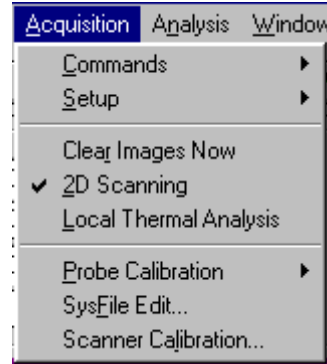


Figure 3.5
Acquisition Menu

Table 3.3 Items on the
Acquisition Menu



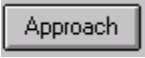
Menu Item	Explanation
Commands Submenu	
Start Scan 	Toggles the microscope to begin scanning (image acquisition) and activates the Display control panel (see Chapter 5).
Oscilloscope 	Opens/closes the Oscilloscope window (see Chapter 5).
Setup Submenu	
Approach 	Automatically lowers the probe until it contacts the surface of the sample.
Calibrate IOT	Opens the I/O-T Offset Calibration window (see Chapter 4).
<i>(table continued on next page)</i>	

Table 3.3
Items on the Acquisition Menu (cont'd)

Menu Item	Explanation
Setup Submenu	
Clear Images Now	Clears all images within all open image windows.
2D Scanning	Selects two-dimensional (2D) scanning (image acquisition) as the current experimental mode.
Local Thermal Analysis	Selects Localized Thermal Analysis as the current experimental mode.
Probe Calibration Submenu	
<i>Load</i>	Opens the Calibration Data window so that you may recall a previously saved thermal probe temperature calibration data file (see Chapter 4).
<i>Generate</i>	Opens the current temperature Calibration window and displays the thermal probe calibration graph currently in use by the μ TALab™ software (see Chapter 4).
SysFile Edit	Opens the System File Edit window (see Appendix B).
Scanner Calibration	For information on scanner calibration, see Appendix E.

Analysis Menu

The **Analysis** menu, shown in Figure 3.6, gives you access to the tools and control panels you can use to analyze and edit the data obtained with scanning.

Please see Chapter 6 for a full explanation of the items available on this menu.

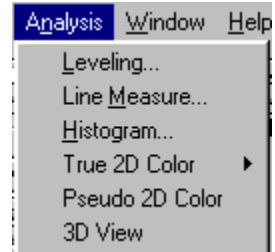


Figure 3.6
Analysis Menu

Using the Tool Bar Buttons

For your convenience and enhanced productivity, commonly used menu items are duplicated and placed on the tool bar, which is located underneath the menu bar and above the control panels (see Figure 3.1). Resting the cursor on any of the buttons will display a Tool Tip, which describes the function of that button. (If this does not happen, check the box “**Show Tool Tips**” in the **Preferences** window, which you can open from the **Options** menu.)

The tool bar is divided into two parts:

- To the *left* of the TA Instruments logo are buttons used specifically for handling files and acquiring images with the μ TA 2990; they are shown in Figure 3.7.
- To the *right* of the logo are buttons for enhancing and analyzing images that have already been acquired; they are shown in Figure 3.8.

Both sets of buttons are described in this section.



Figure 3.7
Tool Bar Buttons to Left of TA Logo



Figure 3.8
Tool Bar Buttons to Right of TA Logo

File Handling and Image Acquisition Buttons

The tool bar buttons to the left of the TA Instruments logo (see Figure 3.7) access the functions you need to handle files and acquire images. They are explained in Table 3.4.

Table 3.4
Tool Bar Buttons for File Handling and Image Acquisition







Button	Explanation
Save As 	Opens the custom Save Data As window. From this window, you can save any or all of the images and/or μ TA™ data displayed within the μ TALab™ window.
File Open 	Opens the custom File Manager window, which displays all image and LTA data files. Same as Open on the File menu.
Print 	Opens the Print Editor so that you may customize your data prior to printing (see Chapter 7).
Tip Up 	Engages the Z-axis motor to raise the probe away from the surface of the sample. The Z motor is on all the time this button is pressed.
Tip Down 	Engages the Z-axis motor to lower the probe toward the surface of the sample. The Z motor is on all the time this button is pressed.
	<p>When lowering the tip to the sample surface, always monitor the process in the monochrome monitor to ensure that the probe is not damaged by hitting the surface.</p>
	<i>(table continued on next page)</i>

Table 3.4
Tool Bar Buttons for File Handling and Image Acquisition (cont'd)


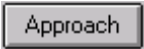






Button	Explanation
<p data-bbox="354 510 509 573">Oscilloscope On/Off</p> 	<p data-bbox="667 510 1073 573">Toggles the Oscilloscope window on and off (see Chapter 5).</p>
<p data-bbox="354 699 477 730">Approach</p> 	<p data-bbox="667 699 1195 793">Engages the Z-axis motor to lower the probe until the set-point (SP) in the Feedback panel value is reached (see Chapter 5).</p>
<p data-bbox="354 856 509 888">Scan On/Off</p> 	<p data-bbox="667 856 1195 951">Starts and stops the scanning process. During scanning, the Display panel becomes active (see Chapter 5).</p>

Image Enhancement and Image Analysis Buttons





The tool bar buttons to the right of the TA Instruments logo (see Figure 3.8) access the functions you need for enhancing and measuring images. They are explained in Table 3.5.

Table 3.5
Tool Bar Buttons for Image Enhancement and Analysis

Button	Explanation
Previous Buffer 	Selects the previous image in the buffer array. This button is disabled ("grayed out") if there are no previous images in the array sequence (see Chapter 6).
Next Buffer 	Selects the next image in the buffer array. This button is disabled if there are no further images in the array sequence (see Chapter 6).
Quick Leveling 	Performs a first-order, two-dimensional leveling on the currently selected image (see Chapter 6).
Left Shading 	Performs left light-source shading on the currently selected image (see Chapter 6).
Right Shading 	Performs right light-source shading on the currently selected image (see Chapter 6).

(table continued on next page)

Table 3.5
Tool Bar Buttons for Image Enhancement and Analysis (cont'd)

Button	Explanation
<p data-bbox="354 554 526 583">Line Measure</p> 	<p data-bbox="667 554 1195 680">Opens the Line Measure window, which enables you to create and compare up to three line profiles from the selected image (see Chapter 6).</p>
<p data-bbox="354 716 483 745">Histogram</p> 	<p data-bbox="667 716 1195 806">Opens the Histogram window, which enables you to edit the image's color histogram (see Chapter 6).</p>
<p data-bbox="354 873 493 903">3D Display</p> 	<p data-bbox="667 873 1203 934">Displays the data in the currently active image window as a three-dimensional image.</p>
<p data-bbox="354 1037 483 1087">Document Info</p> 	<p data-bbox="667 1037 1211 1127">Opens the File Info window, which displays the main acquisition information relating to an image you have selected.</p>

Using Keyboard Shortcuts

Like many Windows® NT and other computer programs, μ TALab™ Software allows you to perform some functions by using combinations of keystrokes, or keyboard shortcuts, instead of using the mouse.

The name of each drop-down menu and each item on the menus contains an underlined letter (for example, on the menu bar: **F**ile, **O**ptions, **A**cquisition, **A**nalysis, **W**indow, and **H**elp), which indicates the keyboard shortcut to that menu or item. To access any item with the keyboard, hold down the ALT key on your keyboard (usually situated to the left of the spacebar) and, *at the same time*, press the letter key on the keyboard that corresponds to the underlined letter in the menu item's name.

For example, if you want to perform a Line Measure on an image, you would hold down the ALT key and press “N” (to open the **A**nalysis menu) followed by “M” (to select **L**ine **M**easure).

Also, you can access the commonly used items in the **F**ile menu (**O**pen, **S**ave, **E**xport, **P**rint, and **E**xit) at any time by selecting CTRL followed by the underlined letter. For example, if you hold down the CTRL key and press the “X” key (for **E**xit), the μ TALab software will close down (after prompting you to save any unsaved data).

As you become familiar with the program, you will probably learn some of the shortcuts for the functions you perform most often. Some people find this method quicker than using the mouse.

X,Y Translation and Z Adjustment

The μ TA 2990 uses manual adjustment to make large Z (height) changes and to accommodate samples of different thicknesses. To accomplish fine Z adjustment, the μ TALab Software interface is used to control the head's Z motor. The sample translator base allows manual X and Y translation of the sample. Follow the instructions found on the next several pages to perform these functions.

X,Y Translation

The μ TA 2990 system includes an X,Y translator base (see the figure below) that can be used both as a scanning platform for the instrument and as a fine manual sample translator.

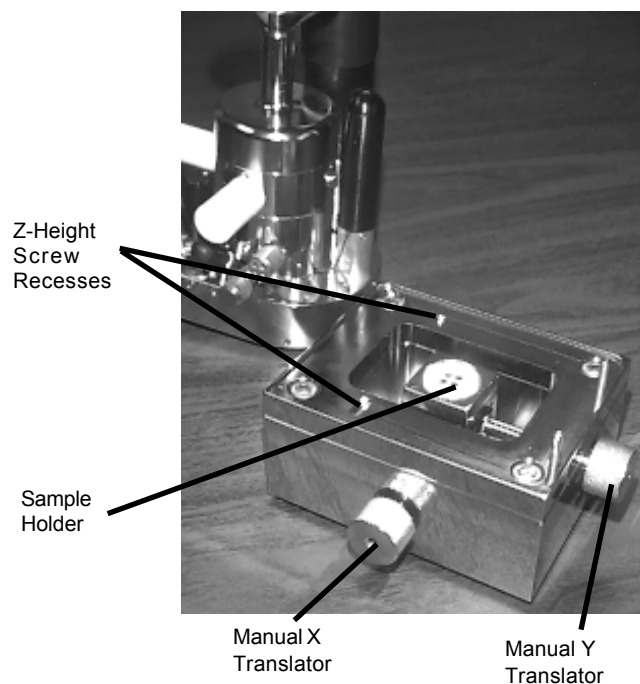


Figure 3.9
 μ TA 2990 Head and X,Y Translator Base

Mount the μ TA 2990 head on the translator so that the two manual Z-height screw ball bearings (on the underside of the head) fit into the two recesses on either side of the sample bay (see the figure on the previous page).

Performing the Translation:

After mounting the sample on the sample holder and the head on the translator platform, you can translate the sample by turning the X and Y knobs—while monitoring the sample's position relative to the probe using the video monitor. The sample holder contains magnets for mounting metallic samples or sample holders. Nonmetallic types of samples can be mounted on the sample holders (mounts) with double-sided tape or an appropriate adhesive.



CAUTION: The tip and Z-axis scanner can be damaged if it collides with the sample surface. Lower the head carefully while directly observing the Z-axis scanner and the sample surface. When the Z-axis scanner gets close to the sample surface, observe the final lowering of the cantilever on the video microscope monitor to ensure that the cantilever does not contact the surface. Use the probe height thumbscrews to bring the cantilever's Z position close to, but not touching, its shadow on the sample surface.

Manual Z Adjustment

Coarse Z adjustment is accomplished by simultaneously rotating the two Z-height thumbscrews (see the figure below) clockwise to raise the head and cantilever and counterclockwise to lower the head.

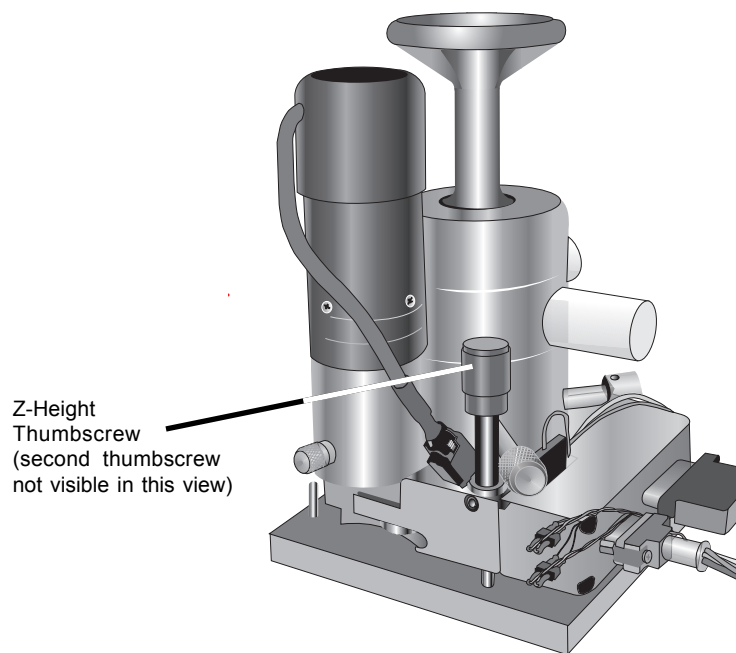




Figure 3.10
Z Height Adjustment Thumbscrews

Fine Z Adjustment of the cantilever / tip is performed with the Tip Up / Tip Down buttons,  , located on the μTALab Software Tool Bar



CAUTION: Use the Tip Down control carefully. DO NOT lower the tip to the point where it is touching the sample surface; damage to the tip, sample and/or Z-axis scanner may result. Use the probe shadow (seen on the sample surface) in the video monitor to judge tip/sample proximity.

Aligning the Laser Beam

Each time a probe is exchanged or any alignment adjustments are altered, the laser must be aligned for proper feedback. Three components must be adjusted:

- Laser
- Photodetector
- Mirror

When properly aligned, the laser light beam bounces off the back side of the cantilever or mirror to the adjustable mirror, then onto the center of the quadrant photodetector (see the figure to the right).

The objectives of the alignment procedure are to maximize the signal generated by the beam at the photodetector and to ensure that nearly equal amounts of light are hitting each quadrant, as seen in the picture below.

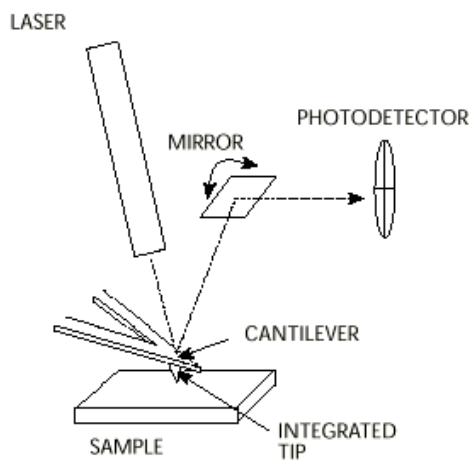
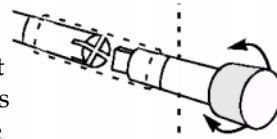


Figure 3.11
Laser, Mirror, Quadrant
Photodetector Configuration



Adjustment Screws

Unless engaged, the laser, mirror, and photodetector alignment thumbscrews (shown here) all rotate freely without affecting adjustments. To engage an alignment screw and make an alignment adjustment, apply slight pressure inward while rotating slowly. When you feel the thumbscrew assembly slip into one of its slots, the knob will move in slightly and there will be more resistance as you rotate. At this point, you have engaged the adjustment mechanism. Once an adjustment has been completed, disengage the thumbscrew by pulling it straight out, ensuring that it rotates freely again. Disengaging a thumbscrew serves two functions: the adjustment screw setting cannot be accidentally changed, and the interior scanning tower is isolated for stability.




CAUTION: When the photodetector thumbscrew is engaged and rotated to either end of its range, a torque-release mechanism will automatically slip, preventing you from over-torquing the photodetector assembly. To avoid damaging the assembly, do not rotate beyond the point at which the torque release stops the adjustment. When engaged, the mirror and laser alignment screws will come to a hard stop at the end of their adjustment range. Do not attempt to rotate a thumbscrews beyond this point.



CAUTION: After any alignment thumbscrew adjustment, always disengage the screw (by pulling it straight out). Failure to do so can prevent the system from scanning properly.

Performing the Alignments

Many of the steps needed to align the laser require use of the μ TALab Software—make sure you have installed the software as directed in Chapter 2.

1. Ensure that a probe is mounted in the μ TA 2990 head. (See Chapter 2 for instructions, if needed.)
2. Press the **Oscilloscope** on/off button, , on the μ TALab tool bar to open the **Oscilloscope** window, shown below.

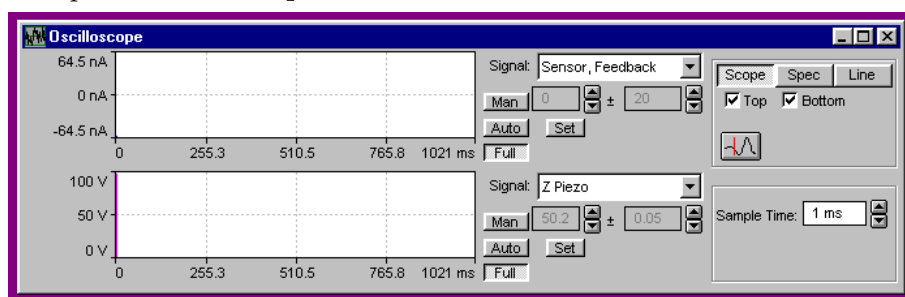


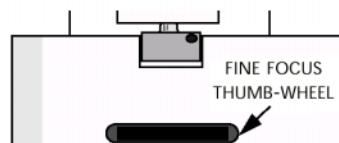
Figure 3.12
Oscilloscope Window

3. Select **Sensor, Feedback** from the drop-down **Signal** list of one of the traces.
4. Then, at the **Sensor, Feedback Signal** window, select **Full**.



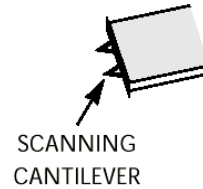
WARNING: Never look directly into the laser beam.

5. If required, adjust the focus thumbwheel (shown in the figure here) on the μ TA 2990 base to achieve optimum focus of the probe for your eyesight. Adjust the focus until the image comes into sharp focus for you.



6. Adjust the laser beam as follows:

- a. *For AFM contact topography probes:* Adjust the laser alignment screws to move the laser beam so it is visible on the tip of the cantilever (as seen in the video monitor). For most applications, alignment and scanning should be performed with the cantilever closest to you as viewed on the video monitor. This is the cantilever that is mounted and tested for optimal alignment.



If you are unable to see the laser beam reflecting off the cantilever, the beam is probably nearby but is difficult to detect because it is not reflecting off the cantilever or its support. If this is the case, it is best to move the laser beam to the cantilever support, then use the laser adjustment screws to move the beam to the apex of the V-shaped cantilever you are using for the scan. You may need to lower the optical microscope's bulb brightness level (with the control on the

Microscope Control Unit electronics) to adequately see the laser beam on the tip of the cantilever. See the figure to the right for the best placement of the laser beam.

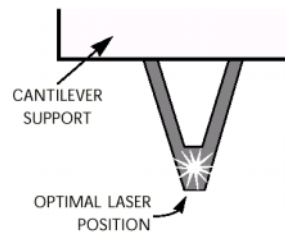


Figure 3.13
Optimal Laser Beam Placement

- b. *For thermal probes:* Adjust the laser alignment screws (shown in the figure on the next page), to move the laser beam on to the mirror located on the top of the thermal probe.

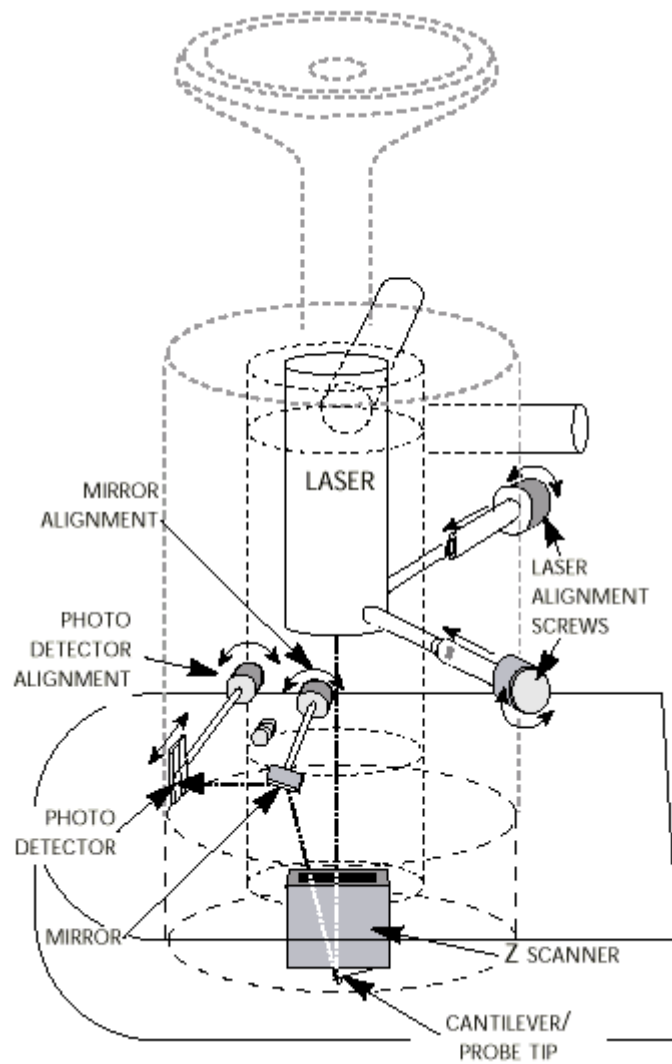


Figure 3.14
 μ TA 2990 Head Showing Main Adjustment Controls

Next you will need to perform the top-to-bottom (T-B) alignment and left-to-right (L-R) steps, found in the next two sections, to place the laser beam in the center of the four-quadrant photodetector.

Top-to-Bottom (T-B) Mirror Alignment

The mirror, shown in Figure 3.11, bounces the laser beam onto the center of the four-quadrant photodetector. Follow the instructions below to properly align the mirror in top-to-bottom orientation.

1. Locate the **Feedback panel** (shown in the figure below) on the window.

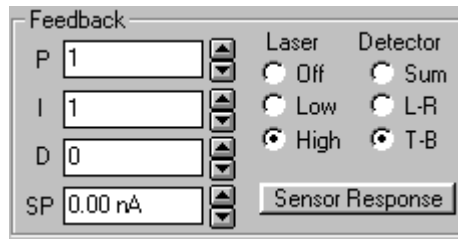


Figure 3.15
Feedback Panel

2. Set the **Laser** setting to **High** and the **Detector** to **T-B** (top-to-bottom). This puts the four-quadrant photodetector in top-bottom alignment mode.
3. Adjust the mirror alignment screw until the **Sensor, Feedback** signal trace crosses the 0 nA level as observed in the **Oscilloscope** window (see Figure 3.12). Then adjust the mirror alignment screw until the **Sensor, Feedback** signal trace is set as close as possible to zero. A reading of less than 5 nA is acceptable.

NOTE: A reading of less than 5 nA for the T-B signal can be obtained under two sets of conditions: The laser either is off of the photodetector completely or is centered between the top and bottom halves of the photodetector. However, the signal trace will be able to "cross 0 nA" only if the laser beam is centered between the top and bottom halves of the photodetector.

Left-to-Right (L-R) Photodetector Alignment

Follow the instructions below to properly center the laser beam on the left and right halves of the photodetector.

1. Locate the **Feedback panel** (shown in the figure below) on the window.

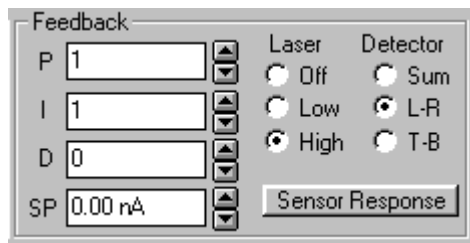


Figure 3.16
Feedback Panel

2. Set the **Laser** setting to "High" and the **Detector** to **L-R** (left-to-right). This puts the four-quadrant photodetector in left-right alignment mode.



WARNING: Never look directly into the laser beam.

3. Adjust the photodetector thumbscrew until the **Sensor, Feedback** signal trace crosses the 0 nA level. Then adjust the mirror alignment screw until the **Sensor, Feedback** signal trace is set as close as possible to zero. A reading of less than 5 nA is acceptable. If you reach one extreme of the photodetector alignment screw's range without being able to bring the signal level into both the negative and positive regions, try adjusting the screw in the opposite direction.

NOTE: A reading of less than 5 nA for the L-R signal can be obtained under two sets of conditions: Either the laser is off of the photodetector completely, or the laser beam is centered between the top and bottom halves of the photodetector. However, the signal trace will be able to "cross 0 nA" only if the laser beam is centered between the top and bottom halves of the photodetector.

The laser beam should now be located in the center of the four-quadrant photodetector. If it is not centered, repeat the alignment procedures beginning in the section "Aligning the Laser Beam."

Troubleshooting

Try one or more of the following solutions to adjust and align the mirror properly:

- If you reach one extreme of the mirror alignment screw's range without being able to bring the signal level into both the negative and positive regions, try adjusting the screw in the opposite direction.
- If mirror adjustment has little or no effect on the T-B signal, the laser may not be properly hitting the apex of the cantilever (for an AFM contact topography probe, or the mirror on the thermal probe). Repeat step 6 on page 82, then repeat step 3 on page 84.
- If after repeated attempts to align the laser signal, you do not obtain an appropriate response, the probe you are using may be damaged or out of alignment. Turn the laser off (using the radio button on the **Feedback** panel shown in the Figure 3.15); install a new probe, and start again from the beginning of the procedure on page 81.

Now you will need to perform the next set of instructions to finely adjust the laser beam.

Fine Adjustments

Fine adjustment of the laser consists of performing three alignments: sum alignment, T-B alignment (repeated), and L-R alignment (repeated).

Sum Alignment (Laser Adjust)

1. Locate the **Feedback** panel.
2. Set the **Detector** to **Sum**. In this mode, the photodetector quadrants are summed, allowing fine adjustments of the laser alignment on the probe to be made.



WARNING: Never look directly into the laser beam.

3. Carefully make very fine adjustments to the laser alignment screws (one at a time) in order to (a) adjust the position of the laser beam on the mirror of the thermal probe and (b) maximize the sensor current as displayed on the **Oscilloscope** window's **Sensor, Feedback** trace. (Typical values are greater than 20 nA.)

Next you will need to perform the T–B alignment again, as instructed in the next section, for fine adjustment.

T–B Mirror Fine Alignment

1. Locate the **Feedback panel** and set the **Detector** to **T–B** again.
2. Finely adjust the mirror until the **Sensor, Feedback** signal crosses as close as possible to the 0 nA level (± 5 nA is acceptable). (You should be able to adjust the mirror to bring the signal level into the negative and positive regions).
3. Adjust the mirror until the signal is at or near the 0 nA level.

Next you will need to perform L–R alignment again, as described in the next section.

L–R Photodetector Fine Alignment

1. Locate the **Feedback panel** and set the **Detector** to **L–R** again.
2. Adjust the photodetector until the **Sensor, Feedback** signal crosses over the 0 nA level. (You should be able to adjust the photodetector to bring the signal level into the negative and positive regions).
3. Adjust the photodetector until the signal is as close as possible to the 0 nA level (± 2 nA is acceptable). This step readjusts the beam to center it between the right and left halves of the photodetector again.
4. Disengage all of the alignment thumbscrews (shown in Figure 3.14) by gently pulling them straight out.

NOTE: The laser should now stay in alignment as long as the probe is in place. Unless you accidentally change the laser, photodetector, or mirror adjustments, raising and lowering the μ TA 2990™ head for sample replacement should not affect laser alignment.

Running a Performance Check

Now that you have installed and aligned your μ TA 2990 system, you can run some simple experiments to make sure that everything is functioning correctly. These experiments are divided into three parts:

- Running a test grid with an AFM probe
- Running a test grid with a thermal probe
- Obtaining localized thermal analysis data on a polymer sample

NOTE: You can use these experiments again later if you find that you are getting strange or inconsistent results with your own samples. Use these experiments to make sure that your system is working correctly.

The procedures found in this section refer to the use of the μ TALab Software without a full description of each function. Detailed descriptions of the individual μ TALab tools and functions are provided in Chapters 4 through 6 of this manual. The objective of this section is simply to provide the essential information you need to perform some successful Micro-Thermal Analysis experiments.

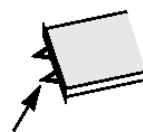
Use the following instructions to test your μ TA 2990 system setup and establish the proper sequence of steps required for the acquisition of images and Localized Thermal Analysis data.

Before you begin, you must have installed your scanner into the μ TA 2990 head, installed the μ TALab™ Software, and aligned the laser as already directed in Chapters 2 and 3 of this manual.

Using the AFM Probe

This section teaches you how to run a test grid using the AFM probe. Follow the instructions below:

1. Locate the Semiconductor standard calibration grid, found in your Micro-Thermal Analysis Sample Kit. This will be your test sample.
2. Using tweezers, mount the calibration grid onto the X-Y sample holder/translator.
3. Using tweezers, mount an AFM contact topography probe onto the magnetic seat of the Z-scanner assembly. (See Chapter 2, "Installing Probes," for instructions.)
4. Align the laser beam onto the closest of the two cantilevers. (See "Aligning the Laser Beam" in this chapter for a detailed procedure.)
5. Translate the calibration grid so the cantilever is positioned over section "E" (See "X,Y Translation/Z Adjustment" in this chapter for a detailed procedure). As viewed on the video monitor, the calibration sample grids can be navigated using the "TopoMetrix" legend as a reference point. See the figure on the next page.



SCANNING
CANTILEVER

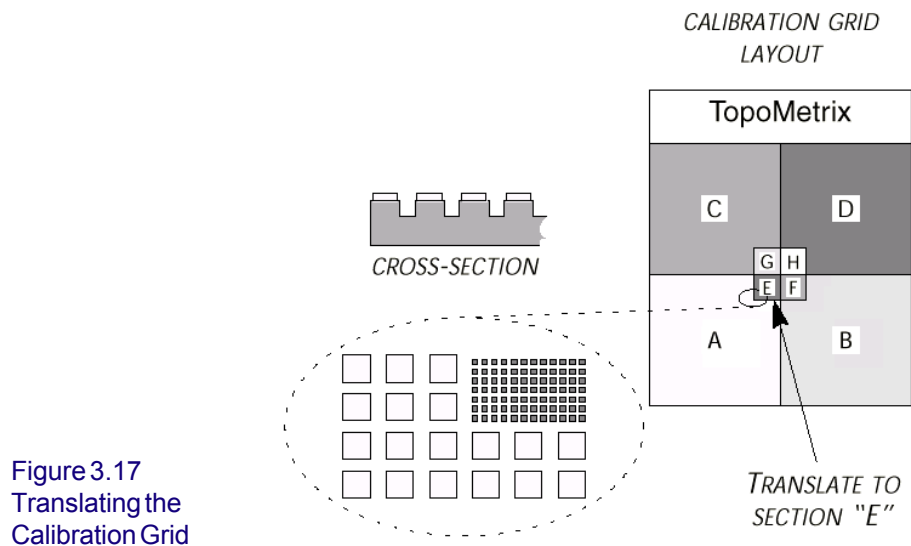
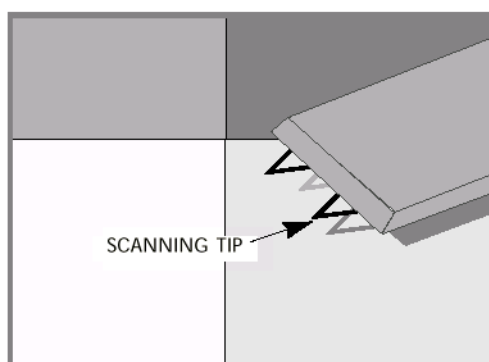
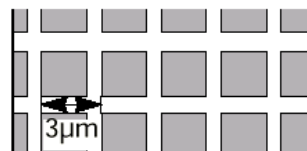


Figure 3.17
Translating the
Calibration Grid

Section E consists of raised SiO_2 squares with a $3\text{-}\mu\text{m}$ pitch (shown here).

When you have completed the setup and alignment, look at the monochrome monitor. You should see an image of both the tip and the sample as shown in the figure below:



SPM TIP AND ITS SHADOW ON SAMPLE

Figure 3.18
Tip and Sample

6. Monitor the cantilevers in the video monitor while you use the manual Z-height adjustment screws to lower the scanning cantilever near the sample surface. This distance is best judged by lowering the tip until it casts a shadow on the sample surface. The distance between the tip and shadow should be adjusted until it appears to be about one-fifth of the length of the cantilever arm. This is the coarse adjustment to bring the cantilever within scanning range on samples of various thicknesses.

NOTE: Make sure that the cantilever base is SLIGHTLY tilted (as set up by the Z-height thumbscrews). The tilt should be great enough so that the unused cantilever is slightly higher than the scanning cantilever. This is determined by observing that the scanning tip's shadow is cast on the sample surface closer than the nonscanning tip's shadow (as shown in Figure 3.18). The cantilever base SHOULD NOT be tilted so much that the corner of the cantilever base touches the sample surface, which would degrade imaging and possibly damage the sample.

7. Open the μ TALab **Oscilloscope** window (shown in Figure 3.12) and ensure that both the **Sensor, Feedback** signal and the **Z-Piezo** signal are displayed at full scale.
8. Access the **Scan panel**; then set the scan parameters to the settings shown in the figure below:

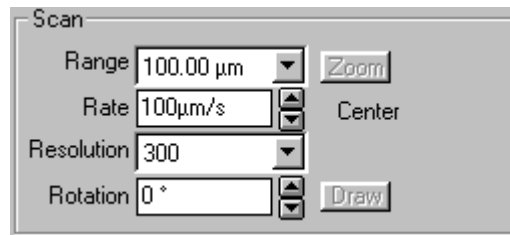


Figure 3.19
Scan Panel Settings

- Access the **Feedback panel**; then set the feedback parameters to the setting shown in the figure below:

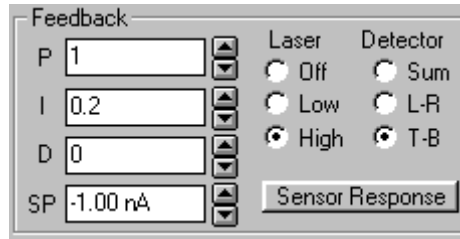


Figure 3.20
Feedback Panel Settings

- Select **Options/Acquire** from the menu bar to open the **Acquisition Setup** window. Then select the following settings (as also shown in Figure 3.21):

- **Topography (Fwd)**: checked on
- **Sensor (Fwd)**: checked on
- **Z Piezo: Hi**
- **Z Setpoint: 50V**

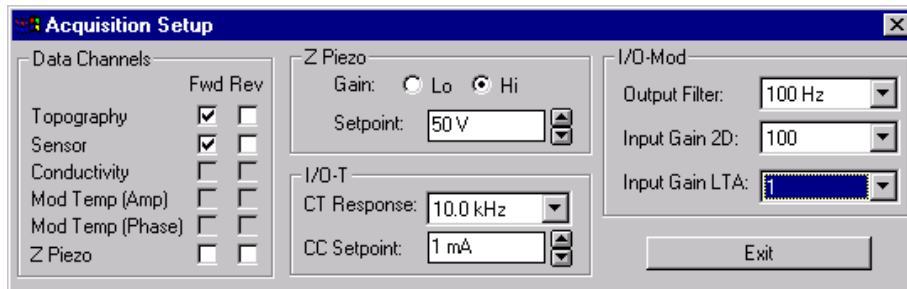



Figure 3.21
Acquisition Setup Window

- Leave all other options at their default settings, and click on the **EXIT** button.
- Select **Options/Preferences** from the menu to open the **Preferences** window, shown in Figure 3.22 on the next page.

- Check the following **Tip Approach** options: **Test for False Feedback** and **Determine Sensor Response**.

- Select the **Display Defaults** options shown in the figure to the right.
- Leave all other options in their default settings as shown.
- Click the **OK** button to close the window.

13. Use the **Tip Down** button, , on the tool bar to lower the cantilever tip toward the sample until the tip is close to, **BUT NOT TOUCHING**, the test grid surface.
14. Monitor the cantilever tip in the video display while you use the cantilever shadow on the test grid surface to judge the distance between the tip and the sample. The scanning cantilever should be close to, but not touching, its own shadow. The final position of the μ TA 2990 head, before the computer-controlled approach is engaged, is as follows: Rear slightly tilted toward the cantilever to be used and front slightly tilted closer to the sample than the rear.

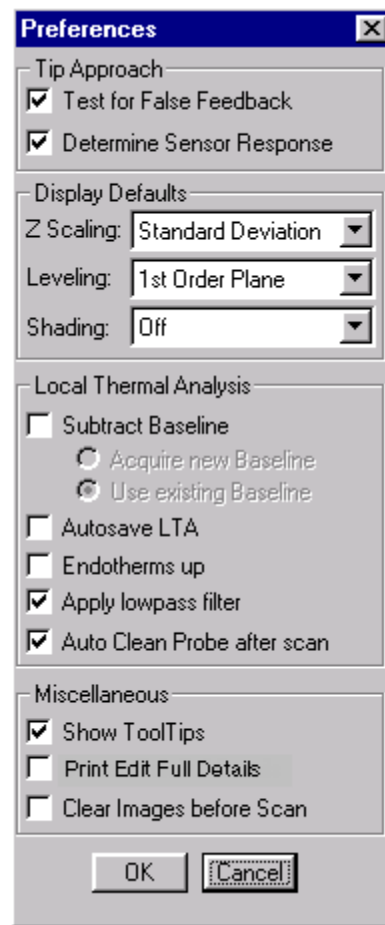
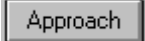


Figure 3.22
Preferences Window


15. Monitor the **Sensor, Feedback** signal in the **Oscilloscope** window while you perform the following steps:
 - Adjust the mirror control on the μ TA 2990 head until the **Sensor** signal is more than 50 nA below the value in the SP (Set Point) field (-1.0 nA). Ensure that the signal has not reached its maximum negative amplitude limit.
 - Disengage the mirror adjustment screw by pulling it straight out. This slightly negative internal feedback sensor signal level will allow positive cantilever deflection to be detected when the tip first touches the sample surface. This, in turn, will help achieve stable feedback.

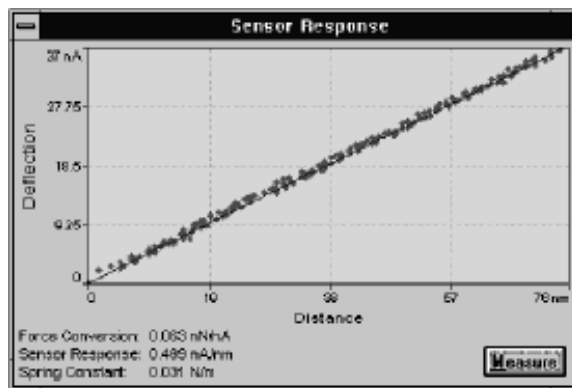
16. Place the Environmental Cover over the μ TA 2990 head.

17. Click on the **Approach** button, :

The tip will move to the sample under software control and the system should go into feedback. As the tip engages and enters feedback, you should notice the following two simultaneous effects in the **Oscilloscope** window:

- The **Sensor Feedback** signal slowly lowers, then quickly jumps back to a midpoint reading (~ 0 nA).
- The **Z-piezo** signal displays a momentary spike.

18. Verify that the system is in feedback by clicking on the **Sensor Response** button, , on the **Feedback panel**. A positive sloping Deflection versus Distance curve should be displayed (as seen in the figure below). If the tip is in feedback, close the window and perform step 1 in the section entitled "Scanning."



