

HYSITRON



# PI 95 TEM PicoIndenter User Manual

Revision 9.2.1211

JEOL TEM Models



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# Introduction

Congratulations on your purchase of a Hysitron nanomechanical testing instrument. Hysitron testing instruments, options and user support have been designed with the user in mind so you can be confident that you are using the most technologically advanced, quality instrument with the highest level of user support available.

This user manual has been constructed to be accessible for any user; from the beginner looking to embark on nanomechanical testing with a Hysitron system to the advanced user looking for complex automation or analysis routines. This user manual also contains the calibrations necessary for maintaining the precision and accuracy of the instrument as well as instructions for performing testing and analysis.

In order to bring attention to important items that may cause damage to the equipment or information that may ease a process, this user manual contains two icons that will be displayed in the margin near the relevant information.

 Warning

 Information

Special attention should be given to any warning information discussed in the user manual as damage to the instrument can result from improper use.

TriboScan 9 and higher is organized into tabs. Tabs, sub tabs, side tabs and buttons are labeled in *ITALICS*. Because of the overall tab/sub tab structure of the TriboScan software there are fewer menu options, nonetheless, menu items, directories and filenames are labeled in **BOLD**.

Following each step in a procedure is important to minimize the possibility of equipment damage and to obtain quality data. For this reason, all procedures are numbered to distinguish procedures from general user manual information.

Hysitron Incorporated  
10025 Valley View Rd.  
Minneapolis, MN 55344

+1-952-835-6366

[support@hysitron.com](mailto:support@hysitron.com)

If a problem or question should arise which is not addressed in this user manual, please contact the Hysitron service department (listed at left) or your local service representative before proceeding.

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# Chapter 1 Hardware

The hardware components that comprise Hysitron nanomechanical testing systems are fragile and should be handled with extreme care. As a direct result of the sensitivity associated with nanoresolution testing it is necessary to ensure that the hardware components are properly calibrated, which will be discussed in the setup and calibration chapter of this user manual.

Although this user manual will emphasize the importance of properly installing and calibrating the hardware it is equally important that the equipment be properly handled and stored when not in use. The specially designed case and stand for the PicoIndenter should be used for storage when it is not in use. Other components that were not supplied with a case are generally less sensitive and should be stored in a low-humidity location where the components will not be subjected to any physical shock or excessive vibration.



**Extreme care should be taken when installing, removing or storing the components associated with the nanomechanical testing instrument. If the user is unsure of how to install, remove or store a component, contact a Hysitron service engineer prior to proceeding.**

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**Hysitron instruments are sensitive to humidity and excessive vibration or shock. For a document outlining the required environmental conditions for optimum performance, contact a Hysitron service engineer.**

---

The basic hardware components of the PI 95 system are listed below. Each of the components listed below will be discussed within the contents of this chapter.

- PI 95 PicoIndenter transducer assembly
- PI 95 PicoIndenter *performech* control unit
- Sample mounting bracket
- Computer and data acquisition system



## Section 1.1 System Requirements

The PI 85/PI 95 SEM/TEM PicoIndenter is supplied with:

- Holder/base with capacitive transducer sensor
- *performech* control unit
- Data acquisition notebook computer
- Cabling
- Video overlay hardware
- TriboScan 9.2 software (or higher)

A desk or cart at least 1 m (40") × 1 m (40") should be used for the computer station and auxiliary electronics. Additional samples, sample holders, probes, or accessories will depend on the individual system.

The system is designed for a specific voltage. Depending on the geographical location of the instrument, the system will run either on 110/120 VAC (60 Hz) or 220/230 VAC (50/60 Hz). The maximum current required at any time will not exceed 15 A for the 110/120 VAC system and 8 A for the 220/230 VAC system.



**Never attempt to connect the system to a voltage supply other than what the instrument has been designed to be use.**

---

## Section 1.2 Transducer Assembly

The core of every Hysitron PI 95 PicoIndenter system is the capacitive force/displacement transducer developed by Hysitron (Figure 1.1). The traditional three-plate capacitive design provides high sensitivity, a large dynamic range and a linear force or displacement output signal with displacements up to 5  $\mu\text{m}$  and loads up to 1.5 mN.

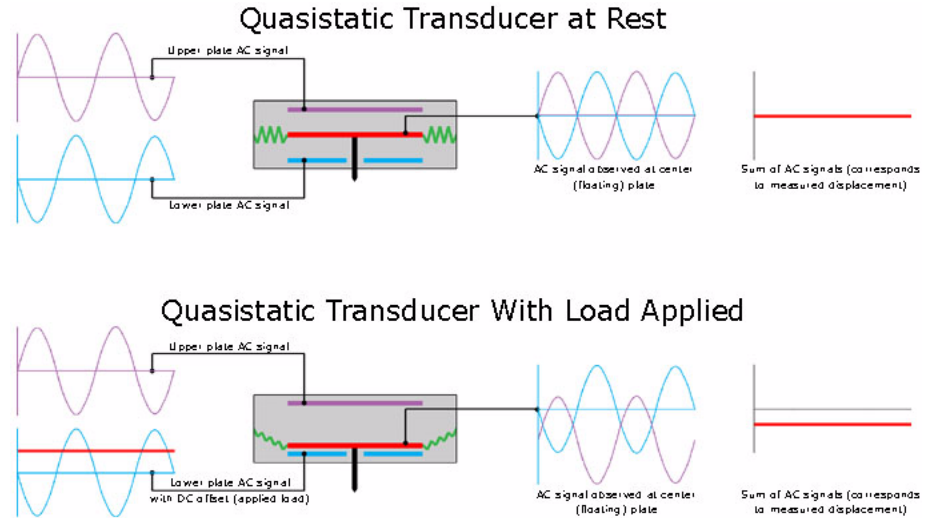
Figure 1.1  
JEOL TEM PicoIndenter



The PI 95 PicoIndenter is a 1D (normal force only) transducer that consists of the force/displacement sensor, drive circuit board and hardware used to mount the transducer to the TEM system.

### Displacement Measurement

The transducer sensor consists of fixed outer electrodes (drive plates), which are driven by AC signals 180 degrees out of phase with adjacent drive plates. Since the drive plates are parallel to each other and closely spaced with respect to the lateral dimensions, the electric field potential between the plates varies linearly. Since the signals applied to the drive plates are equal in magnitude but opposite in polarity at any instant, the electric field potential is maximized (equal to the applied signal) at the drive plates and minimized (zero, the two opposite polarity signals cancel each other out) at the site centered directly between the drive plates (Figure 1.2).

Figure 1.2  
 Transducer actuation diagram


The input impedance of the system is significantly larger than the output impedance of the transducer, so the center electrode will assume the same potential present at its position between the drive plates. The result is a bipolar output signal equal in magnitude to the drive plate at maximum deflection, zero at the center position and varying in a linear manner between maximum displacement and the center position.

## Force Measurement

The force is applied to the transducer electrostatically. To apply a force, a large DC bias (up to 600 V) is applied to the bottom plate of the capacitor. This will create an electrostatic attraction between the center plate and the bottom plate, which will pull the center plate down. The force can be calculated from the magnitude of the voltage applied. The maximum normal force available from the PI 95 PicoIndenter system is about 1.5 mN, however, the maximum available load will vary for each system.

 Table 1.A  
 Specifications of the Transducer  
 Assembly

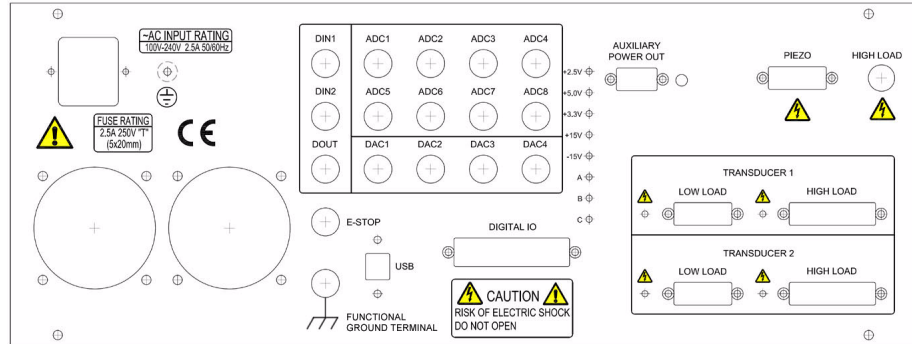
	PI 95 (JEOL)
Maximum Force	1.5 mN
Load Resolution	$\leq 3$ nN
Load Noise Floor	200 nN
Maximum Displacement	5 $\mu$ m
Displacement Resolution	$\leq 0.02$ nm
Displacement Noise Floor	0.4 nm

## Section 1.3

### *performech* Control Unit

Hysitron TI 950 TriboIndenter, TI 750 Ubi, TS 75 TriboScope and PI 95/PI 85 PicoIndenter systems utilize the newest generation of DSP based control units available from Hysitron - the *performech* control unit (Figure 1.3).

Figure 1.3  
*performech* DSP control unit  
back panel



### Section 1.3.1

#### Safety Considerations

Connect an appropriate AC power cord to the AC input connection on the back of the *performech* control unit and then into an available grounded (earth grounded) power outlet. The *performech* control unit accepts line voltages from 100 to 240 VAC, 50/60 Hz, 2.5 Amp maximum, single phase. Do not position the equipment so that it is difficult to disconnect the AC power cord. If mounting in a rack and plugging into a rack power source be sure the main rack power cord or power switch remains accessible for disconnection.

The *performech* control unit uses two 250 V, 2.5 Amp, Type "T" (*time lag*) fuses located in the main power inlet. To inspect or replace the fuses, disconnect the power cord from the back of the control unit and pull out the fuse tray.



**If the equipment is operated in a manner not specified by this user manual, the protection provided by the equipment may be impaired.**

## Section 1.3.2 Ventilation

The *performech* control unit requires adequate ventilation to operate normally. There are two cooling fans located on the rear of the *performech* control unit and one small cooling fan for each DSP or DAQ board located within the *performech* control unit.

Excess heat is expelled out of the vents located on the top of the *performech* control unit. Because of this, it is important that no equipment is placed on top of the *performech* control unit that obstructs these vents. It is also important that if the *performech* control unit is rack-mounted adequate space is given between the top of the control unit and other electronics.

If the vents of the *performech* control unit are obstructed, as the heat builds up, the user may start to notice peculiar operation of the software as the various power supplies begin to operate out of the normal range. Eventually, the 5 VDC power supply may fail, which powers all DSP and DAQ boards so instrument operation will terminate. If this occurs, clear the obstructed vents, allow adequate time for the control unit to cool then attempt to restart the system.



**Do not obstruct the vents on the top of the *performech* control unit or the ventilation fans on the rear of the control unit. Obstructing these vents may cause the unit to over heat can cause damage to the internal components.**

---

### Section 1.3.3 Back Panel Overview

The *performech* control unit is designed to combine many external components into one easy-to-operate, computer driven control unit. The *performech* control unit is equipped with a number of input/output BNC connections that are used for additional options/upgrades. Options and upgrades that utilize these auxiliary connections will be addressed in the respective user manuals.

The *performech* control unit includes two transducer connections (each with a 15-pin and a 25-pin connection). Standard systems should have the transducer connected to *Transducer 1* with the 15-pin connection (the 25-pin connection is used only for MRNP/3D OmniProbe systems). There is one orange LED near each of the transducer connections. When the orange LED is illuminated high-voltage is being supplied to the transducer.

Systems that include TriboScan-based *in-situ* imaging (TI 950 TriboIndenter and TI 750 Ubi systems), or which use a TriboScan-controlled piezo for positioning (such as the PI 95 TEM PicoIndenter system) will connect the instrument piezo scanner to the *Piezo* connector.

The *Emergency Stop* BNC should be connected to the stage controller for TI 950 TriboIndenter and TI 750 Ubi systems and left unconnected for TS 75 TriboScope and PI 95/PI 85 PicoIndenter systems (as there are no Hysitron-supplied automated stages).




There are five green LED lights on the rear of the *performech* control unit. The five lights represent different voltages being supplied by the internal power supply. All five green lights should be illuminated when the *performech* controller is operational.

There are three red LED lights on the rear of the *performech* control unit. The top red light is illuminated if no USB connection is detected between the control unit and the computer. The bottom red light is an emergency stop indicator. If the system is being used with an ESP 6000 stage control unit, a lit LED represents normal operation and an unlit LED represents emergency stop. If the system is being used with an ESP 300/301 stage control unit, a lit LED represents an emergency stop and an unlit LED represents normal operation. If the system is a TriboScope, the status of this light is unimportant.

## Section 1.3.4 Specifications

The specifications for the *performech* control unit are given in Table 1.B

Table 1.B  
*performech* specifications

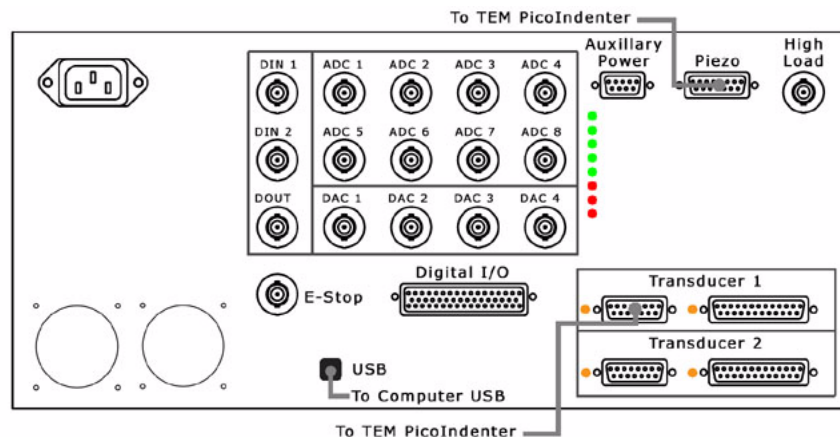
<b>Electrical Requirements</b>	
<b>Voltage</b>	100-240 VAC
<b>Frequency</b>	50/60 Hz
<b>Current</b> (all configurations)	2.5 Amp (max)
<b>Fuses</b> (two located at appliance inlet)	2.5 A, 250 V, Type "T", 5x20 mm
<b>Environmental Conditions (for safe operation of the <i>performech</i> control unit)</b>	
<b>Temperature</b>	5°C - 40°C
<b>Maximum Relative Humidity</b>	80% at 31°C, 50% at 40°C
<b>Altitude</b>	Up to 2000 m
<b>Symbols Related to Safety</b>	
	<i>Warning:</i> Disconnect power input before removing the cover. Only qualified personnel should make adjustments to the instrument.
	<i>Caution:</i> High voltage connection.
	<i>Ground:</i> Indicates the terminal is connected to protective earth ground and chassis ground.

## Section 1.4 Instrument Connections

The Hysitron PI 95 PicoIndenter has been designed to be used with the *performech* control unit (Figure 1.4). There are two connection between the PI 95 PicoIndenter and the *performech* control unit (both being 25-pin cables). Connect the cable labeled *Transducer* to the *Low Load* connection under the *Transducer 1* heading and the *Piezo* cable to the *Piezo* connector.

The DSP *performech* controller connects to the data acquisition computer via a single USB cable.

Figure 1.4  
TI 95 PicoIndenter series  
connection diagram



The transducer and piezo components use similar cable connectors. In order to prevent damage to the TEM PicoIndenter or control unit always verify that the transducer and piezo are connected to their respective locations on the control unit.



Hysitron recommends having the *performech* controller powered on with the TriboScan software running for 20 minutes prior to testing with the PI 95 PicoIndenter.



