MTS®

Nano Indenter® XP

Operating Instructions V1.1

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Read Me First!

While it is difficult to damage the Nano Indenter® XP, it is possible to do so. Thus, before you "jump-in" and begin to operate the instrument, it is important that you understand the ways in which the indenter can be damaged.

The most important sections of this manual in terms of preventing damage to the instrument are Sections 6.0 and 10.0. It is recommended that you read Part One and Part Two of these instructions prior to beginning test set up or using the instrument.

If you have purchased this instrument and have not yet assembled it, please refer to the assembly and start-up instructions prior to reading this manual. The assembly instructions are contained in Appendix F of this manual.

Contents

Preface

Congratulations! You now have the Nano Indenter® XP, the world's most advanced depth-sensing indentation device. As you begin using your Nano Indenter® XP, you will find that the system is designed, from both the software and hardware points of view, to be robust and easy to use, while still retaining the sensitivity required for very demanding experimentation. Whether your use will be in routine testing or more rigorous research, you've made the right decision - the Nano Indenter® XP!

These oper ating instructions contain reference material for users of the Nano Indenter® XP. Additional information is contained in the Appendices, including a step by step procedure for setting up a simple test.

The operating instructions are separated into three parts:

> Part One describes the instrument and provides an overview of the physical characteristics and mechanical operation of the instrument.
>
> Part Two is the reference section for basic operation of the instrument. All of the information contained in part two is relevant to the "short menus."
>
> Part Three is the reference section for advanced operation. This section is intended for users who want to expand the use of the instrument into more flexible research. Consequently, Part Three contains a great deal of information, which can appear intimidating at first glance. You will find, however, that operation of the advanced configuration ("long menus") simply adds more functionality to the operations performed by the "short menus."

While operation of the Nano Indenter® XP is fully explained in this manual, the theory of indentation is not addressed, nor are the mechanics of the indentation system. The list of publications in *Appendix J: Technical References* provides an excellent reference for both topics.

With instruments that are equipped with optional attachments, such as Continuous Stiffness Measurement or Lateral Force Measurement, an additional section to these instructions is provided. An addition to the manual (Part Four) describes the options and the use of the options.

For technical support questions, refer to *Appendix F, Troubleshooting*, before contacting Nano Instruments. Instructions on how to prepare information to expedite the troubleshooting process are contained in this appendix.

1.0 Overview Of The Instrument

1.1 Overview

The Nano Indenter® XP is a complete system, ready to run once it is assembled. This means there is no additional hardware required, other than the air supply required for the vibration isolation table.

1.2 The Nano Indenter® XP System

The Nano Indenter® XP system is composed of a number of "subsystems" that will be explained in detail in Sections 2.0 through 6.0. These subsystems are: the computer system, the motion system, the optics system, the gantry & isolation equipment, and finally the heart of the Nano Indenter® XP, the indenter head.

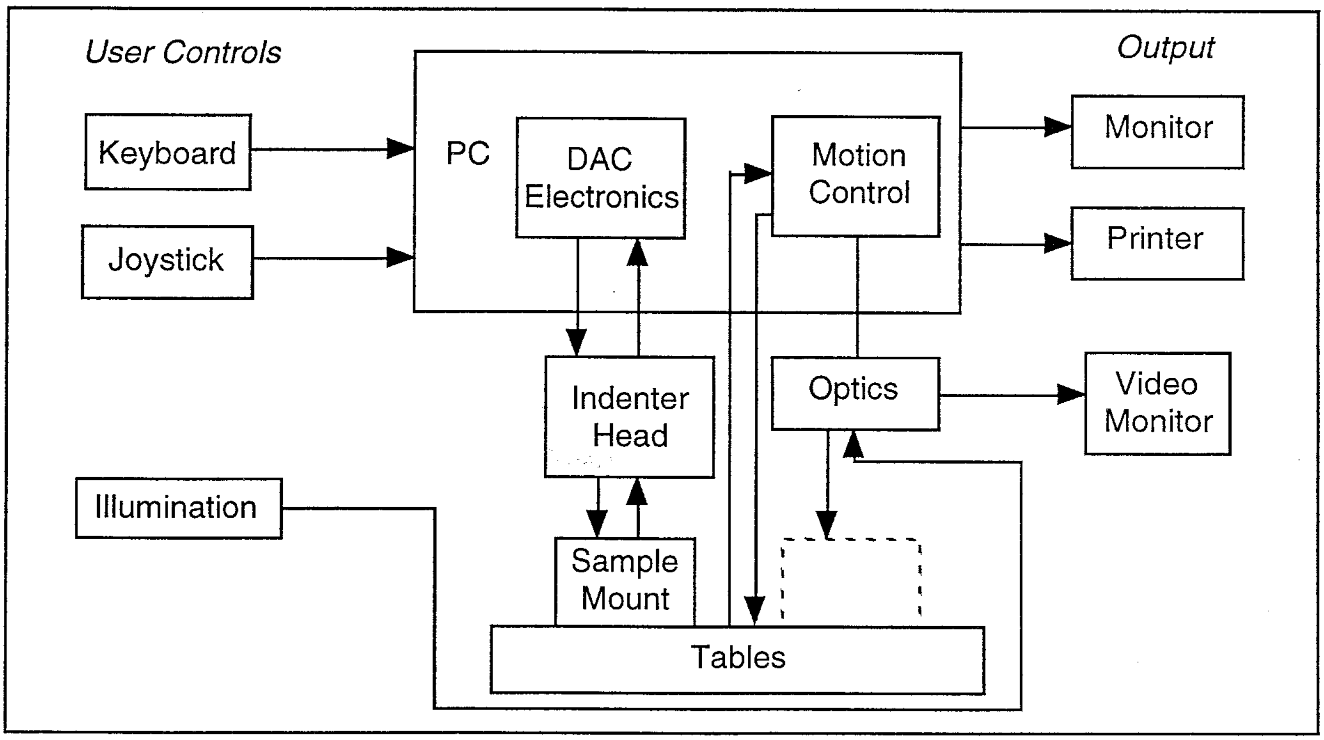

Figure 1.1 Nano Indenter® XP System Diagram

1.2.1 User Interface

The Nano Indenter® XP subsystems are fully integrated, primarily through the computer. All user input and control is remotely managed from the operator station. Only minor adjustments must be made on the instrument itself, such as the polarization on the microscope.