IDEAL SENSOR RESPONSE CURVE — "TRUE FEEDBACK"

Figure 3.23
Ideal Sensor Response Curve

False Feedback

If the system displays the following error message, it is probably in false feedback; the sensor response curve appears as shown in Figure 3.24. The data points are widely scattered and generally have an overall negative slope.

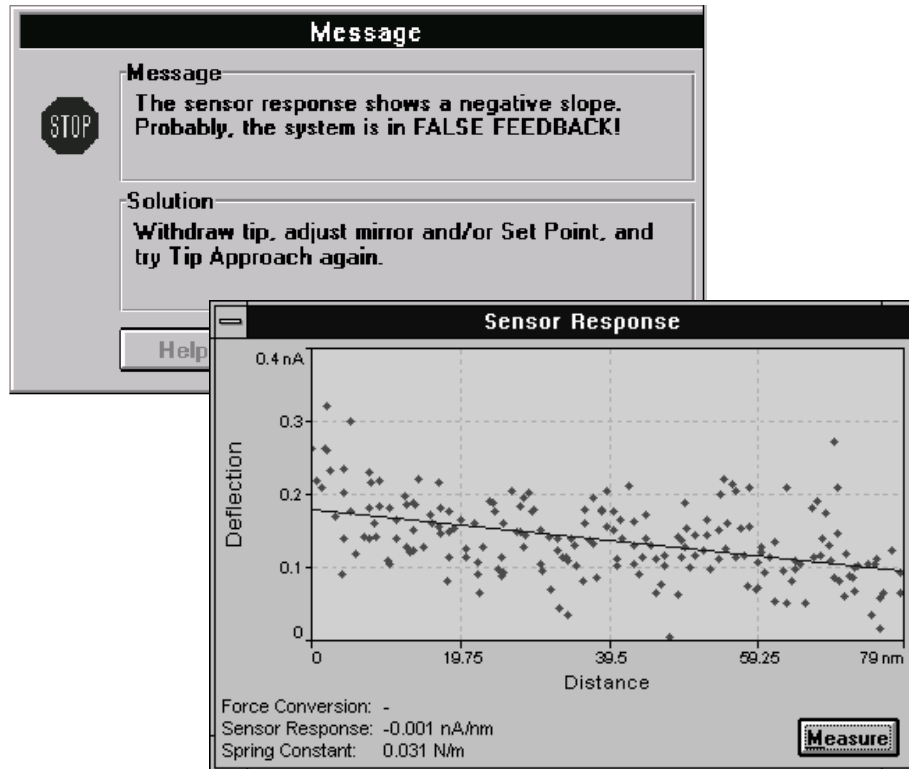



Figure 3.24
False Feedback Error Message and Sensor Response Curve



If this is the case, perform the following actions:

1. Click **OK** to exit the error message.
2. Click once on the **Tip Up** button, , to raise the tip and get out of false feedback.

3. Monitor the **Sensor, Feedback** trace in the **Oscilloscope** window while you adjust the mirror to lower the internal sensor current $\sim 5\text{-}10\text{nA}$ (*i.e.*, more negative).
4. Turn to page 93 and repeat steps 13, 14, 16, 17, and 18 to achieve “true feedback.”

Scanning

After you have followed the procedures needed to run a test grid with an AFM probe and have achieved “true feedback” as shown in Figure 3.23 on page 94, follow the instructions below to scan the sample.

1. Click on the **Oscilloscope On/Off** button, , on the μ TALab tool bar to close the **Oscilloscope** window and allow a clear view of the **Scan** window.
2. Click on the **Start Scan** button, , to start the scan and image acquisition. As each line is scanned, the topography and sensor images (selected in step 10 on page 92) are updated.

Proceed to the next section to set and optimize the feedback parameters and obtain the best image resolution.

Feedback Parameters

Once the system is in feedback and successful scanning is in progress, it may be necessary to optimize the feedback parameters—*proportional*, *integral*, and *derivative gain* settings (PIDs)—to achieve the best possible image resolution.

The proper settings for proportional, integral, and derivative gain are essential to acquire accurate, high-resolution AFM images with the AFM probe. The proper adjustment for each of the PID settings depends on a number of factors, including feature size, tip size, scan method, and scan rate.

PID feedback sums the output of the following three separate feedback loops:

- *Proportional gain* responds quickly to small features but is not optimized to respond to large features. The default value for **P** is 1.

- *Integral gain* controls the response time of the Z-piezo positioner to large, slow sensor changes. If it is set too high, integral gain can result in oscillation or noise in the image. This oscillation will be apparent on the oscilloscope signals. The default value for **I** is 0.2.
- *Derivative gain* tends to reduce oscillation but may amplify high-frequency noise. The default value for **D** is 0.

The PID values can be adjusted while the instrument is scanning; they are located in the **Feedback panel** (see Figure 3.25). For a detailed discussion of these parameters and guidelines for changing them, see Appendix B.

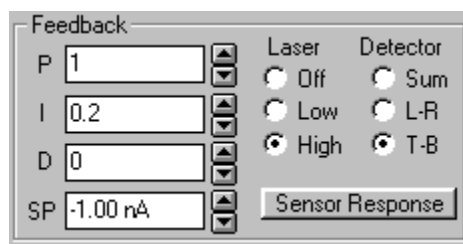




Figure 3.25
PID Settings

Once you have achieved satisfactory settings, you can stop and restart the scan at any time by pressing the **Start Scan** button, , twice.

As each line is scanned, the **Line Remaining** display is updated at the bottom of the μ TALab software window:

Important: At the end of the scan, the tip remains in feedback and in contact with the sample. Click on the **Tip Up** button, , to raise the cantilever tip away from the surface.

NOTE: You cannot raise the tip unless you have stopped scanning by pressing the **Start Scan** button again.

Compare your topography data with the figure shown on the next page to make sure that you have acquired a good image and that your μ TA 2990 system is performing correctly.

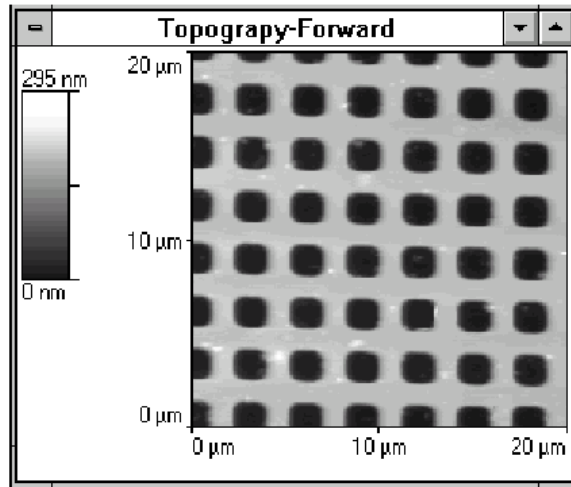



Figure 3.26
Scan Window

If you want to save your images, click on the **Save All** button, , on the tool bar to display the **Save Data As** window shown in the figure below.

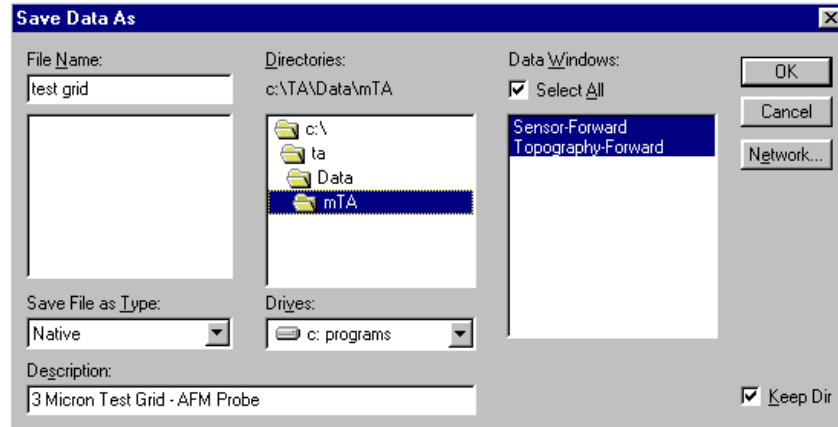


Figure 3.27
Save Data As Window

Follow these steps to save the image(s) in a file:

1. Check the **Select All** box to save both of your images.
2. Select a directory to store your images in, and a filename.
3. Type a more detailed description of your sample or experiment in the **Description** field, if desired.
4. Click **OK** to save your images.

NOTE: The images are saved under the same file name, each with a file extension specific to the scan type. In our example, two data files are stored:

Test grid.sfr = This is the Sensor, Forward,
Raw image

Test grid.tfr = This is the Topography,
Forward, Raw image

Using a Thermal Probe

Before you begin, make sure you have (1) installed your scanner into the μ TA 2990 head, (2) installed the μ TALab Software, and (3) obtained a test image using the AFM probe as directed in the previous section.

This section teaches you how to run a test grid using the μ TA 2990 thermal probe. Follow the instructions below:

1. Select **Options/Acquire** from the menu to open the **Acquisition Setup** window (see Figure 3.21); then check the **Conductivity (Fwd)** and **Sensor (Fwd)** boxes.
2. Locate the standard calibration grid, found in your Micro-Thermal Analysis Sample Kit. This will be your test sample.
3. Using tweezers, mount the calibration grid onto the X-Y sample holder/translator.
4. Using tweezers, mount a thermal probe onto the magnetic seat of the Z-axis scanner assembly and a reference probe on the bottom of the μ TA 2990 head using double-sided tape. (See Chapter 2, "Installing Probes," for instructions.)
5. Align the laser as described in "Aligning the Laser Beam." You will find it much easier to align the laser beam onto the large mirror located on top of the thermal probe than the tip of an AFM cantilever probe. Start with the laser signal in the center of the mirror and slowly work the laser across the mirror while observing the **Sum** signal in the **Oscilloscope** window. Look for the **Sum** signal to reach its maximum. This value should be more than 25 nA (typically ~40 nA). Because of surface irregularities across the mirror, there may be several "sweet spots" that give the localized maximum **Sum** signal.
6. Make sure that you have placed the laser spot at a position on the mirror to obtain a good **Sum** signal. Then, fine tune the alignment of the L-R and T-B signals. Always ensure that the signal can "cross zero" when aligning the L-R and T-B signals. This is an iterative process and may require several cycles to exactly align the laser signal. See the "Fine Adjustments" section in this chapter for more details on how to perform these alignments.

When the μ TALab Software is first loaded on a new system, the thermal probe is turned off and the default temperature calibration file (default.cal) for the thermal probe is loaded. This can be observed in the **Probe** panel shown in the figure below.

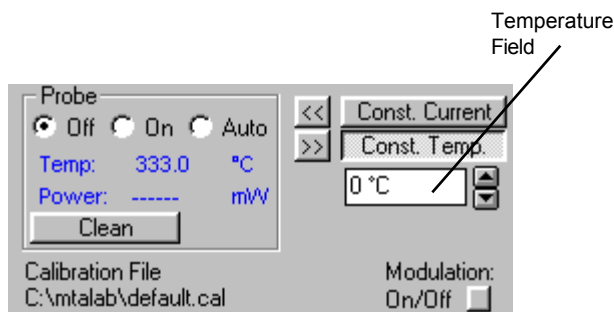


Figure 3.28
Probe Panel

7. Enter -150°C in the temperature field and press **Enter**. The **Power** reading should fluctuate slightly around 0.0, *i.e.*, there should be no power going to the probe and the system should not be in thermal feedback. If there is a power reading, reduce the temperature in the temperature field further, until the probe turns off and the system goes out of thermal feedback.
8. Turn the probe on by selecting the **On** radio button.
9. Enter temperature values in 5°C increments in the **Temperature** field to raise the temperature while you observe the power signal for the point at which the power becomes positive.

The power will become positive when the probe is in *thermal feedback*. The exact temperature value when the system enters feedback varies from probe to probe because of variations in the resistance of each probe's Wollaston wire. At this point, the thermal probe is NOT calibrated, so the observed program temperatures, even when in feedback, are NOT accurate. For the purposes of this experiment, we will continue with an uncalibrated thermal probe. If you wish to calibrate your thermal probe before continuing, refer to Chapter 4 for instructions.

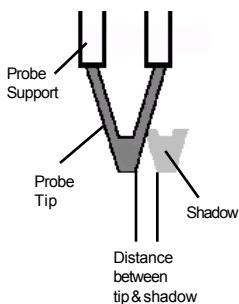
10. Adjust the temperature in 5 to 10°C increments until the heater reaches 1 to 2 mW. This is usually 30 to 35°C *higher* than the temperature at which the probe came into thermal feedback. For example, if the probe

came into thermal feedback when the temperature was increasing from 35 to 40°C, a value of 70°C may be used in the temperature field.

NOTE: If the probe does not come into thermal feedback, either the probe is damaged or there is an open circuit.

11. Translate the calibration grid so the cantilever is positioned over section C (see the figure to the right). See "X, Y Translation and Z Adjustment" in this chapter for a detailed procedure.

12. Monitor the thermal probe on the video display while you use the manual Z-height adjustment screws to lower the probe near to **BUT NOT TOUCHING** the sample surface. You can best judge this distance by lowering the probe until you can see its shadow on the sample surface rising up to meet the thermal probe. The distance between the probe and its shadow should be approximately equal to the width of the probe support (as seen here).



The distance between the probe and its shadow should be approximately equal to the width of the probe support (as seen here).

The final position of the μ TA 2990 head, before the computer-controlled approach is engaged, is as follows: Rear should be level and front slightly tilted closer to the sample than the rear.

13. Access the **Feedback panel**, then set the feedback parameters to the settings shown in the figure below:

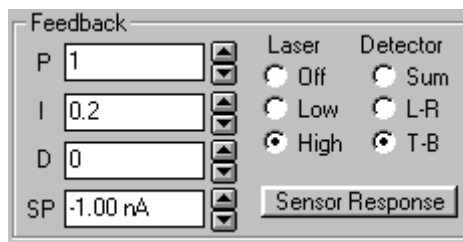


Figure 3.30
Feedback Panel Settings

CALIBRATION GRID
LAYOUT

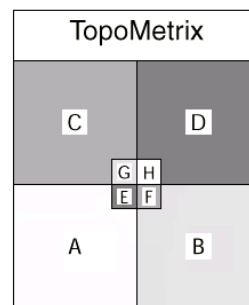


Figure 3.29
Grid of raised squares
with a pitch of 9 μ m

14. Select **Options/Acquire** from the menu to open the **Acquisition Setup** window. Then select the following settings as shown in the figure below:

- **Topography (Fwd)**: checked on
- **Conductivity (Fwd)**: checked on

NOTE: If **Topography** is not available (is “grayed out”), check the **Oscilloscope** window and set the second signal to **Conductivity**.

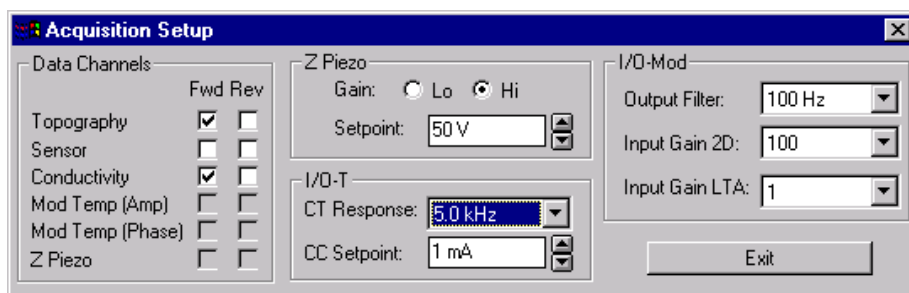


Figure 3.31
Acquisition Setup Window

15. Leave all other options at their default settings, and click on the **EXIT** button.

16. Access the **Scan panel**; then set the scan parameters to the settings shown in the figure below.

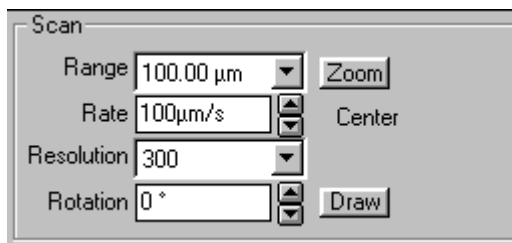


Figure 3.32
Scan Panel Settings

17. Monitor the **Sensor, Feedback** signal in the **Oscilloscope** window while you perform the following steps:

- Adjust the mirror control on the μ TA™2990 head until the **Sensor** signal is more than -10 nA below the value in the **SP** (Set Point) field (-1.0 nA). Ensure that the signal has not reached its maximum negative amplitude limit.

- Disengage the mirror adjustment screw by pulling it straight out. This slightly negative internal feedback sensor signal level allows positive cantilever deflection to be detected when the tip first touches the sample surface. This, in turn, helps achieve stable feedback.

18. Place the Environmental Cover over the μ TA 2990 head.



19. Click on the **Approach** button, .

The thermal probe will move down to the test grid under software control and the system should go into feedback. Perform the test for false feedback, just as you did when you were using the AFM cantilever probe. If the system is in false feedback, raise the probe by clicking once on the **Tip Up**

button, .

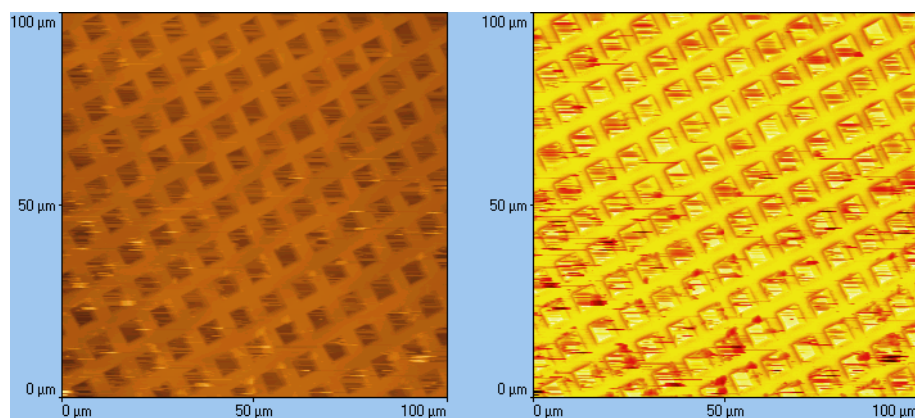
Scanning

After you have followed the procedures needed to run a test grid with a thermal probe and have achieved “true feedback” as shown in Figure 3.24, follow the instructions below to scan the sample:

1. Click on the **Oscilloscope On/Off** button, , on the μ TALab **Main** window's tool bar to close the **Oscilloscope** window and allow a clear view of the **Scan** window.
2. Click on the **Start Scan** button, , to start the scan and image acquisition. As each line is scanned, the topography and conductivity images (selected in step 14 on page 103) are updated.

NOTE: The spatial resolution of the thermal probe is not equal to that of the AFM probe. Consequently, the topography image will be better when you use a dedicated AFM probe than when you use the thermal probe.


After the scan is completed, your images should look similar to the ones shown below.



Conductivity Image

Topography Image

Figure 3.33
Thermal Probe Images

If you want to save your images, click on the **Save All** button, , on the tool bar to display the **Save Data As** window shown in the figure below.

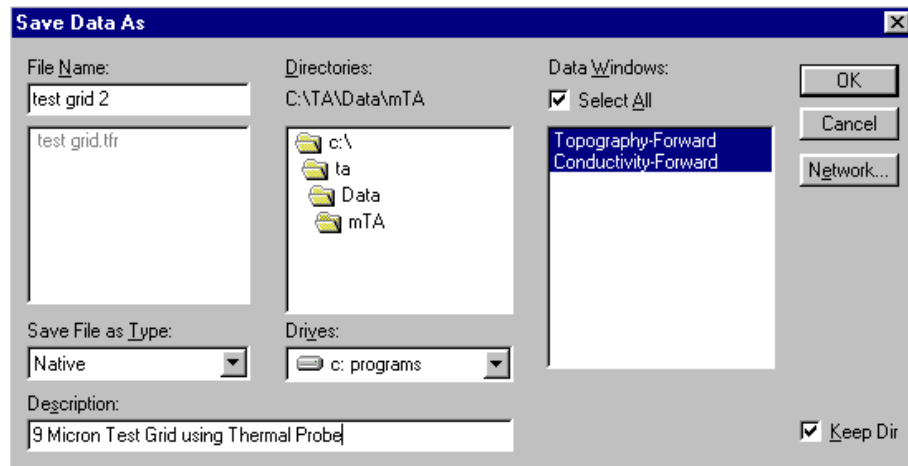


Figure 3.34
Save Data As Window

Follow these steps to save the image(s) in a file:

1. Check the **Select All** box to save both of your images.
2. Select a directory to store your images in, and select or enter a filename.
3. Type a more detailed description of your sample or experiment in the **Description** field, if desired.
4. Click **OK** to save your images.

NOTE: The images are saved under the same file name, each with a file extension specific to the scan type. In our example two data files are stored:

Test grid 2.cfr = This is the Conductivity,
Forward, Raw image


Test grid 2.tfr = This is the Topography,
Forward, Raw image

Obtaining Images and Thermal Analysis Data

Before you begin, you must have (1) installed your scanner into the μ TA 2990 head, (2) installed the μ TALab software, (3) aligned the laser, and (4) successfully obtained images using the AFM and thermal probes as directed in this manual.

1. Locate the polymer test sample (*i.e.*, PET) in your Micro-Thermal Analysis Sample Kit. This will be your test sample.

See Appendix A for instructions on sample preparation.

2. Using tweezers, mount the polymer sample onto the X-Y sample holder/translator.
3. Install the reference and sample thermal probes into the μ TA 2990 head. (See Chapter 2, "Installing Probes," for instructions.)
4. Place the Environmental Cover over the μ TA 2990 head.
5. Select **File/User** from the menu; then enter your name and a suitable description of your sample.
6. Use the same scan, probe, and acquisition settings given in the previous section, "Using a Thermal Probe," to image the sample. After you have obtained satisfactory images, click the **Tip Up** button, , to raise the probe tip.
7. Save the images of the sample surface.

NOTE: Because this sample essentially consists of a single phase, there is little contrast in the thermal conductivity image other than possible artifacts introduced by topography effects. (See Appendix C for an explanation of these effects.) Also, the spatial resolution of the thermal probe is not equal to that of the AFM probe. Consequently, the topography image is better when you use a dedicated AFM probe than when you use the thermal probe.

8. Select **Acquisition/Local Thermal Analysis** from the menu to open the **Acquisition—Local Thermal Analysis** window.

Observe that the **Scan** and **Feedback** panels have been replaced with a **Local Thermal Analysis panel** (shown in the figure below), and a new panel for the temperature program is displayed.

Cursor Location—
Changes as you move
the cursor over
any of the image windows.

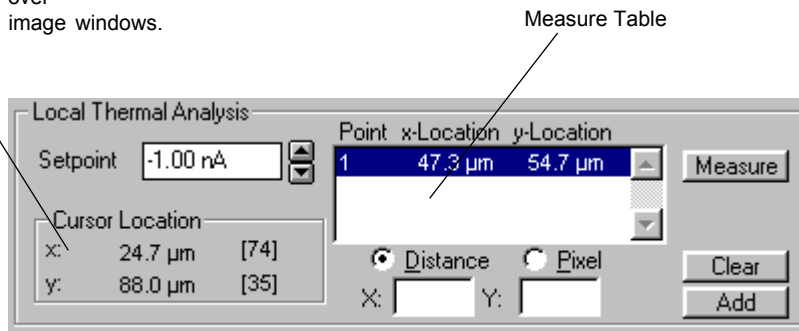


Figure 3.35
Local Thermal Analysis Panel

9. Select locations for your experiment by moving the cursor over the regions/domains of interest and clicking with the mouse.

The **Cursor Location** in the **Local Thermal Analysis panel** is updated as you move the cursor over any of the image windows. See Figure 3.35.

10. Select a single location in the center of one of the image windows by moving the cursor to the center and clicking the mouse. The location information is entered into the **Measure Table**.
11. Select **Options/Preferences** from the menu to open the **Preferences** window, as seen in Figure 3.36 to the right.
12. Make sure that the **Subtract Baseline** and **Auto clean probe** options are *not* selected (are unchecked).

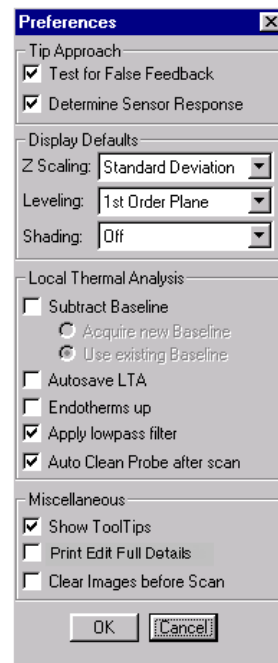


Figure 3.36
Preferences Window

13. In the far left panel, enter the method shown in the figure below for your temperature program:

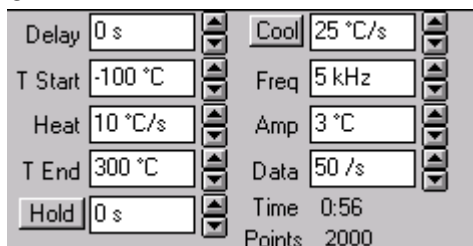


Figure 3.37
Temperature Program for Sample

14. Set the parameters for the real-time display in the **Acquisition—Local Thermal Analysis** window as follows:



Figure 3.38
Parameters for Real-Time Display

15. Click on the **Measure** button, **Measure**, in the **LTA panel** to start your experiment. The thermal probe is lowered onto the sample at the specific location defined in step 9, and the temperature program is initiated.
16. Observe the real-time display of the **Power** signal in the **Acquisition—Local Thermal Analysis** window, as seen in the figure below.

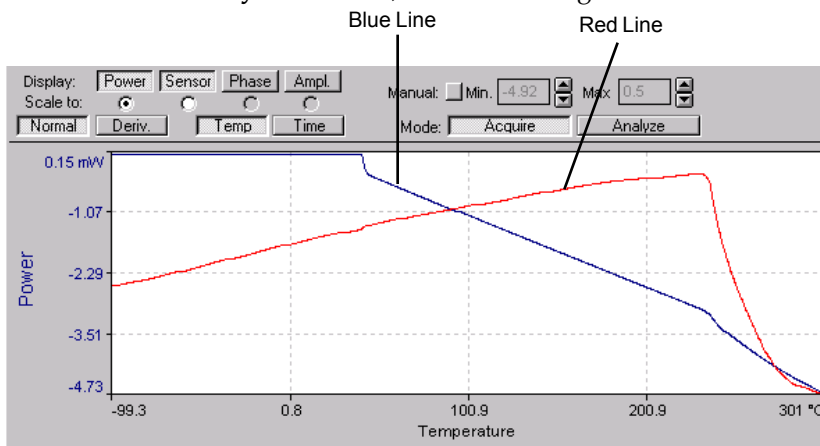


Figure 3.39
Real-Time Display of Power Signal for Nylon Sample

The blue line (shown in Figure 3.39 on the previous page) is the power being supplied to the thermal probe. Note the “kick-in” around room temperature and the change in slope around 240°C owing to the polymer’s melting. The derivative of this curve gives two peaks, one where the probe comes into thermal feedback around room temperature and the other due to the polymer’s melting.

The red line is from the laser movement on the four-quadrant detector. The signal increases with temperature (expansion) until the polymer softens at around 240°C and the probe pushes into the sample.

NOTE: In the example shown here, temperatures are approximate because the thermal probe has not been calibrated.

At the end of the experiment, you will see the message shown in the window below.

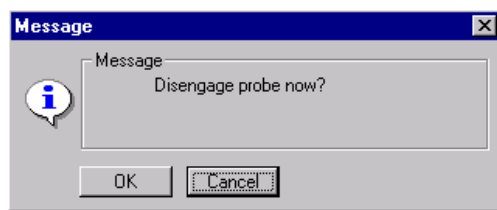


Figure 3.40
Disengage Message

17. Select **OK**, and the probe is retracted from the surface of the sample.

18. Save your images by clicking on the **Save All** button, , on the tool bar to display the **Save Data As** window shown in the figure to the right.

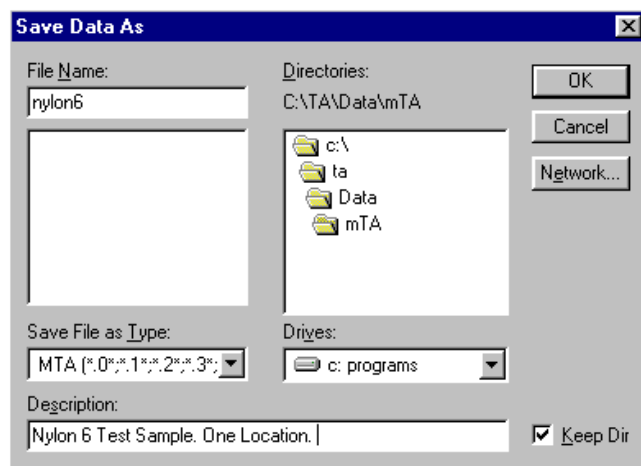

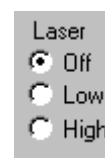
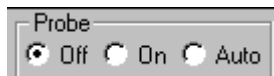


Figure 3.41
Save Data As Window

Shutting Down the μ TA[®] 2990

It is safe to leave the μ TA 2990 on for extended periods, even if it is not in regular use. Doing so leaves the electronics “warmed up” and the system in temperature equilibrium with its environment. You should, however, perform the following steps to put the instrument into a “standby” mode:

- Raise the probe up by pressing the **Tip Up** button, , on the tool bar two or three times.
- Click on the appropriate radio button to turn the laser off.
- Click on the appropriate radio button to turn the thermal probe off.



- *Optional:* Turn off the monochrome monitor; minimize light source by turning the knob on the MCU.
- Close down the μ TALab software.
- Place the Environmental Cover over the μ TA 2990 head.

Full Shutdown Procedures

After you have put the instrument in “standby mode,” you can shut down the entire system, if desired, by following these additional steps:

- Raise the back legs of μ TA 2990 to further lift the entire head away from any sample or surface it is resting upon.
- Turn off the ECU-Plus.
- Turn off the monochrome monitor.
- Place the Environmental Cover over the μ TA 2990 head.

Chapter 4

Calibrating the μ TA[®] 2990

Determining When to Calibrate

As with any analytical instrument, it is important that you calibrate the μ TA 2990 MicroThermal Analyzer under the same conditions as those used for running your samples. The μ TALab 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 either of the sample or reference probes, you should check your temperature calibration and recalibrate as necessary.

This chapter contains procedures for the following types of calibration:

- Temperature calibration
- I/O-TOffset calibration

Appendix contains explanation and procedures for calibrating the scanner and approach speed.

Temperature Calibration

Checking Temperature Calibration

It is advisable to check the temperature calibration of your μ TA 2990 every day. The procedure you use depends on whether you leave your μ TA 2990 on continuously or turn it off when it is not in use:

- If you leave the μ TA 2990 on continuously, the daily temperature calibration check involves only (1) calibrating the I/O-T offset and (2) running a temperature standard.
- If you turn the μ TA 2990 off when it is not in use, you must allow 3 to 4 hours after turning it on for the electronics to stabilize before you check or adjust the I/O-T Offset and temperature calibration.

The μ TA 2990 instrument 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 change in its angle to the surface. This section provides the instructions needed to temperature calibrate the instrument.

Before Calibration

Before you begin the calibration procedure, you need to obtain one or more sample materials with known melting temperatures ($+0.1^{\circ}\text{C}$). The μ TA 2990 accessory kit contains two organic calibration materials and one polymer calibration material. The known melting temperatures for these materials are provided in the certificates of measurement found in the accessory kit.

You also need to know the room temperature in the laboratory or room where the μ TA 2990 resides, to within $+0.1^{\circ}\text{C}$. The best source of this information is a thermometer that is traceable to a national reference laboratory. This type of thermometer is available from most laboratory supply companies.

Make a note of both the melting temperature of your sample and the room temperature. This information is needed later in the procedure.

First Calibration File Setup

When you receive your instrument and install the μ TALab Software, you should find a file called "default.cal" installed with the software. It is very important that you make a backup copy of this original file on a diskette and that you do not alter this file. The default.cal file is used as a starting base for the operation of your instrument and can be used to return the instrument to its original state, if needed.

Follow these steps to set up the calibration file:

1. Click on the **My Computer** icon on your desktop and locate the original default.cal file mentioned above.
2. Create or locate a folder (directory) that you want to use for your μ TA calibration information.
3. Copy the default.cal file into your calibration directory.
4. Right click on the copied default.cal file and select **Rename** from the menu. Rename the file using today's date and keep ".cal" as the extension. (For example, you can rename the file to 121598.cal for December 15, 1998.) This step is important so that you do not accidentally overwrite the original default.cal file.

NOTE: We recommend using the naming convention just described because you should be calibrating your instrument daily. If you name your calibration files in this manner, there will be no confusion about when the instrument was last calibrated.

5. Start up μ TALab Software.
6. Select **Acquisition/Probe Calibration/Load** from the menu bar at the top of the main window. A file selection window is displayed.
7. Locate and load today's calibration file, which you created in step 4.

Now you are ready to proceed with the experimental portion of the calibration.

Performing the Calibration Experiment

This description contains screens from calibrations using anisic acid and a polymer, to show both types of scans.

NOTE: After you have obtained the temperature information needed and loaded your data file, you are ready to perform the calibration experiment as follows.

1. Mount your calibration material following the instructions in this manual. (See the section called "Preparing Calibration Samples.")
2. Set up the initial conditions needed for the calibration scan as follows:

CAUTION: Make sure that the "Auto clean probe after scan" option in the Preferences window is NOT selected. Cleaning an uncalibrated probe could damage or destroy it.



CAUTION: Make sure that the "Subtract Baseline" option in the Preferences window is NOT selected.

- a. Select **Acquisition/Local Thermal Analysis** from the main menu. The **Temperature Program** panel shown in Figure 4.1 is displayed.

Delay	0 s	Cool	10 °C
T Start	-120 °C	Freq	2 kHz
Heat	10 °C/s	Amp	10 °C
T End	300 °C	Data	50 /s
Hold	0 s	Time	0:30
		Points	1375

Figure 4.1

Localized Thermal Analysis Temperature Program Panel

- b. Position the cursor in the **T Start** and **T End** fields to type in the values to be used, pressing **Enter** each time when done. These values vary from probe to probe.

T Start should be some temperature at which the probe is turned off.

T End should be some temperature at which you can be sure that the sample material will have melted.

- c. Position the cursor in the Heat field. Set the heating rate to a value that you will be using for future experiments. For example:

$$\text{Heat} = 10 \text{ }^{\circ}\text{C/s}$$

- d. Set **Data** to four times the ramp rate.

4. Select a location on the image for your temperature calibration measurement by moving the cursor on one of the images to any location and clicking the left mouse button. A red cross should be displayed at the location chosen.

5. Obtain a thermal curve for this location as follows:

- a. Press the Tip Down button, , to lower the probe near to (but not yet touching) the surface of the calibration material.
- b. Click the Measure button, . The probe now automatically approaches the surface. After about one minute, a thermal curve is displayed, as shown in Figure 4.2.

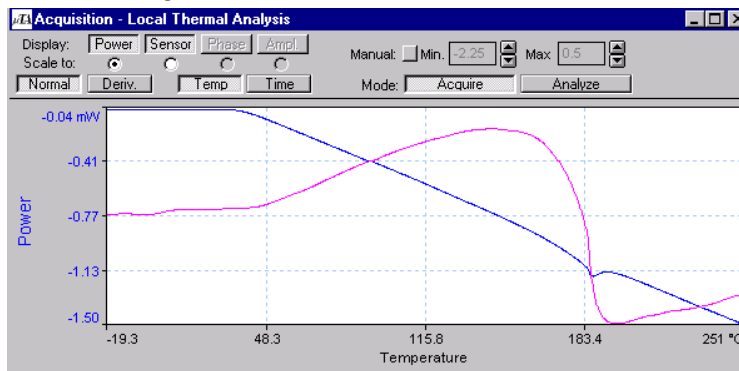


Figure 4.2
Thermal Curve for Anisic Acid

When the experiment has finished, the message **Disengage probe now?** is displayed.

- c. Click **OK**.
- d. Save your data by clicking on the **Save** button on the tool bar. The **Save Data As** window, shown in Figure 4.3, is displayed.

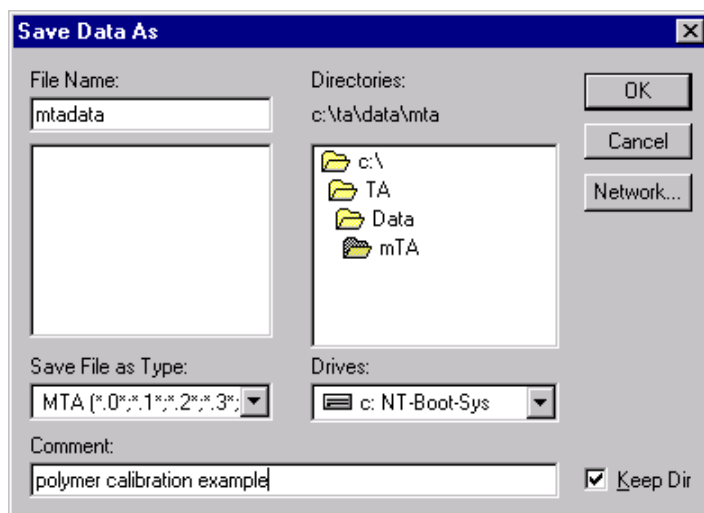


Figure 4.3
Save Data As Window

- e. Make sure that the **Save File as Type** option is set to “mTA.” This will allow you to access this file using the Universal Analysis program for further manipulation and plotting.
 - f. Type a detailed description of your calibration material or calibration experiment in the **Description** field, if desired.
 - g. Select the directory desired and enter a filename. Click **OK** to save your images.
6. Open the Universal Analysis program.
 7. Open the data file created in step 5.
 - a. Use the Universal Analysis program to determine the onset of the transition (see *Universal Analysis Operator's Manual*).

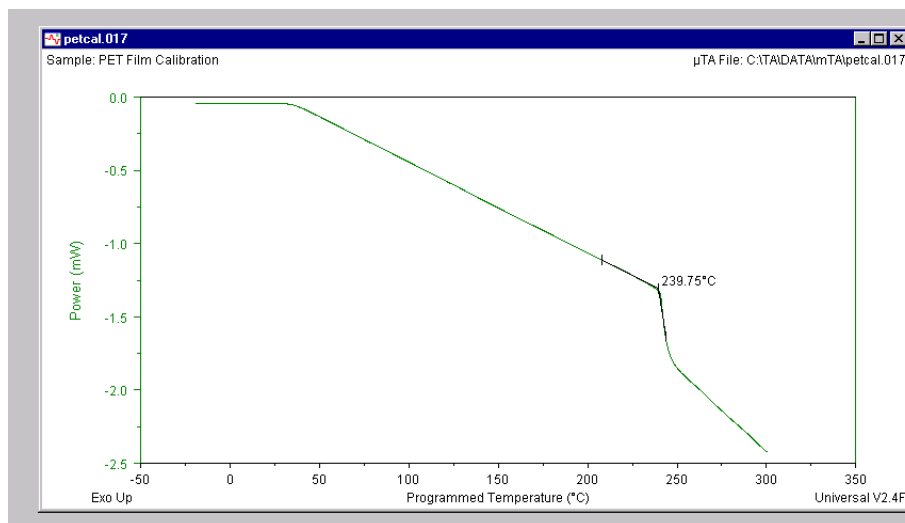


Figure 4.4
Window from Universal Analysis Program
Showing Onset of Transition for a Polymer

NOTE: If no transition is detected, repeat the procedure from step 3, and choose a slightly higher T End value (about 50°C).