Cables should not be connected the PI 95 PicoIndenter holder until the head has been installed into the microscope. It is not necessary to power off the *performech* control unit when connecting or disconnecting cables.



## Section 1.5 Data Acquisition Computer

Hysitron instruments have been designed to be software controlled with the data acquisition computer supplied with the instrument. A new Hysitron system data acquisition computer, as of the time this manual was written, will meet or exceed the following specifications.

- Microsoft™ Windows® XP Professional SP2
- Microsoft™ Office 2010
- Intel® Core 2 Duo E7400 2.8 GHz
- 250 GB hard drive
- 3 GB DDR2 RAM or higher
- CD-R/RW drive
- Onboard LAN
- 19" LCD flat panel monitor




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**PI 95/PI 85 systems will be supplied with a well-equipped notebook computer with identical or better specifications as compared to the desktop computer described in this section.**

---

### Computer Features

A list of standard computer cards and features for the various Hysitron systems as well as cards required for additional options is listed below

Table 1.C  
Computer cards and  
requirements for Hysitron  
instruments

	TI 950 TriboIndenter	TI 750 Ubi	TS 75 TriboScope	PI 95/PI 85 PicoIndenter
<b>IEEE-1394 Port</b>	Required		Optional	
<b>Dual Core Processor</b>	Required			
<b>USB 2.0</b>	Required			
<b>Video Display Graphics Card</b>	Required			Not available
<b>1280 x 1024 Screen Resolution</b>	Highly Recommended			
<b>Microsoft Windows Professional XP</b>	Required			
<b>Acoustic Emissions Measurement PCI Card</b>	Optional			Not available




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**Additional PCI cards may be required for systems running legacy stage control units, optical camera systems, or GPIB devices (lock-in amplifier, nanoECR SourceMeter, etc...).**

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Any computer cards, operating system requirements or hardware that is required for the system will be professionally installed and tested for compatibility at Hysitron and should only be serviced by qualified Hysitron personnel. Future options and upgrades may require additional computer cards.

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## Chapter 2 Setup & Calibration

The Hysitron PI 95/PI 85 PicoIndenter is supplied with the TriboScan software suite. TriboScan has been designed to incorporate all basic instrument operation and analysis as well as the operation of any instrument options and upgraded features.

Although TriboScan has been designed to be fully-automated and incorporate the data collection and analysis for many of the available instrument upgrades and options there may be additional stand-alone software packages for some options. Additionally, there may be communication port settings as well as other system settings that must be entered or verified before the instrument or feature will operate normally.

This chapter will discuss the sample installation, probe and transducer installation as well as the system calibrations to keep the instrument performing at its maximum efficiency.

TriboScan, and any associated Hysitron software, is installed in the **C: → Program Files → Hysitron** directory. Important files such as saved load functions and calibration files can all be found within this directory.



**Installing software or licensing the instrument for components that are not installed on the system is not recommended and may cause the instrument to become unstable. If the user is unsure of what software should be installed on the instrument contact Hysitron before proceeding.**

---



**Do not alter any files located within the Hysitron folder unless directed by the user manual or Hysitron service engineer. Modifications of any files within this directory may cause the software to become unstable.**

---

## Section 2.1 PicoIndenter Axes

When combining the Hysitron PI 95 PicoIndenter and the existing TEM system the axis labeling system becomes convoluted. For users with previous indentation experience the labeling system should be intuitive, however, users who experience is primarily with TEM systems may find the PicoIndenter axis system more difficult to comprehend. Special care should be taken to understand the difference in axis orientation so as the instrument can be properly operated without causing damage.

Table 2.A  
PicoIndenter coordinate system

TEM	PicoIndenter
X	Z
Y	X
Z	Y

There is a large knob on the end of the PicoIndenter that controls the motion in the Z-axis direction; clockwise retracts the probe and counterclockwise approaches the sample.

The two smaller knobs on the bottom and left of the PicoIndenter controls the motion in the Y and X-axis, respectively.




---

**The feedback loop within the software should be disabled prior to using any manual control knobs on the PicoIndenter.**

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**The large knob on the end of the PicoIndenter controls the Z-Axis direction.**  
**Clockwise – retracts probe from sample**  
**Counterclockwise – probe approaches sample.**

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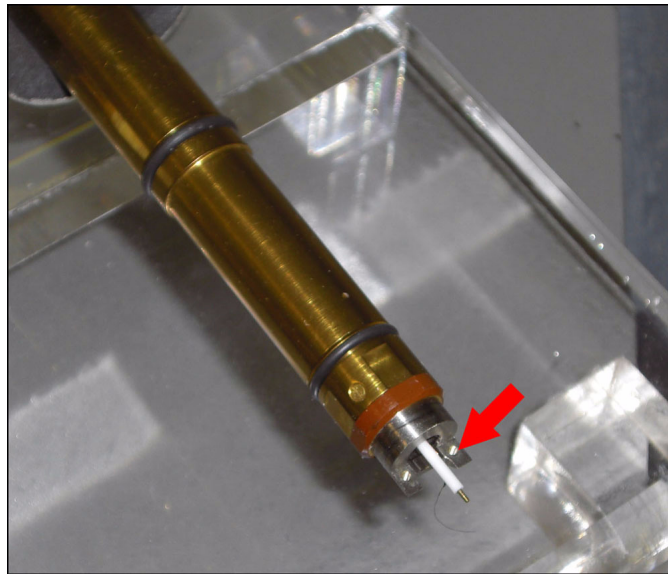
## Section 2.2

### Probe Mounting

Mounting the probe to the transducer is a very delicate process; care should be taken not to over tighten the probe or apply any lateral forces when mounting the probe. The procedure for mounting the probe is outlined below.

1. Turn off TriboScan and disconnect the PI 95 PicoIndenter cables from the *performech* controller.
2. Remove the holder from the TEM system and place the holder on the acrylic resting mount.
3. Wearing rubber gloves and with a small flat-ended screw driver remove the two screws holding the front-end onto the transducer (Figure 2.1).

Figure 2.1  
Location of front-end  
attachment screws




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**Extra care should be exercised to prevent contacting the front-end of the transducer with any foreign objects.**

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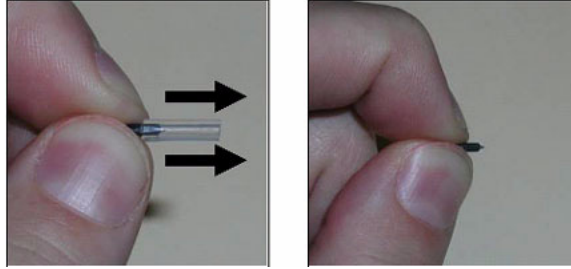

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**Do not modify or adjust any of the O-rings on the TEM PicoIndenter or touch the area between the front O-ring and the probe with ungloved hands as this can compromise the vacuum of the TEM system.**

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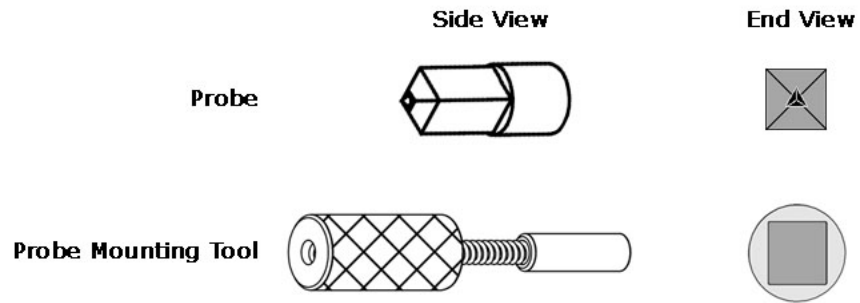
4. Carefully remove the probe from its protective sheath holding the probe in the left hand if the user is right-handed (Figure 2.2).

Figure 2.2  
Removing the nanoindentation  
protective sheath



5. The probe inserts into the probe tool, point first. The probe tool should be held upright at all times. The probe is only held in the tool by gravity and friction, so extra care should be exercised to prevent the probe from falling out of the tool.

Figure 2.3  
Probe mounting tool and probe  
geometry



Use caution when inserting the probe into the probe tool as any contact between the tip of the probe and the installation tool could chip or break the point.

6. Place the threaded end of the probe onto the threads of the TEM PicoIndenter at a slight incline angle and then tilt the tool horizontally to prevent the probe from falling out of the tool.
7. Gently turn the probe counterclockwise until a slight click is felt when the lag of the threads falls into place. This is to avoid cross-threading of the threads of the screw.



Be careful not to cross-thread the probe on the transducer screw. The probe should turn easily. If the probe turns tightly, or becomes stuck, contact Hysitron customer service for advice to avoid damage to the probe or transducer.

8. Gently turn the probe clockwise until the spring of the probe tool begins to deflect.

9. Pull the probe tool straight away from the transducer taking extra care not to contact the tip of the probe with the tool.
10. Reinstall the front-end of the PI 95 PicoIndenter with the two small flat-end screws (Figure 2.1)
11. Follow steps 1 through 10 in reverse order to remove the probe from the transducer.

It is important that the indentation probe is installed fully onto the TEM PicoIndenter. When installed, the probe should be flush against the transducer screw as shown in Figure 2.4b. A probe that was not fully installed is shown in Figure 2.4a.

After the nanoindentation probe has been mounted, the holder can be installed into the TEM (as outlined in the following sections).



**Always perform the probe mounting procedure with the holder laying on its side within the supplied, acrylic resting platform.**

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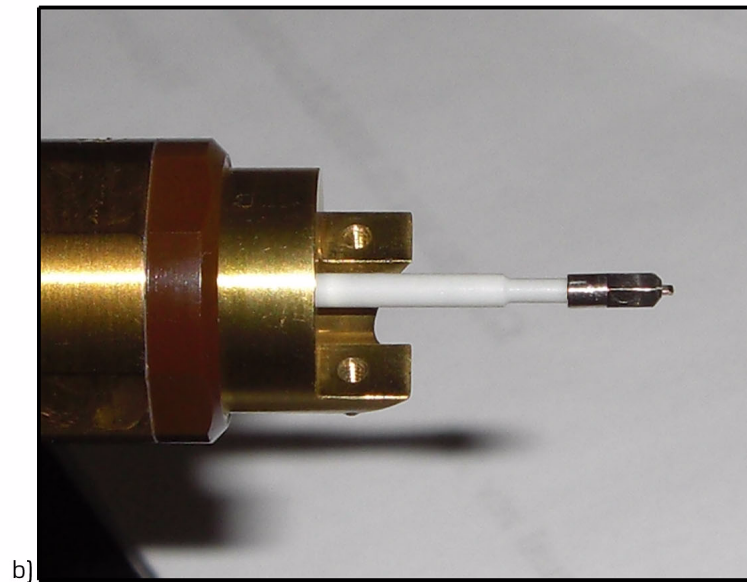
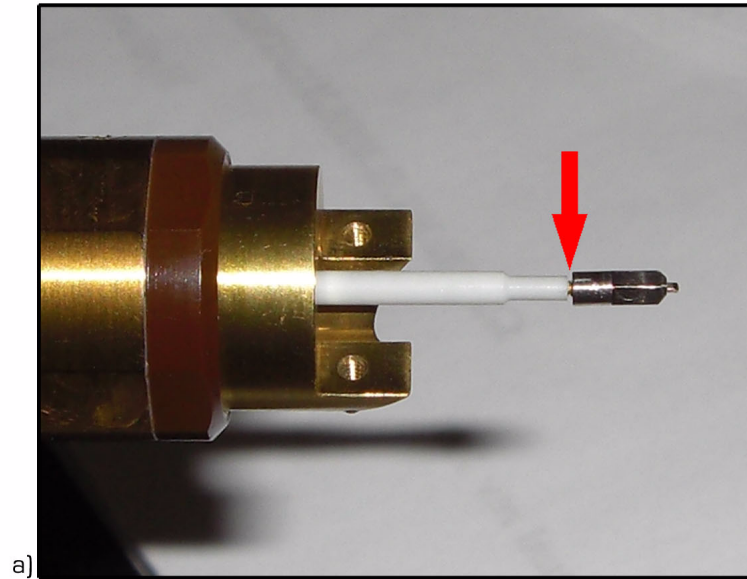


**Never allow the nanoindentation probe tool to hang freely from the transducer. The user should always be supporting the mass of the tool or damage to the finely calibrated springs within the transducer may result.**

---



Figure 2.4  
Improperly (a) and properly (b)  
installed probe



## Section 2.3

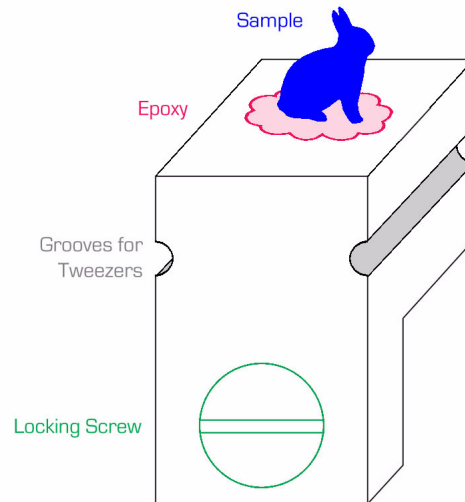
### Sample Mounting

Just as any sample would be prepared for a TEM, the thickness of the area of interest should be less than approximately 300 nm depending on the chemistry of the sample and the accelerating voltage of the TEM. The height of the sample is limited by the travel range of the PicoIndenter axes and if a Hysitron-supplied sample wedge is being used the sample surface should be centered in the field of view. The Hysitron-supplied sample wedges are approximately 0.5 mm thick. Use of thicker sample substrates may cause the sample to be too tall and block the electron beam which would result in no image of the sample surface.

#### Sample Mounting Procedure

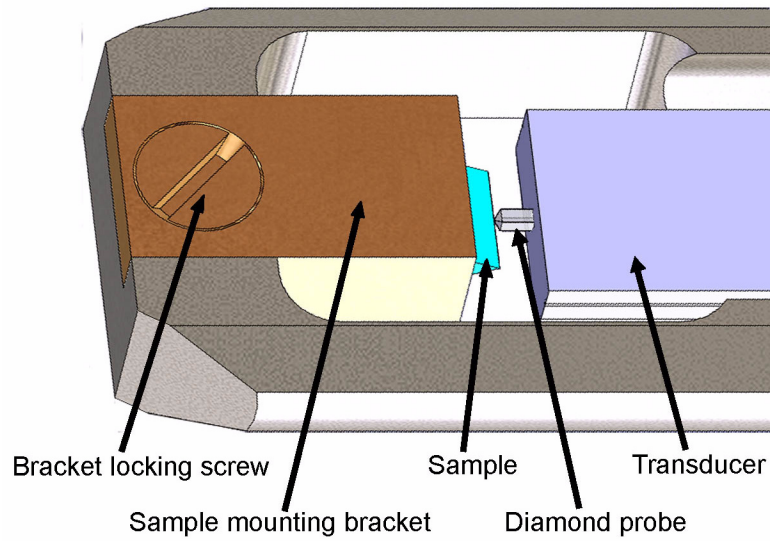
The Hysitron-supplied sample mount consists of a bracket and screw (Figure 3.1). The procedure for mounting samples is given in the procedure below.

Figure 3.1  
PicoIndenter sample mounting bracket



1. Adhere the sample to the bracket. Hysitron recommends using a thin layer of a semi-permanent, adhesive such as Crystalbond™. When adhering the sample to the bracket efforts should be made to center the sample as the X/Y-axis range of the PicoIndenter is limited. The sample should be mounted as level as possible to ensure that the loading axis is normal to the sample surface.
2. Fully retract the indentation probe by turning the knob on the end of the PicoIndenter *clockwise*.
3. Wearing rubber gloves and using tweezers to hold the sample mounting bracket to the front end, tighten the screw that holds the bracket to the front-end (Figure 3.2).