User control is accomplished through the joystick, the computer keyboard, and the fiber optic illumination power source.

1.2.2 Control Modes

There are two modes of control of the Nano Indenter® XP: automated experiment control and direct user control.

1.2.2.1 Automated Experiment Control

Automated experiment control refers to the actual performance of experiments. Once the samples are loaded and the test is defined, the test is automatically performed by the instrument. No user input is necessary during the test. All movement of the positioning stages and the indenter itself are controlled directly by the Nano Software.

1.2.2.2 Direct User Control

Direct control of the Nano Indenter® XP is necessary when loading samples or changing the indenter tip. Direct control is also necessary during the "select position of indents" section of the test set-up procedure. Some other routines, such as calibration of the instrument, provide direct control as well, but it is primarily the "Load Specimens/Manual Control" routine (Section 10.0) which requires and enables user control of the physical system.

2.0 The Computer System

2.1 Overview

The computer is the central control unit for the Nano Indenter® XP. Most of the electronics that drive the system are housed in the PC, as is the operating software and, finally, the resultant test data. The computer system is composed of the PC itself, the internal electronic cards, the printer, the monitor, the joystick, and the software.

2.2 The PC

The baseline PC used in the Nano Indenter® XP system is pre-configured for the Nano Software. There is no need to install any software modules in order to use the instrument.

The locking door on the front of the PC conceals the power and reset buttons, as well as the floppy drive.

When you start the PC, the AUTOEXEC file will automatically load Microsoft Windows[1] (see Section 2.7).

2.3 The Internal Electronic Cards

The internal electronic cards drive the motion system, the indenter head, and the computer/software interface. There is no reason for the user to access the internal electronic cards. If a problem is suspected, please contact Nano Instruments.

All of the cards fit into standard ISA slots. Several of the slots are open. While it is possible to use one of these open slots for ethernet capability, this use is not supported by Nano Instruments.

2.4 The Printer

The printer is a required feature of the Nano Indenter® XP system. After the completion of an experiment run (that is, after the completion of an entire test) data is automatically printed in a pre-formatted report. If the printer is off-line, disconnected, out of paper, or out of ink, the software will pause and wait for the problem to be corrected before allowing you to continue with the Nano Software.

Refer to the printer manuals provided with the instrument for more information about printer operation.

[1]Windows is a trademark of Microsoft Corporation.

2.5 The Monitor

The monitor is connected to a video driver card in the PC. Refer to the manuals provided with the instrument for more information about the monitor.

2.6 The Joystick

The joystick used with the Nano Indenter® XP system is capable of a number of different functions. The stick itself precisely controls the X-Y tables, and can be used for "fast" moves by depressing the trigger button on the joystick. In addition, the joystick buttons and speed control knob can be used to control the optic system focussing and speed of focussing. For more information about the use of the joystick, refer to Section 10.0.

2.7 The Software

There are three significant software packages associated with the Nano Indenter® XP: HTBasic[2], the Nano Software, and Kaleidagraph[3]. Both MS-DOS and Microsoft Windows[4] are also installed on the system. Use of MS-DOS is anticipated and it is possible to "shell-out" of the Nano Software in order to manipulate files via MS-DOS. For file manipulation and running external applications, a menu item in the Nano Software is provided that returns to Windows. Finally, Kaleidagraph is provided as an external plotting package, most useful when performing curve fits associated with tip calibration.

2.7.1 HTBasic

The HTBasic software is necessary in order to run the Nano Software. HTBasic provides the programming environment for the Nano Code. For more information about HTBasic, refer to the manuals that were provided with the instrument.

One important note: HTBasic will not run without the HTBasic "key." This key is installed in the parallel port on the PC. Do not remove this key.

2.7.2 The Nano Software

The Nano Software is activated by double-clicking on the "Nano" icon located in the Applications folder in the Program Manager.

The Nano Software uses a menu-based interface to control the instrument. The specifics of the software are described in Sections 7.0 through 11.0. These sections are the only documentation of the Nano Software you will receive.

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[2] HTBasic is a trademark of TransEra Corporation.

[3] Kaleidagraph is a trademark of Synergy Software.

[4] MS-DOS is a registered trademark, and Windows is a trademark of Microsoft Corporation.

Some useful commands that can be used with the Nano Software are:

ALT-G [5]	Performs a graphic screen dump to the printer, requires form feed on printer
SHIFT-PRINT SCREEN	Performs a text dump to the printer
CONTROL-BREAK	Interrupts the Nano Software
run	To be typed after CONTROL-BREAK in order to restart the Nano Software
continue	Re-enters the Nano Software if possible
quit	Shuts down HTBasic and exits to Windows

Finally, an important feature of the Nano Software is the use of the function keys, which control movement through the menu structure.

2.7.2.1 The Function Keys

An important aspect of the Nano Software operation is the use of the function keys to move between menus and activate menu operations. As shown in Figure 2.1, the functions these keys perform are shown at the bottom of each menu. The function keys, of course, are the physical function keys across the top row of the keyboard.