- b. Record the indicated transition temperatures to within + 0.1°C.
8. Repeat steps 1 through 7 with a second material for a 2-point calibration.
9. Enter the recorded values into the μ TALab software program as follows:
 - a. Click on the **Time** and **Analyze** buttons on the **Acquisition—Local Thermal Analysis** window, shown in Figure 4.5 on the next page.

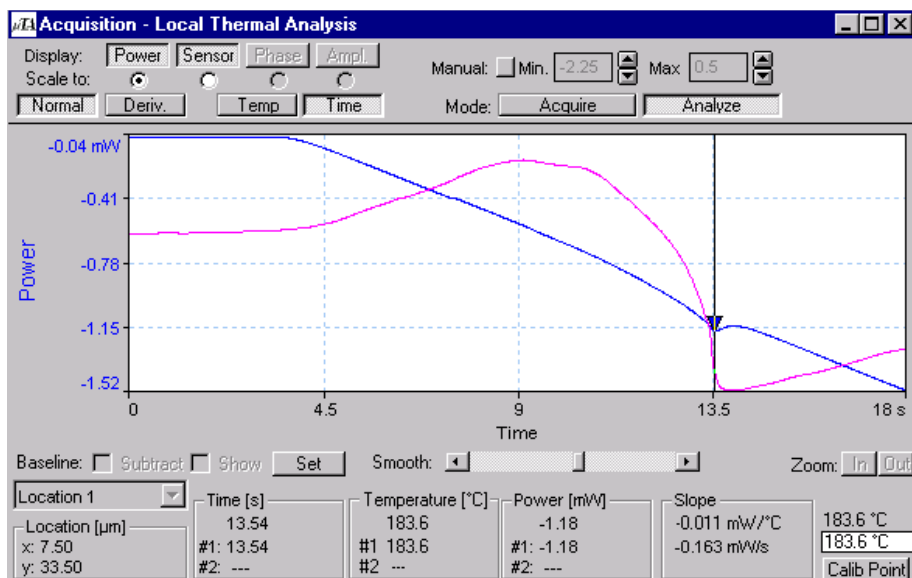


Figure 4.5
Acquisition—Local Thermal Analysis Window Showing
Calibration Scan for Anisic Acid

- b. Move the cursor inside the graph area. Click the left mouse button once to place the cursor on the thermal curve. Move the cursor left or right until the temperature window reads the temperature that you just recorded for the onset of the transition.
- c. Keeping the cursor at the point determined above, click once to create a caret on the thermal curve. Clicking once also enters the temperature in the window in the lower right corner.
- d. Right click to release the cursor; then place the cursor in the lower right corner of the calibration screen and type in the known melting temperature of the sample in the white box below the latter value (see step b).
- e. Click on **Calib Point**.
- f. Repeat steps 9b to 9e to add the second calibration point.

10. Select **Acquisition/Probe Calibration/Generate** from the menu. The **Probe Calibration Generate** window appears. A calibration line should appear next to the two calibration points you have just added.

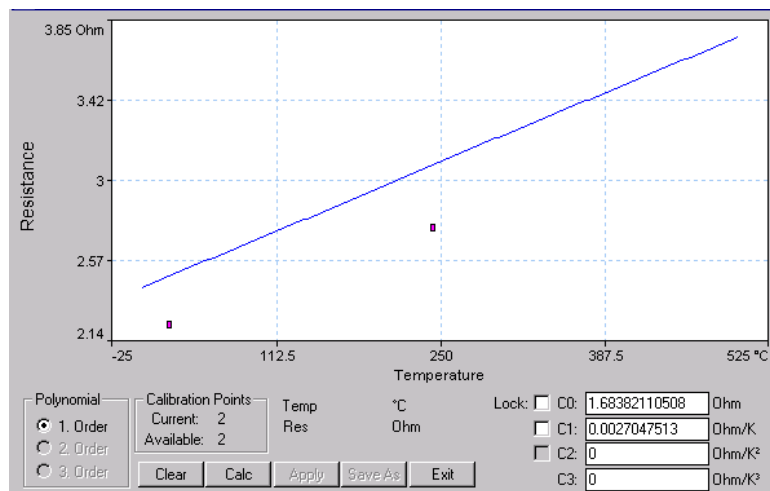


Figure 4.6 Probe Calibration Generate Window

11. Click on **Calc**. The calibration line should now pass through the two calibration points, and the C0 and C1 calibration constants should have been updated. Figure 4.7 shows the **Instrument Calibration** window after calculation.

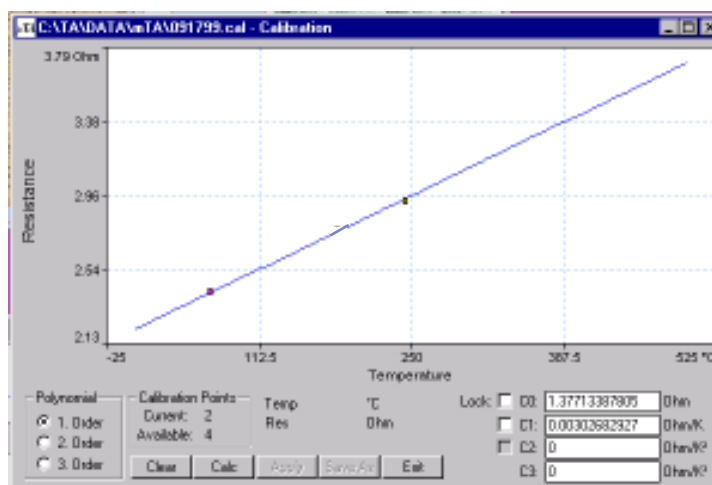


Figure 4.7 Calibration Line Passing Through Calibration Points

12. Click **Save As**. A standard file save window is displayed.
 - a. Select the desired directory and name the calibration file with today's date (*e.g.*, 092599.cal). Click **Save** when completed.
 - b. Click the **Apply** button. This process enters the calibration values into the μ TA operating system.
 - c. Select **Exit**.

Recalibrating a Probe

To recalibrate a probe, first follow steps 1 through 10 as detailed on the previous pages. When opening the **Probe Calibration Generate** window, your old calibration points (pink) will appear along with your new calibration points (yellow). You must remove the old calibration points before clicking on **Calc** to calculate your new coefficients.

Delete the old calibration points as follows:

- a. Position the cursor on the point and right click. A window appears.
- b. Left click on the **Del** (Delete) button on the small window. The window and unwanted calibration point disappear.

Continue your calibration with step 10.

Figure 4.8 shows examples of calibration scans for anisic acid and a polymer.

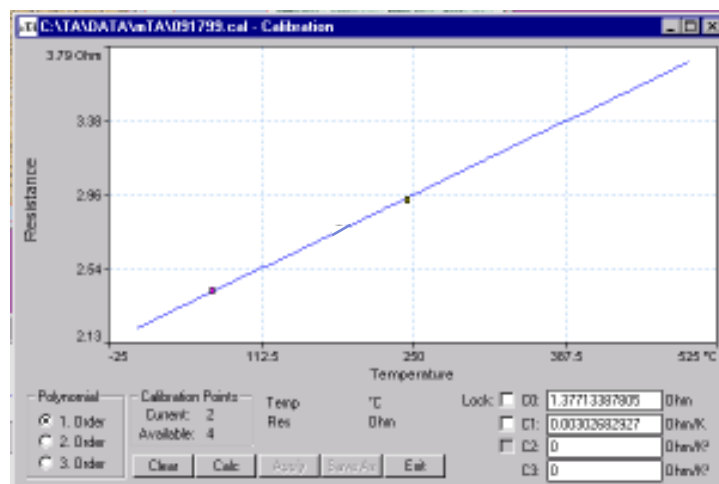
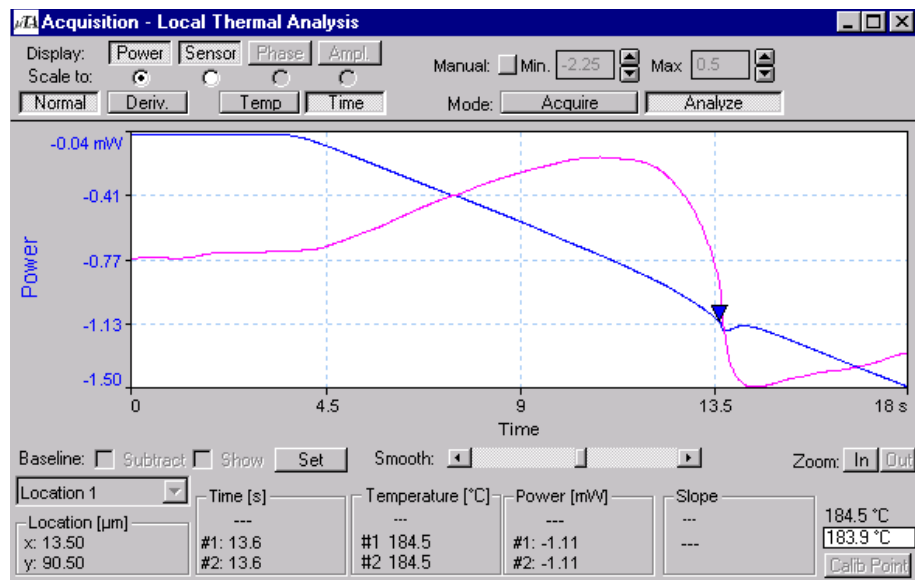


Figure 4.8
Calibration Scans for Anisic Acid (top)
and a Polymer (bottom)

Preparing Calibration Samples

Organic calibration materials are more suitable than metals for calibration in Micro-Thermal Analysis, because (1) they do not react with the platinum probe and (2) the probe can easily be cleaned by “burning off” at 500°C in air at the end of an experiment. You may use any of the six calibration materials listed in Table 4.1, which also gives their nominal melting temperatures.

Table 4.1
Materials for Calibration of μ TA 2990

Material	Temperature (°C)
Biphenyl	69.3
Benzil	94.5
Benzoic acid	122.4
Diphenylacetic acid	147.3
Anisic acid	183.3
2-Chloroanthraquinone	209.6

Biphenyl and anisic acid are provided in the accessory kit in fine powdered form, with crystals too small and rough for use in Micro-Thermal Analysis. You need to prepare a calibration sample by exposing it to a temperature slightly above its melting temperature for a short time, allowing it to form larger crystals but avoiding potential degradation from prolonged exposure to high temperature.

Prepare your calibration samples as follows:

1. Place a few milligrams of your chosen calibration material in a standard 6.3-mm aluminum DSC sample pan. Put the aluminum lid on the pan, but do not crimp it.

NOTE: Using a lid during the process, as described here, results in crystals with the smooth, flat surfaces needed for Micro-Thermal Analysis.

2. Heat the sample in one of two ways:
 - Heat the sample in a DSC (Differential Scanning Calorimeter), using a heating rate of 10°C/min, to 10°C above the material's nominal melting temperature, and then quickly cool it.

- Set a hot plate to a temperature 10°C above the material's nominal temperature, and then put the sample pan on the hot plate for a few seconds.
3. Remove the lid from the sample pan. Prepare the crystals for mounting by cutting down the edges of the sample pan with an X-acto® knife.
 4. Fix the sample to a μ TA 2990 sample mounting stub, using double-sided tape so the individual crystals will not move around during storage and analysis.
 5. Store the mounted samples in small, labeled petri dishes for use as needed.

Calibrating I/O-T Offset

There is a small amount of hysteresis in the temperature control electronics. It manifests as a difference between:

- The temperature at which the system comes into thermal feedback on heating
- The temperature at which thermal feedback is lost on cooling.

In order to compensate for this effect, the **I/O-T Offset** must be calibrated.

We recommend that you check **I/O-T Offset** value at least daily and *always* when you change a sample or reference thermal probe and when the system has been powered off.

The level of hysteresis will be quite high when you first turn on the μ TA 2990 but will decrease over about 60 to 90 minutes as the electronics come into thermal equilibrium with their surroundings. This is the reason that it is recommended that the system be left on at all times.

Procedure to Calibrate I/O-T Offset

The full procedure outlined here should be done once every three months. On a daily basis a simpler procedure can be followed. The calibration is performed entirely within the software program. The calibration screen can be found in the μ TALab software by selecting **Acquisition/Setup/Calibrate IOT Offset**. The window shown to the right is displayed.

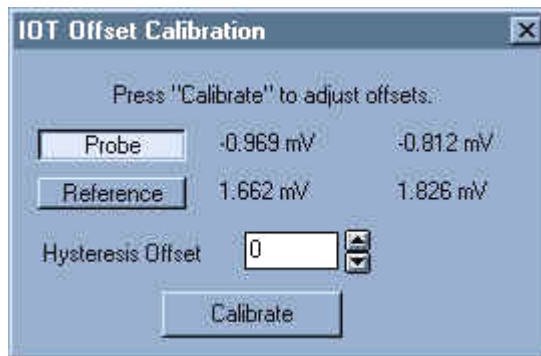


Figure 4.7
IOT Offset Calibration Window

1. Enter the value "0" in the **Hysteresis Offset** field and press the **Calibrate** button.

NOTE: The thermal probe should be "ON" for this entire procedure.

When the mV readings have stabilized at values close to zero (around 0.01 mV), the calibration is finished and the **Calibrate** button reads **Done**.

2. Click the **Done** button to complete the calibration process. Access the **Local Thermal Analysis**.
3. Select **Options/Preferences/Local Thermal Analysis** and uncheck the **Subtract Baseline** option.
4. Mark a point on any image and set up a thermal experiment that will show the turning on and turning off of the probe.
5. Press **Measure** and wait for the **Approach** button to depress, signaling that the tip is approaching to surface.
6. Click the mouse button (left) once to abort this approach. The instrument will begin to collect data with the probe in the air.

Figure 4.8 shows the μ DTA results of an air run after calibration with a **Hysteresis Offset** value of "0." There is a large jump in power at the point where the probe turns on during heat up.

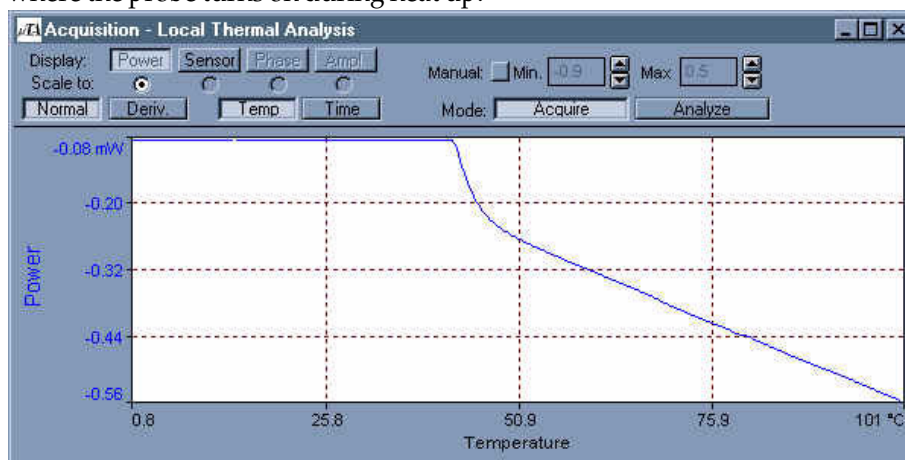


Figure 4.8
 μ DTA Results with Hysteresis Offset = 0

7. After the data has been collected, compare the μ DTA results with those in Figures 4.8 through 4.11. Make the appropriate changes in the Hysteresis Offset value.
8. Repeat steps 1 through 7 until the desired result (Figure 4.10) is obtained.

The procedure outlined states that the **Hysteresis Offset** value should be altered and another air run performed. Usually, the **Hysteresis Offset** value must be increased to eliminate the observed jump in power. Figure 4.9 shows the μ DTA signal after performing a calibration with a **Hysteresis Offset** value of 500. It can be seen that the jump in power has been reduced.

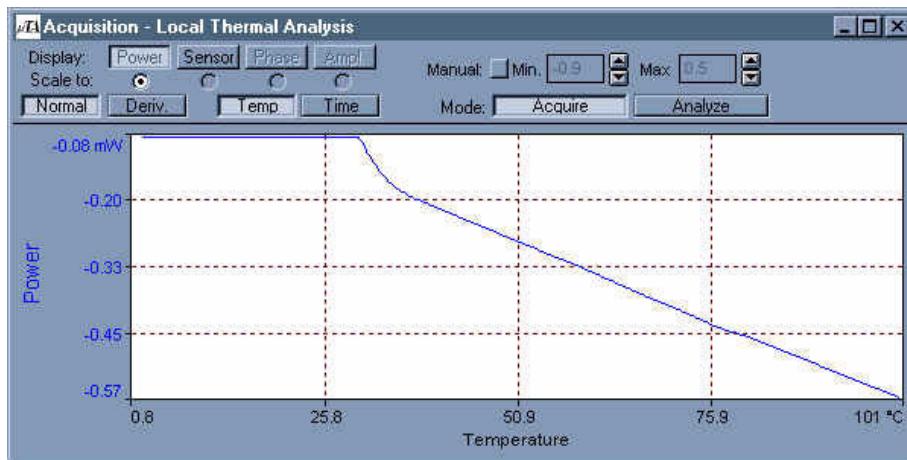


Figure 4.9
 μ DTA Results with Hysteresis Offset = 500

Figure 4.10 below shows the result with a value of 1200 for the **Hysteresis Offset**. Here the heat-up curve is seen with little or no curvature. **This is the desired result**—the probe turns on without a discontinuity.

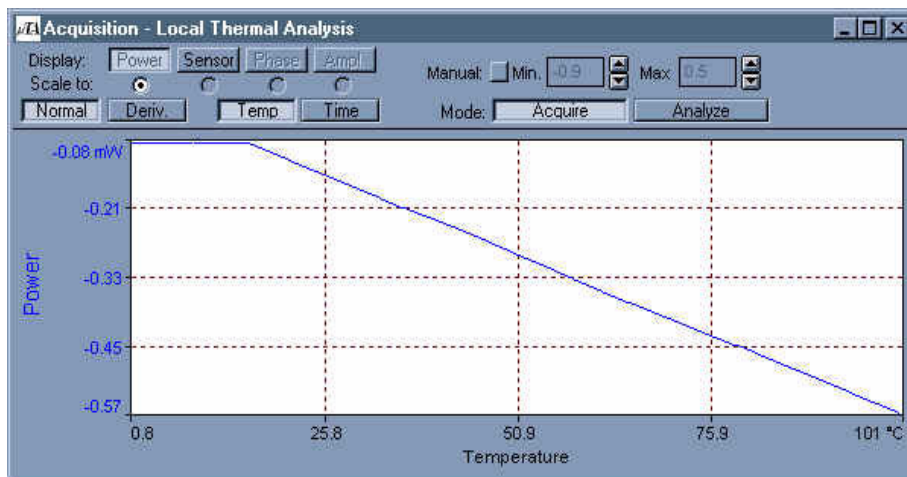


Figure 4.10
 μ DTA Results with Hysteresis Offset = 1200

Figure 4.11 shows what can happen if a value that is too large is entered for the **Hysteresis Offset**. A large curvature in the heat-up curve can result.

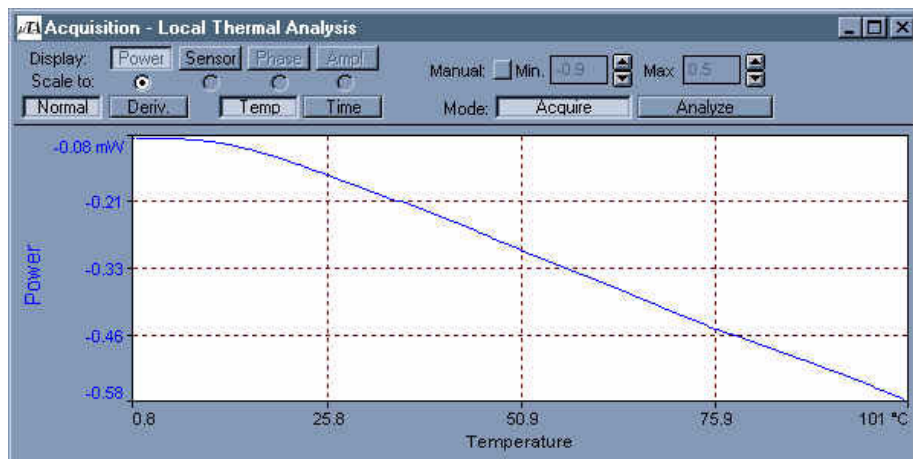


Figure 4.11
μDTA Results with Large Hysteresis Offset

It is recommended that a modified I/O-T Offset calibration be performed every time the instrument is used. Simply access the **Calibration** panel and press the **Calibrate** button. The full calibration to determine the appropriate **Hysteresis Offset** value should be performed once every three months or as necessary.

NOTE: The values used to minimize hysteresis in your system may differ from those used in the example above.

NOTE: If your system appears as seen in Figure 4.11 above after entering the initial value of "0" for the **Hysteresis Offset**, then negative values must be entered to remove this curvature and obtain the desired result.

Chapter 5

Running Samples

Overview

Two basic types of measurement are possible with your μ TA[®] 2990:

- Atomic Force Microscopy (AFM) imaging
- Localized Thermal Analysis (LTA)

It is not essential for you to acquire an image before making LTA measurements. Sometimes it is not possible to obtain an image (the sample surface is too rough) or necessary to do so (the sample is known to be homogenous). Under these circumstances, you can skip the first section of this chapter and begin at "Localized Thermal Analysis."

This chapter describes how to use the μ TALab program's basic software instrumentation controls to acquire images and LTA data.

Selecting Images and Measurements

Before scanning to acquire an image, you must select the type(s) of images (Topography, Thermal Conductivity, etc.) that you wish to measure as well as the measurements you wish to make. To begin doing so, open the **Acquisitions Setup** window (shown in Figure 5.1) by selecting **Acquire** from the **Options** menu.

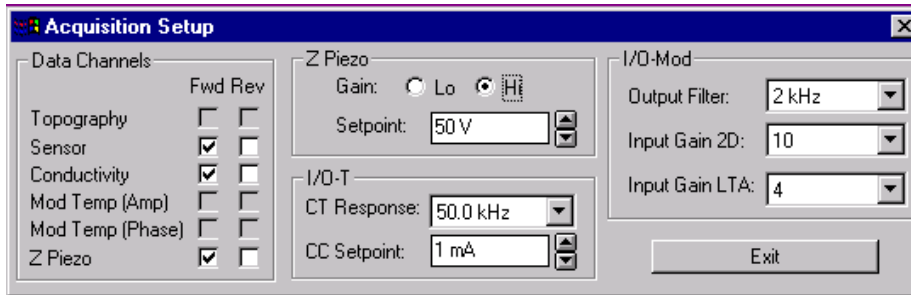


Figure 5.1
Acquisition Setup Window

The **Acquisitions Setup** window contains four panels:

- **Data Channels**
- **Z Piezo**
- **I/O-T** (Input/Output Temperature)
- **I/O-Mod** (Input/Output Modulation)

The settings and choices available on this panel are explained in the following pages.

Once you are satisfied with your choices in this window, press **Exit** to return to the μ TALab **Main** window.

Specifying Images and Scan Direction

The **Data Channels** panel (shown in Figure 5.2) lists the types of images available during forward and reverse scanning of a sample.

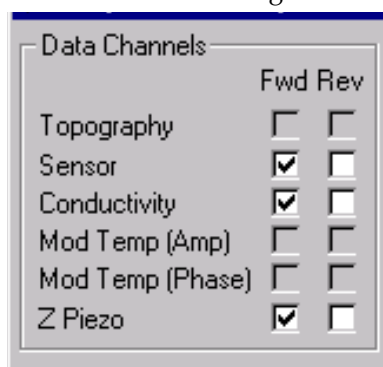


Figure 5.2
Data Channels Panel

To determine which images will be acquired in which scanning direction:

1. Click on the box corresponding to the image types and scan direction you wish. (See page 134 for description of image types and page 135 for an explanation of scan direction.)
2. If a data channel is available for that choice (see below), a check will appear in the box you have chosen, confirming that such images will be acquired during scanning.

Data Channels Available

The μ TA 2990 MicroThermal Analyzer has four data channels that can be used simultaneously during a single scan. Therefore, you may choose up to four types of images for each scan.

The Oscilloscope requires one data channel, however; if that channel is open, three channels are left for image acquisition. Also, the Oscilloscope uses the same data channels as image acquisition. If the Oscilloscope is displaying additional signals on some of the remaining three data channels, the number of channels left for image acquisition is further reduced. You may have to sacrifice displaying a signal on the Oscilloscope during image acquisition so

you have a data channel available to acquire another image type during scanning.

As you select images to be acquired on the **Data Channels** panel, further image selection is reduced according to the number of remaining data channels. Consequently, some image types become “grayed-out” and therefore unavailable on the **Acquisitions Setup** window.

Types of Images

The images you can choose from are as follows:

- **Topography**—A topographic image of the scan area. The quality of the image is affected by the scan and feedback parameters. The topography image is corrected for linearity and hysteresis through the use of the TrueMetrix hardware closed-loop system.
- **Sensor**—An image in which the internal sensor (quadrant photodetector) current is plotted to show topographical features. (See the “Feedback ” section in Appendix B for further explanation.)
- **Conductivity**—A thermal conductivity image generated from the power required to maintain the thermal probe at the probe temperature (or, if modulation is turned on, the average probe temperature).
- **Mod Temp (Amp)**—An image based on the response to the temperature modulation amplitude set in the **AC Mode Setup** panel (see “Using the AC Mode Setup”).
- **Mod Temp (Phase)**—An image based on the phase lag between the applied temperature modulation, set in the **AC Mode Setup** panel (see “Using the AC Mode Setup”), and the observed temperature modulation.
- **Z Piezo**—An image in which the voltage to the Z-piezo positioner is plotted to show topographical features. This function is useful for imaging very flat surfaces in which the feature size is smaller than the noise floor of the scanner's linearizers.

Forward versus Reverse Scanning

As shown on the **Data Channels** panel (Figure 5.2), all of the types of images available can be acquired during the forward scan, the reverse scan, or both. This option can be valuable, because the interaction between the probe tip and the sample may cause forward and reverse scans of the same area to yield different information, as shown in Figure 5.3.

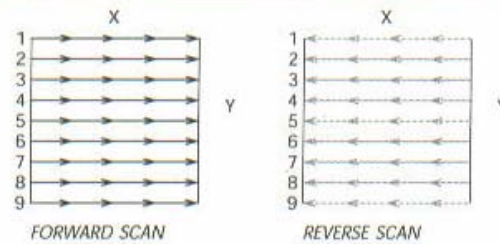


Figure 5.3
Forward and Reverse Scannings*

*From *SPMLab—Release 4.0 Software Reference Manual*, TopoMetrix Corporation, 1996.

I/O-T (Input/Output for Temperature)

The I/O-T (Input/Output for Temperature) panel, shown in Figure 5.4, contains two fields that allow you to determine the constant temperature response and constant current set point for scanning.

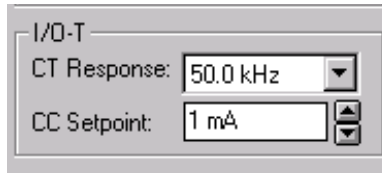


Figure 5.4
I/O-T Panel

Setting the Constant Temperature Response Frequency

Click on the arrow for the **CT Response** field. Choose a value from the dropdown list that appears, to set the **CT Response** value for your scan.

The **CT (Constant Temperature) Response** field determines the resonance frequency (which is the inverse of the response time) of the temperature feedback circuit within the input/output temperature electronics. At higher frequencies, the temperature feedback is faster but potentially unstable. At lower frequencies, temperature feedback reacts more slowly but is more stable.

When the **CT Response** frequency is decreased (slow response, more stable), however, the scan speed must also be decreased, because the feedback loop needs more time to react to a change in the sample's thermal conductivity.

The **CT Response** frequency should be higher than the frequency of thermal features that appear in the image. You can calculate the frequency of thermal features from their distance apart using **Line Measure** and **Scan Rate**.

A default value of 20 KHz for the **CT Response** frequency works for the majority of samples.

The **CT Response** frequency for thermal feedback plays the same role as the PID settings for the force feedback (see Chapter 3).

Choosing the Constant Current Set Point

The **CC Setpoint** field contains a scrollable list from which you can choose the set point value for constant current mode scanning.

Constant current mode enables the probe to sense temperature variation, across the sample, as you image it.

The **CC Setpoint** should be set to a low value (typically, around 1 mA or less) so that the probe does not heat up as it maps temperature variations in the sample.

Setting Up Input/Output Modulation Scans

The **I/O-Mod** (Input/Output for Modulation) panel, shown in Figure 5.5, enables you to change the value for the output filter and to determine the gain for input during scanning or LTA measurements.

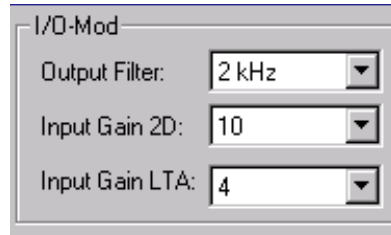


Figure 5.5
I/O-Mod Panel

Setting the Output Filter

The Output Filter removes high-frequency noise from the output signal of the I/O modulation electronics of the μ TA 2990. The value in the **Output Filter** field determines the cutoff frequency of the output filter.

A typical value for Output Filter is approximately 100 Hz.

Click on the arrow next to the **Output Filter** field. Choose a value from the dropdown list that appears to set the cutoff point for the output filter.

Setting the Input Gain for Two-Dimensional Imaging

The **Input Gain 2D** value sets the input gain for the modulation electronics board used in imaging mode (two-dimensional scanning). Raising this number increases the amplification of the thermal imaging signals.

To change this value:

1. Open the **Oscilloscope** window, so that you can monitor the response of the **AC Amp** or **AC Phase** signal to any change you make in the **Input Gain 2D** value.

2. Click on the arrow next to the **Input Gain 2D** field. Choose a value from the dropdown list that appears, to raise or lower the input gain for two-dimensional image acquisition.

Set **Input Gain 2D** to the largest value possible, so that the signal does not go off scale during imaging. You will probably have to experiment to find the value that works best.

Setting the Input Gain for Localized Thermal Analysis

The **Input Gain LTA** field on the **I/O-Mod** panel sets the input gain for the modulation electronics board used in LTA. Raising this number increases the amplification of the LTA signals.

Click on the arrow next to the **Input Gain LTA** field. Choose a value from the dropdown list that appears, to raise or lower the input gain for LTA.

Set the **Input Gain LTA** to the largest value possible, so that the signals does not go off scale during LTA measurements. Determining this value may require some trial and error. You may have to collect a number of LTAs to set the gain correctly. Initially, choose a low value for **Input Gain LTA**. Then try using higher values for subsequent LTAs, until you identify the highest gain value that you can use that does not cause signal saturation.

Setting Up Experimental Parameters

You can use the **Scan** and **Feedback** panels (located underneath the Tool Bar; see Figure 5.6) to change the experimental parameters for two-dimensional (2D) scanning. See Appendix A for a discussion of the effects of scan and feedback settings on image quality.

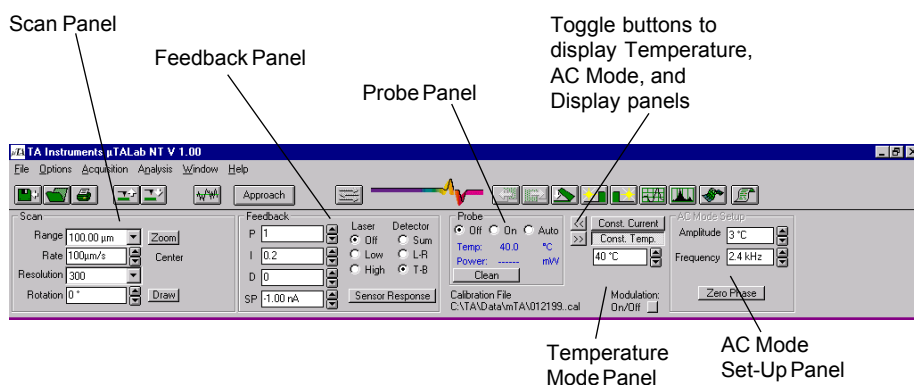


Figure 5.6
Location of Panels on
Main Window in 2D Scanning Mode

Once feedback is established and scanning is in progress, you can optimize the images you are acquiring by adjusting some of the values on the **Scan** and **Feedback** panels:

- Scan **Rate** on the **Scan** panel.
- Set point (**SP**), proportional gain (**P**), integral gain (**I**), and derivative gain (**D**) on the **Feedback** panel.

You can adjust these values while monitoring the associated signals on the **Oscilloscope**, during a **Line** scan, or during image acquisition. For more detailed information, see Appendix B.

Setting Scan Parameters

The **Scan** panel, shown in Figure 5.7, contains fields for selecting and adjusting the area of the sample you wish to image.

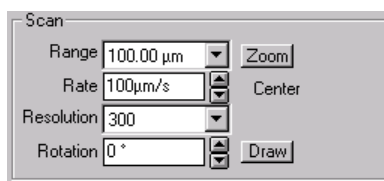


Figure 5.7
Scan Panel

Setting the Range

Click on the arrow for the **Range** field. Choose a value from the dropdown list that appears, to determine the distance of the scan area.

The **Range** field specifies the length of one side of the square scan area. For example, a 50- μm **Range** value is equivalent to a square image area measuring 50 by 50 μm . The dropdown list for this field offers a range of image sizes between 1 and 100 μm . The default condition is 100 μm .

You can also determine the size of the scan area using the **Zoom** button on the **Scan** panel (see next page).

Changing Scan Size and Location

With the **Zoom** button on the **Scan** panel, you can select the area on the image window that you wish to scan, as follows:

1. Click on the **Zoom** button.

The **Zoom** panel appears on the top of the Main window, to the right of the **Probe** panel and under the Image Enhancement part of the tool bar, as shown in Figure 5.8.

The outer (black) square on the panel represents the largest scan area available (100 by 100 μm). The inner (blue) square represents the current scan **Range** (either default or previously set using the **Range** field on the **Scan** panel, as described on the previous page). In Figure 5.8, the blue square represents a 50- μm scan range setting.

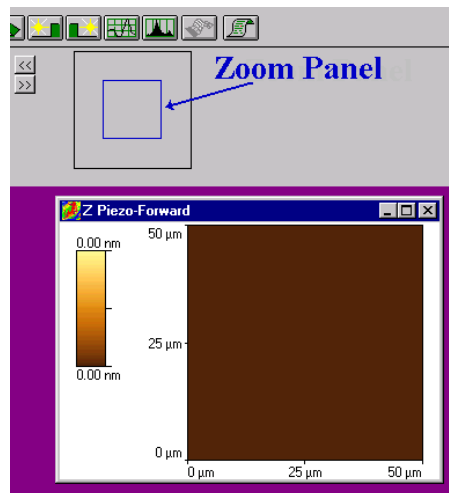


Figure 5.8
Zoom Panel

As the **Zoom** panel opens, coordinates (in μm) for the default location of the square are displayed on the **Scan** panel, below the word "Center."

2. With the mouse, place the cursor over the active image (title bar is blue in active image) whose scan area you wish to change; then left-click and drag the mouse to "draw" a square on the image.

As you do so, observe that the coordinates (in μm) displayed on the **Scan** panel and the size of the blue square on the **Zoom** panel change to match.

3. Release the left mouse button. The new scan **Range** is set.
4. Use the mouse to drag the scan square to the desired location.
5. Press the right mouse button to set the center for the new scan area, and close the **Zoom** panel.

Selecting the Scan Rate

The probe scans the sample surface at the speed specified in the **Rate** field on the **Scan** panel. The default scan speed is displayed in the **Rate** field. Typically, the **Rate** is set to one line/sec. For a 100- μm **Range**, the default **Rate** is 100 $\mu\text{m}/\text{sec}$.

The scan speed is as important as the **PID** settings in acquiring optimum images. It is possible to scan a sample so fast that the Z-piezo positioner cannot react quickly enough to track the surface accurately. See Appendix A for a discussion of the effects of scan and feedback settings on image quality.

The **Rate** field contains a scrollable list from which you can select the new speed.

Changing the Resolution

The default value for image resolution is displayed in the **Resolution** field on the **Scan** panel.

The **Resolution** value determines the number of data points that are collected along a scan line, as well as the number of scan lines that are made to create the image. With the default setting of 300, images that are acquired have 300 pixels per line and 300 lines per image. A higher resolution creates an image file that containing more data points, and the scan takes longer to complete.

See Appendix B for a discussion of the effects of scan and feedback settings on image quality.

If you wish to change the resolution of your scan, click on the down arrow for the **Resolution** field. Choose a new setting from the dropdown list that appears.

Changing the Scan Angle

The **Rotation** field shows the angle at which the probe rasters over the surface; the default value is 0.

Scanning with the probe at a different angle can help you assess whether a feature of an image is real or is an image artifact:

- If the feature is in the correct place and orientation after you have rescanned with a new angle (applying **Rotation**), then the feature is real.
- If the feature is no longer in the correct place and orientation after you have rescanned with a new angle, then the feature on the unrotated scan is an artifact.

Because the thermal probe is asymmetric, it is weaker when scanning at 90 degrees to the normal scan direction. Consequently, some scan angles may cause damage to the thermal probe. If the angle you choose on the **Rotation** field is a potentially dangerous one for the probe, a Warning message to that effect will appear, as shown in Figure 5.9.

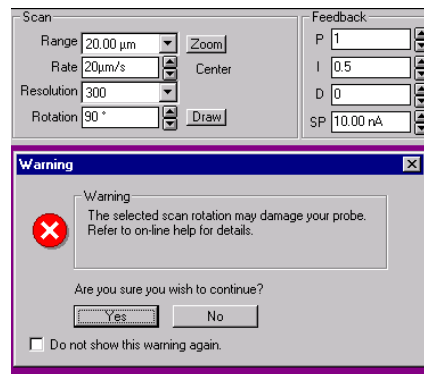


Figure 5.9
Warning Message for a Dangerous Probe Angle

If you are using a standard AFM probe, you can proceed with scanning even after receiving this warning.

If, however, you are using a thermal probe, you might damage the probe if you were to continue with the chosen angle of scan rotation; you should change it.

NOTE: If you check the “Do not show this message again” box on the Warning window, it will be unchecked when you exit the μ TALab™ Software, so that the Warning will be shown the next time a possibly dangerous probe angle is chosen.