Figure 3.2  
PicoIndenter with sample  
assembly installed



4. Turn the knob on the end of the PicoIndenter *anti-clockwise* to move the probe until it is visibly near the sample.



---

**To prevent damage to the TEM system and/or the PicoIndenter the sample must fit fully within the sample bracket edges.**

---

## Section 2.4 Installing the PicoIndenter

The following is the procedure for inserting the PI 95 PicoIndenter into the TEM. It is assumed the sample has been adhered to the sample mounting bracket and the mounted to the PicoIndenter front-end.

1. Power off the *performech* control unit.
2. Examine the O-rings to ensure that they are free of any lint or particles.
3. In order to simplify the sample approach, before installing the PicoIndenter, use the manual Z-axis knob on the end of the PicoIndenter to bring the probe near the sample. Try to position the probe approximately 100  $\mu\text{m}$  from the sample surface (for comparison, the width of the probe is approximately 200  $\mu\text{m}$ ).



---

**Do not contact the probe with the sample during this manual approach as serious damage may result.**

---

4. Load the PicoIndenter into the TEM as any standard TEM holder would be inserted.
5. Connect the piezo and transducer cables to the PicoIndenter.
6. Connect the transducer cable to the *Transducer 1* connection on the rear of the *performech* control unit.
7. Connect the piezo cable to the *Piezo* connection on the rear of the *performech* control unit.
8. Power on the *performech* control unit (and computer if it is not powered on).
9. Start TriboScan by selecting the TriboScan icon on the desktop or by selecting TriboScan from the Windows start menu.



---

**The cables should be connected between the PicoIndenter and the *performech* control unit so that no unnecessary strain is applied to the electronics or cables.**

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**Cables should be routed away from sources that may induce additional noise such as computer monitors or other electronic devices.**

---

## Section 2.5 Calibrations

With the mechanical portions of the instrument set up the system can be powered on. The order to power on the instrument is unimportant, however, all electronics should be powered on prior to starting TriboScan.



---

**The electronics must be powered on before starting the TriboScan software. If the electronics are not powered on before starting TriboScan, the system will not properly initialize the attached hardware components.**

---

### Section 2.5.1 Loading a Transducer Constants File

When a new transducer is first used, a transducer constants file should be created. A new transducer constants file can be created from the *Calibration* tab → *System Calibration* sub tab. To create a new file, select **File** → **Save as** and create a new file name. All transducer calibration files display the normal and lateral force calibration data. If the transducer being used is a 1D (normal force only) the X-axis constants are unused and may be ignored.

If a transducer constants file has already been created for the transducer, it can simply be loaded from the *Calibration* tab → *System Calibration* sub tab, select **File** → **Open**.

After a transducer constants file has been loaded or created, the proper constants can be copied from the transducer constants sheet provided with the transducer and then fine-tuned with the respective calibration(s).

There are three main areas of the *System Calibration* tab:

#### Transducer Constants

Constants in this area are copied directly from the transducer constants sheet or are properties of the transducer/instrument that WILL NOT be adjusted during the calibrations.

#### Transducer Calibrations

Constants in this area are copied directly from the transducer constants sheet or are properties of the transducer/instrument that WILL be adjusted during the calibrations.


#### System Parameters

Variables in this area are intended to match corresponding hardware settings. This area contains gain and attenuations settings for the system.

## Transducer Constants Sheet

A unique transducer constants sheet is produced each time a transducer is calibrated at the Hysitron factory. An example transducer constants sheet for a lateral force transducer is given in Figure 3.3.

Figure 3.3  
Example transducer constants sheet

 NANOMECHANICAL TEST INSTRUMENTS <b>HYSITRON INCORPORATED</b>	
3/4/2003	
<b>Transducer Calibration Constants</b>	
<b>Transducer ID: SN5-138-61</b>	
<b>Z Axis</b>	
Tare (without the tip)	-329 mg
Load Scale Factor (Force)	1 mV/mg
Displacement Scale Factor (Deflection)	18.350 mV/ $\mu$ m
Electrostatic Force Constant	0.03425 $\mu$ N/V <sup>2</sup>
Self Calibration Check	484 mg
Maximum Force	12.330 mN

Every transducer constants sheet will have the following normal axis parameters located under the *Z Axis* heading of the transducer constants sheet:

### Tare

The *Tare* parameter is a value that indicates the rest position of the micromachined comb drive plate. Verifying the tare value is an important step in starting the instrument and is discussed in the next section of this chapter.

### Load Scale Factor

The *Load Scale Factor* is calibrated at Hysitron and should be entered into the transducer calibration file in TriboScan.

### Displacement Scale Factor

The *Displacement Scale Factor* is calibrated at Hysitron and should be entered into the transducer calibration file in TriboScan.

### Electrostatic Force Constant

The *Electrostatic Force Constant* is calibrated during the *Z-Axis Calibration*. However, TriboScan will require an approximate starting value in order to calibrate the transducer properly. This value should be entered into the transducer calibration file and will be updated by the software following the *Z-Axis Calibration*.

### Self Calibration Check

The *Self Calibration Check* is a troubleshooting tool to verify that the springs that suspend the center plate of the transducer have not been damaged since the transducer has been calibrated. The *Self Calibration Check* is performed by viewing the tare value of the transducer then pausing TriboScan, turning the

transducer upside-down, un-pausing TriboScan and viewing the tare value. The *Self Calibration Check* value should be the difference between these two measured tare values. It is unnecessary to perform the *Self Calibration Check* unless instructed by a Hysitron service engineer.

### Maximum Force

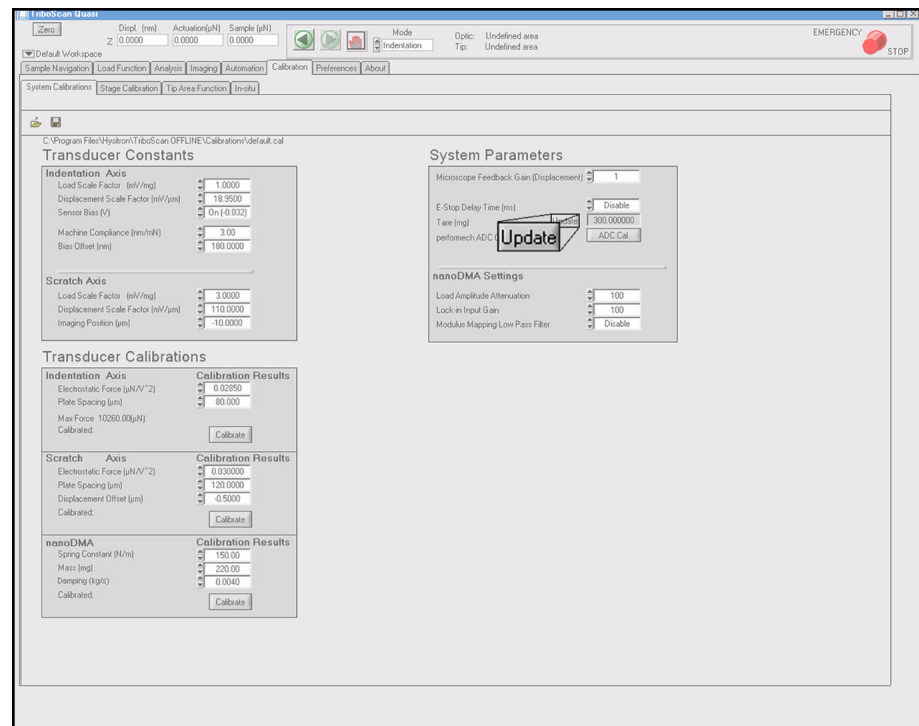
The *Maximum Force* is calculated based on the electrostatic force constant and transducer plate spacing. TriboScan will automatically generate a maximum force following the *Z-Axis Calibration*.

## Section 2.5.2 Checking the Tare Value

The tare value of the system can be read at any time the nanoindentation probe is not in contact with a sample surface. Verifying the tare value is approximately the value given on the supplied transducer constants sheet (typically located within the black transducer case) after the components are installed and the software is started indicates that the transducer and probe are installed correctly and the connections are secure.

To read the tare value of the system, click the *Calibration* tab → *System Calibration* sub tab and click the *Update* button under the *System Parameters* heading (Figure 3.4). The tare value will be displayed in the parameter box to the right of the *Update* button.

Figure 3.4  
Location of *Update* button and  
verification of tare value





The tare value of the system should be verified each time the *performech* control unit is powered on or any hardware components have been removed, replaced or modified.



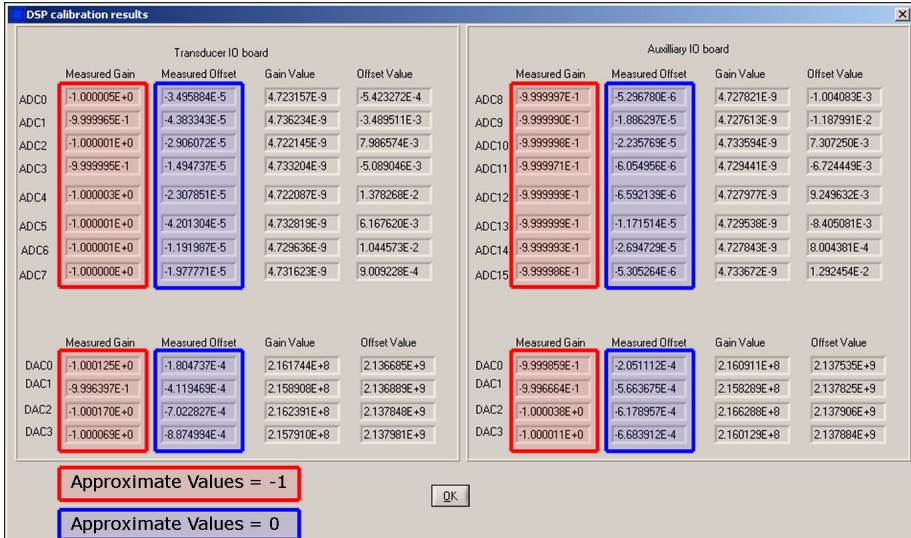
A tare value reading that varies from the transducer constants sheet by more than approximately  $\pm 50$  mg from the recorded value indicates that there may be a problem with the transducer or probe installation.

## Section 2.5.3 ADC Calibration

The *performech* DSP-based controller requires an *ADC Calibration* in order to calibrate the data acquisition boards and the associated gains within the *performech* control unit. The *ADC Calibration* is only required to be performed once for each system configuration (typically performed during the initial installation) but can be performed as frequent as desired to confirm that the gain settings are accurate.

The *ADC Calibration* is accessed from the *Calibration* tab  $\rightarrow$  *System Calibration* sub tab  $\rightarrow$  *ADC Cal* button. After selecting the *ADC Cal* button the system will automatically perform the *ADC Calibration* and generate the *DSP Calibration Results* window (Figure 3.5).

Figure 3.5  
*DSP Calibration Results* window



Transducer ID board				Auxiliary ID board					
	Measured Gain	Measured Offset	Gain Value	Offset Value		Measured Gain	Measured Offset	Gain Value	Offset Value
ADC0	-1.000005E+0	-3.495894E-5	4.723157E-9	-5.423272E-4	ADC8	-9.99997E-1	-5.296780E-6	4.727821E-9	-1.004083E-3
ADC1	-9.999965E-1	-4.383343E-5	4.736234E-9	-3.489511E-3	ADC9	-9.999990E-1	-1.886297E-5	4.727613E-9	-1.187991E-2
ADC2	-1.000001E+0	-2.906072E-5	4.722145E-9	7.986574E-3	ADC10	-9.99998E-1	-2.235769E-5	4.733594E-9	7.307250E-3
ADC3	-9.999995E-1	-1.494737E-5	4.733204E-9	-5.089046E-3	ADC11	-9.999971E-1	-6.054956E-6	4.729441E-9	-6.724449E-3
ADC4	-1.000003E+0	-2.307851E-5	4.722087E-9	1.378268E-2	ADC12	-9.99999E-1	-6.592139E-6	4.727977E-9	9.249632E-3
ADC5	-1.000001E+0	-4.201304E-5	4.732819E-9	6.167620E-3	ADC13	-9.99999E-1	-1.171514E-5	4.729538E-9	-8.405081E-3
ADC6	-1.000001E+0	-1.191987E-5	4.729636E-9	1.044573E-2	ADC14	-9.99993E-1	-2.694729E-5	4.727843E-9	8.004381E-4
ADC7	-1.000000E+0	-1.977771E-5	4.731623E-9	9.009228E-4	ADC15	-9.99998E-1	-5.305264E-6	4.733672E-9	1.292454E-2
	Measured Gain	Measured Offset	Gain Value	Offset Value		Measured Gain	Measured Offset	Gain Value	Offset Value
DAC0	-1.000125E+0	-1.804737E-4	2.161744E+8	2.136689E+9	DAC0	-9.99985E-1	-2.051112E-4	2.160911E+8	2.137535E+9
DAC1	-9.996397E-1	-4.119469E-4	2.159908E+8	2.136889E+9	DAC1	-9.996664E-1	-5.663675E-4	2.158289E+8	2.137829E+9
DAC2	-1.000170E+0	-7.022827E-4	2.162391E+8	2.137849E+9	DAC2	-1.000038E+0	-6.178957E-4	2.166288E+8	2.137906E+9
DAC3	-1.000069E+0	-8.874994E-4	2.157910E+8	2.137981E+9	DAC3	-1.000011E+0	-6.683912E-4	2.160129E+8	2.137884E+9

Approximate Values = -1

Approximate Values = 0

Within the *DSP Calibration Results* window there will be one tab for each of the installed data acquisition cards within the *performech* control unit (the number of data



acquisition cards depends on the instrument options and upgrades). The *Measured Gain* values on all tabs should be  $-1 \pm 0.004$ . The *Measured Offset* values should be near zero (less than  $10^{-3}$ ). The *Gain Value* and *Offset Value* are not used for the *ADC Calibration* and the listed values are unimportant. If any of the values within the *DSP Calibration Result* are not within the given range contact Hysitron for further instruction.



**Do not perform the *ADC Calibration* while the probe is on the sample surface.**

---

## Section 2.5.4 Z-Axis Calibration

The *Electrostatic Force Constant* for the Hysitron micromachined comb drive transducer is determined by the area of the drive plates, the area of the center electrodes and the distances between the two squared. Because the center plate of the transducer is floating on springs, the spacing between the plates can change and thus change the *Electrostatic Force Constant*.

During an indentation test, the center plate (to which the probe is attached) of the transducer is driven into the sample surface. This movement of the center plate will cause a fluctuation in the *Electrostatic Force Constant* and if the transducer has not been properly calibrated a significant amount of error may be present in the results.

TriboScan automatically accounts for changes in the *Electrostatic Force Constant* at large displacements. However, in order to make this correction, TriboScan must know the *Plate Spacing* of the transducer. The *Plate Spacing* and *Electrostatic Force Constant* is given by performing the *Z-Axis Calibration*.

It is important that the *Z-Axis Calibration* be performed every day or each time the instrument is used, whichever comes first. The *Electrostatic Force Constant* is likely to vary slightly from day to day based on factors such as a change in temperature or humidity. Additionally, the *Z-Axis Calibration* is a good indication that the system is working properly and the components are connected securely.