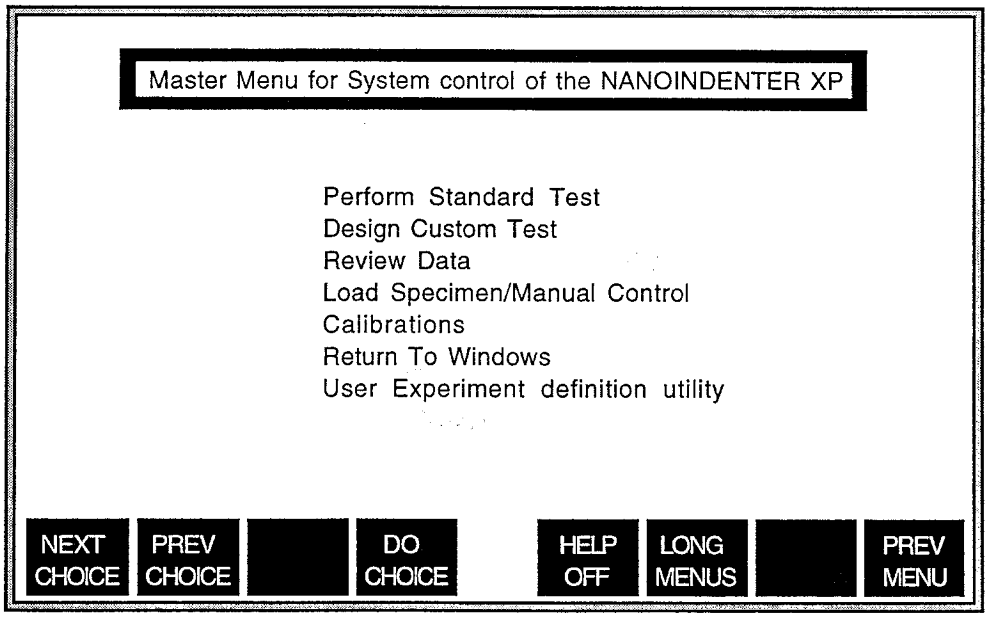

Figure 2.1 The Master Menu & Function Keys

The convention for the use of function keys is that these keys are used only to select menu options. For inputting information, or selecting default text from on-screen prompts, the "Enter" or "Return" key should be used.

There are two other additional methods for selecting menu items. The "Up" and "Down" arrow keys are also activated, and will toggle through the menu options in the same manner as the function keys. The "Enter" or

[5] Combinations of keyboard keys are shown in all caps. Lines to be typed are shown in lower case.

"Return" key functions in the same manner as the "Do Choice" function key.

The second additional method for menu item selection is to use the trackball on the keyboard to move between menu items, and use the buttons on the lower left side of the keyboard to select items.

The more common function keys are listed in the following subsections. For specific information on function keys that appear in specific subroutines (such as "Test Motion of Indenter") see the applicable section describing that subroutine.

2.7.2.1.1 Next Choice

The Next Choice key moves to the next menu item down in the list displayed on the screen. Using this key on the last menu item will move to the top menu item. As described above, you can also use the "Down" arrow key or the trackball for this function.

2.7.2.1.2 Previous Choice

The Previous Choice key moves to the previous menu item up in the list displayed on the screen. Using this key on the first menu item will move to the bottom menu item. As described above, you can also use the "Up" arrow key or the trackball for this function.

2.7.2.1.3 Continue

The Continue key simply resumes a function or operation in progress. This key is most often used with the on-screen help function.

2.7.2.1.4 Do Choice

The Do Choice function key executes a choice selected on screen. As described above, you can also use the "Enter" key or the keyboard trackball buttons for this function.

2.7.2.1.5 Help Off/Help On

Selecting this key will toggle the on-screen help function on or off.

2.7.2.1.6 Long Menus/Short Menu

Using this function key will toggle back and forth from the standard "short menu" to the more advanced "long menu." For more information about this option, see Section 2.7.2.2.

2.7.2.1.7 Previous Menu

The Previous Menu key steps back one level in the software to the previous displayed menu. This key is useful for aborting from specific test set-up procedures, but should certainly not be used if you want to keep information that you have input. In most cases, you will use the ALL DONE, CONTINUE ON menu function to move through the software, as this function stays within the expected path of the code.

2.7.2.2 Standard & Extended Menus

There are two essential modes of operation of the Nano Indenter® XP: the Standard Mode and the Advanced Mode. The Standard Mode is intended for use in laboratory or QC environments where push-button operation is desired and ease of use is more important than flexibility. When the instrument is in Standard Mode, only the "Short Menus" are enabled. These menus contain all options necessary to set up a test, but do not display the more advanced options that are needed less often. By default, the Nano Indenter® XP always operates in Standard Mode.

When you switch to Advanced Mode (using the "Long Menu/Short Menu" function key) more options are displayed for configuration of tests, manipulation of data, and calibration of the instrument. These options are documented in Part Three of the operating instructions. On some instruments, the Advanced Mode may be disabled at the request of the customer, or per specific arrangement for the end use of the instrument. In such cases, Part Three of the operating instructions will not be supplied as a part of this manual.

2.7.2.3 Exiting The Nano Software And Returning To Windows

To exit the Nano Software, simply use the "Return To Windows" option located on the Master Menu screen of the Nano Software (see Figure 2.1).

2.7.3 MS-DOS

To use MS-DOS, you must select the MS-DOS icon in the "Main" folder in Windows.

2.7.4 Kaleidagraph

Kaleidagraph is accesible from the Windows Applications Folder. For more information on the use of Kaleidagraph, refer to the operating instructions for this program.

3.0 The Motion System

3.1 Overview

The sole function of the motion system in basic operation is to move the samples from the microscope focal point to under the indenter, and then to move to each position for indentation. There are three subsystems in the motion system: the positioning tables, the sample mount, and the limit switches.

3.2 The Positioning Tables

The positioning tables are cross-roller bearing supported, lead screw driven stages. The motors, gearboxes, and encoders that interface with the tables are enclosed in the gantry. There should be no reason for the user to access these components.

The Positioning Table subsystem is composed of two stages, the X stage (lower stage) and the Y stage (upper stage). Two rails are mounted on the Y stage. These rails are used to attach the sample mount.

3.3 The Sample Mount

The sample mount system consists of the clamping mechanism and the sample tray. The following directions for the loading of the sample tray into the Positioning Tables is intended as a physical description of the process. For information about using the joystick and keyboard commands to control the motion of the positioning tables (i.e., to position the tables in order to load the sample tray), refer to Section 10.0.

3.3.1 Loading the Sample Tray Into the Rails

The tray slides into the rails on the Y stage, and the clamping mechanism slides into the vertical posts on those rails, and the slot on the sample tray. Turning the clamp screw forces the sample tray into the wedges on the rails, and tightly locks the sample tray in place.

3.3.2 Removing the Sample Tray From the Rails

Removing the sample mount tray from the rails is a simple reversal of the procedure described above. Given the force applied when turning the clamp screw, it will be necessary to loosen the clamp screw before the tray will be able to easily slide out of the wedges on the rails (see Figure 3.1). Simply turn the clamp screw until the tray slides toward you.