You can change the scan angle in one of two ways:

- Select a new setting from the scrollable list in the **Rotation** field.
- Use the mouse to draw the scan angle on the image, as described on the next page.

Drawing the Scan Angle

You can draw the angle for the scan rotation as follows (see Figure 5.10):

1. Press the **Draw** button on the **Scan** panel.
2. Using the mouse, move the cursor to the image window on which you want to change the scan angle.
3. Left click the mouse and then drag it to describe a line at the desired angle on the image.
4. When you have drawn the angle you desire, right click with the mouse to set the angle.

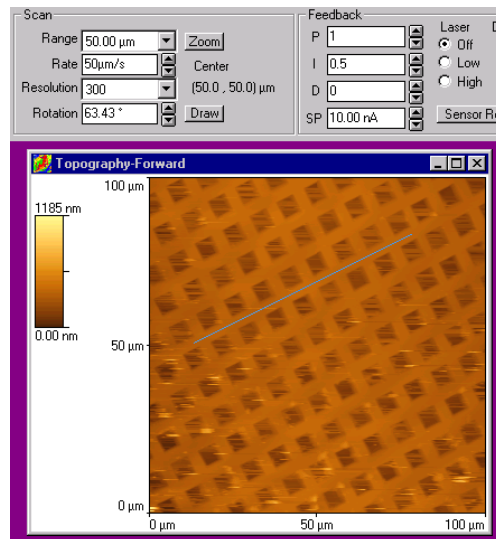


Figure 5.10
Using the Mouse to Draw the Scan Angle

Setting Feedback Parameters

The **Feedback** panel, shown in Figure 5.11, contains fields and buttons you can use to change the feedback settings.

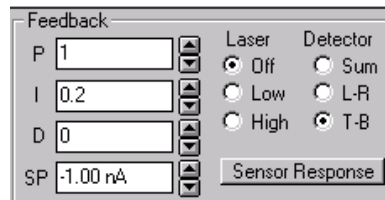


Figure 5.11
Feedback Panel

Figure 5.11 displays the default values for the feedback parameters:

- **P**: proportional gain; default value 1.
- **I**: integral gain; default value 0.2
- **D**: derivative gain; default value 0.
- **SP**: set point; default value -1.00 nA.

See Appendix B for detailed information about the **PID** and **SP** settings and how to change them.

Controlling the Laser

The **Laser** buttons on the **Feedback** panel allow you to control the μ TA 2990's laser detector. The three settings you can choose are:

- Off
- Low
- High

For more information about using the laser, see Appendix B.



WARNING: Never look directly into the laser beam. Always turn the laser off before you invert the head of the μ TA 2990.

Using the Detector Buttons

The three **Detector** buttons on the **Feedback** panel are used in aligning the laser on the μ TA 2990 MicroThermal Analyzer.

Please see Chapter 3 for a discussion of laser alignment.

Checking the Sensor Response

Once the **T-B** (top to bottom) sensor signal reaches the **SP** (set point) value during a tip approach to the sample, the system is considered to be in feedback. Occasionally during tip approach, the sample current can be increased before the tip reaches the surface because of long-range tip-sample interactions, such as static charge and moisture. This can cause the set point value to be reached and the feedback loop to be engaged before the tip actually has reached the surface. This phenomenon is known as *false feedback*.

The **Sensor Response** button on the **Feedback** panel allows you to check for false feedback, and opens the **Sensor Response** window, which displays the results. Figure 5.12 is a **Sensor Response** window from a probe with good force feedback.

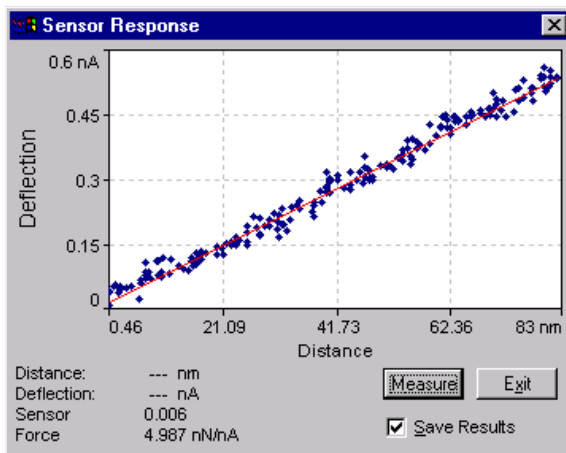


Figure 5.12
Sensor Response from Probe with Good
Force Feedback

Figure 5.13 shows the sensor response from a probe with false feedback and the warning message that is displayed to indicate the situation.

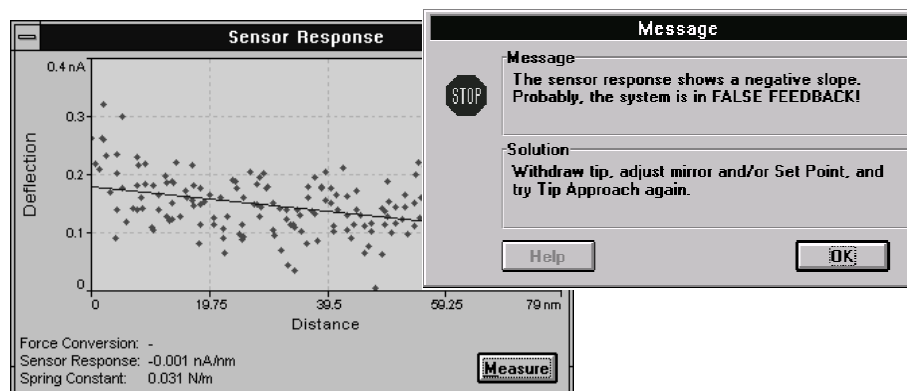


Figure 5.13
Sensor Response from Probe with False
Force Feedback*

When the system checks for false feedback, the Z-piezo positioner is ramped through approximately 10% of its total range while the sensor response (due to probe deflection) is measured. A graph is then plotted of Z-piezo positioner distance against probe deflection. When the system is in true feedback, a straight-line graph with a positive gradient is obtained, as shown in Figure 5.12.

*From *SPMLab—Release 4.0 Software Reference Manual*, TopoMetrix Corporation, 1996.

Using Thermal Feedback Controls

The **Probe** panel, shown in Figure 5.14, is located underneath the tool bar on the **Main** window (see Figure 5.6).

This panel contains a set of radio buttons that indicate the status of the thermal probe and allow you to turn it on or off, put it in automatic mode, or clean it.

Below the **Clean** button is a display showing the current Calibration file. For more information about temperature calibration, see Chapter 4.

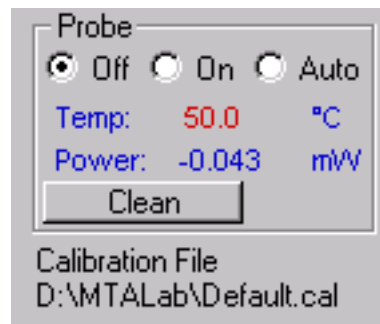


Figure 5.14
Probe Panel

Thermal Probe Settings

The thermal probe has three possible settings, shown as radio buttons across the top of the **Probe** panel:

- On
- Off
- Auto

The default condition for the thermal probe is **On**.

When the thermal probe is turned **Off**, no power is supplied to it. You should ensure that the thermal probe is **Off** before you begin changing probes.

When the thermal probe is turned **On**, the thermal feedback circuit is turned on and the probe is maintained at the current temperature conditions.

When the **Auto** button is activated, the system checks for the balance in the bridge circuit. If the bridge circuit can be balanced, the system will maintain the current temperature conditions. If the system cannot maintain the balance, the probe is turned off.

To choose any of the three probe settings, click on the desired radio button.

Checking Probe Temperature and Power Status

The **Probe** panel (see Figure 5.14) also contains two status displays for the thermal probe:

The **Temp** field on the **Probe** panel displays the current program temperature for the thermal probe.

- If the temperature value is displayed in **blue**, the thermal probe is in thermal feedback.
- If the temperature value is displayed in **red**, the thermal probe is not in thermal feedback; some of the possible reasons for the probe not to be in thermal feedback are that it is turned off, has become disconnected, or is badly calibrated.

The **Power** field on the **Probe** panel displays the amount of power required to maintain the thermal probe in thermal feedback.

- A *positive* **Power** signal indicates that the probe is in thermal feedback.

NOTE: This **Power** value is not a calibrated value. Comparisons of power values should be made only under *identical* experimental conditions.

Cleaning the Probe

When you press the **Clean** button on the **Probe** panel, the temperature of the probe is raised to its maximum value for 3 seconds in order to burn off any contaminants that may have adhered to the probe during imaging or LTA.

The **Clean** button is not available *during* imaging or LTA measurements (its label is grayed-out at those times).



CAUTION: Do NOT clean an uncalibrated probe or select automatic cleaning for it. Cleaning an uncalibrated probe could damage or destroy it.

Using the Temperature Mode Panel

The **Temperature Mode** panel, shown in Figure 5.15, is located to the right of the **Probe** panel on the μ TALab™ Software **Main** window (see Figure 5.6). If this panel and the **AC Mode Setup** panel are not displayed on the **Main** window, use the mouse to click on the toggle buttons to the right of the **Probe** panel; the toggle buttons allow you to toggle (switch) between the **Display** panel and the **Temperature** and **AC Mode Setup** panels.

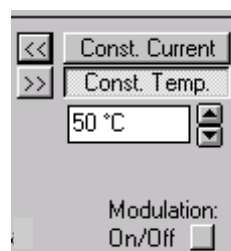


Figure 5.15
Temperature Mode Panel

The **Temperature Mode** panel enables you to choose the temperature mode used for scanning:

- In **Constant Current** mode, the probe is brought into thermal feedback using the **CC Setpoint** value set in **Acquisition Setup**. In this mode, the system maps the changes in the surface temperature of the sample.
- In **Constant Temperature** mode, the probe is maintained at constant temperature (or, if Temperature Modulation is turned on, constant average temperature). In this mode, the system collects thermal conductivity and other thermal images of the sample.

To select the temperature mode for scanning:

1. Turn on the probe so that you can observe the **Probe** field.
2. Select a temperature from the scrollable list that keeps the **Probe** field between 1 and 2 mW of power.

The **Modulation On/Off** button at the bottom of the **Temperature Mode** panel turns the Temperature Modulation circuit on and off. Figure 5.15 shows the button with **Temperature Modulation** turned off.

Using AC Mode Setup

The **AC Mode Setup** panel, shown in Figure 5.16, contains fields that enable you to monitor and change the amplitude and frequency of temperature modulation for your experiment. If this panel and the **Temperature Mode** panel are not displayed on the **Main** window, use the mouse to click on the toggle buttons to the right of the **Probe** panel; the toggle buttons allow you to toggle (switch) between the **Display** panel and the **Temperature Mode** and **AC Mode Setup** panels.

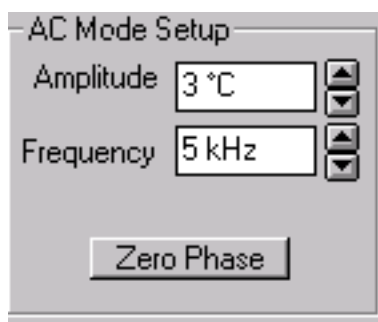


Figure 5.16 AC Mode Setup Panel

The **Amplitude** field allows you to monitor and select the amplitude of the applied temperature modulation. Values from 0.001°C to 100°C are acceptable; you will probably have to experiment to find the best value.

The **Frequency** field allows you to monitor and select the frequency of the applied temperature modulation. Values from 2 kHz to 100 kHz are acceptable; you will probably have to experiment to find the best value.

To change the values in either of these fields, select a new value from the scrollable lists they contain.

NOTE: The temperature modulation is not stable if the **C/T Response** and Modulation **Frequency** values are similar (within a factor of 2). If this occurs, the word "**Frequency**" flashes on the **AC Mode Setup** panel. Modulation Frequency must be a factor of 2 or greater in relation to the C/T response.

Zero Phase

There is a phase lag between the applied and observed temperature modulation. The phase lag is composed of instrumental and sample-related factors.

When you press the **Zero Phase** button on the **AC Mode Setup** panel, the software removes the current measured phase lag from any subsequently measured values.

The phase lag is always automatically adjusted to zero by the μ TALab Software prior to any measurements, to remove instrumental effects.

Using the Display Panel

The **Display** panel, shown in Figure 5.17, contains fields that enable you to adjust **Display** settings while a scan is in progress. The **Display** default values are set in the **Preferences** window.

If this panel is not visible on the **Main** window, use the mouse to click on the toggle buttons to the right of the **Probe** panel (see Figure 5.6); the **Display** panel will appear along with the **Temperature Mode** and **AC Mode Setup** panels.



Figure 5.17
Display Panel

Once you start image acquisition, the **Temperature Mode** and **AC Setup Mode** panels, if displayed, automatically switch back to the **Display** panel.

The **Leveling** field allows you to select the type of leveling function that is applied to the data as it is acquired. (For a full description of leveling functions, see Chapter 6.)

To change **Leveling** value, click on the arrow for the field. Choose a new value from the dropdown list that appears.

The **Shading** field enables you to select the type of shading that is applied to the data as it is acquired. (For a full description of shading functions, see Chapter 6.)

To change the **Shading**, click on the arrow for the field. Choose a new option from the dropdown list that appears.

Z Scaling enables you to control how the color palette represents the surface information at every x, y coordinate point of the image. Correct adjustment can significantly improve the visualization of any surface details in your data.

You can set the default mapping of the colors to the Z data in the **Preferences** window. Other **Z Scaling** options are as follows:

- **Min/Max** linearly distributes the palette from the minimum to the maximum Z values of the previously scanned area (this is updated every 20 line scans). This option optimizes Z scaling most effectively when a histogram of the sample's Z data is evenly distributed across the Z range on the image.
- **Standard Deviation** updates the Z scaling on the basis of the standard deviation of the Z values of the previously scanned area (this is updated every 20 lines).
- **Manual** updates the Z scaling on the basis of the values for **Z Range** and **Z Offset**, which you can enter on the other two fields on the **Display** panel (see below).

Z Range is the range in the Z height to which the histogram will be applied. **Z Offset** is the point in the histogram to which this range will be applied. For example, if the **Z Range** is 20 nm and **Z Offset** is 50 nm, the histogram will be applied to all points within a range from 10 nm below to 10 nm above 50 nm, or 40 to 60 nm.

Using the Oscilloscope Window

The **Oscilloscope** window, shown in Figure 5.18, allows you to monitor two signals simultaneously that relate to the various image signals.

You can access this window in one of two ways:

- Press the **Oscilloscope** button  on the tool bar.
- Select **Acquisition/Commands/Oscilloscope** from the menu bar.

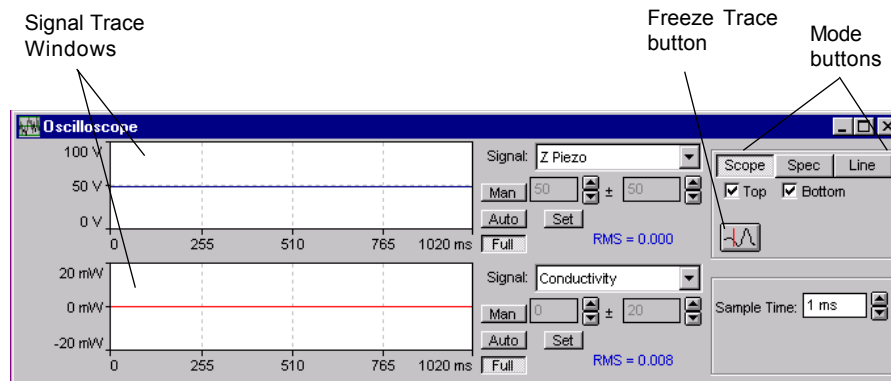


Figure 5.18
Oscilloscope Window in Scope Mode

The Oscilloscope may be used in one of three modes:

- **Scope:** Oscilloscope
- **Line:** Line Scan
- **Spec:** Spectrum


You can select the modes using the **Mode** buttons, located in the top right corner of the **Oscilloscope** window.

Scope Mode

In **Scope** mode (the default mode when the Oscilloscope is first turned on), the **Oscilloscope** window displays time-based amplitude data for the currently selected signals. In default settings for this mode, two Oscilloscope signals are displayed, **Z-piezo** and **Sensor** (as shown in Figure 5.19).

To access other Oscilloscope input signals, click on the down arrow for either of the **Signal** fields. Choose the desired signal from the dropdown list that appears.

The following options or controls are available on the **Oscilloscope** window in **Scope** mode:

- **Manual (Man)** allows you to set the vertical scale to the \pm value entered in the **Scale** field.
- **Auto** automatically sets the vertical scale to a range slightly larger than the highest or lowest value in the previous sweep. This is the optimum setting for viewing small signal variations on the trace.
- **Full** sets the vertical scale to the full range appropriate to the currently selected signal.
- The **Freeze Trace** button  freezes the signal trace so that you can make measurements in the window, as described on the next page.
- The **Top** and **Bottom** buttons allow you to toggle between a dual-channel and a single-channel oscilloscope display.

Top Bottom

Left-click in the either of the Signal Trace windows to attach a vertical cursor to the mouse, as shown in Figure 5.19. With this cursor, you can take an exact signal, time reading anywhere in the window. Right-click the mouse to switch back to the standard mouse cursor.

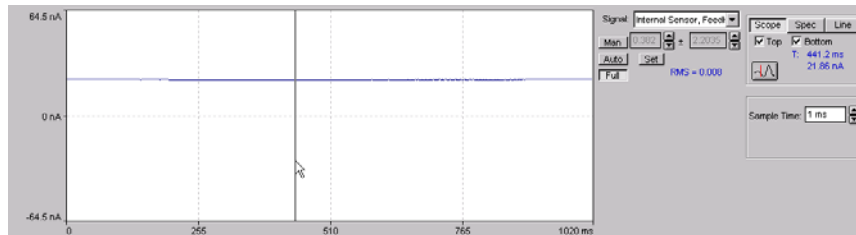
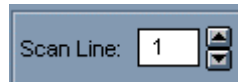


Figure 5.19 Oscilloscope Window
in Scope Mode Showing Vertical Cursor

Line Mode

Selecting **Line** mode initiates a continuous single line scan. The probe can be either (1) raised or (2) in feedback and on the surface of a sample during a line scan.

You can select which signal line will be displayed from the **Scan Line** box, shown below, which appears on the **Oscilloscope** window when you select **Line** mode:



In order to scan a line in the forward or reverse direction, select the appropriate image on the **Acquisition Setup** window (see "Selecting Images and Measurements"). If you selected a reverse scan image on the **Acquisition Setup** window, additional **F** (forward) and **R** (reverse) buttons will appear on the **Oscilloscope** window, as shown below:



Spectrum Mode

Selecting **Spectrum** mode initiates a spectrum analysis of the Z piezo positioner, Sensor or DC (thermal conductivity), and AC (temperature amplitude and phase) thermal signals.

Spectrum mode is typically used to monitor external vibrations and noise that may affect the quality of data that you obtain with your μ TA 2990.

By monitoring the Z piezo positioner and Sensor signals, you can detect mains voltage frequency or any other constant-frequency external vibration.

In Spectrum mode, as shown in Figure 5.20, the **Oscilloscope** window displays three additional options or controls:

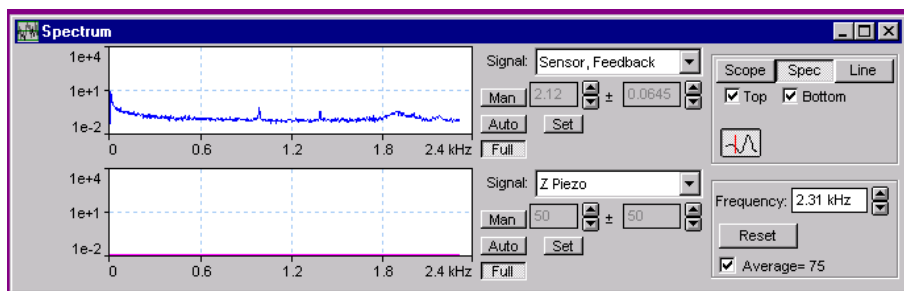


Figure 5.20
Oscilloscope Window in Spectrum Mode

- **Frequency** allows you to set the frequency range of the sweep. The low end of the sweep is set at 0 Hz by default. A frequency range of 0 to 0.5 kHz is useful for monitoring vibration-induced noise.
- **Average** toggles the signal-averaging function on and off. When **Average** is selected, the display shows the continuously calculated average of successive frequency sweeps. Turning the **Average** function on minimizes the effects of random noise on the signal display.
- **Reset** resets the running spectrum average.

Localized Thermal Analysis

Overview

It is not necessary to acquire images of the surface of your sample before acquiring Localized Thermal Analysis (LTA) data.

The basic procedure for acquiring LTA data is as follows:

1. Select Localized Thermal Analysis mode by selecting **Local Thermal Analysis** from the **Acquire** menu on the μ TALab™ **Main** window.

Several new panels appear underneath the tool bar on the μ TALab™ **Main** window, as shown in Figure 5.21. The **Local Thermal Analysis** window also appears, as shown in Figure 5.22 on the next page.

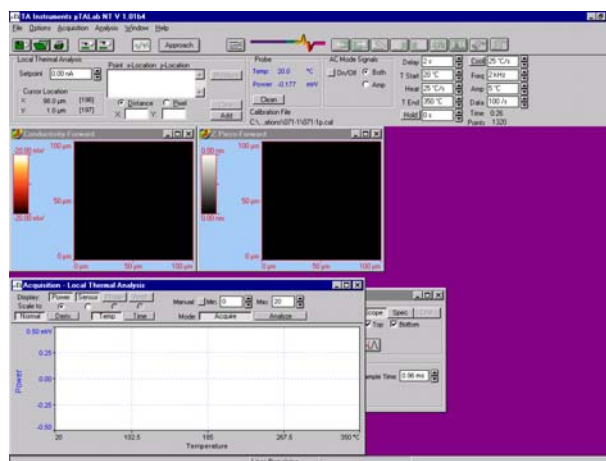


Figure 5.21
 μ TALab Main Window in Localized Thermal Analysis Mode

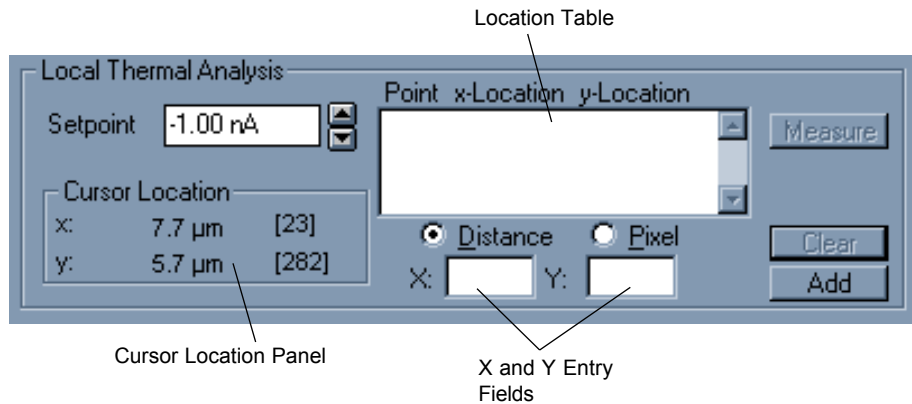


Figure 5.22
Local Thermal Analysis Window

2. Select locations for the LTA experiment by either placing the cursor and clicking on an open image window or entering the x, y coordinates on the **Local Thermal Analysis** window.
3. Enter the temperature program for the experiment on the **Probe** panel.
4. Select **Measure** on the **Local Thermal Analysis** window.

The real-time data for each LTA measurement is displayed in the **Local Thermal Analysis** window. This window also provides basic routines for analyzing your data, selecting LTA baselines, and setting temperature calibration points. Advanced LTA analysis is provided by the Universal Analysis software program.

NOTE: When you click on the **Measure** button, the following Baseline Warning may be displayed if (1) you have chosen the **Subtract Baseline** and **Use Existing Baseline** options in the **Local Thermal Analysis** panel of the **Preferences** window, (2) you are collecting the first LTA since the μ TALab Software was started, or (3) you have changed any of the parameters:

Baseline Warning

The stored baseline does not match your experiment. Do you want to proceed without a baseline?

Three choices are presented:

Proceed: An LTA is collected without a baseline.

Measure: A baseline in air is measured.

Abort: The LTA run is aborted.

Selecting Measurement Locations

You can select the location (up to 9 locations are possible) for the LTA experiments in two ways:

- Using the mouse and cursor.
- Entering coordinates in the **X** and **Y** input fields on the **Local Thermal Analysis** panel.

The x and y coordinates for the measurement locations may be displayed in either distance (μm) or pixels. Select the units for display by pressing either the **Distance** or the **Pixel** radio button located under the **Location Table**.

To select locations with the cursor:

1. Move the cursor into any of the open image windows on the $\mu\text{TALab}^{\text{TM}}$ **Main** window. As you move the cursor across the image window, the **X** and **Y** fields on the **Cursor Location** display constantly change to reflect the position in either μm or pixels.
2. Click on the location that you want analyzed. The coordinates of the location you have chosen are then displayed in the **Location Table**, as shown in Figure 5.23.

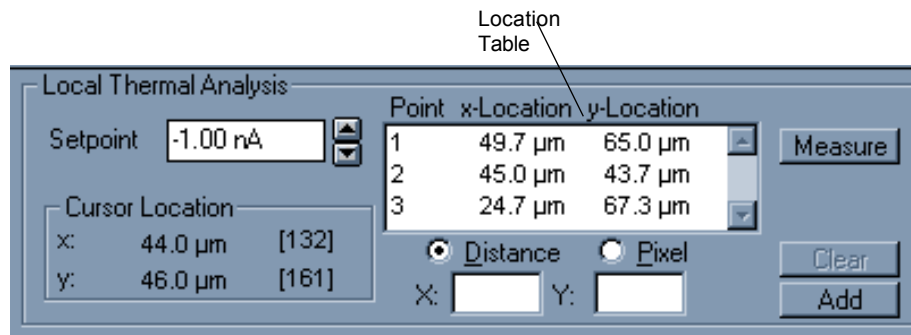


Figure 5.23
Location Table Showing Selected LTA Locations
Using Distance as Display Unit

To enter coordinates on the LTA panel:

1. Enter the x coordinate for the desired location in the **X** field on the LTA panel.

2. Enter the y coordinate for the desired location in the **Y** field on the LTA panel.
3. Click on the **Add** button; the entered coordinates will appear on the **Location Table**.

To remove a location from the Location Table, select the coordinates line with the mouse to highlight it, and then press the **Clear** button.

The number of pixels in the x and y directions on an acquired image depends on the value you chose for **Resolution** on the **Scan** panel before you acquired that image.

Changing the Set Point

You can use the **Set point** field on the **LTA** window (see Figure 5.23 or 5.24) to change the force that the probe applies to the surface during an LTA experiment. More positive set point values apply larger forces, and more negative set point values apply smaller forces.

NOTE: The value in the SP field must never be more negative than the value of the TB signal when the probe is off the surface.

See Appendix B for a detailed discussion of the set point.

To change the set point, select a new value from the scrollable list in the **SP** field.

Preparing the Probe

Figure 5.24 shows the **Probe** panel in Localized Thermal Analysis mode.

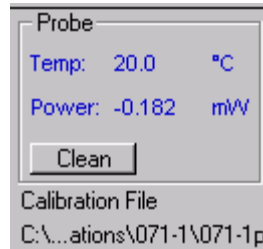


Figure 5.24
Probe Panel
in Localized Thermal Analysis Mode

As described earlier, you can clean the thermal probe (heat to its maximum temperature for 3 seconds) by pressing the **Clean** button on the **Probe** panel. In LTA mode, you can set the probe to be cleaned automatically after each LTA measurement by selecting "**Auto Clean Probe after scan**" in the **Local Thermal Analysis** panel of the **Preferences** window.

Selecting AC Mode Signals

The **AC Mode Signals** panel, shown in Figure 5.25, is located under the tool bar and to the right of the **Probe** panel on the μ TALab **Main** window in Localized Thermal Analysis mode. With this panel, you can select which Modulated (AC) signals (if any) will be collected and stored along with the μ TMA and μ MDTA data.

To collect and store AC signals during your LTA experiment, click the **On/Off** button on the **AC Mode Signals** panel to the On position.

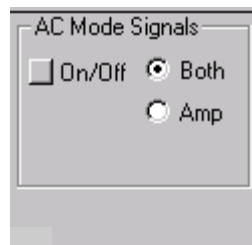


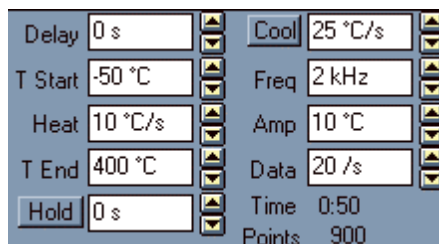
Figure 5.25
AC Mode Signals Panel

You may choose to collect either the amplitude signals only or both amplitude and phase signals. These additional signals will then be available on the **Localized Thermal Analysis** window as well as being stored in the data file with your μ TMA and μ MDTA signals for subsequent formatting and analysis with the Universal Analysis program.

To select or deselect these signals, click on the **Amp** and **Both** radio buttons on the **AC Mode Signals** panel.

Determining the Temperature Program

The **Temperature Program** panel, shown in Figure 5.26, is where you enter the temperature program details for your LTA measurements. Each location in your **Location Table** will be measured under exactly the same conditions.



Delay	0 s	Cool	25 °C/s
T Start	-50 °C	Freq	2 kHz
Heat	10 °C/s	Amp	10 °C
T End	400 °C	Data	20 /s
Hold	0 s	Time	0:50
		Points	900

Figure 5.26
Temperature Program Panel

You can enter or change any value in the **Temperature Program** panel in two ways:

- Enter a new value in the field.
- Select a new value from the scrollable list for the field.

The fields on the **Temperature Program** panel are as follows:

- **Delay:** Once the probe has lowered itself onto the surface of the sample, the μ TALab™ software “turns off” the force feedback loop. The probe is now resting on the surface of the sample with only the temperature feedback loop operational. The program temperature is held at the **T Start** temperature for the **Delay** time (displayed in seconds) before the probe is heated.
- **T Start:** The **T Start** value is the starting program temperature (in °C) for each LTA measurement.

NOTE: Because T Start is a program temperature, it may be below room temperature, even if no cooling device is present. Starting the temperature program below room temperature is useful, in that it allows you to observe the “kick-in” of the power signal when the program temperature exceeds room temperature. This “kick-in” is useful for providing a room temperature calibration point as well as for diagnostic troubleshooting.

- **Heat:** The heating rate for the LTA measurement in °C/sec.

- **T End:** The final program temperature (°C) in for each LTA measurement.
- **Hold:** The hold time (in seconds) at the **T End** program temperature. If the **Hold** button is pressed, LTA data will be stored during this isothermal portion of the each LTA location. The default setting for this field is *not* to store this isothermal data.
- **Cool:** The cooling rate for the LTA measurements in °C/sec. If the **Cool** button is pressed, LTA data will be stored during the cooling portion of each LTA location. The default setting for this field is *not* to store the cooling data.
- **Frequency:** The temperature modulation frequency of thermal program.
- **Amp:** The temperature amplitude of the thermal program.
- **Data:** The data collection rate in points/second.

The **Temperature Program** panel also contains displays of some parameters:

- **Time:** The time for each measurement is calculated from the preceding parameters and displayed here.
- **Points:** The number of data points collected for each LTA measurement is also displayed. The **Points** field changes from blue to red when your file size has exceeded the size of a floppy disk.

Local Thermal Analysis Window in Acquire Mode

The **Local Thermal Analysis (LTA)** window, shown in Figure 5.27, has two modes of operation, **Acquire** and **Analyze**.

Acquire is the default mode. When you first select **Localized Thermal Analysis** from the **Acquisition** menu on the μ TALab™ **Main** window, the window is in **Acquire** mode. In this mode, you can set up how you would like your real-time LTA data to be displayed and scaled, even while the measurements are in progress.

The **Analyze** mode allows you to format and analyze your data and provides the tools required to set LTA baselines and temperature calibration points.

You change between the two modes by clicking on the **Acquire** and **Analyze** mode buttons at the top right of the **LTA** window.

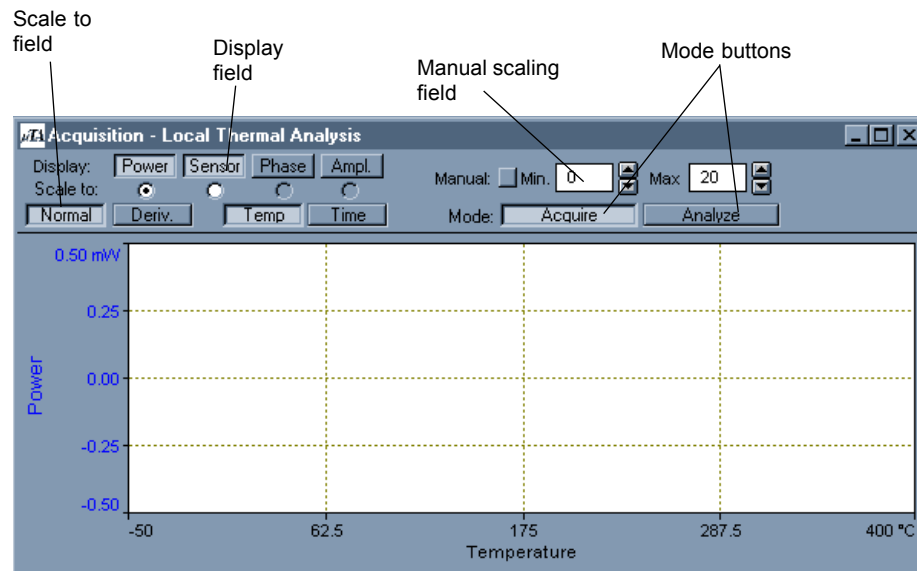


Figure 5.27
LTA Window in Acquire Mode

Signal Selection

In Acquire mode, the **LTA** window displays a graph of signal against program temperature. Up to four signals may be displayed:

- Power
- Sensor
- Phase
- Amplitude

You can select which signals you wish to have displayed by clicking on buttons in the **Display** field, located in the top left corner of the window. In Figure 5.27, only the **Power** and **Sensor** signals have been selected for display.

Signal Scaling

Although you can display up to four signals, only one y axis is displayed. You can use the radio buttons in the **Scale to** field, located under the **Display** buttons, to select which signal (Power, Sensor, Phase, or Amplitude) you wish to see full scale. The y axis then displays the appropriate units for the selected signal.

Figure 5.27 shows an example in which the y axis will be scaled to the **Power** signal, and the y axis units will be mW.

During the real-time collection of data, the y axis is constantly scaled to keep the data on screen. If you wish to impose manual limits on the y range, use the **Manual Scaling** field, located in the top right corner of the LTA window (see Figure 5.29), as described on the following page.

1. Press the **Manual** button.
2. Select the new minimum value for the y axis from the scrollable list for the **Min** field.
3. Select the new maximum value for the y axis from the scrollable list for the **Max** field.

Figures 5.28 and 5.29 are examples of **LTA** windows set up to display **Power** and **Sensor** signals with the scaling selected automatically and manually, respectively.

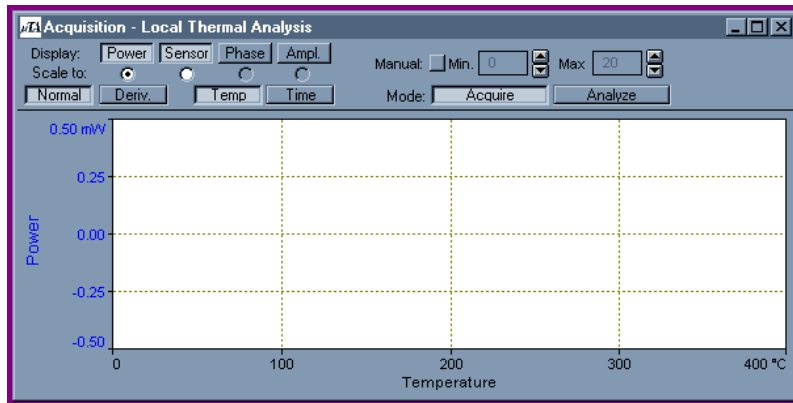


Figure 5.28
LTA Window with Automatic Signal Scaling

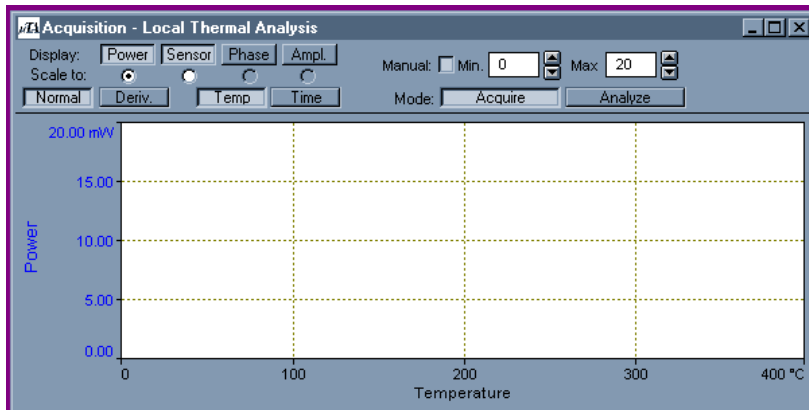


Figure 5.29
LTA Window with Manual Signal Scaling

Signal Display

Four buttons under the **Scale to** field on the **LTA** window allow you to vary the ways in which the selected signals and data are displayed.

You may have the selected signals displayed in two ways:

- Normal (press the **Normal** button).
- As their first derivatives (press the **Deriv** button).

NOTE: It is not possible to display one signal as a derivative and the others as normal.

Data can be displayed as a function of either:

- Temperature (press the **Temp button**).
- or*
- Time (press the **Time button**).

Local Thermal Analysis Window in Analyze Mode

Use the **Analyze** mode of the LTA window to:

- Take a quick “first look” at your LTA data
- Select LTA baselines and temperature calibration points

When you switch the window to **Analyze** mode, a new set of panels appears at the bottom of the LTA window, as shown in Figure 5.30.

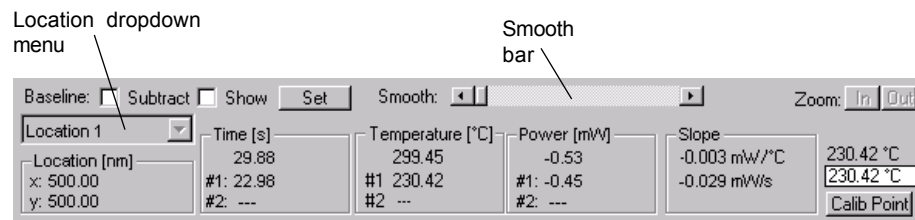


Figure 5.30
Analyze Panels for LTA Window in Analyze Mode

The LTA window in **Analyze** mode makes available two cursors that you can use to scale and mark the LTA data.