### Performing the Calibration

The calibration is performed with the nanoindentation probe installed in the transducer and the transducer installed in the instrument. The *Z-Axis Calibration* will be performed in the air, far from any sample so it is important that there is adequate space between the nanoindentation probe and any samples or the sample stage.

The transducer is carefully calibrated at Hysitron prior to shipment and will be supplied with a constants sheet. The constants given on the supplied transducer constants sheet must be entered into the *System Calibration* tab. A brief definition of the constants within the TriboScan software is listed below:

#### Load Scale Factor

An inherent property of the transducer, the *Load Scale Factor* is calibrated at Hysitron prior to shipment. *The Load Scale Factor* will not change during this calibration.

**Displacement Scale  
Factor**

The *Displacement Scale Factor* is a measurement of the output voltage of the transducer as a function of the displacement of the center plate. The *Displacement Scale Factor* will not change during this calibration.

**Sensor Bias**

The *Sensor Bias* is a set parameter based on the voltage output from the transducer controller. This value should only be changed at the direction of a Hysitron service engineer and will not change during this calibration. The default is *On (-0.032V)*.

**Machine Compliance**

The *Machine Compliance* is a calculated value for a particular instrument/transducer/probe combination. This value is typically about 0.0 for a TEM PicoIndenter system. The *Machine Compliance* will not change during this calibration.

**Bias Offset**

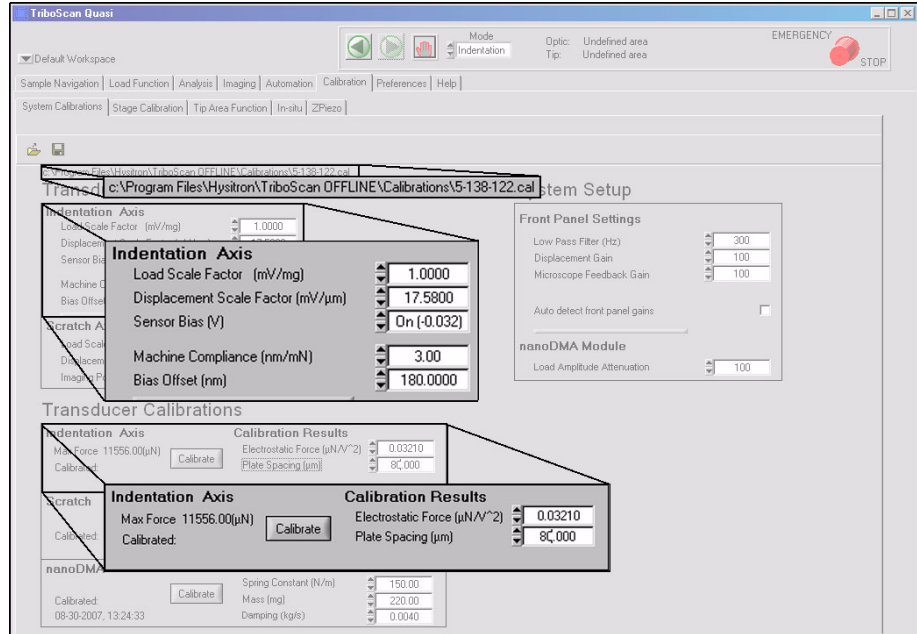
Typically used in cases of adhesion testing or samples that require a large lift-off height. The *Bias Offset* defines the amount of negative displacement available with the transducer. This parameter can be increased, however, increasing the negative displacement will lower the available forward displacement. The *Bias Offset* will not change during this calibration. The default is 100 nm.

**Electrostatic Force  
Constant**

The *Electrostatic Force Constant* is found on the transducer constants sheet and will be a value around  $0.03 \mu\text{N}/\text{V}^2$ . The *Electrostatic Force Constant* must be set to a reasonable value prior to starting the *Z-Axis Calibration* or the calibration may not produce valid results. The *Electrostatic Force Constant* will change during this calibration and may vary from day to day based on environmental conditions.

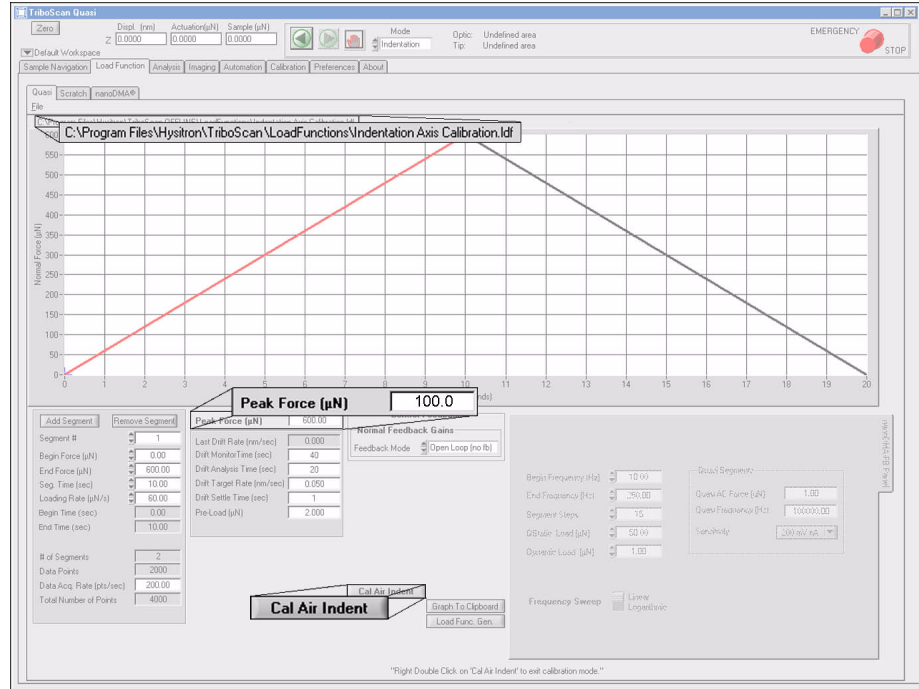
Upon entering or changing any values in the *System Calibration* tab, the file will be saved to the currently open transducer constants file (the file path is given at the top of the *System Calibration* tab). Different calibration files can be accessed by clicking the *Open* icon and a new file can be created by clicking the *Save As* icon.

Figure 3.6  
Verification of Z-axis transducer constants



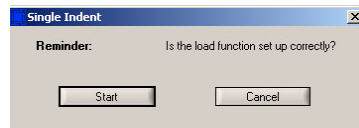
With the transducer constants correctly entered, click the *Calibrate* button located under the *Indentation Axis* heading given in Figure 3.6 to begin the *Z-Axis Calibration* process.

Upon selecting the *Calibrate* button, the system will automatically open the *Load Function* tab → *Indent* sub tab (Figure 3.7).

Figure 3.7  
 Z-Axis Calibration load function


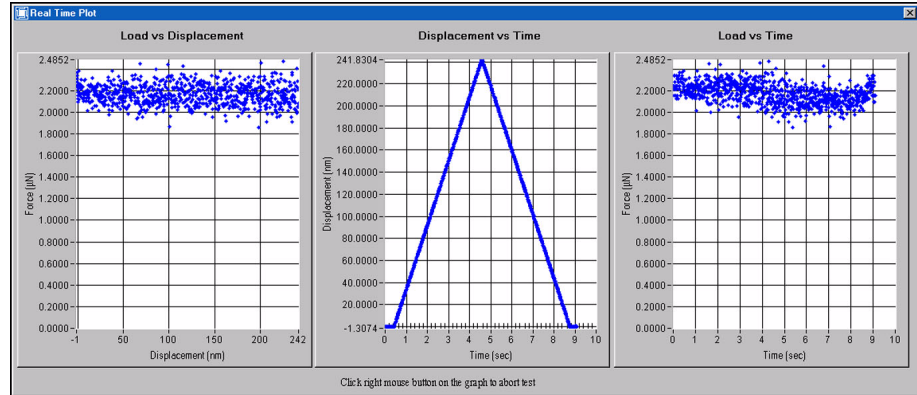
When the system is in the *Z-Axis Calibration* mode, the user can double right-click on the *Cal Air Indent* button to return to normal system operation.

Verify that the current load function is the *Indentation Axis Calibration.ldf* with a *Peak Force* of 600 µN. The maximum required force will vary for different transducers; the given force should achieve a displacement of approximately 3-4 µm. Click the *Cal Air Indent* button and the *Z-Axis Calibration* reminder window (Figure 3.8) will open. Click *Start* to continue with the calibration.

 Figure 3.8  
 Z-Axis Calibration reminder  
 window


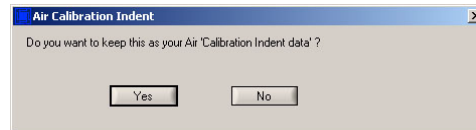
Upon clicking *Start* in Figure 3.8, an indentation test will be performed in the air and the real-time plot will be displayed similar to Figure 3.9

Figure 3.9  
Real-time plot of load vs. displacement, displacement vs. time and load vs. time for the *Z-Axis Calibration*



When the real-time plot has finished, Figure 3.10 will open prompting the user to keep or discard the calibration information. If the calibration has been performed successfully, click *YES*. If a setting was overlooked and the calibration was unsuccessful, click *NO*, locate the source of the unsuccessful calibration and re-perform the *Z-Axis Calibration* starting from the beginning of this procedure.

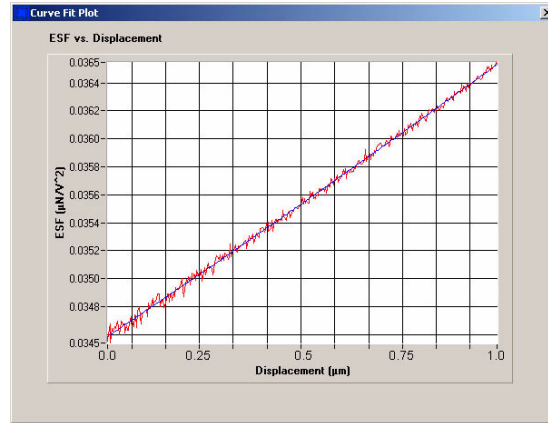
Figure 3.10  
*Z-Axis Calibration* confirmation window



After clicking *YES* in Figure 3.10 the ESF versus displacement plot (Figure 3.11) will open to show the actual data collected during the *Z-Axis Calibration* (shown in red) with respect to the fitted linear plot (shown in blue). The red plot should be very close to the blue plot and the total displacement should be approximately 1 µm. Any significant deviation between the two plots will result in a poor calibration and may indicate further problems within the transducer or other system components.

Displacements greater than about 1 µm may be exceeding the transducer displacement limit and the calibration should be performed at a lower force. Displacements less than about 0.5 µm typically have not exercised the sensor enough to collect a reasonable amount of displacement data for the calibration. Typically, very low displacement calibrations will appear much noisier due to the reduced calibratable range.

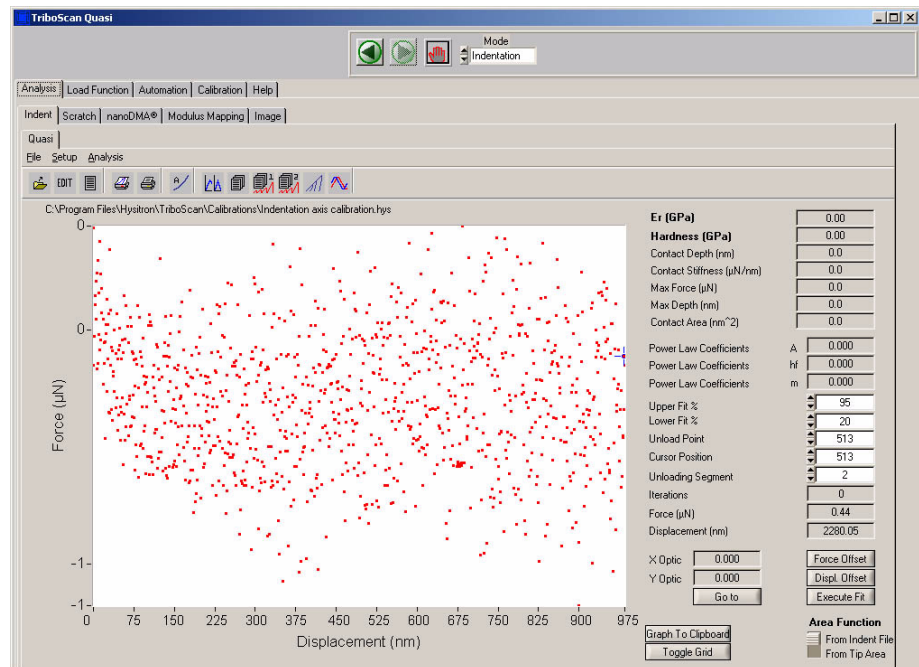
Figure 3.11  
Z-Axis Calibration ESF vs.  
displacement plot



**Do not attempt to approach a sample for testing without first obtaining a satisfactory Z-Axis Calibration and ESF versus displacement plot.**

The Z-Axis Calibration will finish with the result being displayed in the *Analysis* tab → *Indent* sub tab. The result should be a scatter of data points around 0 µN and a displacement of approximately 1 µm (Figure 3.12).

Figure 3.12  
Z-axis calibration result



The Z-Axis Calibration is now complete and the user can proceed with additional calibrations (if necessary, depending on the instrument features) or continue with sample testing.

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# Chapter 3 Software

To start the TriboScan software package, double click the TriboScan icon on the desktop or select the corresponding choice from within the **Start Menu → Programs → TriboScan**. If various features are installed on the system, the software may prompt the user regarding which mode he/she would like to run the software in. After the initialization of the software is complete, the *Analysis* tab will be the active window.

TriboScan version 8 and higher is operated by *tabs* within the TriboScan software. The function of each of these tabs as well as an explanation of the available parameters and features is given in this chapter. The tab scheme was created as an intuitive ‘filter’ method to allow the user to easily find complex features by starting from basic fields.



**Problems starting TriboScan? Confirm that the all electronic components are powered on prior to starting TriboScan.**

---



**Before any testing is begun, the user must be comfortable with the material presented through this chapter of the user manual. Procedures must be followed as outlined in this manual or at the instruction of a Hysitron representative to prevent instrument damage and obtain desirable testing results.**

---

## Section 3.1 The Action Bar

The *Action Bar* contains many of the commonly used global features of the TriboScan software as well as displaying the system status and current testing mode. The *Action Bar* contains:

- *Zero* button and transducer status parameters
- *Back & Forward* shortcut buttons
- *Mode* pull-down menu
- *Emergency Stop* button

Figure 4.1  
TriboScan *Action Bar*



### Section 3.1.1 Zero Button & Transducer Status

The *performech* DSP-based control unit monitors and records the transducer status at all times. The transducer status values display the current status of the transducer at any given time. The *Zero* button must be selected before approaching the sample surface. There are three transducer status parameters and one *Zero* button given on the *Action Bar*:

#### Z Displacement (nm)

The *Z Displacement* status parameter displays the current displacement of the nanoindentation probe in the Z-Axis direction. The *Z-Displacement* status parameter will automatically be zeroed by the software prior to performing any test, however, the user can zero this value at anytime by selecting the *Zero* button.

#### Actuation (μN)

The *Actuation* status parameter displays the current measured force being applied to the transducer Z-Axis. This measured force includes any bias that is applied to the transducer. The *Actuation* status parameter is not affected by the *Zero* button.

#### Sample (μN)

The *Sample* status parameter displays the current Z-Axis force being applied by the system upon a sample for performing a nanoindentation test. The *Sample* status parameter is automatically zeroed by the software prior to performing a test, however, the user can zero this value at anytime by selecting the *Zero* button.