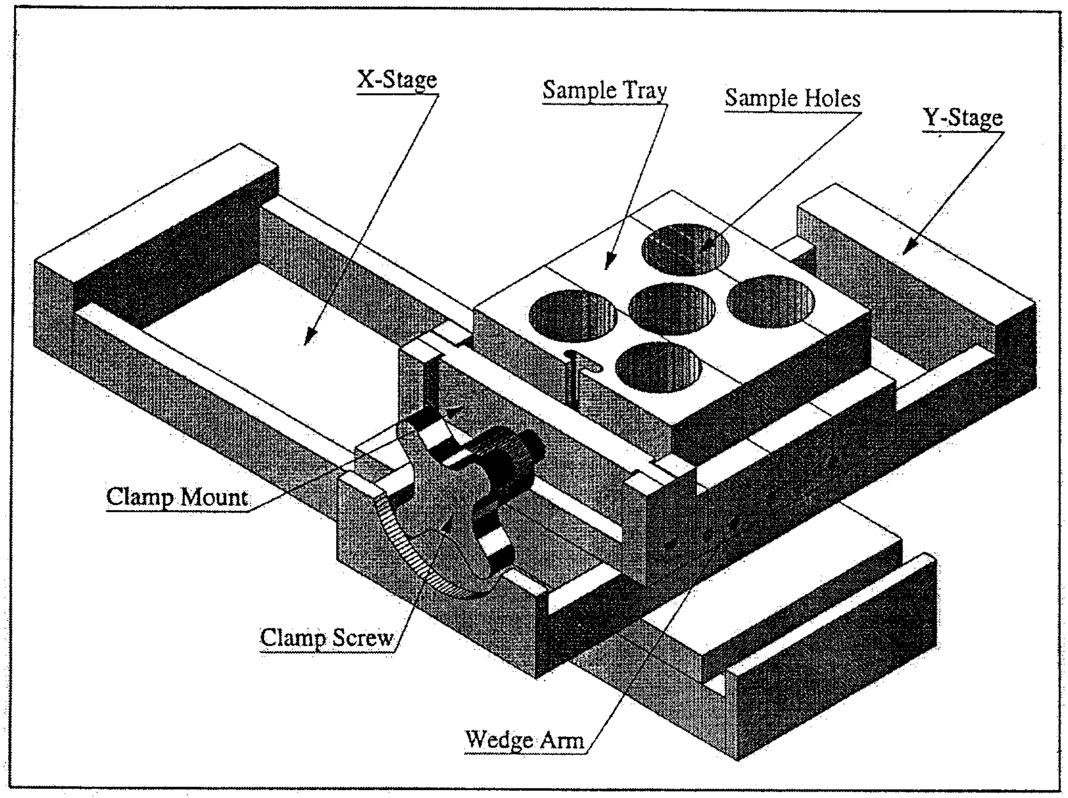

Figure 3.1. The Sample Tray Mounted On The X-Y Stages

3.3.3 Loading Samples Into The Sample Tray

The standard sample tray can accept up to five samples on standard metallographic mounting disks (1.25" diameter, 1" height). To load the samples, insert the sample disk into the bored sample hole and tighten the set screw in place against the sample disk. Repeat this procedure until all desired samples are loaded. Once the samples are loaded, and all are tightened in place, turn the sample mount tray upside down so that the levelling arms are facing downward, and set the sample mount tray on a flat, smooth, and lint-free surface. Loosen all of the sample disks, so that the sample tray rests entirely on the sample levelling arms. Once all of the samples are loose and resting against the "flat surface," retighten the set screws until each sample disk is fixed firmly in place. Turn the sample tray back over, and sight across the levelling arms to ensure that all of the samples are even and level. The sample tray is then ready to load into the rails (see Figure 3.2).

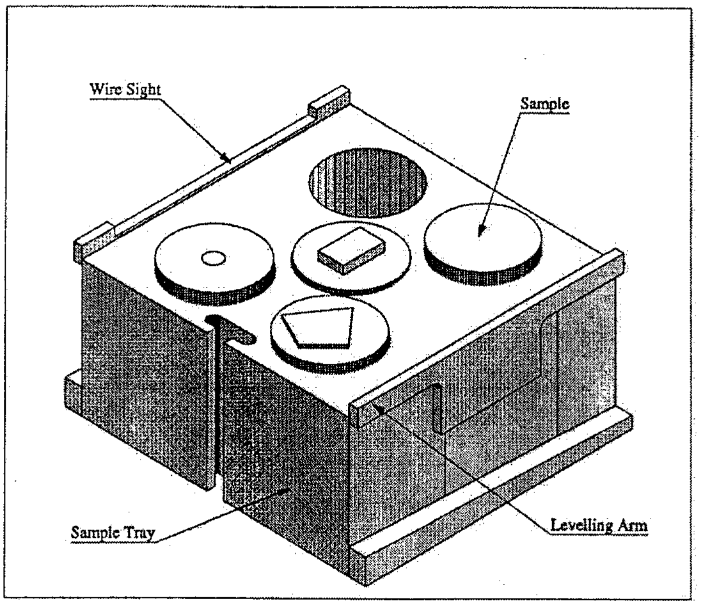

Figure 3.2 The Sample Tray With Loaded Samples

3.4 The Limit Switches

The limit switches are inaccessible to the user. When moving the tables, however, the stage can be driven until the limit switches are tripped. Once they are activated, the limit switch routine will assume control of the positioning tables and move them back and away from the limit switches. This safety precaution is used to prevent "locking" the tables against the end stops, which could result in damage to the gearmotors.

3.5 A Warning Concerning The Motion System

If the indenter is ever damaged, it is most likely the motion system that is the immediate cause of the damage. This can occur in a number of ways. While the Nano Indenter® XP is an extremely robust system, excessive force applied laterally to the indenter shaft can result in internal damage to the displacement sensing system, a misalignment of the load application system, or, most likely, damage to the diamond tip itself. The following warnings should minimize the possibility of accidentally damaging the system.