To obtain a cursor, left-click on the data window; a movable vertical line cursor appears. Move the cursor to the desired location, and left-click again to apply a blue carat to the point chosen. Left-click the mouse again to obtain the second cursor. When you are done, right-click the mouse to make the **Zoom** buttons active.

If you bracket an area of interest, you can expand the region enclosed by the two carats by clicking on the Zoom: **In** button. Pressing the Zoom: **Out** button restores the original scaling.

The values for the cursor positions are constantly updated in the **Time**, **Temp**, **Power**, and **Slope** displays at the bottom of the LTA window.

The buttons across the top of the **Analyze** mode panels enable you to modify how the data are displayed, as follows:

Location

You can choose to display all locations, or a specific location, using the **Location** dropdown menu, as shown in Figure 5.31. After you have selected the location, the coordinates for the selected/highlighted location are displayed in the **Location** field, below the **Location** dropdown window.

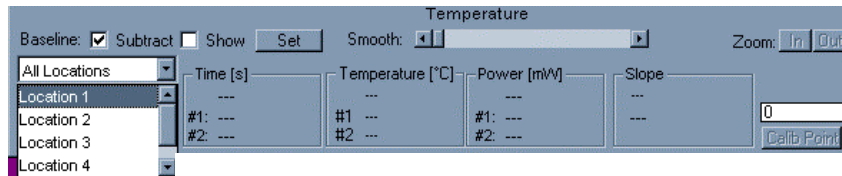


Figure 5.31
Location Dropdown Menu

Smoothing

You can increase or decrease the amount of smoothing applied to the LTA data by using the **Smooth** bar. Move the indicator on the bar to the left for less smoothing or to the right for more smoothing. You can move the indicator in one of two ways:

- Press the left or right arrows at either end of the bar
- Click on the indicator and move/drag it left or right with the mouse.

Baseline Subtract, Show, and Set Options

If you acquired the LTA data with baseline subtraction (either an air or a reference surface), the **Subtract** box is marked to remind you that you are displaying “corrected” data. If you wish to see the baseline data, select the **Show** check box, as shown in Figure 5.32.

If you wish to use the data from one of displayed LTA locations as the baseline for all subsequent LTA measurements, select the location using the **Location** dropdown menu so that it is the only LTA data shown in the window. Then press the **Set** button. You may wish to smooth the data somewhat before selecting the data as the current LTA baseline.

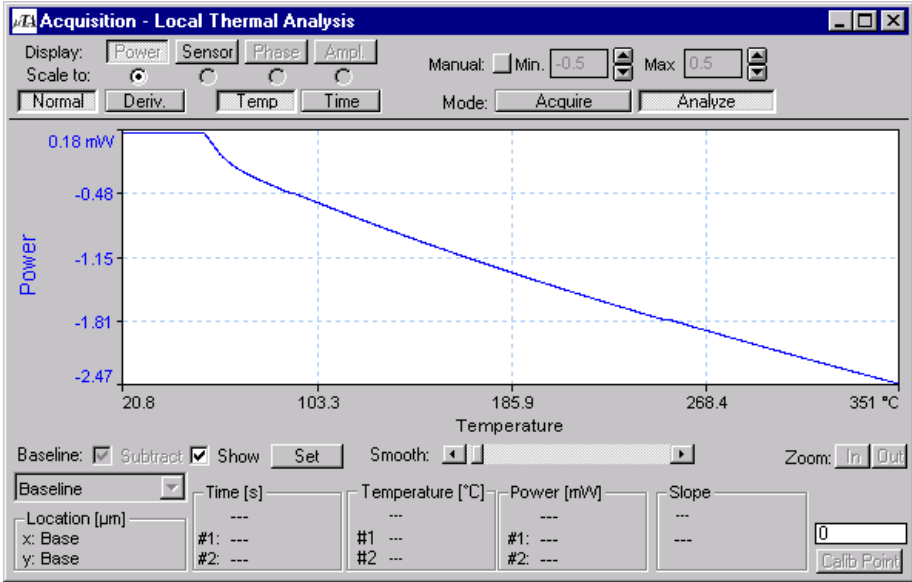


Figure 5.32
LTA Baseline Data Shown

Customizing μ TALab[®] Settings

You can customize many of the default settings for the μ TALab software through selectable preferences. To access the preference window click on **Options** and select **Preferences** from the drop-down menu that is displayed. Three choices are presented: **2D Imaging**, **Local Thermal Analysis**, and **General**. Each choice brings up a window. For each window make your changes and press **OK** to apply them. To abort changes and return to the previous settings, press **Cancel**. The windows that accompany each choice are described below.

2D Imaging

This panel shows the default display parameters used for acquired images:

Display Defaults

- **Z-Scaling** selects the default type of Z-scaling for your acquired images. The default setting is **Standard Deviation**.
- **Leveling** selects the default type of leveling for your acquired images. The default setting is **1st Order Deviation**.
- **Shading** selects the default type of shading for your acquired images. The default setting is **Off**.

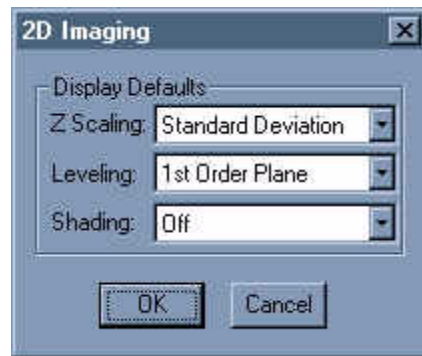


Figure 5.33
2D Imaging Window

LTA Options

The window shown to the right is displayed when you select **Options/Preferences/Local Thermal Analysis**. The following options appear on the window:

Local Thermal Analysis

Subtract Baseline: Causes subsequent LTA measurements to be made using baseline subtraction for each location. There are two subchoices for this setting:

- **Acquire New Baseline:** The system will acquire a new baseline before each set of LTA measurements. The new baseline will be acquired, in air, while the probe is above the sample surface prior to the Approach for the first LTA measurement.
- **Use Existing Baseline:** The existing baseline will be used for each LTA measurement. Any previously measured LTA measurement can be **Set** as the exiting baseline.

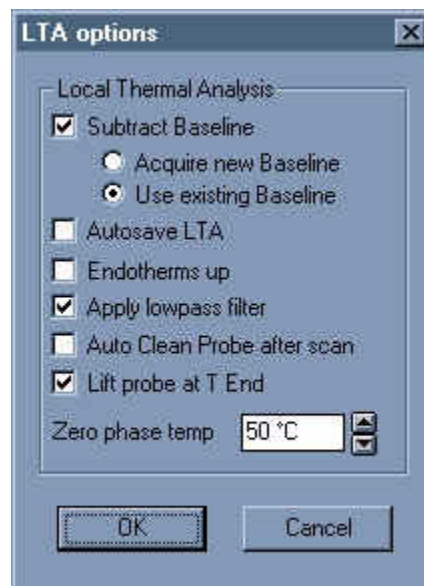


Figure 5.34
LTA Window

NOTE: If the experimental parameters for the LTA measurements are not identical to the baseline parameters, the baseline cannot be used, and the following Baseline Warning will be displayed:

Baseline Warning

The stored baseline does not match your experiment. Do you want to proceed without a baseline?

Three choices are presented:

Proceed: An LTA is collected without a baseline.

Measure: A baseline in air is measured.

Abort: The LTA run is aborted.

Autosave LTA: Prompts you to save the data whenever you are moving between acquiring images and acquiring LTA data, and vice versa.

Endotherms up: The default direction for endotherms is down. This is to ensure consistency of endotherm direction when comparing μ MDTA data with conventional DSC data (in which endotherms are displayed down) in Universal Analysis.

Apply lowpass filter: Applies a low-pass (smoothing) filter to the LTA data.

Auto Clean Probe after scan: Causes the probe to be heated to its maximum temperature for 3 seconds after each LTA measurement.



CAUTION: This box should NOT be checked if you are using an uncalibrated probe. Cleaning an uncalibrated probe could damage or destroy it.

Lift Probe at T End: Check this option to lift the probe from the surface when the T End temperature is reached during a local thermal analysis experiment. This can reduce the amount of surface damage and contribute to the life of the probe. The length of time that the motor runs controls the distance that the probe is lifted. The number entered in the **Hold** field on the Temperature Program panel controls how long the motor runs.

Zero Phase Temp: During AC Local Thermal Analysis operation, if the thermal probe is not in thermal feedback at the start of an experiment, the probe will heat to the **Zero Phase Temperature** and zero the phase of the modulated signals.

General

The window shown to the right is displayed when you select **Options/Preferences/General**. The following options appear on the window:

Tip Approach Settings

Test for False Feedback causes the system to automatically test for false feedback every time the probe comes into force feedback after an Approach.

The default setting for this parameter is **On**.

Determine Sensor Response causes the system to automatically perform a sensor response curve after the system is in force feedback.

The default setting for this parameter is **On**.

Miscellaneous Settings

Show ToolTips: Displays a ToolTip (a brief text description) when the mouse rests over any specific tool bar button for more than a couple of seconds.

Print Editor Full Details: Transfers all elements on the screen to the **Print Editor** when it is opened (see Chapter 7). These elements include signal windows, panels, and images. If this option is not selected, only the image windows are transferred to the **Print Editor** when it is opened.

Clear Images before Scan: Clears (removes all information/image information from) all image windows each time the Scan button is pressed to acquire new images.

Tip Cleaning

Time: This field controls how long the probe is held at the cleaning temperature.

Temperature: This field controls the temperature used for cleaning the probe.

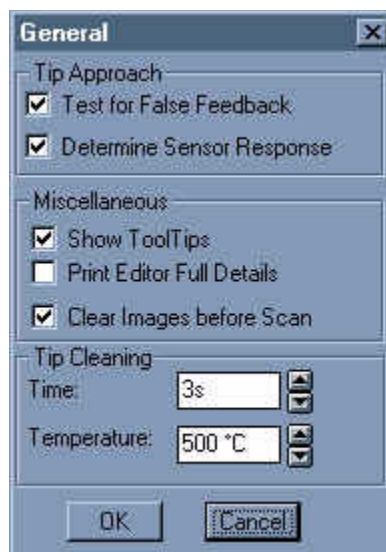


Figure 5.35
General Window

Chapter 6

Image Processing & Analysis

Introduction

The μ TALab® Software provides a range of tools that you can use to display, process, and measure the images collected by your μ TA® 2990. All images are composed of an array of numbers that are collected while the probe tip scans across the surface of the sample. You can manipulate and process this array to enhance the appearance of the image. The tools available for enhancing and measuring your images are described in this chapter.


Using the File Manager

The μ TALab Software's **File Manager** window differs from the standard Windows® **File Open** window in the following ways:

- You can select files (browse) by file name or by seeing a graphic representation of your image (thumbnail).
- You can edit, convert, and copy μ TA™ data files from the **File Manager** window.

The **File Manager** function is a very powerful part of the μ TALab Software, and it is worth spending some time to become familiar with it.

You can open the **File Manager** window in three ways:

- Select **File** from the menu bar on the μ TALab **Main** window; then select **Open** from the submenu that appears.
- Press CTRL+ O keys on the keyboard
- Click on the **File Open** button  on the tool bar.

The **File Manager** window appears, as shown in Figure 6.1 below.

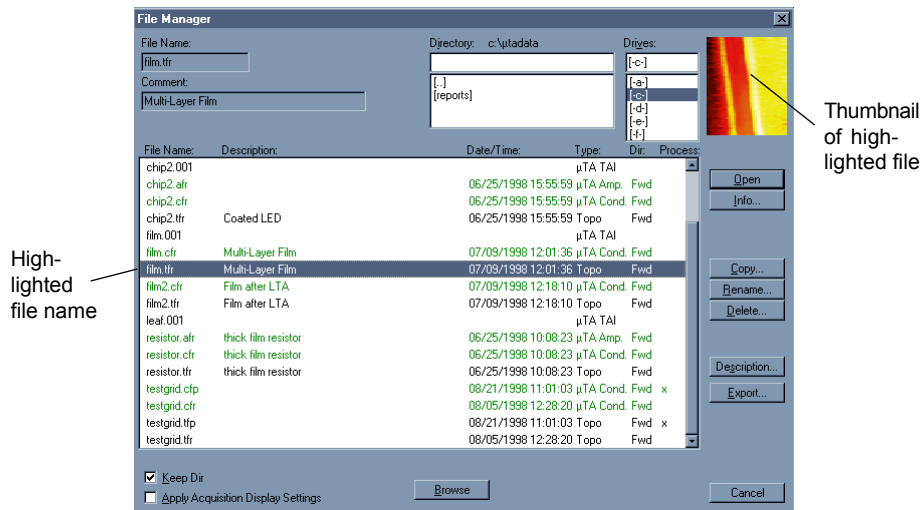


Figure 6.1
File Manager Window in "List" Mode

From any directory on your controller, the **File Manager** window displays only the types of files that μ TALab Software recognizes. These are:

- Image files
- Localized thermal analysis (LTA) files

You can navigate around your controller/network using the **Directory** and **Drives** dropdown menus. The current directory and drive are always highlighted on the **File Manager** window. If a highlighted file is recognized by the μ TALab Software as an image file, a thumbnail of the image is displayed in the top right-hand corner of the **File Manager** window, as shown in Figure 6.1.

The columns on the **File Manager** window are as follows:

- **File Name:** The recognized μ TALab Software files in the current directory.
- **Description:** The sample description that was entered by the last user of the file. You can change this information at any time by clicking on the **Description** button and entering the new text.
- **Date/Time:** The date and time at which the file was last saved.
- **Type:** The type of image file, which is derived from the three-number or three-letter extension in the file's name (after the period).

Files with numerical extensions are Localized Thermal Analysis (" μ TA TAI") files.

The first letter of a three-letter extension indicates the type of image. The second letter indicates the direction in which the probe was traveling when the image was acquired. The third letter indicates whether the file contains raw or processed data. So, for example, a file extension of ".tfr" means that the data file contains an unprocessed (raw) topography image obtained in the forward direction.

Table 6.1, on the next page, summarizes the definitions for the individual letters in a μ TALab file name extension.


Table 6.1
Explanation of File Extensions

Letter	Explanation and Type
First letter:	
a	Modulated temperature amplitude (μ TA Amp.)
c	Thermal conductivity (μ TA Cond.)
p	Modulated temperature phase (μ TA Phase)
t	Topography (Topo.)
Second letter:	Probe Direction:
f	Forward
r	Reverse
Third letter:	Type of data:
r	Raw
p	Processed

- **Dir:** The direction of the probe during the scan (Fwd, forward; Rev, reverse).
- **Process:** An "x" indicates that the image has been processed and is not raw data.

The **File Manager** window contains a set of buttons on the right side and bottom that enable you to perform actions with or learn more about the currently selected file or files. Table 6.2, on the next page, contains explanations of these buttons.

Table 6.2
Buttons on the File Manager Window

Button	Explanation
Open	Opens the selected file(s) for processing. This button is disabled (“grayed out”) if no file is selected.
Info	Opens the File Info window, which displays information regarding the acquisition scan parameters for the selected file.
Copy	Opens the File Copy window, in which you can copy the selected file to a new location.
Rename	Opens the File Rename window, in which you can change the selected file’s name.
	<p>CAUTION: DO NOT change the file name extension of a μTA image. The file extension indicates characteristics of the file for μTALab Software. If you change the extension of a file name, the software will not be able to open or otherwise process the file.</p>
Delete	Permanently removes the selected file from the controller.
Description	Opens the File Description window, in which you can change or add text to the file’s description field.
Export	Creates a new file, from the selected file, that contains ASCII format data. This enables the data to be read into other compatible software programs.

(table continued on the next page)

Table 6.2
(continued)

Button	Explanation
Browse	When the File Manager window is in List mode (see next page), pressing this button displays a thumbnails (preview) of the selected file. If the selected file is an LTA file, which cannot be displayed as a thumbnail, clicking on the Browse button will cause a “No Browse” message to appear. See the next section for a further discussion of Browse mode.

There are also two check boxes on the **File Manager** window:

- **Keep Dir:** When this box is checked, the **File Manager** window will “remember” the last open directory. The next time that the window is opened, it will display the “remembered” directory.
- **Apply Acquisition Display Settings:** When this box is checked, all of the Z-scaling, leveling, and shading that were used when the image was acquired (see Chapter 5) is applied to it as the image file is opened. The original (raw) data is not changed when this option is applied. To recall the original (raw) data, ensure that this option is not selected (*i.e.*, that this box is not checked) when you open an image file.

Browse View

Figure 6.2 shows an example of the File Manager window in **Browse** mode. Note that in this mode, the **Browse** button has changed to a **List** button; clicking on this button returns you to the standard **List** mode of the **File Manager** window.

To open a file for image processing, double-click on the file's thumbnail.

If you have asked for the **Browse** view of an LTA file, which cannot be displayed as a thumbnail, a “No Browse” message appears. Double-clicking on an LTA's file name opens the Universal Analysis software on your TA controller.

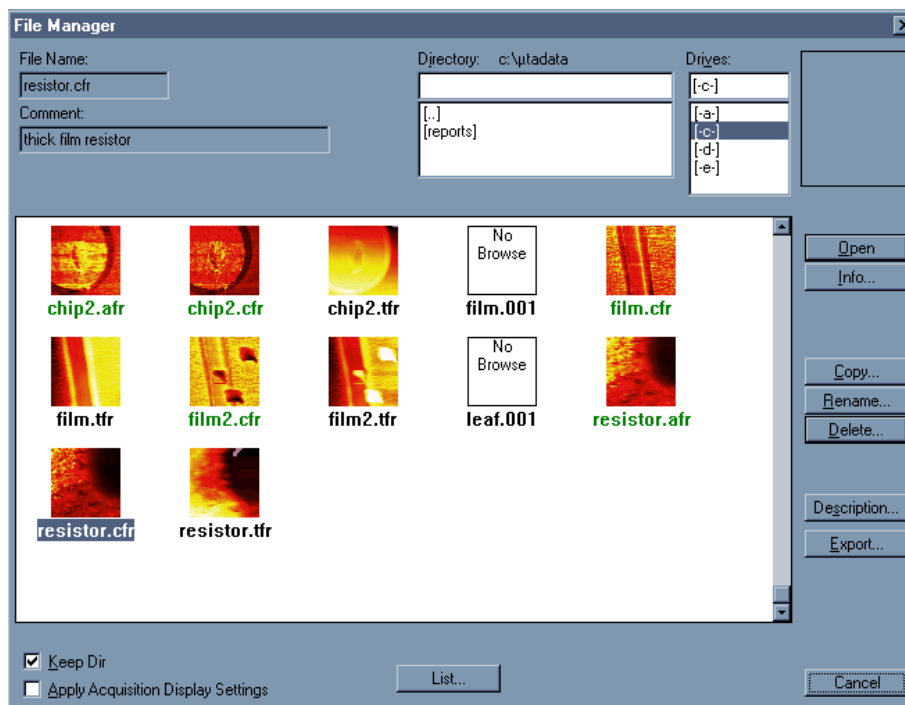


Figure 6.2
The μ TALab File Manager in “Browse” Mode

Image Buffers

Each time you process an image with the μ TALab Software, a copy of it is placed into a buffer. The last 6 image-processing operations are stored in buffers for individual file you open:

- Buffer 1 always contains the original image.
- Buffers 2 through 6 contain processed images.

If all 6 buffers are all full and the image is processed further, the image in buffer 2 is deleted. The image in buffer 3 is moved to buffer 2, and the image in buffer 4 into buffer 3, and so on. The newest image is then placed in the now empty buffer 6.



You can scroll forward and backwards through a sequence of processing steps by using the **Previous Buffer**  and **Next Buffer**  buttons on the tool bar.

Image Display

You can enhance or modify any image to alter the basic visual display characteristics of the image:

- Contrast and shading
- Palettes

Some of the options actually change the meaning of the data you are presenting. Therefore, you should always use display capabilities with the practical significance of the raw data in mind.

Contrast and Shading

On the **Contrast and Brightness** window, as shown in Figure 6.3, you can change the contrast and brightness characteristics of an image. Contrast and brightness adjustments change the distribution of the active palette used to display the image. To open this window, select **Options** on the μ TALab **Main** window's menu bar, and then select **Contrast and Brightness** from the dropdown menu.

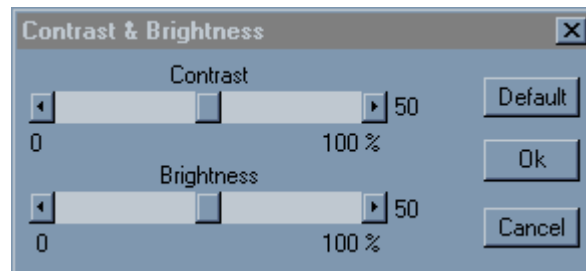


Figure 6.3
Contrast and Brightness Window

You can modify both the contrast and the brightness of your selected image using the **Contrast** and **Brightness** sliders on this window, while monitoring the results on the displayed image. To return to the original values for contrast and brightness, click on the **Default** button.

Changing Palettes

You can change the color palette used to display your image. Viewing data with different color palettes can enhance the features in your images.

Color palettes are not part of the stored data for an image. Any image that is opened (or viewed using the **File Manager**) is always displayed in the currently selected palette—no matter what palette was used for the original data.

To change the color palette of your selected, choose **Options** on the μ TALab™ **Main** window's menu bar, and then select **Palettes** from the dropdown menu. The **Palettes** window appears, as shown in Figure 6.4.

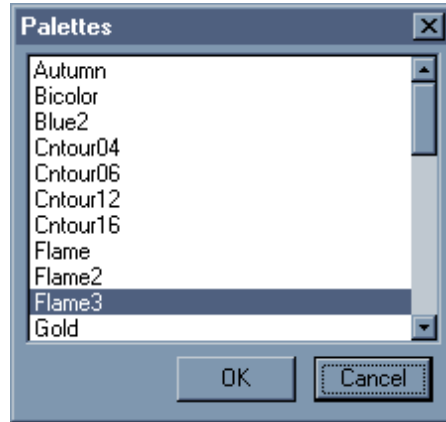


Figure 6.4
Palettes Window

Select a palette and click **OK**. The color palette of the the image you have selected will change to the new palette.

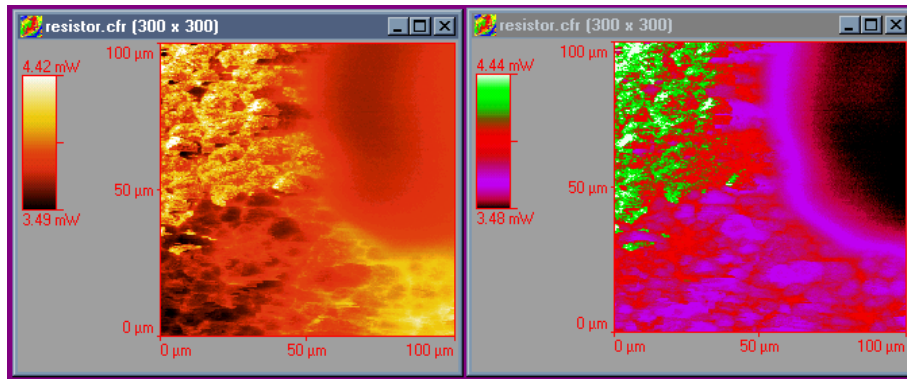


Figure 6.5 Thermal Conductivity Data
Displayed with Two Different Palettes

Leveling

Samples are never mounted without some degree of tilt being present, due to either the sample surface or the mounting of the sample. **Leveling** is a collection of mathematical routines by which you can automatically or manually reduce the sample tilt. Removing sample tilt is important if you wish to make accurate Z-height measurements across a sample.

In some scans, the Z-range of the sample tilt may be close to or greater than the Z-range of the topography of interest. When this occurs, the color scale is distributed primarily over the Z-height of the tilt, leaving relatively little color resolution to resolve the topographical features of the surface. Leveling corrects this problem.

First-order leveling levels the image to a plane, as illustrated in Figure 6.6. For many images, simple one-step leveling can be applied. This process applies a first-order leveling of the entire sample to one plane, using a least-squares algorithm to fit the image to a plane and then subtracting the plane from the image. This “quick leveling” can be applied from the tool bar using the **Quick**

Leveling button .

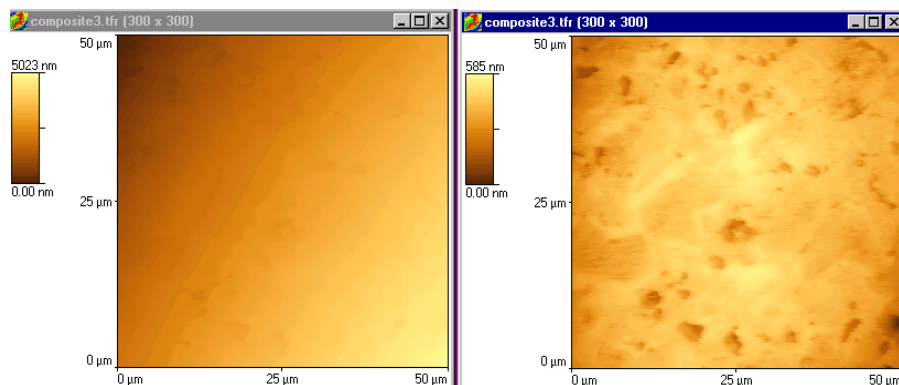


Figure 6.6
Topography Images Before (left) and After (right) Leveling

Second-order or higher leveling corrects for curvature, or bow, in the sample by fitting the data to a curve. To perform more advanced leveling, select **Analysis** on the μ TALab **Main** window's menu bar, and then select **Leveling** from the dropdown menu; the **Leveling** window appears, as shown in Figure 6.7 on the next page.

The **Leveling** window (shown to the right) contains two options, **Auto Leveling** and **Advanced Leveling**. With the **Auto Leveling** function, you can select the type and complexity of leveling to apply to your entire image. The **Advanced Leveling** option allows you to first specify the areas and Z-ranges of your image that are to be leveled. These two options are explained in detail in the following pages.

Auto Leveling

Selecting the **Auto Leveling** option enables you to perform leveling in one of four modes, Horizontal, Vertical, 2D, and 3 Point. In each mode, leveling is performed on the basis of various mathematical models that handle progressively more complex image curvatures and tilts. The process fits each line of the image to a curve. The fitted line is then used to level the line.

On the **Order** field on the Leveling window, you can select from up to six calculations to be used for the leveling. To choose the order, click on the button for the **Order** field, and select an order from the dropdown list. The contents of this list vary according to the mode of leveling you have chosen.

Horizontal leveling applies the leveling function from the top down on the basis of the value selected in the **Order** field on the Leveling window. To use this function, click on the **Horizontal** button on the **Leveling** window.

Vertical leveling applies the Leveling function from side-to-side on the basis of the value selected in the **Order** field. To use this function, click on the **Vertical** button on the **Leveling** window.

Horizontal and vertical leveling can be applied in any one of six orders of calculation. Table 6.3 (on the next page) shows the equations that represent the fitted lines, according to the order of leveling selected, for **Horizontal** and **Vertical** leveling.

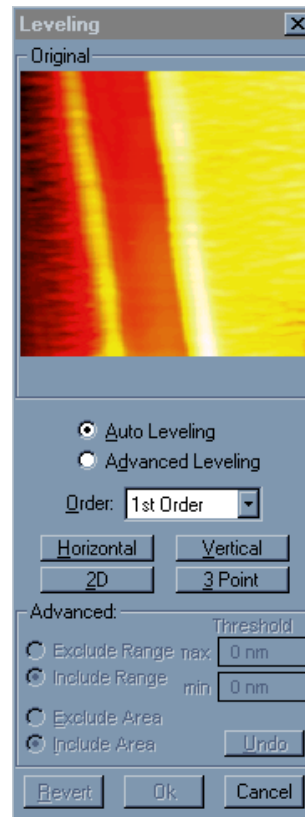


Figure 6.7
Leveling Window

Table 6.3 Equations Used in Horizontal and Vertical Leveling

Order	Equation
1st	$z = ax + b$
2nd	$z = ax^2 + bx + c$
3rd	$z = ax^3 + bx^2 + cx + d$
4th	$z = ax^4 + bx^3 + cx^2 + dx + e$
5th	$z = ax^5 + bx^4 + cx^3 + dx^2 + ex + f$
6th	$z = ax^6 + bx^5 + cx^4 + dx^3 + ex^2 + fx + g$

2D (two-dimensional) or plane leveling levels the data in both the x and y directions. 1st-order through 3rd-order calculations are possible, on the basis of the equations shown in Table 6.4.

Table 6.4
Equations Used in 2D Leveling

Order	Equation
1st	$z = ax + by + c$
2nd	$z = ax^2 + by^2 + cxy + dx + ey + f$
3rd	$z = ax^3 + by^3 + cx^2y + dxy^2 + exy + fx + gy + h$

NOTE: With 2D mode, 1st-order leveling is equivalent to pressing the **Quick Leveling** button on the tool bar.

3 Point leveling allows you to choose three points anywhere on the image that will be used to designate the plane to which the image will be leveled. 3-point leveling is especially good for leveling images in which “steps” occur.

To use this function, click on the **3 Point** button on the **Leveling** window. Then click on the three points on the image itself (not the image displayed in the **Leveling** window) where you want to define the level plane. After you select the third point, leveling is applied.

Advanced Leveling

Advanced Leveling allows you to include or exclude:

- A specified Z-range from the leveling process
- One or more regions within the image

As you define the area or Z-range to be leveled, the selected portions of the scan are highlighted in green on your selected image.

To perform this function, click on the **Advanced Leveling** button on the **Leveling** window. The **Advanced Leveling** subpanel at the bottom will become active; that is, the fields that were grayed out before you selected the function (as shown in Figure 6.7) are now displayed clearly, as shown in Figure 6.8 to the right.

The Z-range fields in this subpanel, **Exclude Range** and **Include Range**, allow you to specify what Z-range within your image will be used for the leveling calculations. You can specify by either (1) excluding a given range from the calculations or (2) including a given range. A certain amount of trial and error may be necessary to define the appropriate range.

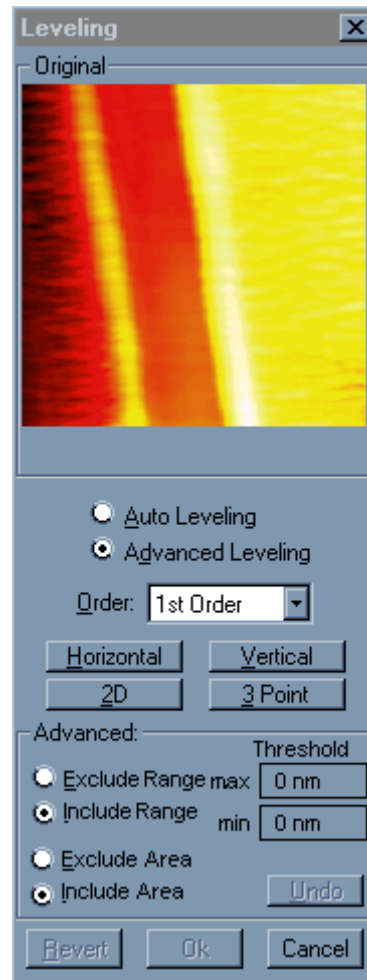


Figure 6.8
Leveling Window with
Advanced Leveling Subpanel
Active

To use this function:

1. Click on either the **Exclude Range** or **Include Range** radio button.
2. Enter the **Min** (minimum) and **Max** (maximum) values for the range in the **Threshold** fields.
3. The area to be leveled will be highlighted in green. Make adjustments to the **Max** and **Min** threshold values, as required.

Area leveling allows you to manually draw a box over any region(s) that you want included or excluded from the leveling process.



To perform this function, use the procedure on the next page.

1. Click on the **Exclude Area** or **Include Area** option.
2. Left-click and drag the mouse to draw a box (or boxes) on the scan image (not the dialog preview image).
3. Right-click to define the area. The area to be leveled will be highlighted in green. Use the mouse to change the area(s) as required.

Once you have selected and defined an area or a Z-range for leveling, select the leveling mode (**Horizontal**, **Vertical**, or **2D**; **3 Point** is not available in **Advanced Leveling**) for your image, as explained under "Auto Leveling."

Shading

Shading can dramatically enhance your images by creating highlights and shadows. You can choose to shade your image with a simulated light source placed to either left or right of the image. The light source is fixed at an angle of 45 degrees from the horizontal.

To apply Shading to your selected image, click on either the **Left Shading** button, , or the **Right Shading** button, , from the tool bar.

It is important to remember that when an image is shaded, the color scale can no longer be used to represent Z-height. Instead, it is used exclusively to represent topographic slope, as depicted by the angle of the light source.

Figure 6.9 shows examples of an original image and the image with shading applied. Note the change in Z axis units on the shaded image.

NOTE: If Z-height is critical information for your application, retain a copy of the original unshaded data.

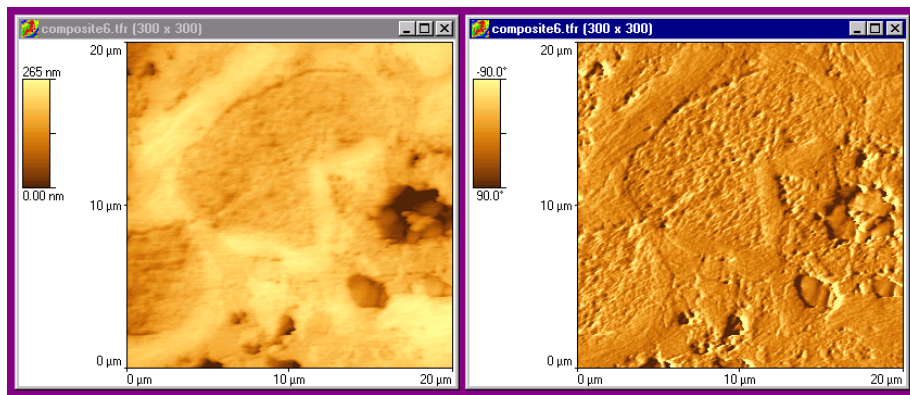


Figure 6.9
Original (left) and
Shaded (right) Topographic Images

Using the Color Editor

As discussed in the previous section, images can be greatly enhanced with the use of shading, but the Z-height information is lost in the shading process. The 2D color functions in the μ TALab software allow you to determine how much of an image's color contrast information will be used to depict the height information and how much will be used to depict feature slopes.

The two color mapping options that can be applied are **Pseudo 2D** and **True 2D**. The different types of 2D processing arise from the differences in the number of colors that various computer graphics cards can display:

- **Pseudo 2D** uses 230 of the 256 colors available from an 8-bit graphics card.
- **True 2D** is available if your computer display adapter is using 16-bit or 24-bit color.

You can set (1) the number of colors used for height and slope and (2) the sharpness used in both of these options on the **2D Color Editor** window, shown in Figure 6.10.

NOTE: Your image must be shaded before you can apply the **Pseudo 2D** or **True 2D** color function.

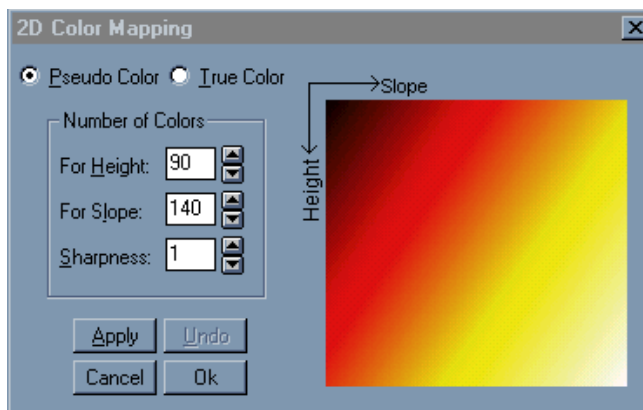


Figure 6.10
2D Color Editor Window in Pseudo Color Mode

To access this window:

1. Select **Analysis** from the μ TALab™ **Main** window.
2. From the dropdown menu that appears, select **True 2D Color**.
3. From the dropdown list that then appears, select **Edit 2D Color**. The **2D Color Editor** window appears.

The **2D Color Editor** allows you to divide the color palette between height and slope information, thereby providing enhancement of shading effects with the height information of a topography-based color scale.

With both **True 2D** and **Pseudo 2D** color modes, results vary dramatically according to (1) the parameters set in the **2D Color Editor** window and (2) the data of a particular image. Experimentation is the only sure way to determine how this function will affect the visualization of your data (images).

Using Pseudo 2D Color

You must use **Pseudo 2D Color** for manual slope/height/sharpness mapping, if you are working in 8-bit (256-color) mode. This function is also available, however, if you are working in 16-bit or 24-bit color mode.

To apply **Pseudo 2D Color** to your image, select **Analysis** from the μ TALab™ **Main** window menu bar. Then select **Pseudo 2D Color** from the dropdown menu that appears.

You can edit the parameters used in **Pseudo 2D Color** editing on the **2D Color Editor** window, as shown in Figure 6.10.

The 2D Color Editor lets you define the distribution of 230 colors (out of 256) between the **Height** and **Slope**.

The **Sharpness** control adjusts the surface reflectivity (shininess) of the image. Higher sharpness values create a more reflective image; lower sharpness values creates a less reflective image. Most color palettes and images are optimized using the default setting of 1 for **Sharpness**.

All values for parameters that you choose on the **2D Color Editor** window are retained for subsequent applications of the **Display / 2D Color / Pseudo 2D** color function, which you can access from the **Analysis** menu on the μ TALab **Main** window's menu bar.

The objective of adjusting these settings is to create a combination that improves the color mapping as it applies to the features of interest in your image. You will have to experiment with the settings to determine the optimum values for your images. As you adjust the settings, you can temporarily see the effect on your image by using the **Apply** button.

The image on the right of the window illustrates the distribution of colors used for height and slope and changes as you change the values in the **Height** and **Slope** fields.

You can reverse all changes made on the **2D Color Editor** window by pressing the **Undo** button.

NOTE: After applying **Pseudo 2D Color** to your image, adjust the contrast and brightness settings to further enhance it.

Using True 2D Color

The **True 2D Color** function evenly distributes the number of colors (for 16-bit color = 65,000; 24-bit color = 16,000,000) between the **Height** and the **Slope**.

To apply **True 2D Color** to your image, select **Analysis** from the μ TALab™ **Main** window menu bar. Then select **True 2D Color** from the dropdown menu that appears.

You can edit the parameters used in **True 2D Color** editing by opening the **2D Color Editor** window, as shown in Figure 6.10, and selecting the **True Color** option.

In **True 2D Color** mode, the **2D Color Mapping** editing consists of adjusting only the image's **Sharpness** value. The **Sharpness** control varies the distribution of color intensity between height data and shading data.

The **Sharpness** control adjusts the surface reflectivity (shininess) of the image. Higher sharpness values create a more reflective image. Lower sharpness values create a less reflective image. Most color palettes and images are optimized using the default setting of 1.

All values for parameters that you choose on the **2D Color Editor** window are retained for subsequent applications of the **Display/2DColor/True 2D** color function, which you can access from the **Analysis** menu on the μ TALab **Main** window's menu bar.

The objective of adjusting these settings is to create a combination that improves the color mapping as it applies to the features of interest in your image. You will have to experiment with the settings to determine the optimum values for your images. As you adjust the settings, you can temporarily see the effect on your image by using the **Apply** button.