#### Zero Button

The *Zero* button can be used to bring the *Z Displacement* and *Sample* parameters to the zero position. The *Zero* button should be selected each time before a sample is tested to zero the Z-Axis displacement.

### Section 3.1.2 Back & Forward Shortcut Buttons

The *Back* and *Forward* shortcut buttons operate similar to many of today's popular internet browsers. The *Back* and *Forward* shortcut buttons are intended to allow the user to easily navigate common and frequently used tabs by allowing simple, chronological navigation.

### Section 3.1.3 Mode Pull-Down Menu

The *Mode* pull-down menu determines what type of test the system is capable of performing at any given time. Depending upon how the instrument is licensed there will always be the option for *Indentation* and there may be the option for *Tensile*, *nanoDMA*, *nanoECR*, etc...

### Section 3.1.4 Emergency Stop Button

The software emergency stop is located at the far right of the *Action Bar* in TriboScan. Clicking the *Emergency Stop* button will halt all motion in the X/Y and Z-axis (for all PI 85 systems and PI 95 systems with motorized movement). When the software emergency stop is activated (depressed), a button labeled *Enable Motors* will appear over the top of the *Emergency Stop* button. The motors must be enabled by selecting the *Enable Motors* button before the user can continue to operate the software.



---

**For PI 85/95 PicoIndenter systems the emergency stop can only be initiated by the user and there is no system automated emergency stop.**

---

## Section 3.2 The Analysis Tab

The *Analysis* tab contains the analysis tools and functions for most data collected with Hysitron systems. Depending on the options and upgrades that the instrument has been equipped with, additional sub tabs may be available from the *Analysis* tab, each of which would be discussed in the respective option or upgrade user manual.

The *Analysis* tab contains the following sub tabs:

### Indent

The *Indent* sub tab is composed of two additional sub tabs:

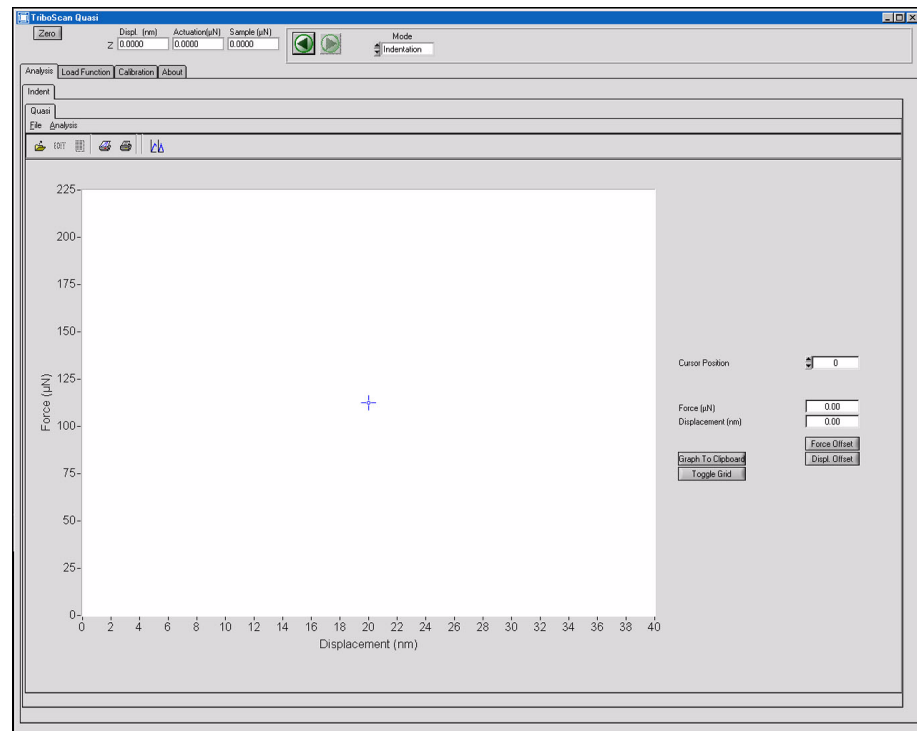
- *Quasi*

All quasistatic indentation analysis occurs from within the *Indent* sub tab. This sub tab is standard on all instruments.

- *nanoECR*

All quasistatic indentation data the contains nanoECR information is analyzed from this sub tab. This sub tab is optional and will be available only on instruments equipped with nanoECR.

Figure 4.2  
*Analysis* tab



## Section 3.2.1 The Indent Sub Tab

The quasistatic indentation analysis (accessed from the *Analysis* tab → *Indent* sub tab → *Quasi* sub tab) is used for all open-loop, load and displacement controlled indentation analysis. Many of the shortcut icons on the menu bar at the top of the *Indent* sub tab → *Quasi* sub tab are also located in the command bar and are defined below:



*Open File* (also available from **File** → **Open**)

The *Open File* command allows the user to open any previously saved \*.hys quasistatic nanoindentation file.



*Edit File* (also available from **File** → **Edit File**)

The *Edit File* command allows the user to view and/or edit any previously saved \*.hys quasistatic nanoindentation file constants values. Editable values include:

- Spring Force Compensation
- Drift Rate
- Load Scale Factor
- Displacement Scale Factor
- Electrostatic Force Constant
- Machine Compliance
- Displacement Gain
- Plate Spacing
- Number of Data Points to Average



*Export Text File* (also available from **File** → **Export Text File**)

The *Export Text File* command creates a tab delimited text file of the current \*.hys file so that the data can be viewed by third party programs. The text file includes the load and displacement information with respect to time.



*Print Graph* (also available from **File** → **Print Graph**)

The *Print Graph* command prints the currently displayed plot as viewed on the monitor.



*Print Window* (also available from **File** → **Print Window**)

The *Print Window* command prints the currently displayed window including any fitting information and calculated values.



*Plot vs. Time* (also available from **Analysis** → **Plot vs. Time**)

The *Plot vs. Time* command displays the currently open \*.hys file as force and displacement over time. This is a useful troubleshooting tool as well as useful for certain analysis routines.

The *Indent* sub tab has additional options that do not have a corresponding icon:

**File → Export Multiple Text Files**

The **Export Multiple Text Files** command allows the user to select several \*.hys files to export as tab delimited text files to be used by third party programs.

**File → Update Multiple Files**

The **Update Multiple Files** command works similar to the *Edit* icon but will update several files at the same time. To use the **Update Multiple Files** command:

1. Open one of the \*.hys file to be updated.
2. Go to the *Calibration* tab → *System Calibration* sub tab.
3. Enter ALL transducer constants as desired for the files to be updated.
4. Go to the *Analysis* tab → *Indent* sub tab → *Quasi* sub tab → **File → Update Multiple Files**. When prompted select the files to be updated.
5. Open a \*.hys file that was updated and click the *Edit* icon, the *Current Parameter Values* should read the desired updated values.




---

Using the *Update Multiple Files* command will modify the \*.hys file(s) with all parameters listed in the *Calibration* tab → *System Calibration* sub tab. This includes all transducer constants, *Plate Spacing*, *Electrostatic Force Constant* and all gain settings which typically vary with environmental changes or testing routines.

---

The *Analysis* tab → *Indent* sub tab → *Quasi* sub tab also contains six buttons and one toggle switch:

**Force Offset**

The *Force Offset* button allows the user to select a new point to be the origin of the force axis. This option is primarily intended for use with feedback-controlled tests that have a pre-test lift height and may incorrectly register the point where the probe contacts the sample surface as the zero force value.

**Displ. Offset**

The *Displ. Offset (Displacement Offset)* button allows the user to select a new point to be the origin of the displacement axis. This option is primarily intended for use with feedback-controlled tests that have a pre-test lift height and may incorrectly register the point where the probe contacts the sample surface as the zero displacement value.

**Graph to Clipboard**

The *Graph to Clipboard* button copies the currently displayed plot onto the computer clipboard so that it may be copied into a third party program.

**Toggle Grid**

The *Toggle Grid* button cycles through the available grid options for the displayed plot. Options include: no grid, vertical grid, horizontal grid and horizontal/vertical grid.

**Cursor Position**

The *Cursor Position* is given as the currently selected point on the plot as chosen by the user.

**Force**

The *Force* parameter displays the force for the currently selected point in the plot.

**Displacement**

The *Displacement* parameter displays the displacement for the currently selected point in the plot.



## Section 3.3 The Load Function Tab

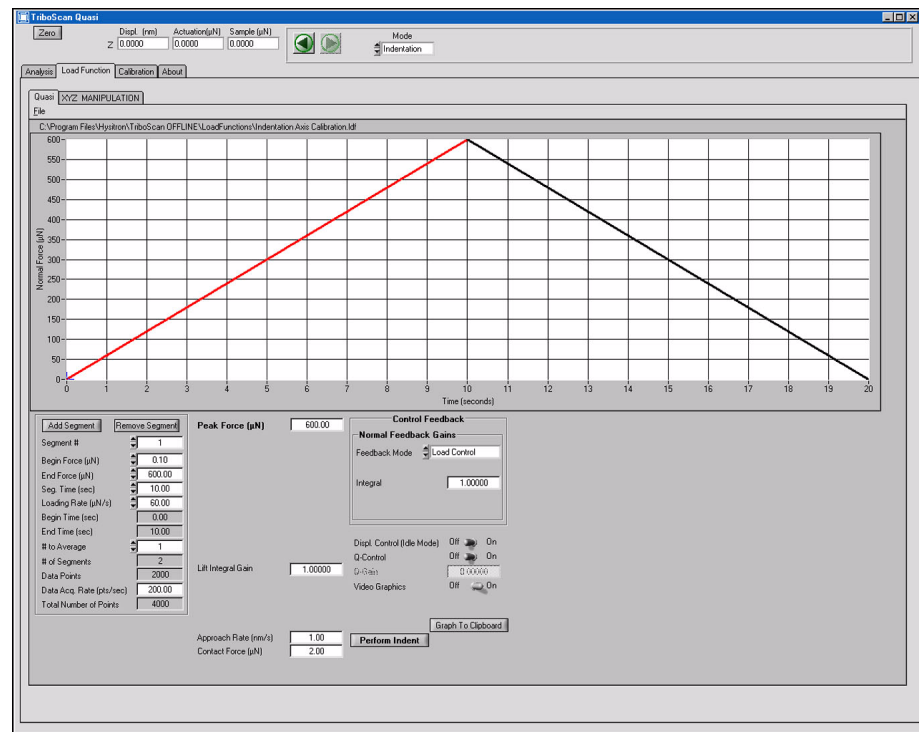
The *Load Function* tab contains the tools necessary for creating any of the available nanomechanical tests available with the given instrument. Depending on the options and upgrades that the instrument has been equipped with, additional sub tabs may be available from the *Load Function* tab, each of which would be discussed in the option or upgrade respective user manual.

The *Load Function* tab contains the following sub tabs:

### Indentation

Quasistatic nanoindentation load function editor. This sub tab is standard on all instruments.

Figure 4.3  
Load Function tab



### Section 3.3.1 The Indentation Sub Tab

The quasistatic load function editor (accessed from *Load Function* tab → *Indentation* sub tab) is used to create all standard indentation tests. The load function can be saved at any time by clicking **File** → **Save**, likewise, a previously saved file can be opened by clicking **File** → **Open**. Triboscan is pre-loaded with some basic load functions located at C: → **Program Files** → **Hysitron** → **Triboscan** → **Load Functions**.



Save time by modifying pre-made load functions to meet your testing needs. TriboScan comes loaded with several standard quasistatic nanoindentation load functions. Open any of these load functions and modify the segment times, loads and number of segments instead of creating load functions from scratch.

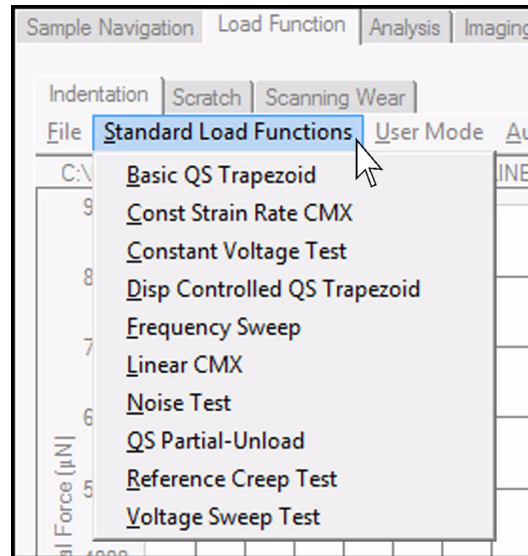
### *Standard Load Function menu*

The *Standard Load Function* menu (Figure 4.4) allows easy access to some of the most commonly used load functions including a basic quasistatic trapezoid, and noise test.



The *Standard Load Function* menu is only intended as a shortcut to some of the most commonly used load functions. Users can continue to create, edit, or modify load functions as discussed in later sections of this user manual.

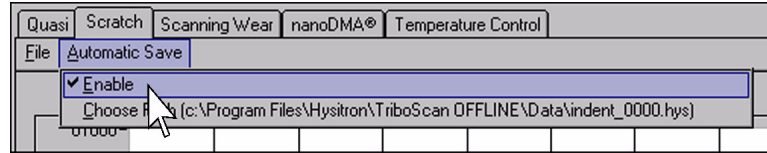
Figure 4.4  
*Standard Load Function menu*



### *Automatic Save menu*

The **Automatic Save** menu item allows the user to define a path and base file name for the tests that will be performed. With the *Automatic Save* feature activated (with a check, similar to Figure 4.5) when a test has been performed it will automatically be saved to the given directory with the given base file name and a test number (i.e., test\_000, test\_001, test\_002, etc...). This feature prevents the user from having to name each individual file following a test and will speed the individual test process.

Figure 4.5  
Automatic Save menu



### Indentation sub tab parameters

The normal force vs. time plot will be updated after any change have been made in the parameters below the load function plot. When a segment is selected by a single left-click, the parameters below the plot will adjust to reflect the properties of that particular segment.

There are several buttons on the *Indentation* sub tab as shown in Figure 4.3. The buttons are described below:

#### Add Segment

Clicking the *Add Segment* button will add a ten-second, constant load or displacement segment immediately to the right of the currently selected segment. The user can select a segment by clicking on the segment in the normal force or displacement vs. time plot.

#### Remove Segment

Clicking the *Remove Segment* button will remove the currently selected segment. The first and last segment can not be removed. There must always be at least two segments and the beginning and ending force for the entire function must be 0.0  $\mu\text{N}$ . The software will not allow the user to remove a segment that violates these truths.

#### Graph to Clipboard

Clicking the *Graph to Clipboard* button copies the currently displayed normal force or displacement vs. time plot to the computer clipboard so that it may be pasted into any other available program.

#### Air Indent

The *Air Indent* button will perform the currently defined test a the current probe position (no drift correction and no sample approach is performed).

#### Perform Indent

Clicking the *Perform Indent* button will perform the defined test at the current probe position.