3.5.1 Mounting Samples At Improper Height

It is important that the proper height for the exposed portion of the sample be established. When the instrument arrives at your site, the levelling arms will have been adjusted for this height. Depending on sample use and mounting procedures, it is possible for you to change this "default" sample height. Although the indenter is retracted into the indenter housing, you should take care that the samples are not mounted too high in the sample tray. The maximum height of the samples must be lower than the bottom of the indenter housing.

If the samples are mounted too low in the sample tray, the indenter will not be able to reach the surfaces, causing the experiment run to abort, but no damage to the system will ensue.

3.5.2 Mounting Samples At Unequal Height

Not only must the samples be mounted at the proper height, but all of the samples' surfaces must be at the same height. If there is a large deviation in height from one sample to the next, problems can arise during indentation.

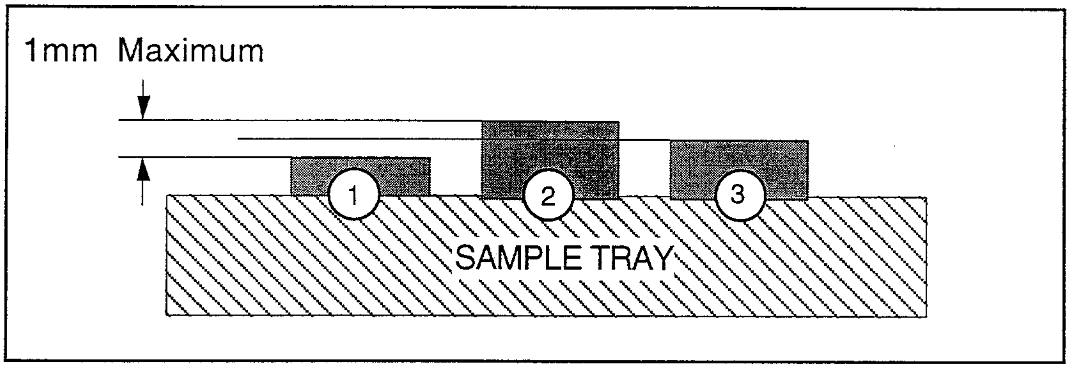

Figure 3.3 Maximum Sample-To-Sample Height Deviation

The maximum allowable deviation between sample height is 1mm. This deviation is measured from the "highest" sample to the "lowest" sample.

3.5.3 Samples With Surface Height Variation.

Some samples themselves will have unusual geometries that require care in setting up the test. A good example of this type of sample is the micro-cantilever beam. It is important to select the proper indentation site so that the indenter is not struck from the side during a repositioning operation.

3.5.4 Surface Slope

While the indenter will automatically compensate for significant sloping of the sample surface, care should be taken that the surface slope does not exceed about 3°. The maximum height compensation the indenter can achieve during a single array of indentations is 1mm.

4.0 The Optics System

4.1 Overview

The Optics system is almost entirely enclosed inside the gantry. However, adjustments to the optics system can be performed without the need to open the body of the gantry. These adjustments include insertion of different objectives, parcentricity adjustment, polarization, iris diaphragm adjustment, and intensity control. Re-alignment of the optic system requires access to the inside of the gantry, and is not recommended without specific instructions from Nano Instruments.

The major components of the optics system are the microscope, the video monitor, and the illuminator.

4.2 The Microscope

The microscope body is fully contained with the Nano Indenter® XP gantry. The only exposed region is at the base of the microscope, in the working area of the gantry. It is in this region that removal of the objective occurs, and all adjustments to parcentricity are made here as well.

There should be no need for the user to access the video camera, microscope alignment screws, or optic focus motor, all of which are housed inside the gantry.

One particular aspect of the microscope's operation is worth noting here. The microscope moves in the Z-direction on an eccentric bearing. Thus, moving the microscope continuously "downward" will result in an upward motion once the bearing rotates past the point of maximum eccentricity. See Section 10.0 for more information about operating the microscope focussing.

4.2.1 Inserting And Removing Objectives

Objective lenses for the Nano Indenter® XP optics system are mounted on precision slides so that they can be interchanged easily. Parcentricity adjustment of the objectives is also enabled by these slides, so that each objective can be aligned and interchange can be accomplished without a need to re-find positions (see Section 7.0). To insert an objective mounted on a slide, simply grasp the objective itself, turn the slide so that the parcentricity adjustment screws point to the front and right sides of the slide, and insert the slide into the mounting rails on the microscope until the slide snaps into place (see Figure 4.1).