You can reverse all changes made on the **2D Color Editor** window by pressing the **Undo** button.

Figure 6.11 shows an image before and after **2D True Color** mapping has been applied.

NOTE: After applying **True 2D Color** to your image, adjust the contrast and brightness settings to further enhance it.

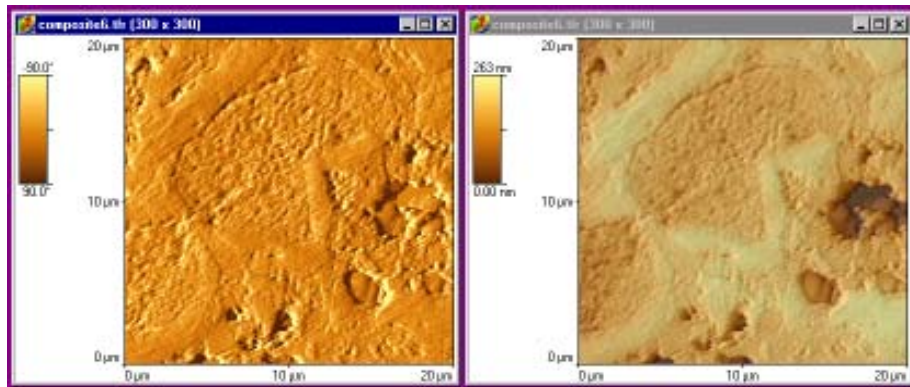


Figure 6.11
Shaded (left) and True 2D Color (right) Images

Data Histogram: Color Distribution

The color histogram function of the μ TALab software allows you to spread most of your palette's color distribution over the Z-range of interest in your data. By default, the color palette is evenly distributed across the entire Z-range. For many types of scans, this is acceptable. However, on some scans—a planar surface containing a deep hole, for example—much of the color distribution is taken up by the walls of the hole, making it difficult to resolve fine details of the surface.

After you select the image you wish to use the histogram function on, access the function from the μ TALab **Main** window in two ways:

- Select **Analysis** from the μ TALab **Main** window menu bar, and then select **Histogram** from the dropdown menu that appears.
- Press the **Histogram** button on the tool bar.

The **Histogram** window appears, as shown in Figure 6.12. The top of the window shows a copy of your original selected image; the image window on the screen outside the **Histogram** window will show results of changes you make with the tools available on the **Histogram** window, allowing you to apply them to the original image or to cancel or revise them before applying them.

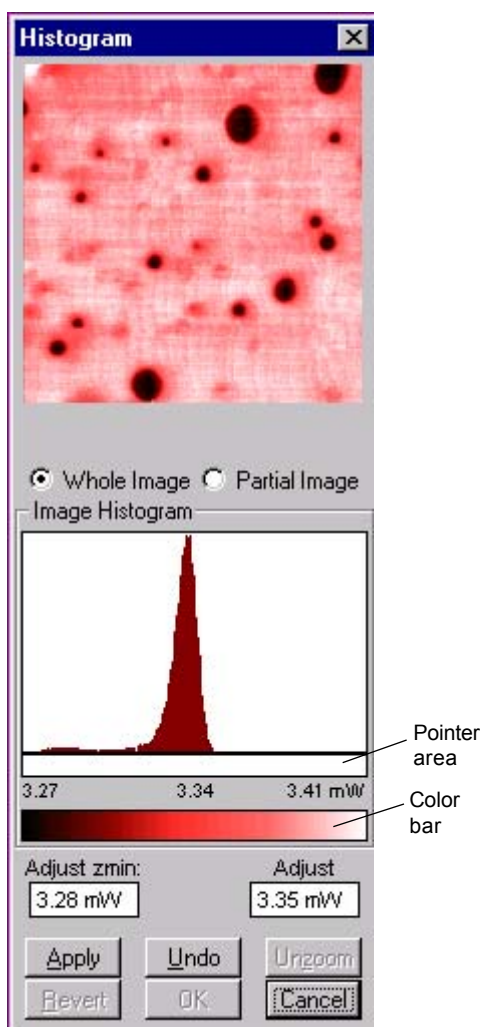


Figure 6.12
Histogram Window

Underneath the image is a panel with a data histogram of frequency vs. height for the selected image. The color bar on the **Histogram** window shows the color distribution of the data points displayed, just like the vertical bar on any image window.

The next section explains how to apply the color histogram using the radio buttons and other buttons shown on this window.

Applying the Histogram

You can redistribute the color histogram over the entire image, or just a portion of the image, using

one of three options available on the Histogram window to:

- Redistribute color over the whole image
- Redistribute color over a portion of the image (partial image)
- Redistribute color over a portion of the Z-range

Figure 6.13 is a thermal conductivity image of a polymer blend in which the color distribution is uneven.

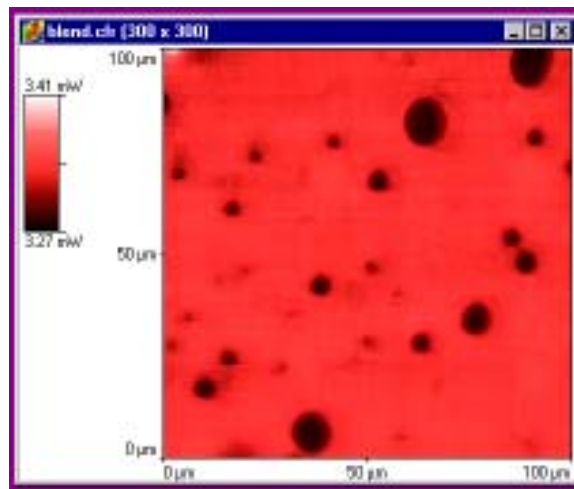


Figure 6.13
Thermal Conductivity Image
Before Application of Histogram

To redistribute the color palette over the entire image:

1. Click on the **Whole Image** check box under the selected image.
2. Click and hold the cursor in the **Pointer** area below the histogram, at the left edge of the data you want to highlight. A pointer appears under the histogram (see Figure 6.14).

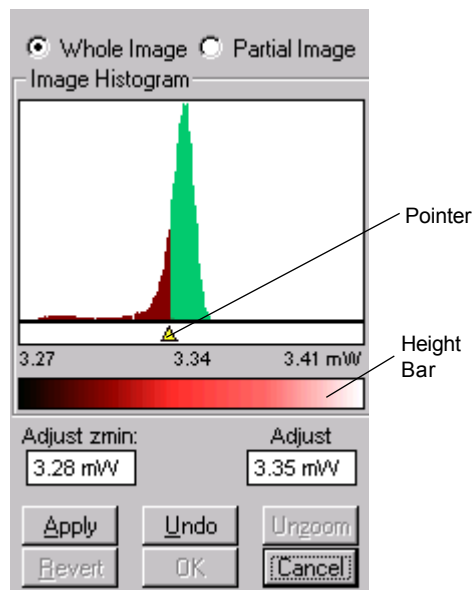


Figure 6.14
Highlighting the Data Range

3. Drag the pointer to the right until you have highlighted the appropriate data range (as shown in Figure 6.14), and release the cursor. The values in the Adjust zmin (Z minimum) and Adjust (Z maximum) fields change to match the new limits you have chosen.
4. To delete the change you have made, click on the **Undo** button, and try again.
5. To see the effect of the color palette distributed over the range you have selected, click the **Apply** button. The changes will appear on the original image (the image on the **Histogram** window does not change).
6. To delete the change and return to the original image, click on the **Revert** button.

Figure 6.15 shows the thermal conductivity image from Figure 6.14 with the color redistributed over the entire image.

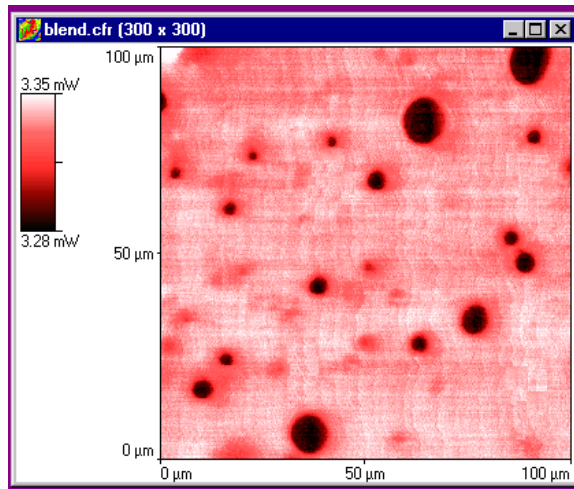


Figure 6.15 Color Palette
Redistributed over Whole Image

To redistribute color over a portion of the image:

1. Click on the **Partial Image** check box.

A cross-hair cursor appears on the full display of your image (not on the preview image on the **Histogram** window).

2. To select the area of this image for color redistribution, left-click and drag the cross-hair cursor to draw a box around the area of interest. Release the mouse to finish drawing the box.

Once you have drawn the box, you can move it around the image.

3. Right-click when you are happy with the placement of the box.
4. Click and hold the cursor in the Height bar on the **Histogram** window, to the left edge of the data you want to highlight. A point appears below the histogram (see Figure 6.14).
5. Drag to the right until you have highlighted the appropriate data range, and release mouse (see Figure 6.14).

6. Click on the **Apply** button.

The image show at the top of the **Histogram** window will change to show the color palette distributed over your selected range and image portion. Figure 6.16 shows an image before and after color distribution has been applied.

You can go back to the original image, at any time, by clicking on the **Revert** button.

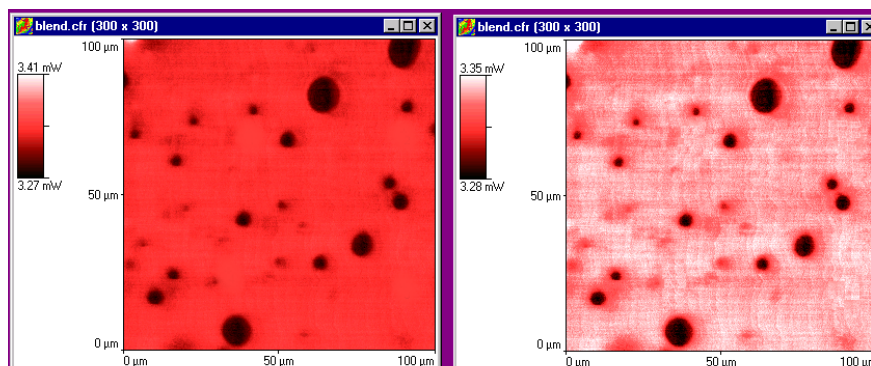


Figure 6.16
Before (left) and After (right) Color Distribution

You can zoom in on a portion of the Z-range by defining the upper and lower limits of the range, as follows:

1. Enter the minimum mV value for the Z-range in the **Adjust zmin** field on the **Histogram** window.
2. Enter the maximum mV value for the Z-range in the **Adjust** field on the **Histogram** window.
3. Click on the **Apply** button. The software zooms in on the defined portion of the histogram and redistributes the color palette over that range.

After applying the function, you can click on the **Unzoom** button to return to the original full histogram color distribution.

Using the Image Processing Tools Collectively

You should use the image enhancement and processing tools collectively to best display your data. In a typical image processing session, you would use the tools in the following order:

1. Remove z tilt by using **Leveling**.
2. Ensure that the palette has optimum distribution by using the **Histogram** window.
3. Shade your image to enhance any topographic features.
4. Use **True 2D Color** to create an enhanced image with accurate z data. For presentations and reports, **3D View** can be very effective.

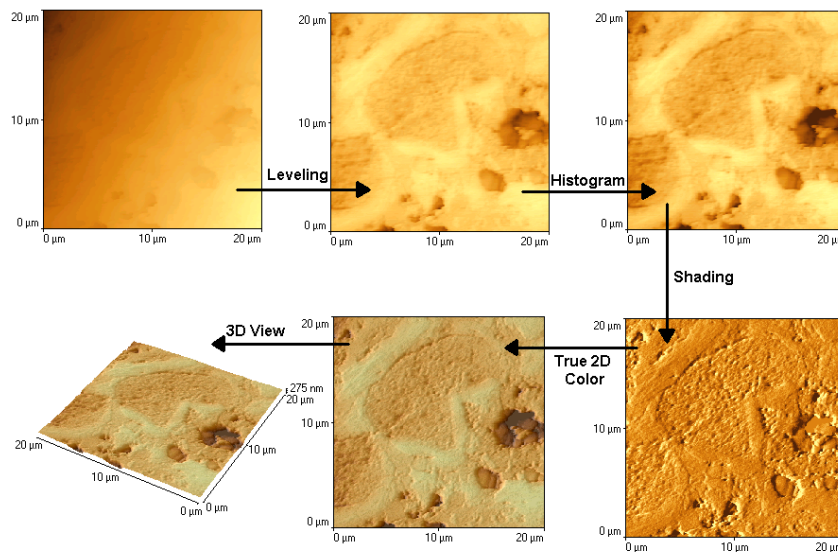



Figure 6.17
Summary of Typical Image Processing Steps

Line Measure

The **Line Measure** function allows you to make a cross-sectional measurement along a user-defined line on the selected image. You can then obtain x, y, and z data for any position along a line's profile, together with point-to-point distance and angle information.

You can access this function in one of two ways:

- Select **Analysis** from the μ TALab **Main** window menu bar, and then select **Line Measure** from the dropdown menu that appears.
- Press the **Line Measure** button, , on the tool bar.

The **Line Measure** window appears, as shown in Figure 6.18.

The top of the **Line Measure** window displays summary file information, which consists of:

- Z max
- Z max
- Scan Range
- Resolution

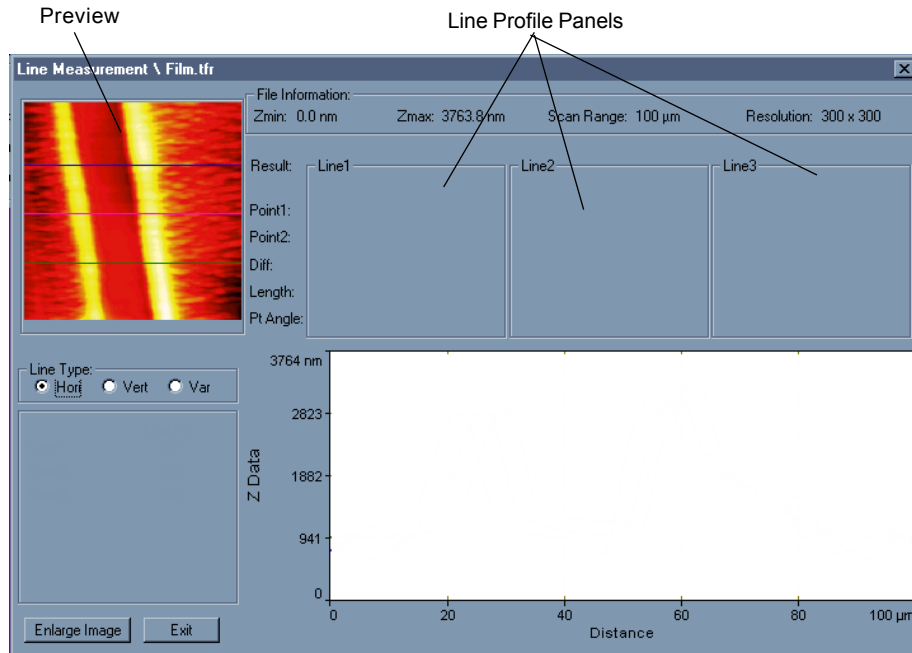


Figure 6.18
Line Measure Window

Drawing Lines

As you move the mouse over the **Preview** panel in the top left corner of the **Line Measure** window, the mouse pointer changes to a line marker. You can use the marker to draw up to three lines of the following type on your image, as described on the following pages:

- Horizontal
- Vertical
- Variable (*i.e.*, lines at any angle and of any length)

NOTE: You may find accurate positioning of lines easier if you work on a larger view of the image. To obtain a larger view, click on the **Enlarge Image** button at the bottom of the **Line Measure** window.

Horizontal Lines

To put a horizontal line on your image:

1. Click on the **Hori** (Horizontal) radio button on the **Line Measure** window.
2. Place the cursor (which is now a line marker) at the spot on the image in the **Preview** panel where you want a horizontal line.
3. Click on the location.

A horizontal line through the line marker appears on your image. Note that the scan line number is displayed underneath the **Preview** panel.

Figure 6.19 shows an image with three horizontal lines drawn on it.

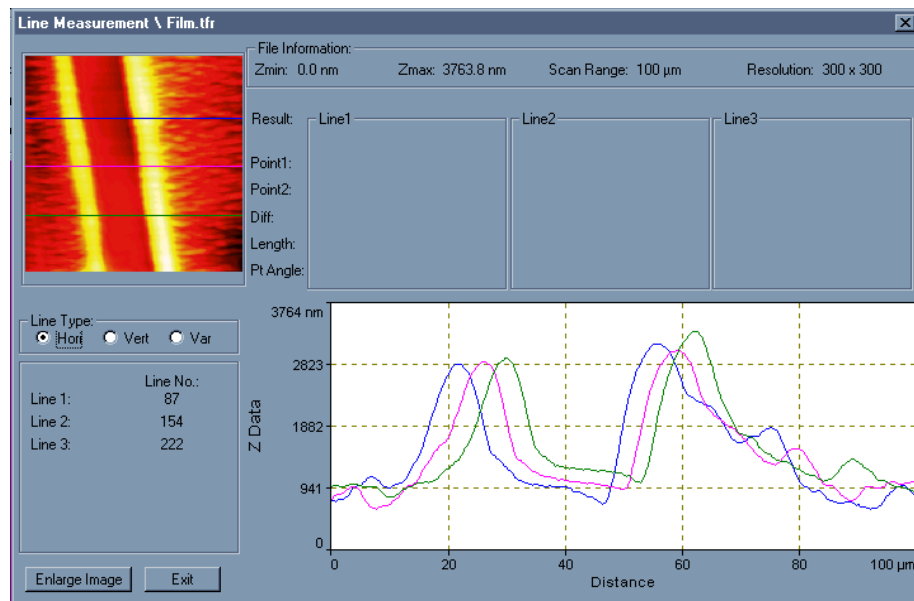


Figure 6.19
Line Measure Window
with Three Horizontal Lines Drawn

Vertical Lines

To put a vertical line on your image:

1. Click on the **Vert** (Vertical) radio button on the **Line Measure** window.
2. Place your cursor (which is now a line marker) at the location the image in the **Preview** panel where you want a vertical line.
3. Click on the location.

A vertical line through the line marker appears on your image. Note that the scan line number is displayed underneath the **Preview** panel.

Variable Lines

To place a variable line, that is, a line of any length and any angle, on your image:

1. Click on the **Var** (Variable) radio button on the **Line Measure** window.
2. Place your cursor at the location on the image in the **Preview** panel where you want the line to begin.
3. Click and drag on the image to draw a line of any angle or length, and release the mouse button.
4. The line appears on the image. You can now move the line across the image by moving it with the mouse.
5. Once you have accurately positioned the line, right click the mouse to place the line.

Note that the angles between variable lines are displayed below the **Preview** panel.

As each line is added to the image, the relevant line profile is added to the **Line Profile** panel.

Making Measurements

You can make measurements on the lines you have drawn on your image. To do so, follow this procedure.

NOTE: You may find accurate positioning of lines easier if you work on a larger view of the image. To obtain a larger view, click on the **Enlarge Image** button at the bottom of the **Line Measure** window.

1. Click in the **Line Profile** panel on the **Line Measure** window. The mouse cursor changes to a vertical line marker.
2. Position the vertical line marker on the first point you wish to measure, and left-click the mouse.
3. Position the vertical cursor at the second point you wish to measure, and left-click the mouse.

As you move and select points with the vertical marker, note that each point selected along the profile line is also shown as an "X" marker on the **Preview** panel. See the example in Figure 6.20.

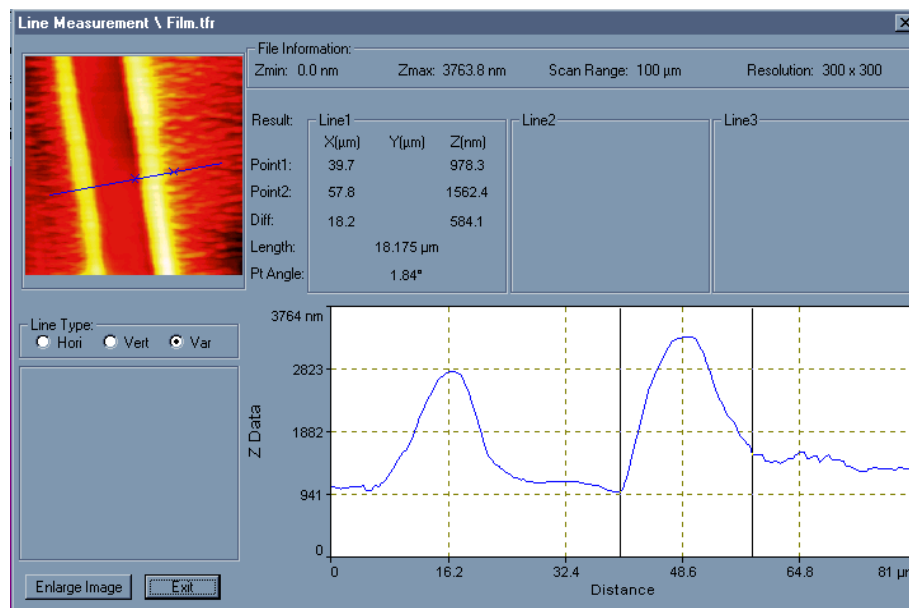


Figure 6.20
Variable Line Profile with Measured Features

The following information is displayed for each analysis line profile:

- x, y, and z locations for both points
- The differences in x, y, and z between the two points
- The line length between two points
- The angle of the slope between the two points


Chapter 7

Using the Print Editor

Introduction

The **Print Editor** in the μ TALab software is a powerful function that gives you a great deal of flexibility in optimizing and printing the data you obtain with your μ TA 2990. The **Print Editor** functions as a free-form presentation generation tool, allowing you to manipulate images, signal windows, and control panels together with text, arrows, and other graphic objects.

You can access the **Print Editor** in either of two ways:

- Select **File** from the μ TALab **Main** window menu bar, and then select **Print Editor** from the dropdown menu that appears.
- Click on the **Print** button, , on the μ TALab **Main** window tool bar.

The **Print Editor** window appears. Figure 7.1 shows the menu bar and tool bar that appear across the top of the **Print Editor** window.

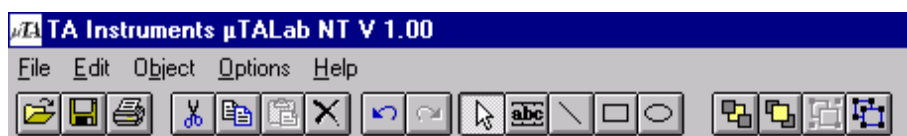


Figure 7.1
Menu Bar and Tool Bar of Print Editor Window

Initially, the rest of the **Print Editor** window contains elements of the μ TALab software: images, control panels, or signal windows. Exactly what the window displays depends on whether the **Print Edit Full Details** option is selected in the **Preferences** window (which is accessed through the **Options** menu; see Chapter 5).

If the **Full Details** option is selected, all images, control panels, and signal windows are displayed on the **Print Editor** screen; if **Full Details** is not selected, only the images and Localized Thermal Analysis (LTA) graphs are displayed.

You can select any object in the **Print Editor** window by left-clicking with the cursor in the middle of the object. Once you have selected the object, you can drag, stretch, or delete it using standard Windows® NT mouse controls. (Please refer to your Windows NT documentation for complete information on how to select, stretch, move, and delete objects with a mouse.) These standard mouse controls give you great flexibility in optimizing the presentation of your data prior to printing.

The following section details how you can further enhance your data using the text and formatting tools of the **Print Editor**. Many of the commonly used functions are available from both the menu and the tool bar. The discussion follows the organization of the menus. Many of the functions in the **Print Editor** are the same, in part or whole, as functions with the same name in any Windows® applications (Open a file, Print, Save as..., *etc.*). This chapter describes only the μ TALAb-specific parts of a common printer or printing related menu item.

File Menu

Access the **Print Editor File** menu, shown in Figure 7.2, by clicking **File** on the **Print Editor** window menu bar.

Most of the items on this menu are the same as standard Windows® File menu items. They are explained, when necessary, beginning on the next page.



Figure 7.2
Print Editor File Menu



Clicking on **File/Open** or the **File Open** button accesses the **File Open** window. This window allows you to open and/or import graphic files from other programs. Table 7.1 lists the file types or formats (along with their file name extensions) that you can import or open with μ TALab **Print Editor**.

Table 7.1
File Types That Can Be Opened or
Imported with the Print Editor

File Type	File Extension
Windows & OS/2 Bitmap	*.bmp
LEAD	*.cmp
Encapsulated Postscript	*.eps
JPEG	*.jpg
Macintosh Picture	*.pic
Zsoft PC Paintbrush	*.pcx
SUN Raster Image	*.ras
Truevision Targa	*.tga
Tagged Image File	*.tif
Windows MetaFile	*.wmf
WordPerfect Vector	*.wpg

Opening and Importing Scanning Images and Localized Thermal Analysis Data

To create printouts containing images and LTA data, follow this procedure:

1. Format LTA data using the *Thermal Solutions* Universal Analysis program.
2. Save your plot as a bitmap (*.bmp) using the **File/Export Plot** function of the Universal Analysis program.
3. Import the bitmap created by Universal Analysis into the **Print Editor** using the **File/Open** function.

Save As

Clicking on **File/Save as** or the **Save as** tool bar button accesses the **Save File** window, shown in Figure 7.3.

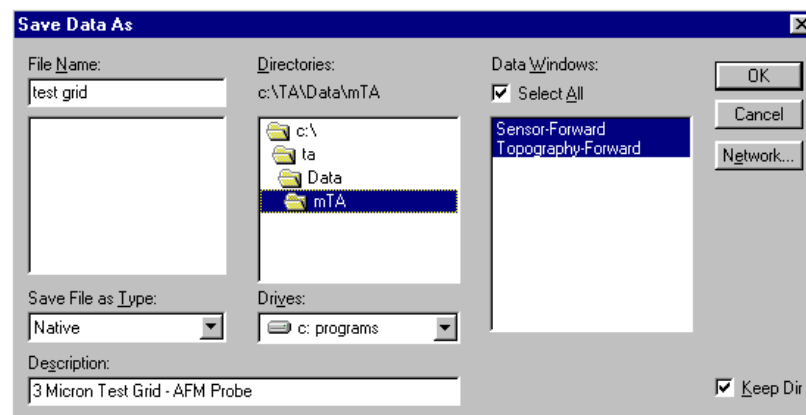


Figure 7.3
Save As Window

You can this window to save a selected item in any of the graphics file formats supported by the μ TALab Software. (If none or all of the items on the active screen have been selected, all of the items will be saved.) Table 7.2 lists the graphic file formats that are supported by the μ TALab Software.

Table 7.2
Supported Graphic Formats

Supported Format	File Extension
Encapsulated Postscript	*.eps
Macintosh PICT8	*.pct
Zsoft PC Paintbrush	*.pcx
Tagged Image File	*.tif
WordPerfect Vector	*.wpg

Convert

Clicking on **File/Convert** opens the **Convert** windows, which allow you to change files from one supported file format to another.

The first window that appears, The File Convert—Source window (see Figure 7.4), prompts you for the source file.

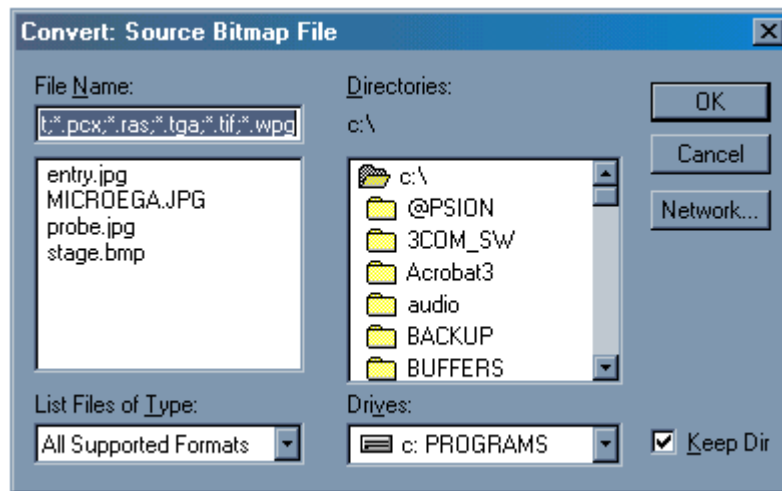


Figure 7.4
File Convert—Source Window

Use the scrollable lists to specify the location and name of the file, and press **OK**.

The **File Convert—Destination** window appears, as shown in Figure 7.5.

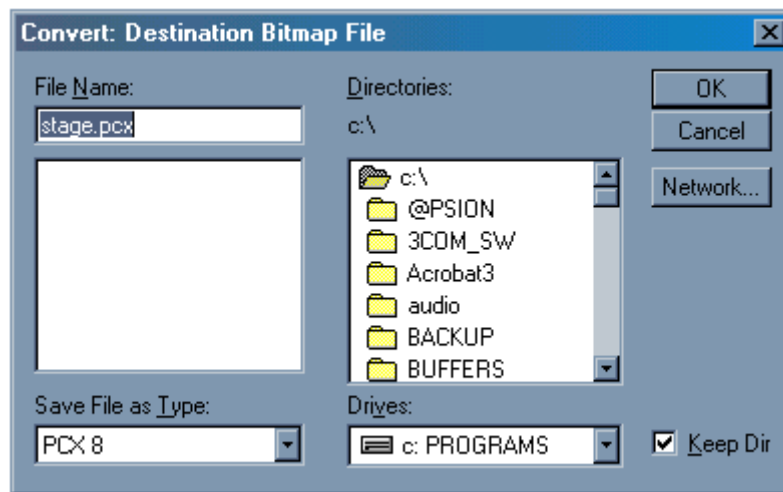


Figure 7.5
File Convert—Destination Window

This window prompts you for the destination file name and file type:

1. Type in the destination file name.
2. Choose the destination file type.
3. Press **OK**.

Edit Exit



Selecting **Edit Exit** on the **File** menu closes the **Print Editor** and returns you to the point in μ TALab Software at which you opened the **Print Editor**.

The Edit Menu

Access the **Edit** menu, shown in Figure 7.6, by clicking **File** on the **Print Editor** window menu bar.

Many of the items on this menu are found on any standard Windows® Edit menu (Undo, Redo, Cut, Paste, Delete, Select All) and are not described here.

The last item on the **Edit** menu, however, accesses the **Insert** submenu, which you can use to customize your μ TALab images and data by adding any of the following items to it:

- Text
- Line
- Rectangle
- Ellipse

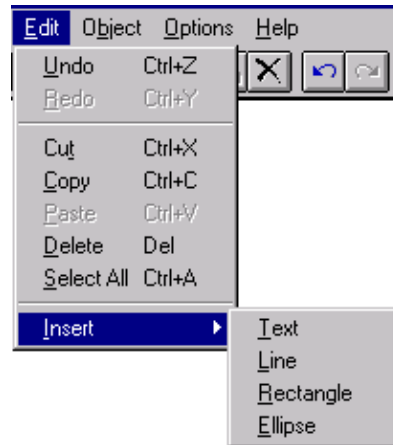


Figure 7.6
Print Editor Edit Menu

Adding Text to Your Image

Selecting **Insert/Text** on the **Edit** menu or clicking on the **Insert/Text** button



on the μ TALab **Print Editor** window menu bar enables you to add text to your image.

With the cursor, click and drag in a white space area of your image to create a text field. The **Text Properties** window appears, as shown in Figure 7.7.

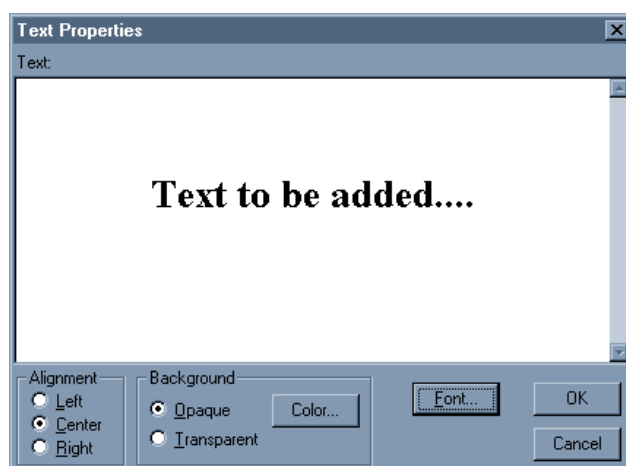


Figure 7.7
Text Properties Window

1. Type the text you wish to add.
2. Use the buttons in the **Alignment** subpanel to choose the alignment of your text.
3. Specify in the **Background** subpanel whether the background for your text should be **Transparent** (whatever is behind your text will show behind it) or **Opaque**.

The default background color is black. To choose another color, press the **Color** button. Choose from the colors panel that appears when you press the **Color** button.

4. Choose a font or typeface for your text by pressing the **Font** button. A **Font Properties** window appears, as shown in Figure 7.8.

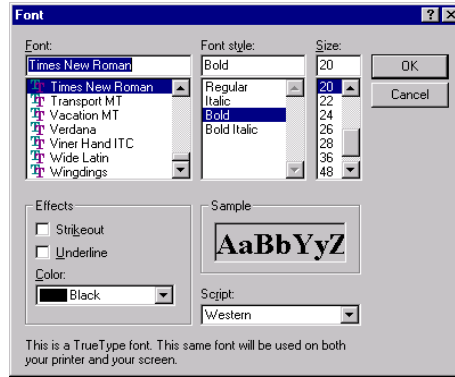


Figure 7.8
Font Properties Window

This window contains buttons and fields that allow you to change the following characteristics of the text you are adding:

- Font (typeface) and script (English, Cyrillic, Greek, etc.)
- Type size and style (normal, bold, italic, etc).

NOTE: These fonts are provided by third party vendors and are not supplied by TA Instruments, Thermo Microscopes, or IBM.

Press **OK** to apply the choices you have made.

Double-clicking on any text already added with the **Print Editor** also opens the **Text Properties** window so that you can change or add to the text.

Adding a Line to Your Image

Selecting **Insert/Line** on the **Edit** menu or clicking on the **Draw Line** button,



on the μ TALab **Print Editor** window menu bar enables you to add a line to your image.

Click and drag the cursor to draw a line and add it to the screen.

Double-click on any line that you have added to your image to open the **Line Properties** window, as shown in Figure 7.9.

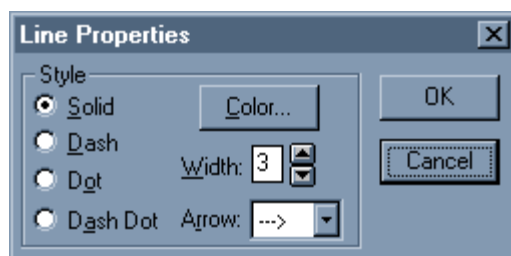



Figure 7.9
Line Properties Window

Use the buttons on this window to customize your line by choosing:

- **Style:** solid, dashed, dotted, or dashed-dotted.
 - **Color:** Choose from the colors panel that appears when you press the **Color** button.
 - **Width:** Choose from the scrollable list, which specifies the relative line width.
 - Add an **Arrow:** Choose from the scrollable list.
2. Press **OK** when finished.

Adding a Rectangle to Your Image

Selecting **Insert/Rectangle** on the **Edit** menu or clicking on the **Draw Rectangle** button, , on the μ TALab **Print Editor** window menu bar enables you to add a rectangle to your image.

Click and drag the cursor to create a rectangle and add it to the screen.

Double-click on any rectangle that you have added to your image to open the **Rectangle Properties** window, as shown in Figure 7.10.

Use the buttons on this window to customize the rectangle's frame (outside line) and fill (inside area).

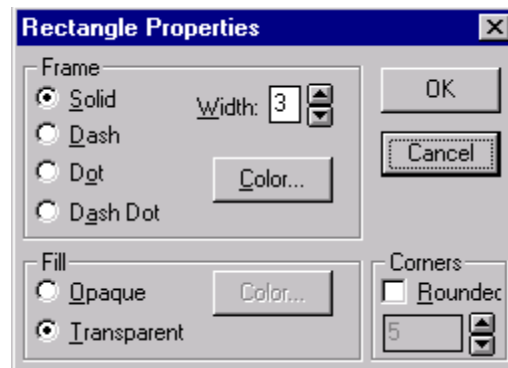


Figure 7.10
Rectangle Properties Window

Press **OK** when finished.

Use the **Frame** subpanel on this window to specify the properties of the line that surrounds or frames the rectangle you have drawn:

- **Style:** solid, dashed, dotted, or dashed-dotted.
- **Color:** Choose from the colors panel that appears when you press the **Color** button.
- **Width:** Choose from the scrollable list, which specifies the relative line width.

The default **Corners** style is square. To select rounded corners, check the **Rounded** box.


Use the **Fill** subpanel on this window to specify the properties of the inside or fill of the rectangle you have drawn:

The default setting for the **Fill** of a rectangle is for it to be transparent (the frame is shown but the image behind it shows inside the frame).

To fill in the rectangle, click on the **Opaque** radio button. The default fill is black.

When the **Opaque** radio button is selected, the **Color** button on this panel becomes active. To change the color of the **Fill**, click on the **Color** button. Choose from the colors panel that appears when you press the **Color** button.

Adding an Ellipse to Your Image

Selecting **Insert/Ellipse** on the **Edit** menu or clicking on the **Draw Ellipse** button, , on the μ TALab **Print Editor** window menu bar enables you to add an ellipse to your image.

Click and drag the cursor to create an ellipse and add it to the screen.

Double-click on an ellipse that you have added to your image to open the **Ellipse Properties** window, as shown in Figure 7.11.

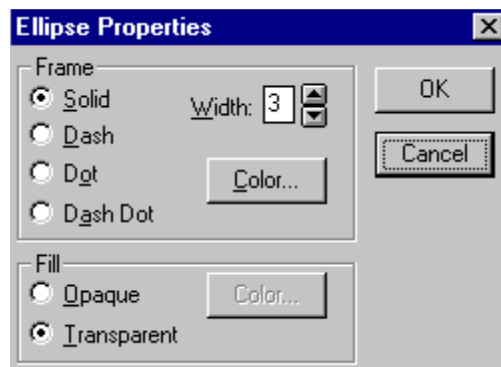


Figure 7.11
Ellipse Properties Window

Use the buttons on this window to customize the ellipse's frame (outside line) and fill (inside area) as specified on the following page.

Press **OK** when finished.

The **Frame** subpanel on the **Ellipse Properties** window allows you to specify the properties of the line that surrounds or frames the ellipse you have drawn:

- Click a radio button to specify the type of line (solid, dashed, dotted, or dashed-dotted).
- Choose the relative width of the frame using the **Width** scroll buttons.
- Choose a color (other than black, which is the default color) for your frame by clicking on the **Color** button.