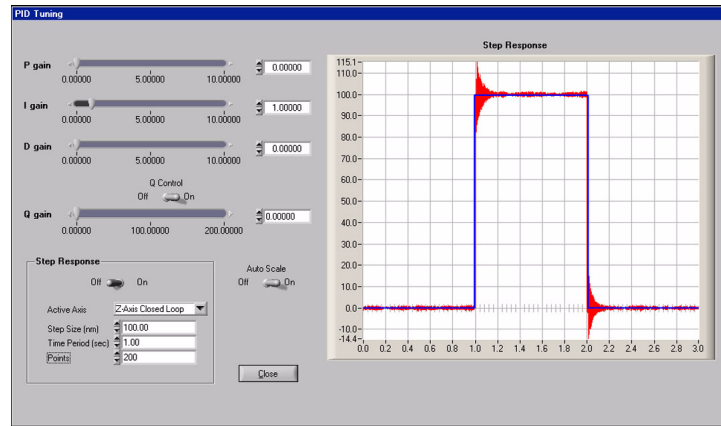
#### Tune Displ. PID

(displacement control testing only) The *Tune Displ. PID* button opens the *PID Tuning* window (Figure 4.6). The *PID Tuning* window is used to tune the proportional, integral and derivative gain settings properly. The *PID Tuning* window is also used to tune the *Q Control* gain setting.

The *PID Tuning* window allows the user to modify and toggle the step response on and off. When the step response is enabled a desired blue step response will appear in the *Step Response* plot and a red (measured data) plot will appear over the plot. It is desirable for the red and blue plots to follow closely (as shown in Figure 4.6). The PID gains can be modified in real-time while the result continues to update on the *Step Response* plot. When satisfied with the gain settings, click the *Close* button and the software will prompt the user if they

would like to save the current gain settings. The gain setting will automatically be populated in the *Load Function* tab.

Figure 4.6  
PID Tuning window

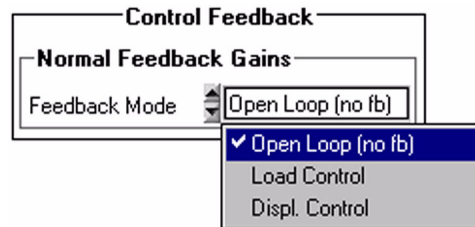


**Load Func. Gen.**

Clicking the *Load Func. Gen.* button will launch the load function creator software which allows the user to create complex partial unload functions. The *Load Function Generator* is discussed further in the *Testing* chapter → *Partial Unload & Constant Strain Rate* section of this user manual.

In the lower center section of the *Indentation* sub tab there is a *Control Feedback* pull-down menu that the user can select different feedback controlled options (Figure 4.7) including *open-loop*, *load* and *displacement* control testing.

Figure 4.7  
Control Feedback pull-down menu



By selecting the different types of quasistatic nanoindentation tests from the *Control Feedback* pull-down menu (*Open Loop*, *Load Control* or *Displacement Control*) the load function parameters will change. A description of the parameters follows:

**Proportional Gain**

(displacement control only) Displacement control tests are inherently more difficult in forcing the test results to follow the desired testing load function. The *Proportional Gain* looks at where the actual result is in relation to the desired result at any given time (present error). Increasing the *Proportional Gain* may result in a quicker response but increased too much may cause oscillations. Default value is 0.00.

**Integral Gain**

(load and displacement control only) This field allows the user to define the *Integral Gain*. This gain has the biggest effect on how well the test follows the

load function. The *Integral Gain* looks at where the actual result was in relation to the desired result (past error) and uses this information to make corrections and bring the actual result closer to the desired result. Increasing the *Integral Gain* forces the actual result to follow the desired result more closely, however, too much *Integral Gain* may cause instabilities in the system and too little will result in a sluggish response time. Default value is 1.00 for both load and displacement control testing.

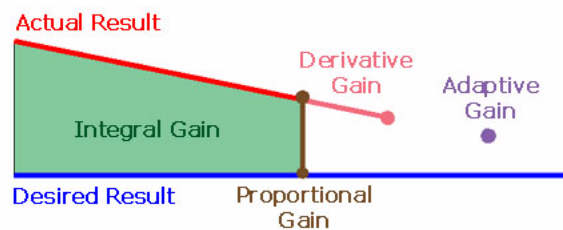
#### Derivative Gain

(displacement control only) Displacement control tests are inherently more difficult in forcing the test results to follow the desired testing load function. The *Derivative Gain* looks at where the actual result is going to be in relation to the desired result at any given time (prediction of future error based on current rate of change). The *Derivative Gain* works to limit the amount of overshoot; however, increasing the *Derivative Gain* by too much may result in a slower response. Default value is 0.00.

#### Adaptive Gain

(displacement control only) Displacement control tests are inherently more difficult in forcing the test results to follow the desired testing load function. The *Adaptive Gain* is a feed-forward control that can quickly adapt to changes in the test to regain stability. Decreasing the *Adaptive Gain* may result in a slower response for complex load functions and an increased *Adaptive Gain* may will force complex load functions to follow the desired result more closely. Default value is 0.00.

Figure 4.8  
Illustration of PID gain operation



#### Q-Control

In a high vacuum/low air damping environment, there is an increase in the mechanical amplification at the resonance frequency and an increase in the overall settling time of the transducer. In addition to long settling times while operating in open-loop control mode, the closed-loop control of the test may become quite unstable due to this effect. To solve the problems inherent in performing nanomechanical testing in a high vacuum environment, increasing the system damping is highly desired. The Hysitron PI 95 has a Q value (mechanical quality factor) of ~6000 in high vacuum and it is recommended to decrease the Q value by 1000 times to achieve reasonable control.

To effectively use the *Q-Control* mode, the *Q-Gain* must be tuned in a manner similar to tuning the PID gains for closed-loop displacement control mode. From the PID Tuning panel, change the active axis to *Z-Axis Open Loop*. In open loop mode, the transducer will begin to oscillate due to the low damping vacuum environment. Turn on the *Q-Control* switch and increase *Q-Gain* until the desired performance is achieved. *Q-Control* will actively dampen the transducer oscillation. Note that if the *Q-Gain* is set too high the transducer may become unstable. Once you have tuned the *Q-Gain*, it is recommended to do a few air

indents to make sure there are no visible issues with transducer instability. If the transducer appears to become unstable during the indent the *Q-Gain* should be decreased.

#### Q-Gain

The *Q-Gain* is the gain setting used when the *Q-Control* is enabled. The *Q-Gain* can be tuned by selecting the *Tune Displ. PID* button when the system is set in displacement control mode.




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**For MEMs-based PI\_95 systems the resonance frequency of the system is much higher and the *Q-Control* and *Q-Gain* is typically not needed while working in vacuum.**

---

#### Video Graphics

The *Video Graphics* toggle switch enables the indentation video overlay so that the indentation plot and SEM video can be viewed (more information can be found in the *Analysis* chapter). Setting the *Video Graphics* toggle switch to *Off* will save only a static \*.hys indent plot and will have no references to the SEM video. Setting the *Video Graphics* to *On* will save a static \*.hys file as well as an \*.avi video file with the same file name in the same directory (if connected).

#### Lift Integral Gain

(load control only) The *Lift Integral Gain* field allows the user to adjust the gain for the system while the probe is approaching the sample surface to perform a load control test. If the *Lift Integral Gain* is set too high, the system may become unstable during the lift height, however, if the *Lift Integral Gain* is set too low the probe may not lift fully from the sample surface. Default is 1.0.

#### Approach Rate

(load control only) The *Approach Rate* field allows the user to set the rate for the system to approach the sample while performing a load control test. When the PicoIndenter performs a load control test, the system approaches the sample in displacement control mode at the defined *Approach Rate* and stops when the defined *Contact Force* is sensed at which time the load control test is begun. The default value of 1 nm/sec is typically too slow for most tests and it is recommended to increase this based on the distance the probe is starting from the surface (typically 10-50 nm/sec).

#### Contact Force

(load control only) When a load control test is performed, the system approaches the sample in displacement control mode until the defined *Contact Force* is sensed. When the *Contact Force* is sensed, the displacement control approach stops and the defined load control test begins. The default *Contact Force* of 2  $\mu\text{N}$  will work well for a quiet system but if there is excessive instrument, stage, or building noise the *Contact Force* may need to be increased.

#### App. Timeout

(load control only) The *App. Timeout* parameter allows the user to define a time to start the test if the approach time is too long. The default value of 1.0 sec is typically much too short (as the test would typically start in the air above the sample) so it is recommended to set the *App. Timeout* to a much larger value like 200 sec to ensure the probe has adequate time to contact the sample surface.




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**For more information regarding the integral, proportional, derivative and adaptive gains, refer to the *Testing* chapter of this user manual.**

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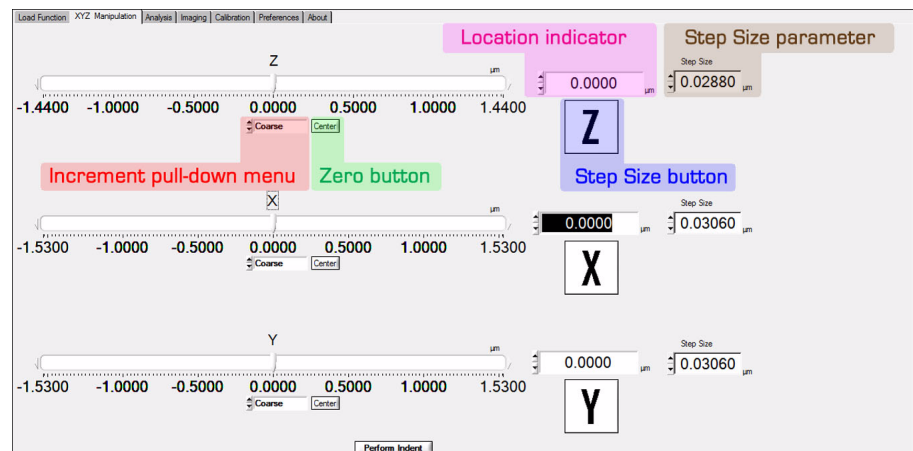
Peak Force/Peak Displacement	The <i>Peak Force</i> or <i>Peak Displacement</i> field is used to enter the load function maximum force (open loop or load controlled test) or displacement (displacement controlled test) for the entire load function.
Begin Force/Begin Disp.	The <i>Begin Force</i> or <i>Begin Displacement</i> field is used to enter the force or displacement for the selected segment to begin. The software will require that the user define the same value as the ending force or displacement for the previous segment (or zero for the first segment of the load function).
End Force/End Disp.	The <i>End Force</i> or <i>End Displacement</i> field is used to enter the force or displacement for the selected segment to end. The software will require that the user define the same value as the beginning force or displacement for the following segment (or zero for the last segment of the load function).
Segment Time	The <i>Segment Time</i> field allows the user to define a length of time for the selected segment. The loading rate will be automatically calculated.
Loading Rate	The <i>Loading Rate</i> field allows the user to define a loading rate for the selected segment. Segment time will automatically be calculated.
Begin Time	The <i>Begin Time</i> is the time that the selected load function segment will begin. This field is automatically populated and cannot be edited.
End Time	The <i>End Time</i> is the time that the selected load function segment will end. This field is automatically populated and cannot be edited.
# to Average	The <i># to Average</i> field allows the user to define how to average the collected data. <i>One</i> corresponds to no averaging (raw data). <i>Two</i> corresponds to every two data points being averaged, yielding less data but a smoother plot, and so on.
# of Segments	The <i># of Segments</i> displays the number of segments for the open load function. This field is automatically populated and cannot be edited.
Data Points	The <i>Data Points</i> parameter displays the number of data points that will be collected during the currently selected load function segment. The <i>Data Points</i> is calculated based on the defined <i>Data Acq. Rate</i> and the time for the selected load function segment.
Data Acq. Rate	The <i>Data Acq. Rate</i> parameter allows the user to define how many data points to collect per second for the duration of the test. The default is 200 pts/sec.
Total Number of Points	The <i>Total Number of Points</i> parameter displays the number of data points that will be collected for the entire test. This is calculated based on the <i>Data Acq. Rate</i> and the total test time.

## Section 3.4 XYZ Manipulation tab

The *XYZ Manipulation* tab (Figure 4.9) controls the position of the X, Y and Z-axis piezo elements within the PI 95 PicoIndenter. The *XYZ Manipulation* tab is used for precisely positioning the probe of the PicoIndenter with respect to the sample prior to running a test.

The X, Y and Z-axis piezo can be manipulated by clicking the small arrows on either side of the slider bar, by clicking and dragging the slider bar or by right and left-clicking the large *Step Size* button.

Figure 4.9  
*XYZ Manipulation* sub tab



Each of the X, Y and Z axes has an identical set of controls including:

**Increment pull-down menu**

The *Increment* pull-down menu allows the user to select the scale of the positioning slider bar. Selecting coarse offers the full range of the piezo element. Selecting medium or fine offers a smaller range with greater accuracy.

**Zero button**

The *Zero* button is used while operating the slider bars in medium or fine mode. Because the entire range of the piezo is unavailable while using the medium or fine increment, clicking the *Zero* button establishes the current cursor position as the new origin thus extending the available range for the piezo elements to move in medium or fine mode. The entire range of the piezo element is available in coarse mode so the *Zero* button has no function while the axis is set in coarse mode.

**Location indicator**

The *Location* indicator displays the current value of the slider bar.

**Step Size button**

The *Step Size* button moves the piezo the defined *Step Size*. The button is intended to be used by placing the mouse on the button, then left-clicking to move negative and right-clicking to move positive. The large, easy to locate button is intended to help users operate the piezo positioning while focusing on the sample or probe approaching the surface.



**Step Size parameter**

The *Step Size* parameter is the user-defined amount that left or right-clicking on the *Step Size* button will move the piezo element in either the positive or negative direction.

**Perform Indent**

Clicking the *Perform Indent* button will perform the defined test at the current probe position.



---

**Extra care should be taken to prevent the probe from contacting the sample or other surfaces while operating the X, Y and Z-axis piezo elements.**

---

## Section 3.5 The Calibration Tab

The *Calibration* tab contains the calibrations that are required for keeping Hysitron systems running properly. Depending on the options and upgrades that the instrument has been equipped with, additional sub tabs may be available from the *Calibration* tab, each of which would be discussed in the option or upgrade respective user manual.

The *Calibration* tab contains the following sub tabs:

### System Calibrations

The *System Calibrations* tab is available with all Hysitron instruments and is used to perform the calibrations necessary to verify the transducer is operating properly and account for small changes in transducer properties that may be caused by temperature, humidity or changing probe mass.

### *in-situ*

The *in-situ* tab contains the tools for calculating, saving and opening calibration files for the piezo scanner that is used for fine positioning of the probe. The *in-situ* tab also offers *X/Y/Z Piezo Correction Factors* to help account for offsets in different directions when activating a specific axis.

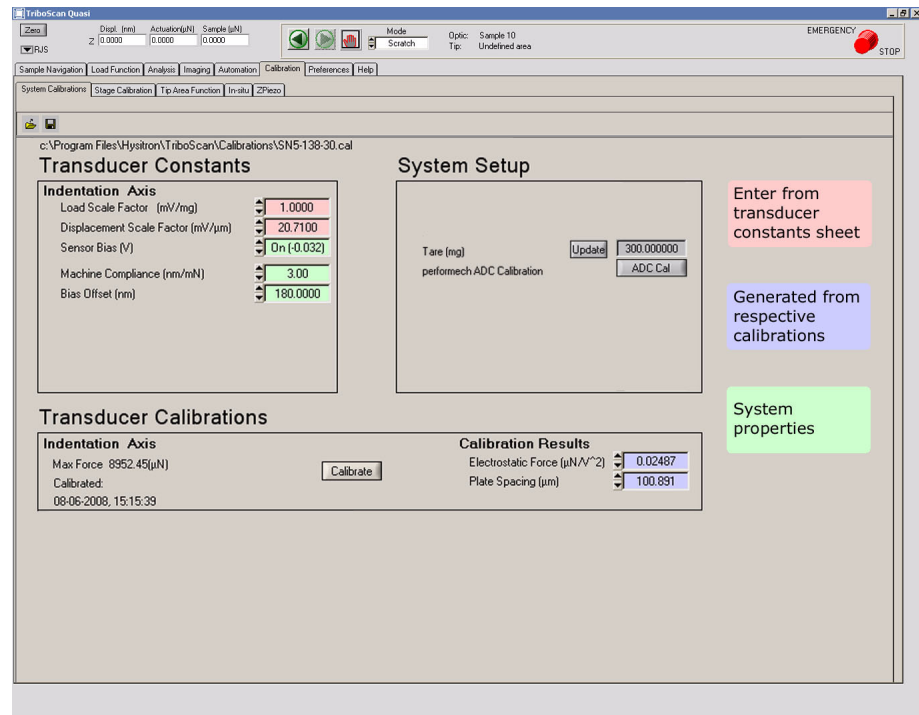
### Frame Grabber

The *Frame Grabber* sub tab is available with all Hysitron PI 95 PicoIndenter systems utilizing the enhanced video overlay feature. The *Frame Grabber* sub tab allows the user to select the appropriate video device as well as settings and drivers for the selected device.