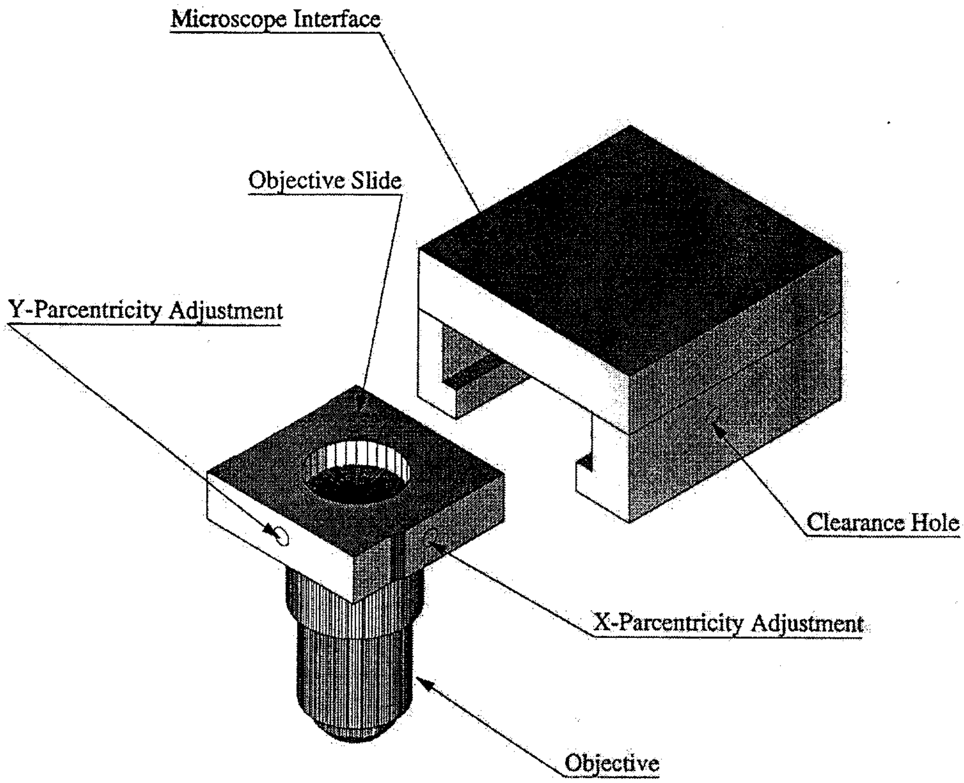

Figure 4.1 Inserting the Objective Slide

To remove the objective, simply reverse the procedure, grasping the objective and sliding it out of the mounting rails.

Because of the nature of the infinity-corrected optics, some height deviation exists in the focal points of the objectives. While this height deviation is insignificant between the higher power objectives, the deviation between the 5X and 20X objectives is large enough to require additional height adjustment of the objective. This height adjustment is accomplished with a spacer ring, which is added to all objectives except the 5X. Make sure that this spacer ring (threaded between the objective and the slide) is in place on higher power objectives.

4.2.2 *Adjusting The Parcentricity*

Adjustment of the parcentricity, or the X-Y focal location, is an iterative process. While the adjustment screws themselves are oriented in the X-Y directions, turning the screws results in an "angular" movement of the focal location. Obviously, the travel enabled on the 5X objective will be less "effective" in terms of screen location than the travel enabled by the higher power objectives. Some adjustment of all objectives will probably be necessary. That is, for an 80X objective to be aligned to the same point as the 5X objective, the 5X objective may need to be set to a different focal location so that the 80X objective adjustment range can meet with that of the 5X objective (see Figure 4.2).

Adjustment is performed with the objective mounted on the microscope. The parcentricity adjustment screws themselves are mounted in the objective slide.

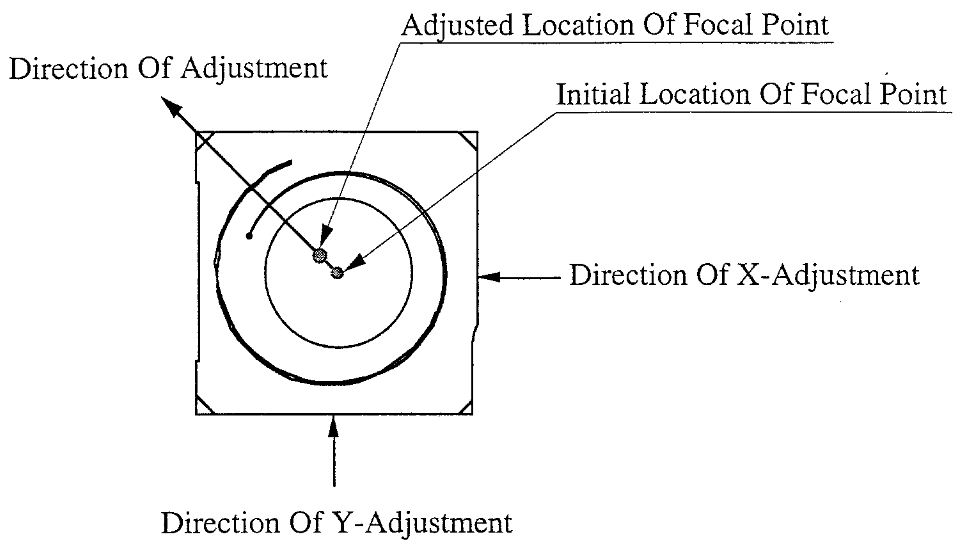

Figure 4.2 Adjustment Of Parcentricity

There is a clearance hole drilled through the side of the microscope mounting rails so that the "X" direction adjustment screw can be reached while the slide is in place. To turn the adjustment screw, use a metric 1.5mm allen wrench.

4.2.3 Adjusting The Polarization

The optic system polarizer is located inside the gantry, and is adjusted by means of a push rod that extends through the front of the gantry. This push rod simply rotates a polarized lens relative to a fixed polarized lens.

4.2.4 Adjusting The Iris Diaphragm

The iris diaphragm is located inside the gantry, and is adjusted by means of a push rod that extends through the front of the gantry. This push rod simply opens and closes the iris.

Centering of the iris is performed at Nano Instruments. There should be no need to adjust the iris center once the instrument is at your site. Contact Nano Instruments if you have questions about the center point of the iris.

The iris diaphragm is sometimes useful in finding the surface of very smooth or translucent specimens. By partially closing the iris, then focussing on the iris itself, you can get a very good estimate of the focal point on the sample.

In addition, opening or closing the iris can enhance the contrast on the sample surface.

The best iris position should be determined through use of the instrument.

4.3 The Video Monitor

The video monitor included with the system is the only output location for the optics system camera. Refer to the manual included with the instrument for more information about the video monitor.

4.4 The Illuminator

The fiber optic illuminator is located with the PC and other remote operating equipment. The illuminator features a power switch and an intensity control knob. The intensity is adjustable from 0 to 100%. The fiber optic cable from the illuminator to the microscope system plugs into the front of the illuminator.

The illuminator uses a halogen bulb, USHIO part number EKE 21V 150W. If you need replacement bulbs, contact Nano Instruments, or your local representative.

5.0 The Gantry & Isolation Systems

5.1 Overview

The Nano Indenter® XP is a complete system, including all components necessary for operation of the instrument. These components include the gantry housing for the primary components of the instrument and the isolation systems that enable operation in otherwise unsuitable environments.

5.2 The Gantry

The Nano Indenter® XP gantry provides isolation of the internal components of the indentation, positioning, and optic systems. All areas of user interface are left exposed, while the cables, electronics, and mechanical parts are enclosed in either the gantry itself or the protective back cover.

There should be no need for the user to access the inside of the gantry. All cable connections are provided on the outside surface of the gantry. There are no user-serviceable parts inside the gantry body.