Use the **Fill** subpanel on this window to specify the properties of the inside or fill of the ellipse you have drawn:

The default setting for the **Fill** of an ellipse is for it to be transparent (the frame is shown but the image behind it shows inside the frame).

To fill in the ellipse, click on the **Opaque** radio button. The default fill is black.

When the **Opaque** radio button is selected, the **Color** button on this panel becomes active. To change the color of the **Fill**, click on the **Color** button. Choose from the colors panel that appears when you press the **Color** button.

The Object Menu

Access the **Object** menu, shown in Figure 7.12, by clicking **Object** on the **Print Editor** window menu bar.

Many of the items on this menu are found on any standard Windows® **Object** menu that allow you to change the relative position of objects on the screen (Move to Front, Move to Back, Move Forward, Move Backward) and are not described here.

The remaining items on the **Object** menu, however, have specific functions that you can use to customize your μ TALab images and data:

- Properties
- Group
- Ungroup

To use these functions, you must have selected or highlighted an image or a part of an image on the screen that you wish to change.



Figure 7.12
Print Editor Object Menu

Viewing or Changing an Object's Properties

Clicking on the **Properties** option on the **Object** menu allows you to access the Properties window for your selected object. For example, if you have selected an image, clicking on the **Properties** option opens the **Image Properties** window, as shown in Figure 7.13.

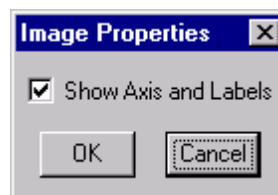


Figure 7.13
Image Properties Window


The default setting on this window is to show the axis and labels on an image.


To remove the axis and labels from an image, click in the **Show Axis and Labels** check box to remove the check; after you click on **OK**, the axis and labels are removed from the image.

To restore the axis and labels on an image, open the **Image Properties** window and click in the check box to add a check; when you click **OK**, the axis and labels reappear on the image.

Grouping and Ungrouping Objects

Like a similar function in other Windows® programs, The **Group** and **Ungroup** options on the **Print Editor Object** menu are like similar functions in other Windows® programs.

To group two objects, click on the **Group** tool bar button, , on **Print Editor** window or select **Group** from the **Object** menu.

To disconnect or ungroup two or more objects, click on the **Ungroup** tool bar button, , on the **Print Editor** window or select **Ungroup** from the **Object** menu.

The Options Menu

Access the **Options** menu, shown in Figure 7.14, by clicking **Options** on the **Print Editor** window menu bar.

The items on this window enable you to further customize and groom your image and data in preparation for printing. There are no tool bar buttons for these functions.

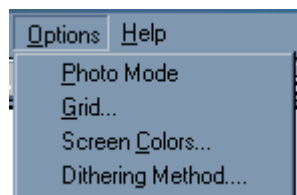


Figure 7.14
Print Editor Options Menu

Using Photo Mode

When you select **Photo Mode**, all extraneous μ TALab information normally displayed on your computer screen (title bar, menu bar, tool bar and scroll bars) disappears, so that you can perform a screen capture or screen photo of the **Print Editor** window using other software.

Clicking anywhere on the screen in **Photo Mode** returns the **Print Editor** to the standard display mode.

Customizing the Print Editor Grid

Selecting **Grid** from the **Options** menu opens the **Screen Edit Options** window, shown in Figure 7.15.

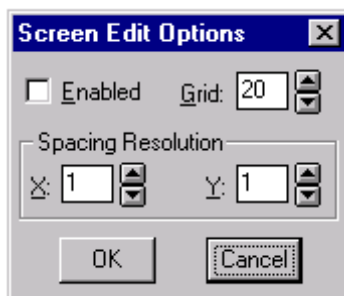


Figure 7.15
Screen Edit Options Window

The **Print Editor Grid** makes some annotations easier. With a grid of squares overlying the image, you can precisely place your annotation on the editor page.

With this window, you can enable and disable the **Print Editor** grid and define the grid's configuration and placement resolution (snap spacing). Use the grid and placement resolution to align and space items on the screen.

To enable or disable the **Print Editor Grid**, click on the **Enabled** check box in the upper right of the window. The name of the box changes to reflect the current status of the **Print Editor** grid.

Choosing Color Settings

Selecting **Color Settings** from the **Options** menu opens the **Color Settings** window, shown in Figure 7.16, which enables you to change the colors used in the **Print Editor**.

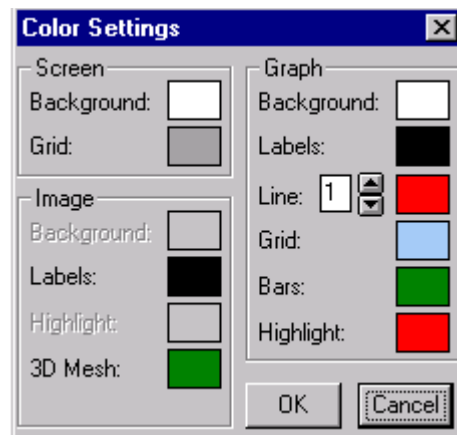


Figure 7.16
Color Settings Window

This window contains several panels with fields showing the colors used in the various displays in the **Print Editor**.

The **Screen** panel allows you to change (1) the colors of the background or “paper” behind your images and the (2) the colors of the **Grid** lines.

You can use the **Image** panel to change the colors of the following areas on the image you have selected to display:

- **Background** (area behind the graph lines)
- **Labels** (axis labels)
- **Highlighted** material
- **3D** (three-dimensional) **mesh**.

With the **Graph** panel, you can change the colors on a graph image:

- **Background** (area behind the graph lines)
- **Labels** (axis labels on a captured image)
- **Line**; select each line with the scrollable list.
- **Grid**
- **Bars**
- **Highlighting**.

Press **OK** when you have made your choices.

Choosing a Dithering Method

Selecting **Dithering Method** from the **Options** menu opens the **Dithering Method** window, shown in Figure 7.17.

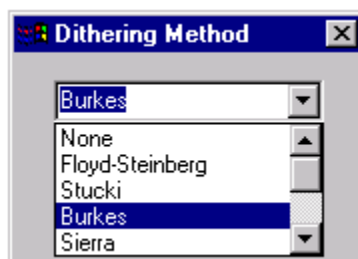


Figure 7.17
Dithering Method Window

From this window, you can select the dithering method used to display images that contain more colors than the screen can display. The default **Dithering Method** for the μ TALab is "None."

Using Universal Analysis with the μ TALab[®] Print Editor

You can combine images and Localized Thermal Analysis (LTA) data formatted within the *Thermal Solutions* Universal Analysis program within the **Print Editor** to create a “composite” output. For example, you can create a plot that contains visualization (images) and characterization (LTA) information on single page.

To do this, you first must optimize and analyze your LTA data with the Universal Analysis program. After you have done so, follow this procedure:

1. Select **File/Export Plot** from within Universal Analysis to open the **Export Plot** window, and make the following selections:
 - a. For “Destination:” select “File,” and enter an appropriate file name.
 - b. For “Format:,” select “Bitmap.”
 - c. For “Size,” select 530 by 720 pixels. This reduces the height and width of the image by approximately 70%.
 - d. Click on “Export” to save your new file.
2. Open the μ TALab Software program, and then open the **Print Editor** (select **File/Print** from the μ TALab **Main** window menu bar or click the **Print** button on the μ TALab **Main** window tool bar), and then open the **Print Editor**.
3. In the **Print Editor**, open the file you saved in Step 1 by using the menu commands **File/Open**.
4. Use the **Print Editor** functions to format your images and Universal Analysis bitmap as needed. Figure 7.18 is an example of a bitmap created in the Universal Analysis program and imported into the μ TALab **Print Editor**.

Appendix A

Sample Preparation

Overview

One of the most important things that you need to do when running experiments on the μ TA 2990 is to store and handle samples in such a way that the surface is not degraded or damaged in any way. Micro-Thermal Analysis 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 make localized thermal analysis (LTA) experiments.

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.

NOTE: Asterisks (*), double asterisks (**), and triple asterisks (***) appear after some of the material names in this discussion. Refer to "Obtaining Materials," on page A-17, for sources of these materials.

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 objective of μ TA[®] sample preparation is to hold the sample 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-piezo positioner.

NOTE: For the μ TA 2990 system, the range of the Z-piezo positioner is 10 μ m.

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 apply 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. To level a sample, follow these suggestions:

- 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.
- 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 nonconductive, make a path from the top of the sample to the sample mount using carbon paint.*
- Wipe the sample surface with water, using a cotton swab. The water will dissipate the charge. Allow for time for the sample to dry before imaging or you may obtain false feedback.



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

Debris on the Sample Surface

The μ TA 2990 instrument acquires images by scanning a thermal probe very close to the sample surface and determining changes in the surface topography and thermal properties. The the surface must remain constant during the analysis. 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 slightly larger and heavier. Later, the small particles may fall off. This results in streaks across the sample surface 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 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. Click the **Clean** button on the **Probe** Panel to remove possible contamination after imaging or LTA experiments.
- 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. Make sure that you clean the probe by clicking on the **Clean** button on the **Probe** Panel between obtaining images.

Soft Samples

Soft samples, such as polymer films, 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

Polymer Films

Use either double-sided tape found in the accessory kit 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 to push the sample down onto the adhesive. Gently push down on the sample in order to firmly attach it, without causing surface roughness to be added to the sample.

Tips for Soft Samples

Some guidelines to keep in mind when using 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 tend to collect condensation, 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.

Polymer Sheets

If you want to analyze large polymer sheets, you can place the μ TA 2990 on top of the sample. However, this positioning can make alignment of the thermal probe over specific features quite difficult. Also, as the Z-motor raises and lowers the head, the μ TA 2990 may tend to slip across the surface of the polymer sheet. For “homogenous” samples, however, in which little information would be obtained from the images, placing the μ TA 2990 on top of the sample is a quick way to obtain LTA data.



IMPORTANT: This technique can damage the surface of many soft samples. The sample can also be pulled taut and placed on tape. This must be done carefully, however, so as not to stretch the sample. Stretching the sample would affect the quality of the images as well as potentially orienting the polymer and changing its crystallinity. An increase in crystallinity might increase transition temperatures as well as peak heights in LTA experiments.

Polymer Pellets

You can use double-sided tape, glue, or modeling clay to easily mount polymer pellets onto sample mounts. However, because it is usually necessary to adjust the back legs of the μ TA 2990 when you move between large and small samples, you may wish to cut a thin slice off the pellet. 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 or modeling clay (as directed earlier in this chapter) onto a sample stud.

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 the TA Instruments μ TA 2990 system. You can image fiber surfaces as well as their cross-sections. Your 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.

TEMPFIX*** or 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 also need to tack the center of the fiber to reduce vibration of the fiber during scanning.

The use of carbon paint* also helps eliminate reflection of the laser from the sample back into the detector, which could interfere with the imaging of highly reflective surfaces.

Tips for Fiber Samples

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 (see the next paragraph for an exception), which can be cleaned with acetone.
- *Do not* clean hair samples. Hair samples are generally imaged to observe surface treatments, and cleaning would change the effect.
- Only the top 1/4 to 1/3 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 Z-piezo positioner displacement to less than 10 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 by only microtoming, 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 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:

Optical Glue* Method

1. Place the sample mount on a hot plate, and warm the mount (stub) so that it is slightly warm to the touch.
- b. 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; otherwise, the solder joint holding the ground wire to the sample mount may melt.
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.

Carbon Paint*/Wafer Chip Method

1. Cut a piece of silicon wafer 1 cm x 1 cm.
2. Put carbon paint on the sample mount and place the wafer chip on the paint so that the paint squeezes out the side.
3. Make a carbon paint path from the top of the wafer chip to the grounding wire to allow static to drain.
4. Put a small amount of the powder sample into a test tube with water or other solvent.
5. Sonicate or stir to make a slurry.
6. 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.
7. Place the slurry sample on the wafer chip, covering the spot in the center.
8. Allow to dry. Capillary surface adhesion will hold the powder particles for scanning.

Tips for Powder Samples

A tip to keep in mind when using powder samples is:

- Mica can be used in place of silicon wafers for the wafer chip method (found on the previous page). Mica has steps that can hold powder particles. This can help in some applications by preventing powder particles from moving. However, because the mica steps may be imaged, using mica can also interfere with imaging, confusing image interpretation. If the objective is to obtain good LTA data, however, the mica step interference should not be a problem.

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, P.O. Box 656, West Chester, PA 19381.

** Superglue is a product of the Loctite Company.

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

Appendix B

Optimizing μ TA[®] Data

Overview

If you have followed the instructions contained in the first three chapters of this manual, you should be familiar with the way the μ TA[™] 2990 system operates. This chapter will give you guidelines that TA Instruments recommends in order to give you the best results possible when you use your μ TA 2990 system.

As already mentioned in the first chapter, this system can produce two types of measurement: Atomic Force Microscopy (AFM) images, also called *visualization*, and localized thermal analysis data, also called *characterization*. These two types of information, when obtained and used together, constitute the technique of Micro-Thermal Analysis.

You do not need to be an expert in AFM or thermal analysis to get good results from the μ TA 2990. However, a reasonable understanding of both disciplines is desirable in order to get the most out of your instrument. This chapter will introduce you to the specific concepts and constraints to bear in mind as you run your instrument. For detailed technical information, refer to Appendix C.

Optimizing Visualization Parameters

To help you produce high-quality AFM images when you use your TA Instruments μ TA 2990 instrument, we have provided guidelines in this section that specifically target the parameters associated with imaging (*visualization*) of your samples.

Thermal probes have a high spring constant compared with the AFM probe. This feature, coupled with the asymmetry of the thermal probe, means it is not desirable to obtain images other than rastering parallel to the scanner. The μ TALab Software does support scan rotation. However, a warning sign is displayed if the rotation option is chosen, to remind you that this may damage your thermal probe.

Image Size versus Sample Roughness

The *maximum image size* obtainable with the μ TA 2990 is 100 μm by 100 μm . The *maximum displacement* that can be measured in the Z-axis is specified as 10 μm .

NOTE: The exact displacement in the Z-axis varies from scanner to scanner. The exact figure is marked on the underside of your scanner.



CAUTION: You may damage your system if you image a sample that exceeds the scanner Z-axis specification.

Rough samples are defined as those samples with a Z displacement exceeding 10 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 10- μm limit. 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.

If you need Localized Thermal Analysis (LTA) data only and not images from “rough” samples, select **Acquisition/Local Thermal Analysis** from the menu, and then choose the locations for LTA measurements on any of the blank image windows. Then observe the thermal probe on the video monitor to ensure that it is not damaged during the experiment.

NOTE: Click on the **Measure** button during an LTA experiment to abort the measurement for that specific location.

Effects of Topography on Thermal Image Contrast

The thermal images obtained with the μ TA 2990 are affected by the topography of the sample. Consider the three samples shown in Figure B.1.

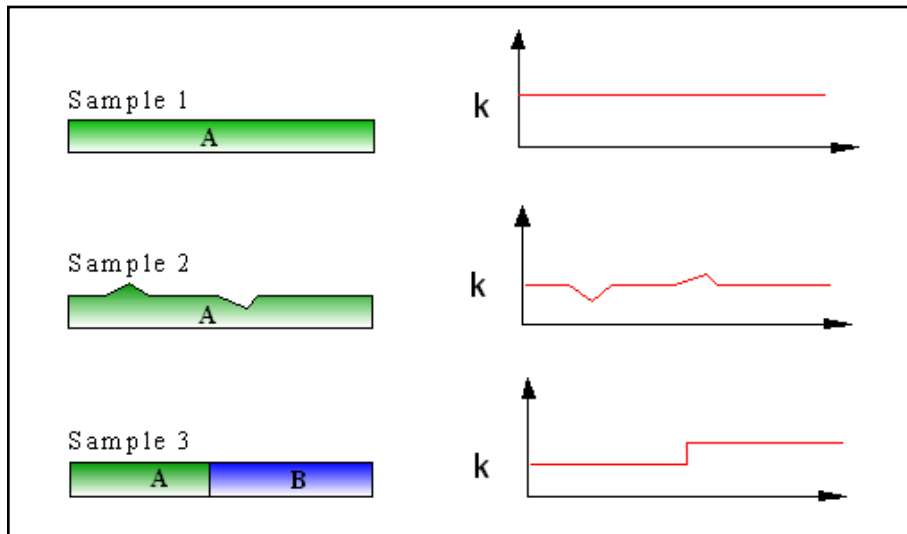


Figure B.1
Topography vs. Image Contrast

- Sample 1 is flat and consists of a single phase.
- Sample 2 has some topographical features and is also a single phase.
- Sample 3 is flat and contains two phases that have different thermal properties.

As the thermal probe travels over Sample 1, a constant current is required to maintain the probe temperature.

As the probe travels over Sample 2 (shown in Figure B.1), the current changes to maintain the probe at constant temperature. When the probe is at the top of the "mountain," it is surrounded by less sample and more air than when it is on the "plain." Air has a lower thermal conductivity than the sample; therefore, the apparent thermal conductivity (k) measured by the probe is lower. When the probe is in the "valley," it is surrounded by more sample and less air

than when it is on the “plain.” Consequently, the apparent thermal conductivity increases. These observed changes in the thermal conductivity signal (and, therefore, in the images) are due entirely to topographical effects—not to changes in the thermal properties of the sample.

As the probe travels over Sample 3 (shown in Figure B.1 on the previous page), the measured change in thermal conductivity is due to a change 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 thermal images.

In conclusion: When possible, use sample preparation techniques that produce flat surfaces in order to make image interpretation easier.

Interpreting the Thermal Image versus Topography

Notice that the effect on the thermal conductivity image due to the topography (shown in Figure B.1 on the previous page) is the *inverse* of the topography—*i.e.*, the “mountains” in the topography image appear as “valleys” in the thermal images, and vice versa. This means that you can image a sample and then determine which

features in the thermal images are due to material properties and which are due to topographical features.

For example, if a feature exists in the thermal image with no corresponding feature in the topography image, it is likely to be due to a difference in thermal properties at that location. Conversely, if a “high” feature exists in your thermal image and a corresponding “low” feature exists in your topography image, it is likely that this is a topography effect and not a change in the thermal properties of your sample.

You can change the palettes (colors) used to display each image. If you use this feature, you can choose color palettes to help you differentiate between topographical and thermal properties when you compare the images. See Chapter 6 for instructions.

Function of Thermal Images

As a result of the topographical effects, the thermal images obtained with the μ TA 2990 are not quantitative. The functions of the thermal images are to provide image contrast based on the thermal properties of the phases or components in your sample, and to aid you in assessing locations for LTA experiments.

Effects of Scan and Feedback Settings on Image Quality

The proper settings for the proportional (P), integral (I) and derivative (D) gain—the PID settings—are essential for producing high-resolution AFM images. Their importance in MicroThermal Analysis is attenuated by the spatial resolution of the thermal probe and its high spring constant. However, you need to have understanding the effects of the PID settings as well as of the scan rate and set point values on the images. This section covers the methodology for feedback optimization.

NOTE: The **Scan** and **Feedback Panels**, shown in the figure below, are displayed only if acquisition is set to 2D imaging (select **Acquisition->2D imaging** from the **Main** window menu bar).

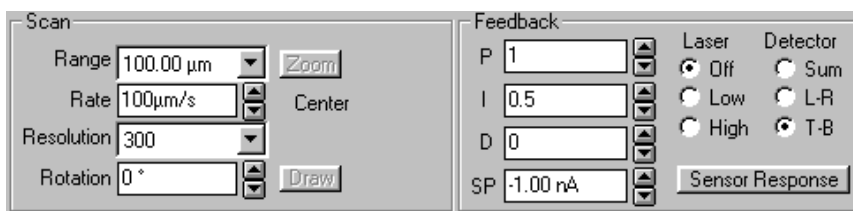


Figure B.2
Scan and Feedback Panels

The parameters shown in Figure B.2 are good default settings for acquiring images with the thermal probe. When you analyze an unknown sample for the first time, we recommend that you use these settings and then optimize them from the values given in this section. The following pages provide more information on the parameters shown in Figure B.2.

Set Point

The **Set Point** (**SP** in Figure B.2) affects the tracking force or load on the thermal probe. The recommended setting for the **T-B** value is between 10 and 11 nA, with a **Set Point** of -1.00 nA. *The difference between these values is equal to the force applied to the probe.*

During image acquisition, the system extends or retracts the Z-piezo positioner to maintain the “force” feedback current at the value specified in the **SP** field. The **SP** value sets the amount of photodetector current used during the probe approach. As the probe tip gets closer to the surface, the deflection of the probe (cantilever) causes the sensor current (**T-B**) value to increase. Once the current reaches the defined set point, (1) the controller considers the system to be in force feedback, (2) the approach motor is turned off, and (3) the feedback loop is activated to keep the sensor at the **SP** value. The default **SP** value is -1.00 nA.

- Increasing the **Set Point** can improve resolution of fine features but may also damage soft samples or bend the platinum tip of the thermal probe.
- Decreasing the **Set Point** will result in poor topographical image quality but has less chance of damaging the probe or sample.

The **Set Point** also affects the load on the probe during an LTA experiment. The **Set Point** value used to obtain images is retained when moving between the 2D scanning and LTA parts of your experiment. You can change the **Set Point** in the LTA experiment in much the same way as you would vary the force upon the TMA probe in a TMA experiment. However, if you use a

Set Point that is too high, it can bend and /or damage the probe. Use only low forces (**Set Points**) in the LTA experiments, such as those used for imaging.

NOTE: The amount of force applied by the probe to the sample surface is proportional to the difference between the **Set Point** and the **T-B** value with the probe raised. With a larger difference, more force is applied; with a smaller difference, less force is applied. When the thermal probe is used, the typical difference is between 10 and 20 nA, with the **Set Point** value more positive than the **T-B** value.

Scan Rate

The *Scan Rate* setting (**Rate** in Figure B.2) is a compromise among image quality, time, and possible damage to the probe. Start with a **Rate** equal to your image size—*i.e.*, if the Range is set to 100 μm , then set the **Rate** to 100 $\mu\text{m/s}$.

- The flatter the sample, the more you can increase the scan rate without fear of damaging the probe by scanning too quickly over a rough sample.
- Decreasing the rate may improve the quality of the images, but at some point, the resolution of the probe will become the limiting factor for improving image quality in this manner.

Resolution

The value in the **Resolution** field (shown in Figure B.2) determines the number of sampling points acquired for a given scan range. For example, a setting of 300 sets up the acquisition of a 300 pixel-per-line by 300-line image within the given Scan Range.

- The *higher* the **Resolution** number, the more data points are taken and longer the scan takes to acquire. Settings of 300 or higher produce images that are of publication quality.
- To speed up the image acquisition time or to obtain a fast “survey image” of your sample, *lower* the **Resolution** value.

The most efficient method is to first use a lower **Resolution** value to quickly obtain the initial “survey image.” Then, once you have optimized all of the **Scan** and **Feedback** parameters to your satisfaction and obtained an image of the correct part of your sample, go back and increase the **Resolution** value to obtain your final image.

PID Settings

The **PID** (proportional, integral and derivative) feedback control system uses an equation that combines terms proportional to the error signal, the integral of the error signal, and the derivative of the error signal to control the response of the Z-piezo positioner to the probe deflection. See the Figure B.3 for a schematic of the feedback.

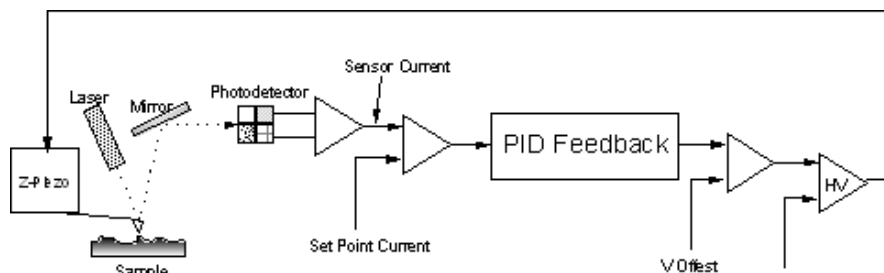


Figure B.3
Feedback Parameters

The error signal is the difference between the internal sensor and the set point. The optimum settings for the PID coefficients largely depend on the sample properties, scan rate, and the probe tip geometry. Therefore, they need to be determined experimentally.

Proportional Gain (P Setting)

The high-frequency response of the Z-piezo positioner is controlled by the proportional gain. In practical terms, this equates to how the piezo positioner responds to fine structural details on the sample surface. Optimization of this setting is crucial to proper imaging of topography. Adjustment of this parameter for large scan ranges may have little noticeable effect.

The value of the proportional gain setting should be at least that of the integral setting. Setting the proportional gain too high causes oscillation.

Typical values for the thermal probe are between 1 and 2.

Integral Gain (I Setting)

The low-frequency response of the Z-piezo positioner is controlled by the integral gain. In practical terms this equates to how the piezo positioner responds to large topographical features as well as the roughness and tilt of the sample. This is the most sensitive and most important of the three PID parameters:

- Lowering the integral gain too much “smooths out” topographical features such as the grid pitch of the test grid.
- Increasing the integral gain too much causes oscillation, because the Z-piezo positioner overcompensates for topographical deflections in the probe.

Typically, the **I** value is 0.3 to 0.5 times the **P** value. Typical **I values** for the thermal probe are 0.5 to 1. The default **I** value is 0.2.

Derivative Gain (D Setting)

The value in the derivative field should initially be set to zero, which is also the default value. Raising the value from zero may reduce unwanted oscillation, allowing higher values for the integral gain to be used; however, doing so may amplify high-frequency noise.

The derivative gain acts as a stabilizing parameter when the feedback loop is taxed to the maximum degree and is close to oscillation. The derivative gain helps the feedback loop respond to large topographical features, without causing overshoot or oscillation.

A rough sample, if scanned quickly, often benefits from a derivative gain set somewhere between the proportional and integral gain values. The derivative gain has little effect on scanning of flat or smooth samples and in most cases can be set to zero.

Tips for Setting PID Parameters

You may find it helpful to note the following suggestions when setting your PID parameters on the **Feedback** Panel:

- If you observe sudden bursts of sinusoidal noise on the **Sensor** (when viewed on the **Oscilloscope** window), you need to reduce the integral gain value (**I** setting).
- When you are using the thermal probe, good starting values for the PID settings are: $P = 1$, $I = 0.5$, and $D = 0$.
- When you are using the AFM contact topography probe, good starting values for the PID settings are: $P = 0.3$, $I = 0.15$, and $D = 0$.
- Generally, you should use a value for the proportional gain (**P** setting) about two to three times larger than that of the integral gain (**I** setting).

Setting Up the Z-Piezo Positioner

The **Z Piezo** panel, shown in Figure B.4, allows you to set the gain and set point for the Z-piezo.

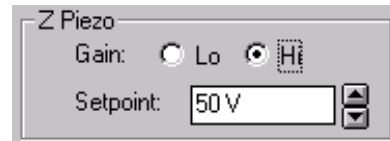


Figure B.4
Z Piezo Panel

Setting the Gain

The Z-piezo positioner has two gain ranges, which you choose by clicking on the radio buttons on the **Z Piezo panel** for that range:

- The **Hi** setting uses a 0–100 volt range, which is best for rough samples.
- The **Lo** setting uses a 0–40 volt range, which is best for flat samples.

Selecting the **Gain** setting requires consideration of the tradeoff between Z range and Z resolution. For optimal resolution, the general rule of thumb is as follows:

- If a sample's maximum feature height is less than 10% of the Z range of the scanner (for the μ TA 2990, the Z range is 10 μ m), select **Lo** gain.
- If the sample's maximum feature height is within 10% to 90% of the scanner's Z range, select **Hi** gain.

If you are running an unknown sample, always use the **Hi** gain first (*i.e.*, the default condition is **Hi** gain) and check the sample's roughness on the Oscilloscope by monitoring the Z-Piezo positioner signal using **Line** mode. You can then change to **Lo** gain, if required, before starting image acquisition.

Choosing the Set Point

The **Set Point** field contains a scrollable list from which you choose a set point for the Z-piezo positioner.

Usually, you should choose a set point value that is at the midpoint of the Z-piezo positioner range (*i.e.*, 50V when using **Hi** gain [the default value is 50V] and 20V when using **Lo** gain), to give equal range in both up and down directions. There are two exceptions to this general rule:

- If you know that your sample has a “significant depression,” or hole, change the set point to a lower value. This will give you more range in the down direction.
- If you know that your sample has high features, change the set point to a higher value. This will give you more range in the up direction.

Optimizing Characterization Parameters

To help you produce high-quality thermal analysis results when you use your TA Instruments μ TA 2990 instrument, we have provided guidelines in this section that specifically target the parameters associated with localized thermal analysis (*characterization*) of your samples.

Select **Acquisition/Local Thermal Analysis** from the menu bar to display the AC Mode Signals panel, which is specific for localized thermal analysis (LTA) experiments.

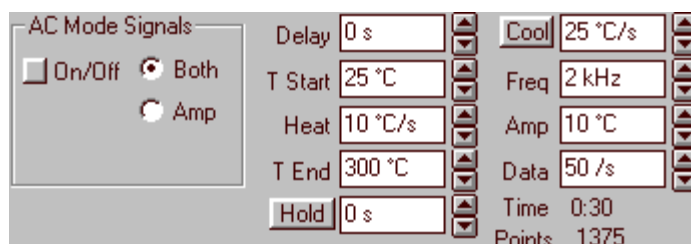


Figure B.5
AC Mode Signals Parameters

The procedure for optimizing parameters in LTA experiments is similar to those for traditional thermal analysis experiments.

NOTE: Please remember that the LTA data is used primarily to obtain transition temperatures and is not otherwise quantitative.

Heating Rate

When you want to obtain thermal analysis data using the μ TA 2990, 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 Micro-Thermal Analysis 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 5 to 10 °C/sec usually yield the best overall results.

Temperature Modulation Conditions

To set up the temperature modulation conditions for LTA experiments, follow these recommendations:

- Lower frequencies are more sensitive to transitions, and values of 5 to 15 kHz give the most easily seen transitions.
- Similarly, amplitudes of several degrees give the best signal to noise, typically 2 to 10 degrees.

Baseline Subtraction

You can perform baseline subtraction using the μ TALab software; however, only μ -MDTA signals are baseline subtracted. Two types of baselines are possible:

- **Air:** This type of baseline is taken with the thermal probe above the sample, when not in thermal or force feedback.
- **Reference Surface:** This type of baseline is taken with the thermal probe on a reference surface. A reference surface is one that is inert over the temperature range that you plan to use for your sample measurements. A sapphire disk, mounted onto a sample mount (stud), is supplied in your Micro-Thermal Analysis Sample Kit.

The objective of baseline subtraction is to remove any instrument (thermal probe) characteristics from your LTA measurements. Baselines are specific to the sample–reference probe pair used for the measurement, together with the exact temperature program and modulation conditions. If any of these are changed, you will need to rerun your baseline experiment. Refer to Chapter 5 for details on how to run and use baselines.

Reproducibility

Variation in the MDTA™ or TMA™ curves, obtained at multiple locations using the same sample, can be due to either the sample itself or the μ TA 2990 system. 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. The LTA data are obtained from a cubic sample about 10 μ m in size. Local variations across the surface of your sample can be measured by the μ TA 2990. 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, do not change when moving from conventional to MicroThermal 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™ 2920 DSC or your μ TA 2990 .

Typically, when you are working with single-phase materials, TMA results agree within $\pm 0.3 \mu\text{m}$. Transition temperatures for calibration materials agree within $\pm 1 ^\circ\text{C}$.

You need to assess the reproducibility for your own types of samples before drawing conclusions about the similarity or difference between two samples.

Analyzing Localized Thermal Analysis Data

TA Instruments' *Thermal Solutions* software includes a data analysis program called Universal Analysis. For in-depth information regarding this program, refer to the *Universal Analysis* manual. This section provides you with some specific information as it relates to using LTA data files with the Universal Analysis software.

- Because localized thermal analysis data files are not quantitative, the sample size is always set to zero when the data are stored. This ensures, for example, that quantitative MDTA™ peak areas can not be calculated, nor can expansion coefficients from TMA™ curves.
- If data are obtained from multiple locations on single sample, all locations are stored in a single data file. Up to 9 locations can be measured and stored in a single data file. The x, y coordinates (in μm) of each location are stored within the data file parameter block to aid traceability.
- The filename for the image data is also stored as part of the parameter block of the LTA data file. This feature helps file location and offers the ability to work back from stored LTA data.
- It is possible to run both μTALab and Universal Analysis programs at the same time. You can keep the Universal Analysis window in the foreground over the μTALab window by selecting **Window / Stay on Top** from the Universal Analysis menu.
- If you use a baseline is used when making LTA measurements, the baseline data are included in the sample data file. The baseline is stored in Location 0. The default setting suppresses the display of the baseline data when you recall an LTA data file
- You can use either the Multiplex (Mpx) button on the tool bar or **Location List** on the Universal Analysis menu to display a list of locations within your LTA data file. You can turn specific locations (including Location 0, the baseline) on or off for viewing by checking the appropriate box.
- No method log is created for LTA data files. However, the method name is created from the temperature program values used to obtain the data from within the μTALab Software.

System File Edit

Recalibration of your scanner is indicated when topographic measurements, cross-talk, and linearity do not meet specifications for your applications. Calibrating your scanner via the **SysFileEdit** function of the μ TALab software involves three basic steps:

1. Prepare and scan a standard calibration sample grid to acquire a topographic image. (An equivalent sample with a periodic structure that has known dimensions can also be used.)
2. Use the μ TALab software to examine the image on the basis of your criteria and then to compute new calibration, linearity, and cross-talk coefficients. You may have to edit the image to obtain well-defined structures for the calibration.
3. Once you have obtained acceptable results for scanning, save the new calibration coefficients to the scanner (.SYS) file. (The program automatically backs up the existing .SYS file, giving it a .SBK extension.)

The following pages describe the process of scanner calibration in detail.

Preparing the Sample and Acquiring the Image

1. Obtain a (clean) standard calibration grid (Si test grid, found in your MicroThermal Analysis Sample Kit) or an equivalent sample with a known periodic pattern.
2. Using tweezers, mount the calibration grid onto the X-Y sample holder/translator.
3. Translate the sample to locate an appropriate area of periodic structures.
4. On the **Scan** panel (located on the left of the μ TALab **Main** window, under the menu bar), select or enter a setting for the **Range** that is appropriate for the sample area to be measured.


The scan range value divided by the number of periodic structures should be greater than 4 (Range/Pitch > 4).


The optimum number of periodic structures within scan range is 8 to 12 (*e.g.*, when calibrating a 25 μm scan range, use a calibration standard with a pitch of 2 or 3 μm).

5. On the **Scan** panel, set the **Rate** to 4 times the scan **Range** value you selected in step 4.
6. On the **Scan** panel, set the **Resolution** to 500.

You can use a lower **Resolution** setting, but it may result in poor boundary definition, reducing the accuracy of calibration.

7. On the **Scan** panel, set the **Rotation** field to 0°.
8. Make sure that the laser is properly aligned on the scanning probe (see Chapter 2).

9. Press the **Approach** button, .

10. Once the tip is in feedback, press the **Scan** button, .

The sample is scanned and an image is acquired. You are now ready to calibrate and linearize the results.

Calibrating and Linearizing the Image

1. Select **Acquire, Sys File Edit** from the μ TALab **Main** window. The **SysFileEdit** panel appears, as shown in Figure B.6, on the top of the **Main** window, to the right of the **Probe** panel. It replaces the **Temperature** and **AC Mode Setup** panels (see Figure 5.6) when the **SysFileEdit** function is selected.

NOTE: Unless the window for the image you have acquired is active (highlighted), the **SysFileEdit** option on the **Acquire** menu is unavailable (grayed out).

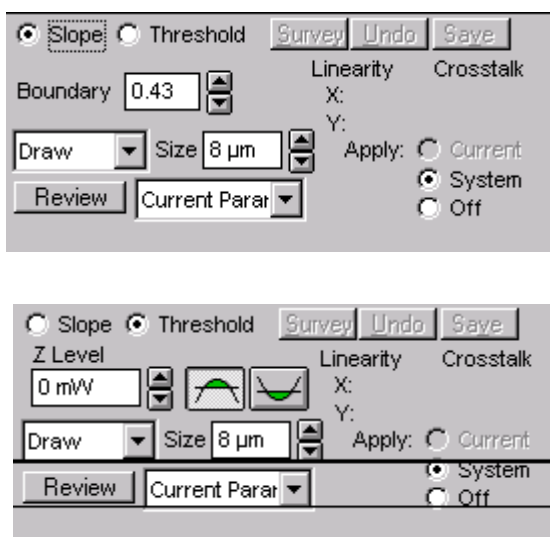




Figure B.6
SysFileEdit Panel in Slope Mode (top)
and Threshold Mode (bottom)

The top of Figure B.6 shows the **SysFileEdit** panel in **Slope** edge detection mode, which is the default setting. (For information about when to use the **Slope** mode for calibration, contact TA Instruments.)

2. To access **Threshold** edge detection mode, which you will use for this calibration procedure, click on the **Threshold** radio button. The **SysFileEdit** panel changes to **Threshold** mode, as shown in the bottom of Figure B.6.

The **Review** button on this panel accesses a window displaying the contents of the file selected on the scrollable list next to the button. Usually it is the calibration file currently being used by the system. For more information about this window, contact TA Instruments.

Most of the other buttons and fields on this panel are explained at the points they are used in the calibration procedure.

3. In the **Z Level** field on the **SysFileEdit** panel, accept the default value (the approximate equivalent of 1/2 the maximum **Z Height** [in nm] of the sample structures).
4. Press one of the two threshold tool bar buttons on the **SysFileEdit** panel according to the following criteria:
 - If you wish to highlight and use the data *above* the **Z Level** setting (when measuring structures that have a positive **Z Height**, such as columns, posts, and mesas), press the **Above Threshold** button, .
 - If you wish to highlight and use the data *below* the **Z Level** setting (when measuring structures below the surface, such as holes and trenches), press the **Below Threshold** button, .
5. In the **Size** field, enter the pitch size (in μm) of the structures being used for calibration.

The pitch size of the calibration samples that come in your Micro-Thermal Analysis Sample Kit are listed on the standard calibration specification sheet that accompanies the samples. If you use a nonstandard sample, you must know its pitch size.

6. Ensure that the **System** radio button (rather than the **Off** button) is toggled on.
7. Press the **Survey** button.

The acquired image is leveled, and the structures are detected with the threshold criterion. Structures with a Z-height above (or below) the **Z Level** threshold are highlighted.

If necessary, adjust the value in the **Z Level** field to help the system properly detect the structures, and press the **Survey** button again.

Editing the Image and Drawing the Lines

In order to obtain well-defined structures for the calibration, you may have to use the image editing tools to fill in or erase parts of the highlighted area. The purpose of editing is to obtain clear, uniform periodic structures that can be accurately measured. The editing tools are available from the drop-down menu containing the **Draw** tool (see Figure B.6).

The editing tools function as follows:

- **Paint Line** paints a missing line in the highlight color.
- **Paint Area** paints an area in the highlight color.
- **Erase Line** erases a line from the highlighted area.
- **Erase Area** erases a defined area from the highlighted area.
- **Draw** enables you to highlight a row or column of periodic features in the image once you have edited it.