## Section 3.5.1 System Calibrations Sub Tab

The *System Calibrations* sub tab is used to perform the calibration necessary to verify the instrument is operating properly and to account for small changes in transducer properties that may be caused by temperature, humidity or changing probe mass. The *System Calibrations* sub tab is given in Figure 4.10. A description of the parameters given in the *System Calibrations* sub tab is given below.

Figure 4.10  
*System Calibrations* sub tab with  
color-coded parameters



Indentation Axis Load  
Scale Factor

The *Load Scale Factor* is measured in mV/mg and is tuned during the initial transducer calibration at Hysitron.

Indentation Displacement  
Scale Factor

With the *Load Scale Factor* tuned, the resulting *Displacement Scale Factor* is measured (prior to shipment) based on the position of the center plate of the transducer with a specified applied voltage.

Sensor Bias

The *Sensor Bias* is a property of the transducer controller and should read *On(-0.032)* at all times unless otherwise advised by a Hysitron service engineer.

Machine Compliance

The *Machine Compliance* is a calculated value that represents the compliance of the entire system including the sample, probe, transducer, scanner, X/Y and Z-Axis stage assemblies and any associated instrument base. The default *Machine Compliance* for the PI 95 PicoIndenter system is 0.0 and should only be changed if instructed to do so by a Hysitron service engineer.

Bias Offset

The *Bias Offset* is the amount of offset applied to the center plate of the transducer to allow for bi-directional movement of the nanoindentation probe. The bi-directional movement is of most importance during the *lift height* before performing a feedback control nanoindentation test or measuring the drift rate

before a test is begun. The default is 180 nm, however, this value can be turned up to 2000 nm if larger bi-directional movement is required. Keep in mind, however, that the transducer has a maximum range of 5  $\mu\text{m}$  and any value entered into the *Bias Offset* field will be subtracted from the total available forward range of the Z-Axis displacement.

#### Tare

The *Tare* parameter displays the tare value of the transducer (indicator of the rest position of the center plate of the three-plate capacitive sensor). The *Tare* of the transducer, as measured at Hysitron with no probe installed, is given on the supplied transducer constants sheet. The value given in the software will be slightly more negative due to the mass of the probe but should be similar to the value given on the supplied sheet. The *Tare* value can be updated at anytime the system is not performing a test or in contact with a sample surface by clicking the *Update* button.

#### *performech* ADC Calibration

The *performech ADC Calibration* is used to calibrate the data acquisition boards and the associated gains within the *performech* controller. The *ADC Calibration* is only required to be performed once for each system (typically during the initial installation) but can be performed as frequent as desired to confirm that the gain settings are accurate.

#### Indentation Axis Electrostatic Force

The *Electrostatic Force* is a property of the transducer that is calibrated during the *Indentation Axis Calibration*. The *Electrostatic Force* will be approximately 0.03  $\mu\text{N}/\text{V}^2$  but varies by transducer model.

#### Indentation Axis Plate Spacing

The *Plate Spacing* is the distance between the center plate of the transducer and the lower drive plate. The default setting is 80  $\mu\text{m}$  but will be calibrated during the *Indentation Axis Calibration* and will vary slightly with ambient temperature/humidity as well as probe mass.

Calibrations for the individual components of the transducer are performed by selecting the appropriate corresponding *Calibrate* button under the *Transducer Calibrations* heading. Transducer calibration procedures are discussed in the *Calibration* chapter of this user manual.

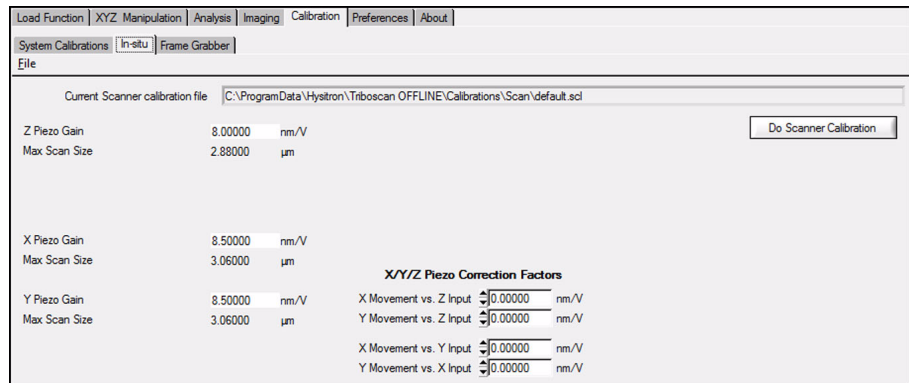
## Section 3.5.2

### The *in-situ* sub tab

The *in-situ* sub tab displays the currently selected scanner calibration as well as the tools to load a new scanner calibration. The *in-situ* sub tab also contains the piezo gain settings for the X, Y and Z-axis piezo actuators.

The PI 95 PicoIndenter will be supplied with a piezo calibration sheet listing an X, Y, and Z-axis *Piezo Gain* as well as correction factors for the X, Y, and Z vs. each axis piezo actuation. The user can modify the *Piezo Correction Factors* until the piezo movement is accurate in the X, Y and Z-axis.

Figure 4.11  
*in-situ* sub tab

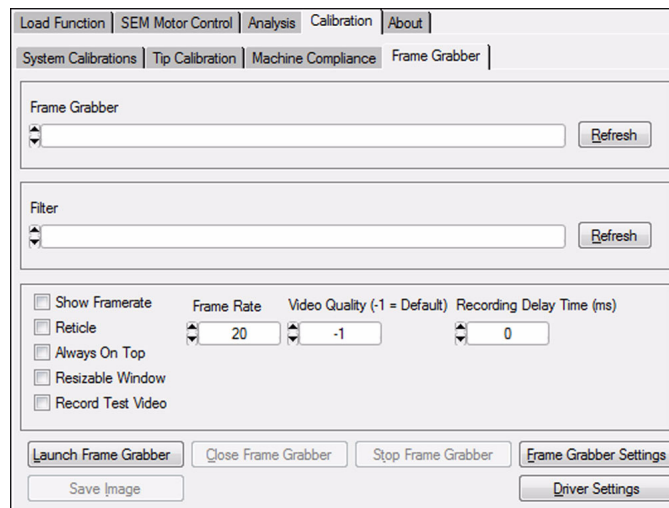


### Section 3.5.3 Frame Grabber Sub Tab

The *Frame Grabber* sub tab (Figure 4.12) is used to select the video source (used for the enhanced video overlay feature) as well as to configure the settings and driver for the video capture device.

A description of some of the parameters given in the *Frame Grabber* sub tab are given below, however, more advanced configuration will be performed during the installation with the Hysitron service engineer.

Figure 4.12  
*Frame Grabber* sub tab



#### Frame Grabber

The *Frame Grabber* pull-down menu allows the user to select the video capture device connected to the microscope. All video capture devices connected to the computer will be displayed so it is important that the video capture device connected to the microscope be selected.

#### Filter

The *Filter* pull-down menu allows the user to determine the encoding method for creating the \*.avi video file.

#### Refresh

The *Refresh* button updates the available parameters on the *Frame Grabber* and *Filter* pull-down menus. The parameters will only change if a computer setting or hardware has been modified without restarting the TriboScan software.

#### Show Frame Rate

The *Show Frame Rate* check box will display the current frame rate in the lower left corner of the video window.

#### Reticle

The *Reticle* check box will display a reticle (cross-hair) on the video window.

#### Always On Top

The *Always On Top* check box will display the frame grabber window (usually only used during a test) on top of the other TriboScan windows.

The frame grabber video window may need to be moved off screen to access features within TriboScan when the *Always On Top* check box is selected.

<a href="#">Resizable Window</a>	The <i>Resizable Window</i> check box will enable the ability for the user to resize the video window.
<a href="#">Record Test Video</a>	The <i>Record Test Video</i> check box will start a sample recording of the current video window. This is usually used to verify the driver and settings.
<a href="#">Frame Rate</a>	The <i>Frame Rate</i> parameter allows the user to determine the number of frames per second to display. The default setting is 20.
<a href="#">Video Quality</a>	The <i>Video Quality</i> parameter allows the user to determine the quality of the recorded video. If the video quality is set too high the software may start to perform slowly. The default setting is -1, which is an automatic detect mode. The user can set the <i>Video Quality</i> from 0 to 100 with 100 being the highest quality.
<a href="#">Recording Delay Time</a>	The <i>Recording Delay Time</i> allows the user to apply a delay in the recorded video to compensate for any lag in the video between the test result and the video movement.
<a href="#">Launch Frame Grabber</a>	The <i>Launch Frame Grabber</i> button will open the video window for the user to view the current video feed from the microscope.
<a href="#">Close Frame Grabber</a>	The <i>Close Frame Grabber</i> button will close the video window (if it is open).
<a href="#">Stop Frame Grabber</a>	The <i>Stop Frame Grabber</i> button will stop collecting video on the frame grabber window but leave the video window open.
<a href="#">Save Image</a>	The <i>Save Image</i> button will save an image of the current video image.
<a href="#">Frame Grabber Settings</a>	The <i>Frame Grabber Settings</i> button has multiple settings to modify the video window. The settings include things like color, tint, resolution, etc... The <i>Frame Grabber Settings</i> will be configured by a service engineer for the particular microscope during the installation and the user may need to contact Hysitron for assistance in modifying these parameters.
<a href="#">Driver Settings</a>	The <i>Driver Settings</i> button has multiple settings relating to the video capture device drivers and settings. The driver will be configured by a service engineer for the particular microscope during the installation and the user may need to contact Hysitron for assistance in modifying these parameters.

## Section 3.6

### About tab

The *About* tab contains information about the TriboScan software version number, any associated National Instruments data acquisition version numbers, stage controller, and software engine revision numbers. The information provided in the *About* tab (especially the TriboScan version number) is often very useful to provide to the customer service engineer when calling for assistance.

Hysitron customer support email: [support@hysitron.com](mailto:support@hysitron.com)

Hysitron customer support phone: +1-952-835-6366

Hysitron now offers online customer support! Go to:

<http://www.hysitron.com/>

to request a login username and password to access the Hysitron user support center. The Hysitron user support center offers live online chat with support representatives, downloadable documentation, and hundreds of knowledgebase articles to assist with common issues and testing routines.

Figure 4.13  
Hysitron customer support  
center website



Double left-clicking on the *TriboScan* in the first line of the information in the *About* tab will display the current features installed and licensed on the system.



# Chapter 4 Testing

TriboScan is a full-featured software package that combines both testing and analysis into one easy-to-use software suite. The *Testing* chapter will cover common testing methods equipped on most systems as well as basic testing example routines that beginning users can refer to as a simple step-by-step method of performing a specific test.

The *Testing* chapter assumes that all calibrations listed in the *Setup & Calibrations* chapter have been satisfactorily completed. If the user is unfamiliar with the hardware setup and calibrations required of the system either refer to the *Setup & Calibrations* chapter or contact a Hysitron service engineer.



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**It is dangerous to perform procedures within the *Testing* chapter without first completing or verifying the hardware is correctly setup and calibrated as given in the *Setup & Calibration* chapter. Failure to follow the preceding procedures, as given in the *Setup & Calibration* chapter can result in damaged hardware, probes or samples.**

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Detailed descriptions of the various TriboScan software tabs is given in the *Software* chapter and will not be covered in the *Testing* chapter. For information on the particular function, button or parameter that is mentioned but not discussed within the *Testing* chapter refer to the appropriate tab within the *Software* chapter for a more thorough description.

Hysitron systems are designed for superior indentation testing. There are three feedback modes to perform nanoindentation and nanoscratch testing. Depending upon what the instrument was configured and licensed for, the testing modes are:

- Open-loop
- Load Control
- Displacement Control

Load and displacement controlled testing utilizes a feedback loop between the transducer controller and transducer to precisely achieve the desired force or displacement. Besides the ability to obtain precise forces or displacements accurately during a test, load and displacement controlled testing is very beneficial when measuring sample adhesion or stress-relaxation properties.

Open-loop testing is similar to load control testing, except that no feedback loop is used. Open-loop is a useful testing mode when a load controlled test is required but contact with the sample prior to testing is undesirable.

## Section 4.1 Loading the Holder

With the indentation probe and sample mounted to the PI 95 PicoIndenter, the holder can be loaded into the TEM. The following procedure is recommended for loading the PI 95 PicoIndenter holder into the TEM:

1. Power off the *performech* control unit.
2. Check that the four screws that hold the front end of the holder (near the O-ring) are tight.
3. Check the O-rings and remove any lint or particles.
4. Use the manual Z-axis control knob to bring the probe close to the sample. Try to position the probe about 100 $\mu$ m from the sample surface. As a guide for your eye, note that the width of the shaft of the diamond tip is about 200 $\mu$ m.



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**Be careful not to get too close; ramming the tip into the sample can seriously damage your transducer.**

---

5. Load the sample holder in the microscope as you would a standard holder for your TEM.
6. Connect the transducer signal cable and piezo cable between the PI 95 PicoIndenter and *performech* control unit. Cables should be routed to prevent unnecessary strain and away from electrical devices such as computer monitors to minimize interference.
7. Power on the *performech* control unit.
8. Start the TriboScan software.
9. Verify the tare value of the PI 95 PicoIndenter by selecting the *Calibration* tab  $\rightarrow$  *System Calibration* sub tab and clicking the *Update* button near the *Tare Value* parameter on the right side of the window.
10. Verify that the proper transducer calibration constants are entered in the *Calibration* tab  $\rightarrow$  *System Calibration* sub tab.
11. Run the *Z-Axis Calibration* as outlined in the *Setup & Calibration* chapter of this user manual.

## Section 4.2 Mechanical Testing

The general procedure used for performing an indentation test with the TEM PicoIndenter is outlined below:

1. Before performing a test both the sample and the probe must be located. DO NOT attempt to adjust the probe location (except to retract, if desired) unless the probe is visible—otherwise you risk ramming the probe into the sample.



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**DO NOT attempt to adjust the probe location (except to retract, if desired) unless the probe is visible. Attempting to do so may cause serious damage to the PicoIndenter.**

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2. Go to the *XYZ Manipulation* sub tab and move Z-Axis of the piezo element to approximately 80% retracted. This will allow for more movement in the forward Z-Axis direction when the sample test is ready to be performed.
3. Locate the very end of the probe and slowly approach the sample, using the Z-Axis knob on the end of the TEM PicoIndenter while simultaneously using the TEM stage to track the probe movement. Continue approaching until both the probe and the sample can be seen in the field of view.
4. Manually adjust the sample and probe heights: First set the sample height to the eucentric height, then adjust the probe height using the manual control knob so that it has the same focus as the sample (and therefore lies in the same plane).
5. Using the piezo controls in the *Load Function* tab → *XYZ Manipulation* tab, bring the probe close to the sample using Z, position laterally using X, and adjust the probe height with Y (such that the probe and the sample are in the same focus).
6. Choose a testing mode (open-loop, load control or displacement control) from the *Load Function* tab → *Quasi* sub tab and setup the desired load function.
7. Click the *Zero* button in the upper left of the TriboScan software.
8. Click the *Perform Indent* button to run the test.