If you suspect there is a need to access the inside of the gantry, please contact Nano Instruments first in order to avoid damage to the internal components.

5.3 The Isolation System

The isolation system for the Nano Indenter® XP is divided into two major components: the environmental isolation cabinet and the vibration isolation table.

5.3.1 The Environmental Isolation Cabinet

In order to prevent thermal or acoustic disturbance of the system during operation, the Nano Indenter® XP is provided with a cabinet that fully encloses the system. The cabinet is constructed of wood and lined with eggshell foam so that it provides maximum internal damping of air movement.

For best results, the instrument should always be operated with the cabinet closed.

A useful technique for taking advantage of the thermal isolation provided by the cabinet is to leave your samples in place for several hours prior to testing, so that the samples reach equilibrium with the thermal mass inside the cabinet.

5.3.2 The Vibration Isolation Table

The vibration isolation table is separated from the isolation cabinet for maximum protection against transmitted mechanical vibrations. The isolation table is constructed of two primary components: the legs and the table top. The legs contain pneumatic pistons that support the laminated granite table top. When these pistons are properly levelled and provided with the correct air supply, the table can attenuate at least 90% of all vibrations above 7 Hz.

6.0 The Indenter Head

6.1 Overview

The Indenter Head is the heart of the Nano Indenter® XP. All of the other subsystems can be considered as support systems for the indenter head. While the body of the indenter head is enclosed within the gantry, the active area is exposed. This active area is limited to the indenter's diamond tip, which is only exposed during operation of the instrument, or when no power is supplied to the indenter's loading system.

6.2 The Diamond Tip

While operation of the indenter is achieved entirely through the Nano Software interface, there are some occasions when you will work directly with the indenter head itself. The most notable occasion is during the tip change procedure (see Appendix A).

The most common diamond tip geometry used with the indenter head is the Berkovich diamond. The Berkovich diamond is a three sided pyramid-shaped indenter, whose area follows the same function as the Vickers indenter.

Any diamond indenter geometry that is desired can be used with the indenter head, provided that the diamond mount is configured to fit into the indenter shaft. Contact Nano Instruments for more information about alternative diamond tip geometries.

6.3 Working With the Indenter Head

Any time you are working near or with the diamond tip, you should be careful not to accidentally strike or move the indenter, as it is possible to damage the indentation system.

You should be most careful to avoid applying lateral forces to the indenter shaft, as this is the most likely way to cause damage to the displacement sensing system.

It is useful to occasionally run diagnostic routines to test the indenter head. The most useful of these routines are the DAC calibration (Section 11.2.2) and the Test Motion of Indenter function (Section 11.2.3).

If you suspect that the indenter head has been damaged or is not functioning correctly, it is likely that the results of the DAC calibration or the Test Motion procedure will be required in order to correctly diagnose the problem. Thus it is worthwhile to familiarize yourself with these routines.

7.0 Perform Standard Test

7.1 Overview

The result of the "Standard Test" option is dependent upon the standard test defined in the software. Typically, this experiment is defined when the instrument is produced at Nano Instruments, according to your specifications. If you would like to change the Standard Test, contact Nano Instruments. The Standard Test option is simply selected from the Master Menu, which appears when the instrument is first powered on.

The entire procedure for performing a Standard Test can be itemized as:

Select "Perform Standard Test" from the Master Menu
Press the "DO CHOICE" key
Press the "CONTINUE" key
Select the start point of the array using the joystick
Press the "E" key to exit manual control.
Enter the operator name.

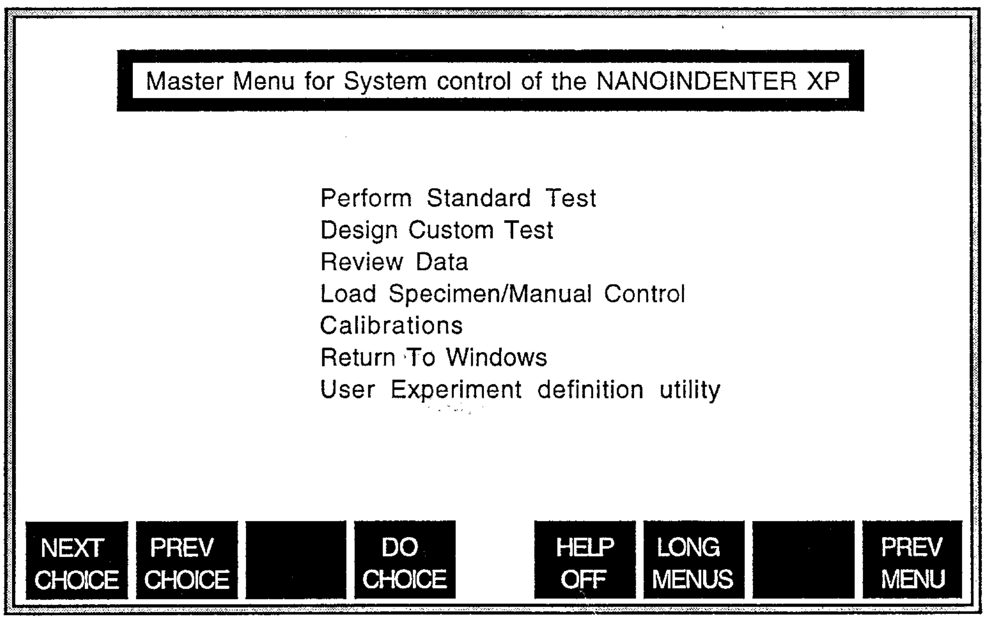

Figure 7.1 The Master Menu

7.2 Select "Perform Standard Test"

To run a standard test, simply select the line "Perform Standard Test" and use the "Do Choice" function key to enter this routine.

Once you have selected and executed the Perform Standard Test option, the following text will appear on the screen:

Please select the start point for this experiment run.

Hit CONTINUE when ready.

The only user input that is necessary in order to set up the standard test is the X-Y position of the first indent in the experiment positional array. To select this point, the joystick is enabled so that you can find a suitable location on the sample surface, using the video system. To enter the manual control utility, simply press the CONTINUE function key[6].