To use each of the editing tools:

1. Click on the tool you wish to use.
2. Move the cursor to the location, and then left-click and drag the mouse to draw the line or define the area. Release the mouse button when done.
3. Right-click to apply the chosen tool and exit the command.

Once you have cleaned up your image, use the following method to draw a horizontal line along a row that contains at least four consecutive, well-defined periodic structures:

1. Select the **Draw** function from the drop-down menu, and then left click and drag to draw a horizontal line long enough to intersect as many whole structures as possible (at least four). Release the mouse button.

2. Use the cursor to position the line appropriately.
3. When you are satisfied with the position of the line, right click to exit the function.

The selected row is now highlighted.

Use the same method to draw a vertical line along a column that contains as many consecutive, well-defined structures as possible (at least four).

NOTE: Avoid drawing lines on partial structures, such as those cropped by the boundary of the scan **Range**.

NOTE: Sometimes you may observe small areas that are highlighted outside the periodic structures. These may be caused by debris on the sample. Avoid selecting a row or column that contains such artifacts, or use the image editing tools to erase them.

NOTE: If more than one row or column of structures is highlighted, click on the **Undo** button. Then adjust the **Z Level** or use the image editing tools to erase areas that should not be highlighted.

Calculating the Coefficients

Once one row and one column have been successfully highlighted, the system computes linearity coefficients, cross-talk coefficients (orthogonality between X and Y), and scan range. It then reports the nonlinearity percentage and angle between X and Y (cross-talk) in the fields displayed on the **System File Edit** window.

If (1) the value reported in the **Crosstalk** field is greater than ± 10 degrees or (2) the values for X and Y reported in the **Linearity** field are greater than 10%, you must click on the **Undo** button and repeat the image editing and line drawing procedures.

Errors as great as those listed above cannot be corrected by the software. They indicate an erroneous measurement, severe drift, or a damaged scanner. To check for erroneous measurement, visually inspect the sample and determine whether you should scan a different, cleaner area.

When you have obtained values for the cross-talk and linearity coefficients and scan range that are within acceptable limits, proceed to the next page to verify the results.

Verifying Coefficients

To verify the newly computed calibration coefficients, you will need to scan the sample again, applying the new coefficients.

CAUTION: DO NOT scan a sample if the new coefficients are erroneous, i.e., if the Cross-Talk value is greater than +10° or the Linearity values are greater than 10%. Tip damage could result.

When the scan is complete, inspect the new image to confirm that calibration and linearity are correct. For example, if you use a scan **Range** of 75 μm to perform a scan of five structures, each of which has a known pitch of 15 μm , the image should display the equivalent of the pitch of exactly five structures.

When you have verified that the new coefficients are within acceptable limits, you can save them in your system; for instructions, see the next page.

Saving the New Coefficients

Once you have verified that the new calibration coefficients are within acceptable limits, you need to save them to the system in order for them to be used. To do so, click on the **Save** button on the **SysFileEdit** window.

The **Save** function writes the calibration parameters to the system file on your hard drive (*i.e.*, in the scanners directory), under the same name as the file that was shipped with your system.

If you need to go back to the previous system file data, there are two places to locate the stored files:

- The system backs up the “old” system file (that is being replaced) by renaming it with the extension .SBK.
- The original calibration .SYS file for your scanner is stored on the floppy disk that was shipped with the system.

Appendix C

Technical Reference

Overview

This discussion introduces some of the basic concepts and principles of scanning probe microscopy (SPM), upon which your μ TA 2990 is based. There are two basic modes of SPM—scanning tunneling microscopy (STM) and atomic force microscopy (AFM). STM is discussed in detail here. The basic functionality of an SPM instrument is outlined, including Z-piezo positioner, scanning in feedback, and imaging resolution.

NOTE: The magnification and resolution attributed to SPM in the following discussions are possible only with use of specific AFM probes, and are not possible with use of the thermal probe.

SPM Theory

Several techniques are available for magnifying the microscopic features of a surface—optical microscopy, scanning electron microscopy (SEM), and scanning probe microscopy.

Methods for magnifying surface features originated with magnifying lenses and optical microscopy in the late 18th century. Optical instruments are limited by the wavelength of visible light and can resolve objects down to approximately $0.5\ \mu\text{m}$.

During the 20th century, methods for magnification based on electron and ion beams were developed. Operating in a vacuum, the scanning electron microscope (SEM) can resolve features to the level of approximately $0.3\ \text{nm}$, if the specimen's atomic number contrast is high. SEM microscopy often has destructive effects on the sample, and SEM techniques do not readily yield depth information.

Scanning probe microscopy (SPM) is a recent, innovative technology that relies on a mechanical probe for generation of magnified images. An SPM instrument is operable in ambient air, liquid, and vacuum and resolves features in three dimensions—down to a few picometers (pm). The table on the next page compares current magnification techniques with regard to magnification, operating environment, type of image, and damage to the sample. The first four techniques rely on focusing particles, such as photons, electrons, and ions, to resolve an image. SPM microscopy resolves a magnified image using data obtained by scanning the sample surface with a sharp, microscopic mechanical probe.

Table C.1
Comparison of
Magnification Techniques

Technique	Mag. Factor	Medium	Image	Damage
Optical	10 ³	Air, liquid	2D	None
Confocal	10 ⁴	Air	2D/3D	Minimal
Ion beam	10 ⁵	Vacuum	2D	Severe
SEM	10 ⁶	Vacuum	2D	Some
SPM	10 ⁹	Air, liquid, & vacuum	3D	Minimal to none

Table C.2
Standard Microscopy
Units of Measure (Distance)

Unit	Value
centimeter (cm)	1 cm = 1/100 meter
millimeter (mm)	1 mm = 1/1,000 meter (10 ⁻³)
micrometer (μm)	1 μm = 1/100,000 meter (10 ⁻⁶)
nanometer (nm)	1 nm = 1/1,000,000,000 meter (10 ⁻⁹)
angstrom (Å)	1 Å = 1/10,000,000,000 meter (10 ⁻¹⁰)
picometer (pm)	1 pm = 1/1,000,000,000,000 meter (10 ⁻¹²)

Scanning Probe Microscopy

A scanning probe microscope (SPM) is composed of a sensing probe, piezo positioners for adjusting the probe, an electronic control unit, and a computer for controlling the scan parameters as well as generating and presenting images. Each of these subcomponents is discussed in the following sections, followed by a functional description of the entire instrument. An essential component in the SPM is a sensor with very high spatial resolution. These sensors can routinely sense height changes as small as 100 pm. Two common types of sensors are the tunneling sensor and force sensor.

Scanning Tunneling Microscopy

Scanning Tunneling Microscopy (STM) uses a tunneling sensor (shown in the figure below) to measure the current passing between a metal probe and a conductive sample in extremely close proximity. Because of the quantum mechanical tunneling effect, electrons can be transferred from the probe to the surface, even though the two materials are not in physical contact.

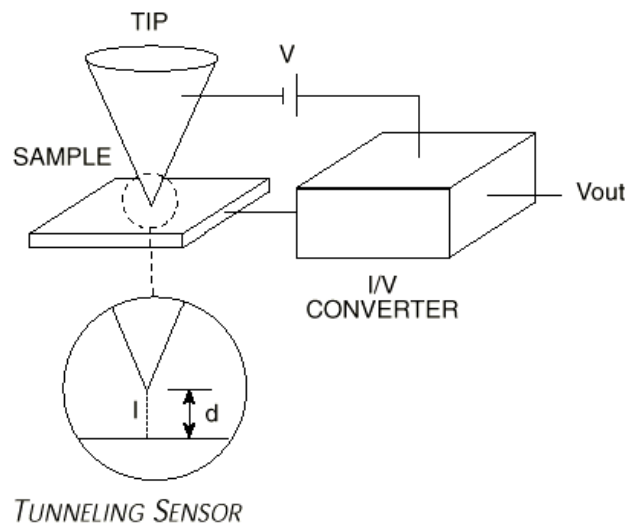


Figure C.1
Tunneling Sensor

The tunneling current (I) is directly proportional to the applied voltage (V) and exponentially proportional to the distance separating the tip and sample. This relationship is expressed as follows:

$$I = aV e^{-b\phi^{1/2} d}$$

Where:

- I = tunneling current
- V = bias voltage
- ϕ = barrier height (a function of the tip and sample materials, representing the minimum energy needed by an electron to tunnel through the vacuum gap)
- d = tip-to-sample gap
- a, b = constants

A small decrease in the tip-to-sample distance (d) will result in a large increase in the tunneling current (I), assuming a constant applied voltage. The sensitivity of this relationship between current and distance allows for very precise control of tip/sample separation.

MicroThermal Analysis Theory

When you perform MicroThermal Analysis studies, the standard AFM probe is replaced by an ultraminiature temperature probe on the μ TA. This thermal probe not only provides the heat source but also measures the thermal response, providing information similar to that from conventional thermal analysis. The thermal probe is made from a tiny platinum filament surrounded by a silver sheath. The wire is formed into a probe and the silver is etched away to reveal the platinum tip. The probe acts as a resistive heater and provides the means of measuring temperature. The temperature program applied to the probe can be isothermal, dynamic, or modulated.

The thermal probe can be used in two ways: to obtain surface images and to run localized thermal analysis experiments. This section provides some technical information on these two types of techniques.

Imaging with the Thermal Probe

The μ TA 2990 thermal probe can provide information by maintaining a constant temperature and thermal current across the surface of the sample under investigation.

- **Constant Temperature** maps heat flow (thermal conductivity) into the surface by (1) maintaining the thermal probe tip at a constant, elevated temperature and (2) measuring the current required to maintain that temperature offset as the tip scans across varying materials on the sample surface. Superimposing a temperature modulation allows additional AC images (both amplitude and phase) to be obtained, which are related to the thermal diffusibility of the varying materials in the sample. Varying the modulation conditions allows the depth (and volume) of material imaged to be changed. Consequently, three thermal images (in addition to other contact images) can be obtained simultaneously:
 - Thermal Conductivity
 - Modulated Temperature (Amplitude)
 - Modulated Temperature (Phase)

- **Constant Current** maps thermal variations (hot spots) across the surface by maintaining a constant current flow through the thermal probe as it scans across the varying temperature regions of the sample's surface. As the temperature of the surface changes, the resistivity of the tip changes, requiring an adjustment in the bias voltage to maintain the constant current. This voltage change is measured to calculate the temperature of the surface under the tip.

Both modes of imaging produce relative thermal property results, not absolute values, in any measurement unit.

Localized Thermal Analysis

The images obtained from the sample surface can then be used to differentiate phases, domains, features, and contaminants within the sample. Individual locations on the surface may be measured using the thermal probe as a tiny thermal analyzer.

The temperature of the tip can be scanned just as in any thermal analysis experiment. The use of temperature modulation is crucial to this type of experiment because it limits the volume of the sample that is analyzed—the AC signal is sensitive only to changes close to the resistive heater. The volume analyzed is controlled by the frequency of modulation.

A number of signals are stored during a localized thermal analysis (LTA) experiment. As the probe heats the sample, *expansion of softening* can be measured by the movement (in the T-B signal) of the laser signal on the photodetector. This measurement is analogous to conventional thermomechanical analysis (TMA) and can be used to determine expansion, softening, melting, and glass transition. We call this technique *Micro-Thermomechanical Analysis* or TMA™.

The power required to maintain the modulated temperature program (compared to a reference probe) and the phase and amplitude of the response to the modulation are measured. The derivative of power or phase gives rise to curves that are analogous to those in Modulated DSC and can be used to determine melt onset, peak and glass transition temperatures. We call this technique *MicroModulated Differential Thermal Analysis* or MDTA™.

Because the mass of material measured during an LTA experiment is unknown, quantitative data is not obtainable. However, the qualitative transition temperatures measured by TMA™ and MDTA can be readily compared with their conventional thermal analysis counterparts.

Appendix D

Calculating Approach Speed

You can adjust the speed at which the Z motor moves when (1) the **Tip up** or **Tip down** button is pressed or (2) the **Approach** button is pressed. To do so, bring up the **Z Motor** window, in one of two ways:

- Select **Setup** from the **Acquisitions** menu on the μ TALab **Main** window; from the **Setup** submenu, select **Approach**.
- Press the **Approach** button on the μ TALab **Main** window's tool bar.

The Z Motor window appears, as shown in Figure D.1.

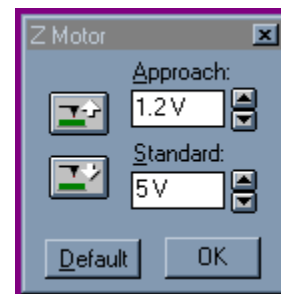


Figure D.1
Z Motor Window

The **Approach** field in this window displays the current value for the voltage applied to the Z motor when the **Approach** button is pressed on the tool bar. The default value is 1.2 V. To change this value, select a new value from the scrollable list for the field, and press **OK**.

The **Standard** field displays the current value for the voltage to the Z motor when the **Tip Up** and **Tip Down** buttons on the tool bar or **Z Motor** window are pressed. The default value is 4.9 V. To change this value, select a new value from the scrollable list for the field, and press **OK**.

Increasing the voltage in either field will increase the speed of the Z motor.

Pressing the **Default** button selects the default values for both fields: 1.2 V for **Approach** and 4.9 V for **Standard**.

Appendix E

Scanner Calibration

Introduction

After you have installed and used your μ TA 2990 instrument, you will need to check that it is operating accurately by performing a scanner calibration check every one to two months.

This section provides information needed to first check the current scanner calibration and then perform the scanner calibration, if needed.

Calibration Overview

The calibration details for each scanner you have are stored in a *system file* with a .sys suffix in the **Program Files\TA Instruments\μTALab\Scanners** directory. This file contains voltage–displacement information for the X and Y piezo's for each of the preset image scan sizes (100μm, 80μm, 50μm, 20μm, 10μm, 5μm and 1μm).

You can view the contents of any system file by selecting **Acquisition/SysFile Edit** from the menu. When this option is selected, the far right panel changes (as seen in the figure below).



System File Name

Figure E.1
The System File Panel

Scanner calibration involves imaging a sample with features of known size (usually the supplied test grid) and updating the voltage–displacement values so that future measurements are accurate. You can update the .sys file with new calibration information two ways—it can be done manually or through the μTALab software. This appendix provides information on how to perform either method.

Understanding the System Files

You have a .sys file for each scanner that you have. The currently active .sys file depends on the scanner that you select when the μTALab software starts up.

NOTE: If you only have one scanner file, you are not offered a selection when the μTALab software loads. The filename of your only .sys file is displayed.

The filename of the currently used system file is displayed. In the example shown in the figure below the filename is EX499803.SYS.

Pressing the Review button to the left of the filename displays the calibration information contained within the .sys file. Scroll down to see the information relating to Range and Crosstalk.

For each scan range there is a pair of voltage (for both the X and Y direction) as well as voltages to center the probe. In the figure on the next page the Range data for the 20 μ m scan range are highlighted. Beneath the Range data are the Crosstalk values, one for each of the scan ranges. The Crosstalk value is in degrees and is the correction to be applied to ensure that the X and Y scanners work at 90° from each other. The values are also displayed in the panel beneath the scroll window, where they can be edited to change the calibration data. For the moment, just press Cancel.

NOTE: The hysteresis and other values are related to non-linearized scanners. The scanner provided with the Micro-TA system is a hardware linearized scanner and these values may be ignored.

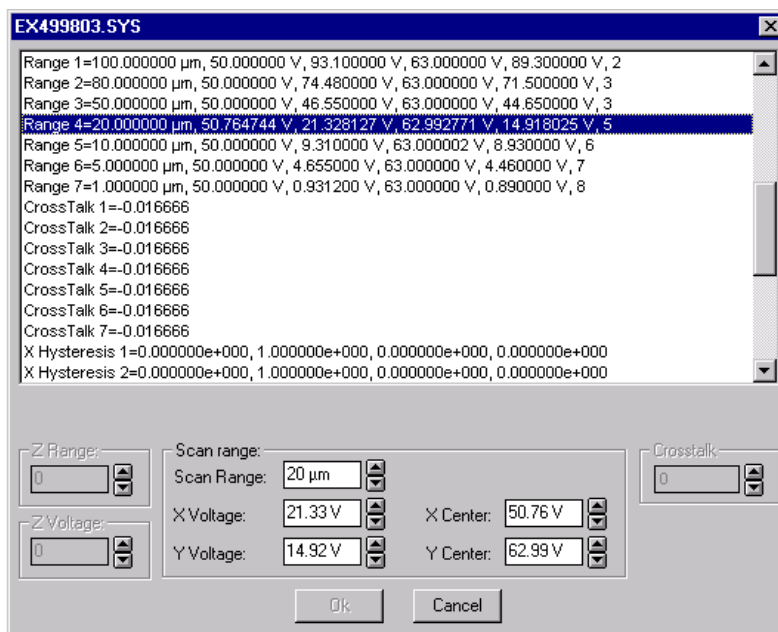


Figure E.2
Example System File Review Window

Checking Your Scanner Calibration

The first step in determining if your scanner requires calibration is to check the current calibration. This is easily done by checking the accuracy of your scanner calibration by imaging the supplied test grid using the contact mode AFMprobe.



CAUTION: Do not calibrate your scanner using the thermal probe.

NOTE: Please refer to Chapter 3 of this manual for information on making contact mode AFM measurements, if needed.

Once you have imaged the test grid, you can compare the size of the measured features, using the **Line Measure** function, to the actual size of the test grid features. Choose an area of the grid that will result in 5–7 regular features being imaged over the scan range.

For example:

- If you are imaging the grid using the 100 μ m scan range, then the 8 μ m pitch area of the grid is ideal.
- If you are imaging the grid using the 20 μ m scan range, then the 3 μ m pitch area of the grid is ideal.
 - If you choose too large a pitch for your scan range, you will not image enough features and the calibration may be in error.
 - If you choose too small a pitch, you will not have enough data points in your image to accurately describe each feature. Images should be taken with at least 400 line resolution.

Figure E.3, on the next page, for example, shows a 20 μ m scan of the 3 μ m pitch area of the test grid with 400-line resolution. Note also that the test grid is roughly perpendicular to the scan direction. If the grid is at a significant angle (greater than 10°) to the scan direction, then physically rotate the test grid and re-image.

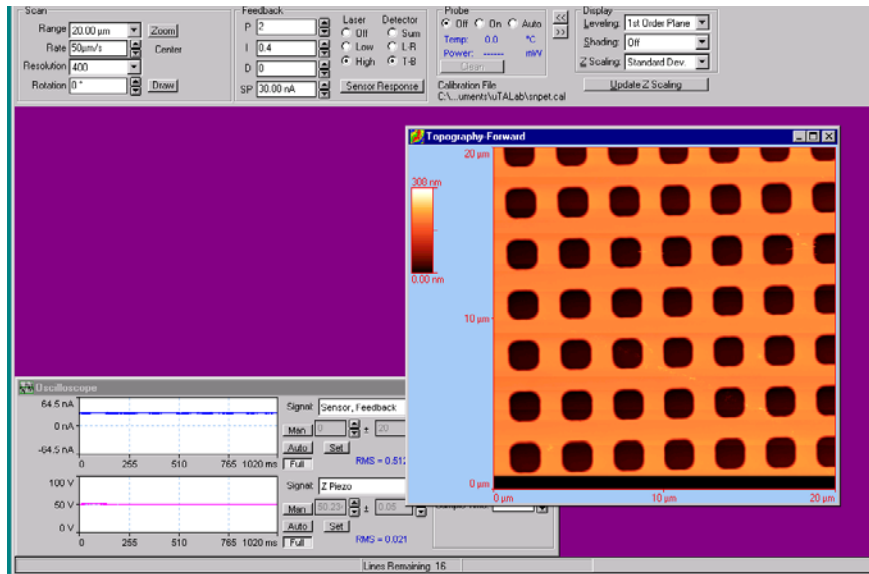


Figure E.3
20-µm Scan, 3-µm Pitch Area with 400-Line Resolution

After you obtain a complete image, select **Analysis/Line Measure**. Using the variable line type (Var) draw a line across five of the features in the X direction and measure the distance between the features.

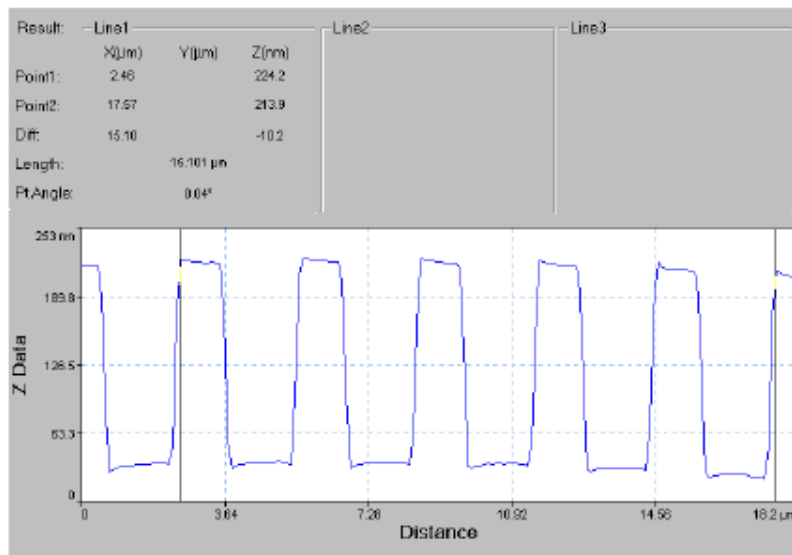


Figure E.4
Length Calculation Between Five Test Grid Features

Figure E.4, on the previous page, shows a Line Measure across five of the features. Note that the length of the line is measured at 15.101 μm . For each feature the pitch is 3.020 μm . This is in good agreement with the 3- μm pitch of the test grid. Typically, you should be able to achieve around 1% accuracy with a calibrated system. (The system specification is <2%.) If your scanner has not met the specifications above, calibrate the scanner as directed in the next section.

Calibrating Your Scanner

Should you find a significant error after making the previous measurements, you should re-calibrate your scanner. You can choose to calibrate the scanner manually, or calibrate it through the μ TALab software. This section describes both methods.

Manual Calibration

Follow these steps to manually change the values in the .sys file:

1. Select **Acquisition/SysFile Edit**. Select the system file of interest from the drop-down list and click on Review to open the **Sys Review** window (see the figure below). In this figure the 20- μ m scan range is highlighted. A voltage of 21.33 V is the current value for the X piezo (left – right raster) at this scan range.

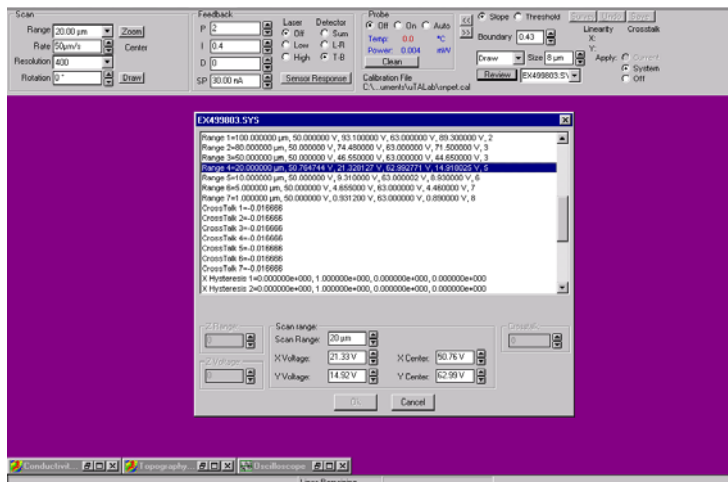


Figure E.5
System File Review Window

2. Calculate the new voltage by dividing the measured pitch by the known pitch and multiplying by 21.33 V, see the equation below:

$$\text{New Voltage} = (\text{measured pitch} / \text{known pitch}) * \text{current voltage}$$

For example: If we had measured a pitch of 3.020 μm, the new voltage would be:

$$(3.020 / 3.000) * 21.33V = 21.47V$$

2. Enter the new voltage (21.47 V) into the **X-Voltage** box of the **.Sys Review** window (see the figure on the previous page for the location of the box).
3. Repeat the above procedure in the Y (top – bottom) direction and calculate the new **Y-Voltage**. Enter this value in the **.Sys Review** window.
4. Press **OK** to accept the new values. You will now need to restart the μTALab software so that the new values are used for subsequent measurements.
5. Repeat the scan of the test grid to ensure that your scanner is calibrated.

The above procedure has only calibrated the scanner at the 20-μm scan range. You should repeat this procedure for the other scan ranges.

For scan ranges that are 5 μm and lower, you can linearly scale the values from the 10-μm scan range.



For example: If the voltages are increased by 1% for the 10-μm range, apply a similar 1% increase to the voltages used in the lower scan ranges.


Software Calibration

Follow these steps to calibrate the scanner using the software:

First scan the test grid with the instrument in contact mode using at least 400-line resolution. The grid should be roughly perpendicular to the scan direction, just as you did for the manual calibration. Once the image has been acquired, raise the probe tip and with the image as an active window select **Acquisition/SysFile Edit**. The **Sys Review** window is displayed.

The panel to the far right of the screen changes. Follow these steps to use the μ TALab software to automatically analyze the image and calculate the X and Y voltages required.

1. Locate the **Size** box and select the pitch size of the region that has just been scanned. In this case it is 3 μ m.
2. Select the  (**Above Threshold**) button, if the imaged region of the grid contains raised features. Select  (**Below Threshold**) button, if the imaged region of the grid contains recessed features.
3. Ensure that **Threshold** is selected (as opposed to **Slope**) so that the features on the grid can be automatically detected by the software.

With the settings set as described above, press the **Survey** button, .

The software will then attempt to recognize the repeat features of the test grid and highlight them as seen in the figure to the right.

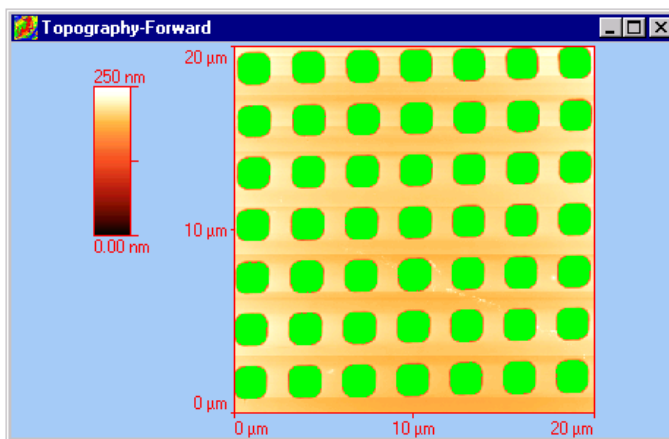



Figure E.6
Highlighted Test Grid

4. Ensure that **Draw**, , is selected in the drop-down list on the .Sys Panel and draw a line (left to right), using the mouse, across the several of the highlighted features. (Select the start point with the cursor, click the left mouse and drag to the right, release left mouse button and click the right mouse button when complete.)

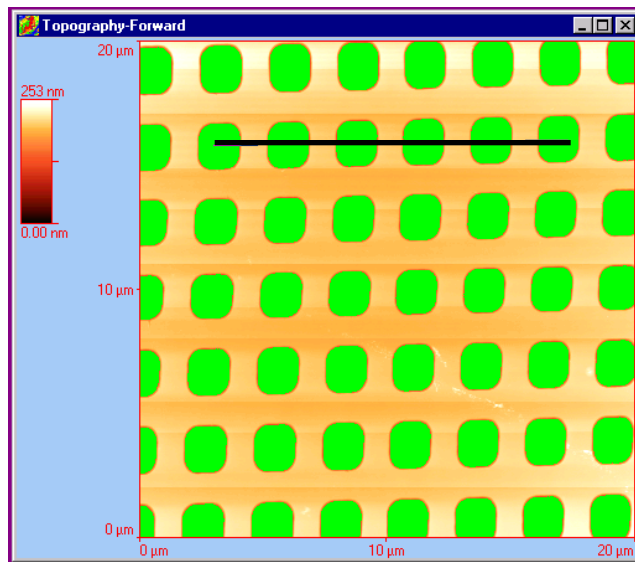


Figure E.7
Line Drawn Across Features

Once the right mouse button has been clicked the row of features will be highlighted as seen in the figure to the right.

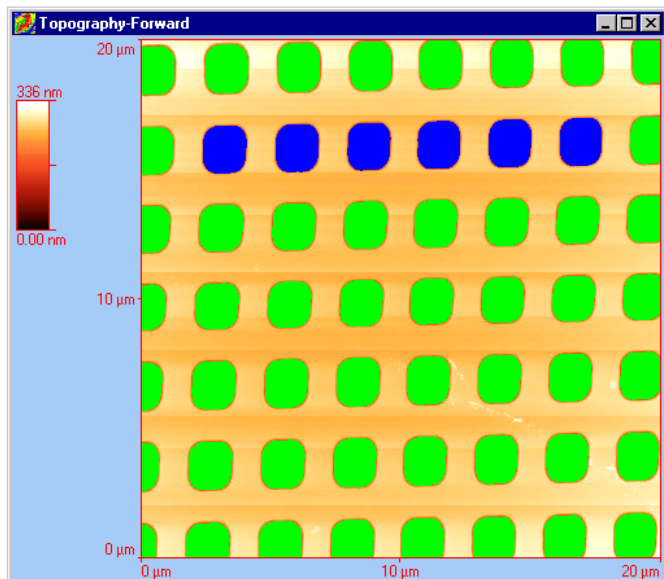


Figure E.8
Highlighted Row of Features

5. Repeat the line draw from top to bottom, starting with the first feature already highlighted.

Once the right mouse button has been clicked the column will be highlighted (as seen in the figure below).

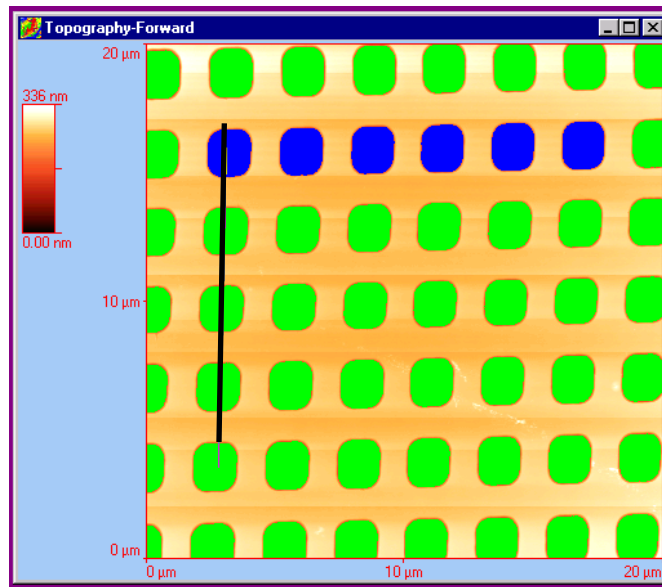


Figure E.9
Line Drawn Top to Bottom

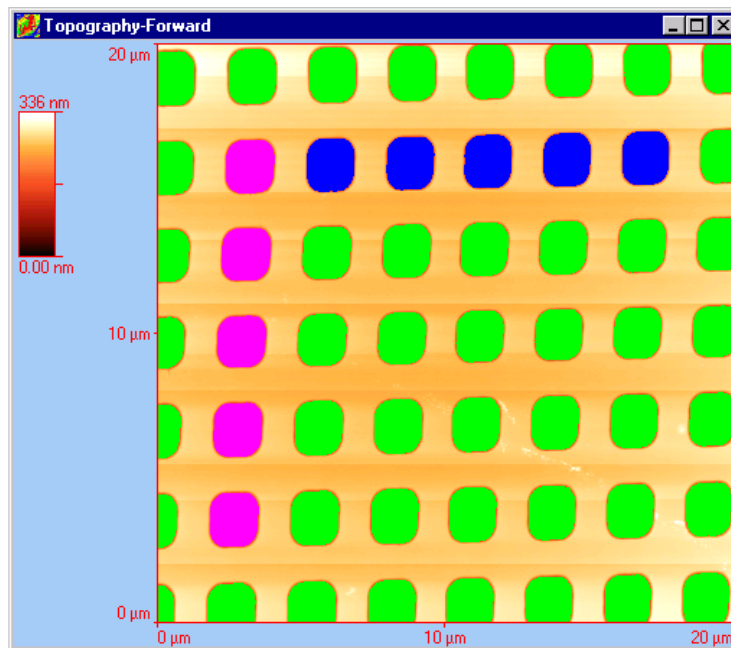


Figure E.10
Highlighted Column of Features

6. Look at the **System File** panel. The values for X and Y Linearity and Crosstalk for this scan range have been calculated (as seen in the figure below). See the next section for a more complete explanation of this panel.

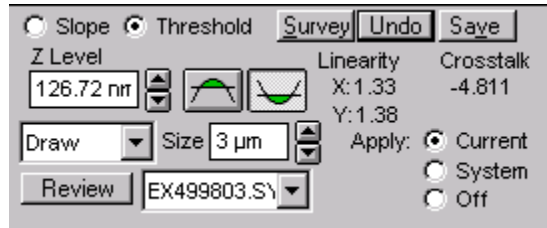


Figure E.11
Sys Panel After Calibration

NOTE: If the value in the Crosstalk is greater than $\pm 10^\circ$, or the value in the Linearity (%) is greater than 10 (X or Y values), click on the UNDO button and repeat your calibration. Errors this great cannot be corrected by the software and indicate an erroneous measurement, severe drift, or a damaged scanner. In the case of an erroneous measurement, visually inspect the sample and determine if a new, cleaner area of the sample should be scanned.

7. Press the **Save** button to update the .sys file with the new calculated values. You will now need to restart the μ TALab software so that the new values are used for subsequent measurements.
8. Repeat the scan of the test grid to ensure that your scanner is calibrated.

Understanding the System File Panel

For the majority of users and applications the calibration procedures already described will be sufficient to check and calibrate a scanner.

This section provides a more detailed explanation of the functionality of the **System File** panel, which is referred to in the calibration procedures.

When reading through these descriptions, refer to the figure below for an example of the items listed.

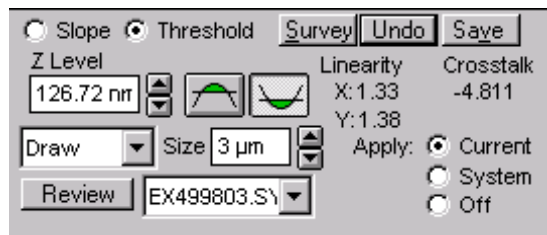


Figure E.12
System File Panel



Slope/Threshold

Slope and threshold shown on the **System File** panel are two methods used by the μ TALab software to detect the edges of features (also called structures). The acquired image is examined by the software in order to detect the edges and analyze the periodicity of the test grid.

When using a test grid that has well-defined height transitions along the edge features, use the *threshold* method of edge detection.

If a sample is not flat or cannot be leveled or does not have very clear well-defined edges, use the *slope* detection.

- **Slope** detects the edge of the structure by calculating the slope and slope width. Any slope that is narrower in width than the value set in the **Boundary Thickness** field is defined as an edge. The accuracy of the calibration process is dependent upon the definition of the structures (features) that are detected in this step. Structures that are well-defined and have clean, closed boundaries will result in a more accurate calibration.

- **Threshold** detects the structures by setting a Z threshold and analyzing structures (features) above or below this horizontal threshold. The Z-Level field defines the location of the horizontal threshold. Selecting the  button considers data *above* the Z level in the calibration (hills).
Selecting the  button considers data *below* the Z level in the calibration (holes).

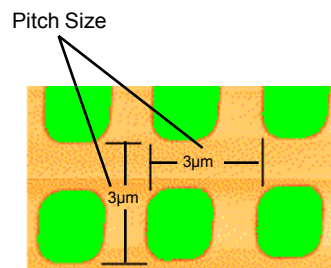
Editing Tools List

Contained on the **System File** panel is a drop-down list of image editing tools. These are used in order to get well-defined structures for the calibration. You will need to obtain clear, uniform structures for accurate measurement. If you make a mistake, use the erase tools to delete the mistake and start over.

- **Draw** is used to drag-and-drop a line along a row or column of features that contains consecutive well-defined structures. After the line is drawn it can be finely positioned using the cursor.
- **Paint Line** is used to paint a missing line in the highlight color.
- **Paint Area** is used to paint an area in the highlight color.
- **Erase Line** is used to erase a line from the highlighted area.
- **Erase Area** is used to erase a defined area from the highlighted area.

Size Field

The **Size** field is used to enter the pitch size (in μm) of the features (structures) being used for calibration. See the figure here for a diagram of pitch size. The pitch size(s) of the supplied calibration grid can be found on the calibration specification sheet that comes with the Micro-TA system. For non-standard samples, pitch size must be known.



Review Button

Click the **Review** button on the **System File** panel to display the **Review Calibration Information** window. This window displays the nominal pitch size and the mean, maximum, and minimum pitch sizes for both the X and Y directions.

Highlighting any line that can be edited causes the corresponding field(s) in the dialog to become active. Parameter values can be edited within a pre-defined limit range. Values outside of these limits cannot be entered. Three files are available for review and/or editing from the Review Files drop-down list: the current .sys file, the back-up (.sbk) file, and the Current Parameters file. Back-up (.sbk) and current parameters files can only be reviewed; you cannot edit these file types.

Applying Calibration Coefficients

A component of the **System File** panel is a set of radio buttons that allow you to apply specific types of calibration coefficients to the succeeding scans or disable the coefficients.

- Selecting the **Current** button applies the current scan's calibration coefficients and cross talk to the next scan.
- Selecting the **System** button applies the calculated calibration coefficients to the next scan.
- Selecting the **Off** button disables all calibration coefficients from being applied to the next scan.

Survey/Undo/Save Buttons

The set of three buttons on the **System File** panel are used as follows:

- **Survey** is used to level the acquired image and the detect the regular structures (features) using the currently selected threshold criteria (**Threshold** or **Slope**).
- **Undo** is used to reset the calibration coefficients to those previously stored.
- **Save** is used when you are satisfied that the calibration coefficients you have calculated. Click this button to update the .sys file. The .sys file is located in the **\Program Files\TA Instruments\uTALab\Scanner** directory. In case you need to revert back to the previous system file data, this is saved in a back-up file with a .sbk extension. Also, the original calibration .sys file for each scanner is provided on a floppy disk that comes with the system.

Linearity X/Y & Crosstalk

Under the set of buttons on the **System File** panel are a set of values described below:

- **Linearity:** Once one row and one column of features have been highlighted, the linearity is displayed for both the X and Y directions as a percentage.
- **Crosstalk:** Once one row and column have been highlighted, the crosstalk (orthogonality) between X and Y is displayed in degrees.

NOTE: If the value in the crosstalk is greater than $\pm 10^\circ$, or the value in the Linearity (%) is greater than 10 (X or Y values), click on the UNDO button and repeat your calibration. Errors this great cannot be corrected by the software and indicate an erroneous measurement, severe drift, or a damaged scanner. In the case of an erroneous measurement, visually inspect the sample and determine if a new, cleaner area of the sample should be scanned.

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