## Section 4.2.1

### Testing Suggestions

- The probe may jump when the system is toggled into load or displacement control mode (when the feedback is enabled). Make sure the probe is not too near the sample before enabling feedback to prevent the probe from hitting the sample.
- Never use the manual axis controls when the feedback loop is enabled.
- Before you load the holder into the TEM, adjust the Y-axis height using the manual axis control. Once inside the TEM it can be difficult to determine in which direction to move the probe (up or down). Adjust the probe height such that it is roughly in the center of the sample.

## Section 4.2.2 Feedback Gain Settings

It is important, when testing with load or displacement controlled testing, to ensure good feedback control during the indentation. There are several gains (located within the *Load Function* window) to control but for most testing needs only a few of the gains are used.

Table 4.A  
Default gain settings

Default Gain Settings	
	Standard System
<b>Integral Gain</b>	1.0
<b>Proportional Gain</b>	0.0
<b>Derivative Gain</b>	0.0
<b>Adaptive Gain</b>	0.0
<b>Lift Integral Gain</b>	1.0

The *Integral Gain* has the biggest effect on the feedback controlled testing and will determine how strongly the force or displacement signal will react to varying sample properties. If the *Integral Gain* is too low, the force or displacement may not change quickly enough when there is an error from the requested load. If the *Integral Gain* is too high, the force may react too quickly to small changes caused by noise and oscillations will begin to appear on the plot.

The *Pre-Load Integral Gain* setting is the integral gain setting during the drift monitor time. The force is not changing during this hold, so the gain setting must be lower to avoid oscillations. This is typically set at 0.2.

The *Lift Integral Gain* is the gain setting when the tip is in the air during the pre-indent lift height movement or after the indent has come out of contact with the sample at the end of the indentation. During these times, the stiffness of the contact is much less, and there is no damping from the sample. As a result, the *Lift Integral Gain* setting is typically about 10 times less than the *Integral Gain* during while in contact with the sample.

In order to tune the gains for any particular sample a few practice indentations at similar loads and loading rates should be run. The *Integral Gain* can then be increased until oscillations occur. The *Integral Gain* should then be reduced by about

30% for the best results. When the gains are tuned properly, the acquired data will plot directly over the requested ramp on the real-time data plot. Examples of feedback control real-time plots are given in Figure 4.14.



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**If large oscillations occur, due to improperly tuned gains, the displacement may exceed 5  $\mu\text{m}$  and the user may receive a *Displacement Limit Exceeded* error.**

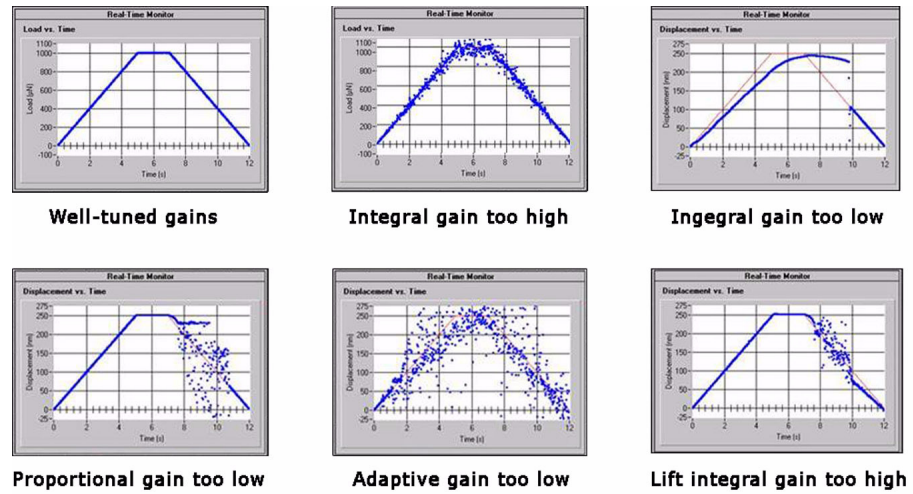
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Suggestions for tuning the gains are given below.

- Try testing with the default gain settings first. The default gain settings work well for most samples and typically require very little adjusting.
- In general, higher gains will increase noise-induced oscillations, and lower gains will cause the displacement to deviate from the requested ramp.
- Keep the adaptive gain near zero. Larger values will cause oscillations in most materials.
- Turn the proportional and derivative gains to zero to start. Use only the adaptive gain and integral gain to force the displacement to follow the load function then use the other gains to track small changes.
- Keep the derivative and proportional gains at a similar ratio when changing them. The derivative gain will act as a stabilizing factor to damp oscillations, but can also increase the effect of noise.
- If oscillations are observed at the point where the tip leaves the surface, lower the *Lift Integral Gain*, as this causes the oscillations in the air.
- Slow the nanoindentation test down. In some very stiff metal samples, the load has to change very quickly for a very small change in displacement. The feedback loop may have a difficult time in ramping the force this quickly to maintain the proper displacement rate so increasing the time will help.

Examples of incorrect gain settings for indentations on single crystal aluminum are given in Figure 4.14.

Figure 4.14  
Feedback control gain  
suggestions



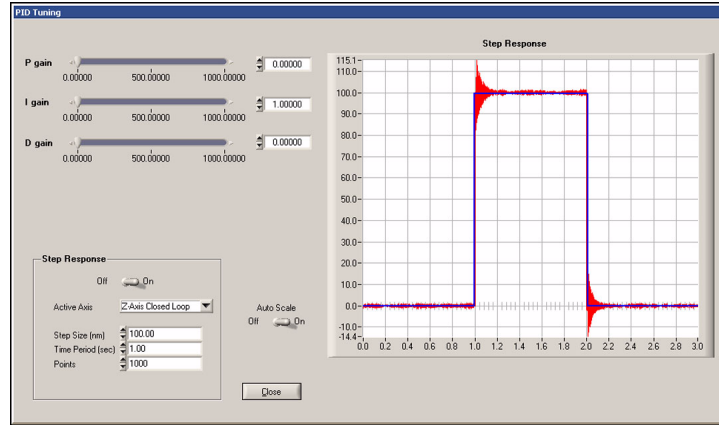
## Displacement Controlled Testing

For displacement controlled nanoindentation tests, it is best to start tuning the gains by running practice tests in the air. If the gains are tuned properly in the air, they will be close to the correct settings on a sample. After setting the gains from the air tests practice several nanoindentation tests on the sample, if possible, in order to fine tune the feedback gains.

Displacement controlled testing has a program to assist the user in tuning the PID gain settings. When a displacement controlled indentation has been selected from the *Load Function* tab, an additional *Tune PID Gains* button will appear. Selecting the *Tune PID Gains* button will open the *PID Tuning* window (Figure 4.15).

The *PID Tuning* window allows the user to modify and toggle the step response on and off. When the step response is enabled a desired blue step response will appear in the *Step Response* plot and a red (measured data) plot will appear over the plot. It is desirable for the red and blue plots to follow closely (as shown in Figure 4.15). The PID gains can be modified in real-time while the result continues to update on the *Step Response* plot. When satisfied with the gain settings, click the *Close* button and the software will prompt the user if they would like to save the current gain settings. The gain setting will automatically be populated in the *Load Function* tab.



Figure 4.15  
*PID Tuning window*


The desired gain settings are typically very near the default settings. Over-tuning the gains will cause severe oscillations within the transducer and cause an emergency stop loop that requires a software restart. Although the PID gains have slider bars that go from 0.0 up to 1000, the gains should be adjusted slowly and monitored to prevent any severe oscillation.

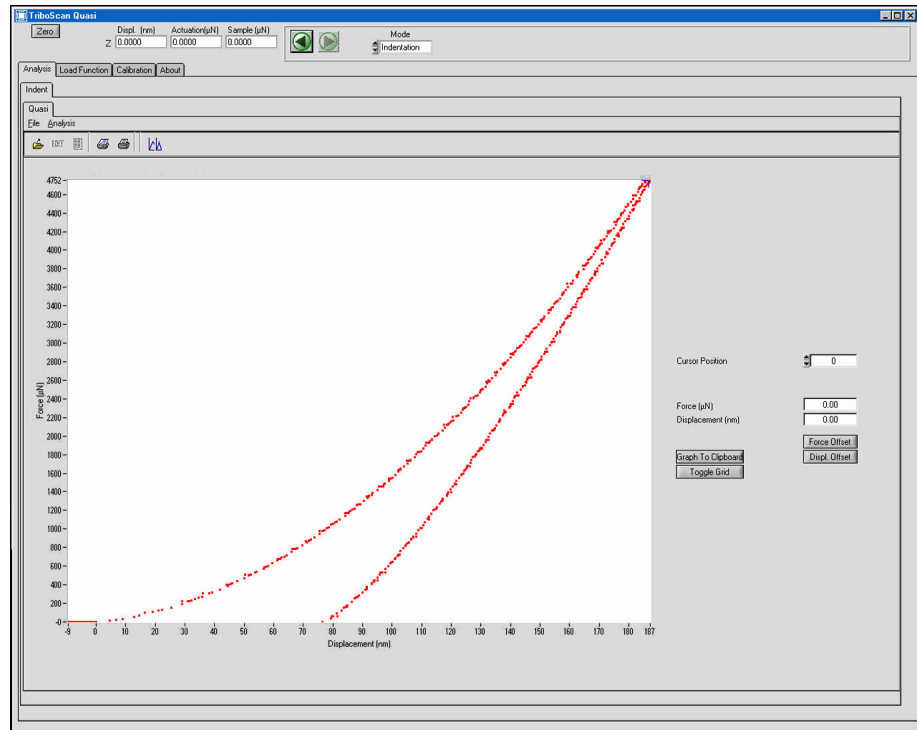
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# Chapter 5 Analysis

## Section 5.1 Indentation Analysis

Analysis performed with the PI 95 PicoIndenter system is typically performed by analyzing the indentation video in conjunction with the real-time indentation plot. After each test, the system will prompt the user to save the file as a \*.hys file. \*.hys files can be read by TriboScan from the *Analysis* tab → *Quasi* sub tab → *Quasi* sub tab (Figure 5.1).

Figure 5.1  
*Quasi* sub tab



To open an existing \*.hys (Hysitron indentation file) click the *Open* icon at the top of the window or select **File** → **Open**.

The *Force Offset* and *Displ. Offset* buttons allow the user to make any point on the plot the origin. This is helpful in accounting for setpoint or lift height artifacts, or if the test began with the probe out of contact with the sample surface. To use these options, select a new point on the plot to be the zero force or zero displacement and click either *Force Offset* and/or *Displ. Offset*.

## Section 5.1 Enhanced Video Overlay

The enhanced video overlay functionality is one of the most important analysis tools available to the PicoIndenter system. TriboScan 9.2.12 (and above) incorporate the video from the SEM/TEM system into the TriboScan software. In order to access the video overlay functionality within TriboScan the test must be performed with the *Video Graphics* toggle button (on the *Load Function* tab) switched to the *On* position.



**The *Video Graphics* toggle switch must be set to *On* before performing the test in order for the user to offline access to the video overlay features.**

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**Previous versions of the video overlay used a blue screen overlay with a ‘virtual’ monitor and third party frame grabber software. For information on upgrading your system to the enhanced video overlay functionality contact a Hysitron service engineer.**

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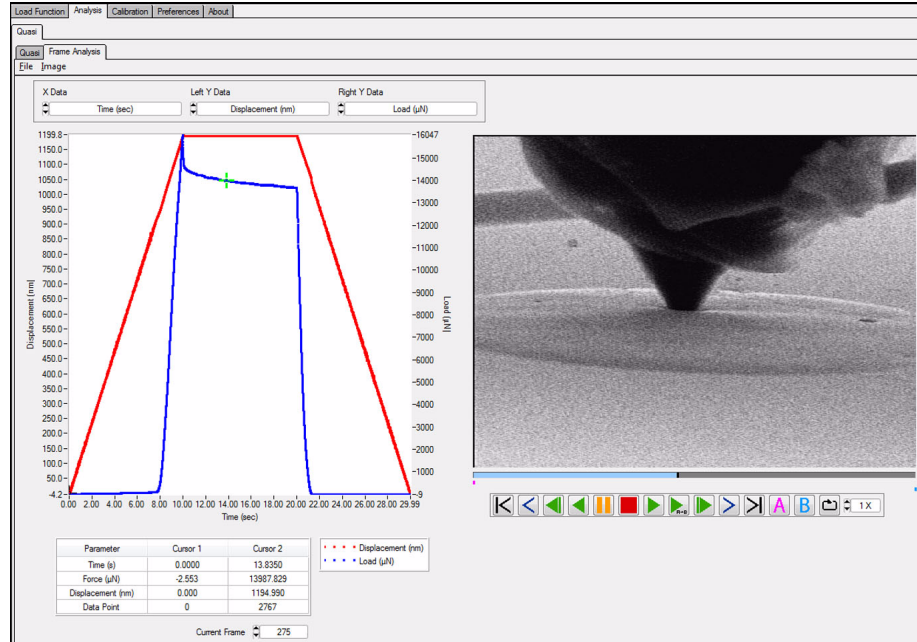
During the test the *Frame Grabber* window may appear during the test and will display the video that is captured during the test. Typically the system will be set up to capture a video of the probe contacting the sample via one of the following methods:

- VGA monitor splitter cable (with RCA out) and USB frame grabber
- Microscope BNC video output and USB frame grabber
- Ethernet frame grabber hardware and appropriate cabling

The hardware required will vary for each microscope and the frame grabber hardware will be installed by the service engineer during the instrument installation.

The system will capture the standard *\*.hys* file and a *\*.avi* file with the same file name and the files will be saved in the same directory. The user can open the *\*.hys* file or the *\*.avi* file independently for analysis or the two files can be opened in the same window for analysis from the *Analysis* tab → *Quasi* sub tab → *Frame Analysis* sub tab (Figure 5.2).

Figure 5.2  
*Frame Analysis* sub tab



Within the *Frame Analysis* sub tab there are several viewing options as well as video playback options.

The menu items (*File* and *Image*) to allow the user to save the indent and video as a side-by-side an \*.avi movie, save the indent curve as an \*.avi movie, rotate the image, and set an offset for the movie (to help sync the video with the indent curve if there is a delay between the two systems). The delay can be minimized by starting with the probe out of contact and synchronizing the start of the probe movement with the \*.hys and \*.avi file.

There are three pull-down menus at the top of the *Frame Analysis* sub tab that allows the user to choose the data to plot on the X and Y axes as well as a *Current Frame* parameter at the bottom of the window that allows the user to set jump to a desired frame.

There are multiple buttons under the video window that control the video. The operation of these buttons (in order) are:

- Jump to start
- Back one frame
- Rewind
- Play backward
- Pause

- Stop
- Play forward (normal play)
- Play between **A** and **B** markers
- Fast forward
- Forward one frame
- Jump to end
- Set **A** marker (for selective play between **A** and **B**)
- Set **B** marker (for selective play between **A** and **B**)
- Loop video
- Change video speed (down to 1/10x normal speed and up to 10x normal speed)

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