7.3 Select The Start Point

A modified "Manual Control" screen will appear once you have pressed the CONTINUE function key.

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* * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * *
*  MANUAL CONTROL functions:                                                                         *
*  The joystick controls table position                                                              *
*                                                                                                    *
*                        P or p  Moves to a requested position                                       *
*                        X or x  Resets the USER X position counter                                  *
*                        Y or y  Resets the USER Y position counter                                  *
*                        U or u  to change units of display                                          *
*                                                                                                    *
*                          ***E or e to exit manual control***                                       *
*  USER COUNTERS- Position (um) X=  1763.84  Y= -454.2                                               *
*  SYS. COUNTERS= Position (um) X=  1763.84  Y= -454.2                                               *
*  Indenter Disp. (V)= -4.99985   Load (V)= 1                                                        *
* * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * *

WAITING FOR NEXT COMMAND
```

Figure 7.2 The Modified Manual Control Screen

For information about the command functions displayed on the screen, refer to Section 10.0. Of course, it is not necessary to use any of these commands. Simply use the joystick to move the sample and focus the microscope, and once you have found the desired location for the first indent, hit the "E" button on the keyboard to exit manual control. The position under the cross-hairs on the video screen will be the start point for the Standard Test indent array.

7.4 That's It!

It is still necessary, however, for you to enter the "operator name" at the prompt:

[6] At this level in the software, the function keys *f*1 through *f*4 will all enable the CONTINUE function.

Who is the operator?

Generally, entering the initials of the operator is acceptable, although more characters can be used if desired (up to a limit of 100).

This is the last user input that is required. Once you exit the manual control screen, the software takes control of the system, performs the tests, calculates the data, and prints a report to the printer. The data in the report is also stored on the hard drive of the computer.

7.5 The Abort Run Function Key

Once a test begins to run, one of the function keys (f1) will display the option to abort the run. This is an extremely useful option, far preferable to performing a shut-down of the instrument (although the shut-down method is still prefered in the case of emergencies).

The Abort Run key must be pressed repeatedly until the key changes to "Remove Abort," as displayed on the computer screen. Once this change is made, the run will be aborted upon completion of whatever operation the instrument is executing.

Thus, if the Abort Run key is used during an indent, upon completion of theindent the computer will calculate the data and return the sample to its position under the microscope.

8.0 Design Custom Test

8.1 Overview

The "Design Custom Test" option adds flexibility to the Nano Software that enables the user to define experiments beyond the "standard experiment" (see Section 7.0).

Although in most cases, tests will be performed using the standard experiment, the additional flexibility provided by the custom test definition extends the Nano Indenter® XP into more detailed research, allowing the modification of the depths, loads, or indent positions of an experiment.

The Design Custom Test option is selected from the Master Menu (Figure 8.1) using the function keys (for a description of the function key operation, see Section 2.7.2.1).

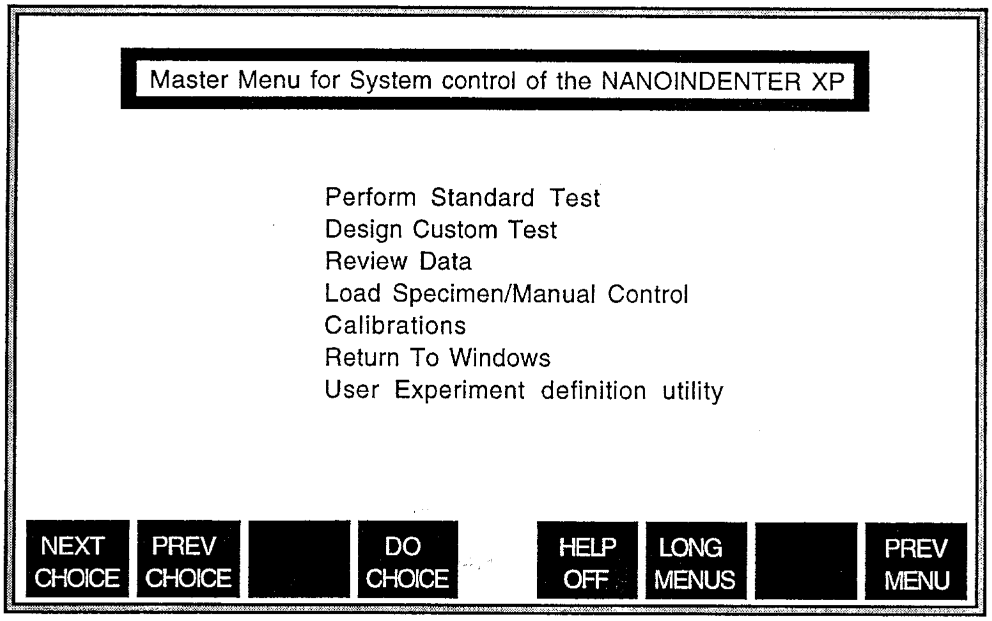

Figure 8.1 The Master Menu

8.2 The Custom Test Menu

When "Design Custom Test" is selected from the Master Menu, a second menu appears, displaying the various options that define the custom test (see Figure 8.2).

8.2.1 Test Set-Up Explained

A test is defined by a number of parameters. These include the position at which indents are to be placed, the indentation procedure to perform at those locations and the data filing information to be included with the final test report (and sometimes in the data files as well).

8.2.1.1 Indent Positions

Indent positions are selected in a number of ways, but a start point is always required. In most cases, the start point is selected by using the joystick to move the sample under the microscope and then selecting the start point manually. Once the start point is selected, an array of indents can be performed, with the first indent placed at the selected start point. You can then go on to define additional arrays.

Alternatively, you can select each indent's position individually, simply using the joystick to move the sample and selecting each position manually.

These two methods (arrayed and individual positions) can be combined in a single test. See Sections 8.2.6 and 8.2.7 for the specific instructions for selecting positions.

You can re-enter either of these two menu options (array of positions or individual positions) as many times as necessary to define the positions you need. Each time you enter one of these routines, you will be defining a "subshape." You can add as many subshapes as you like, up to the limit of 999 positions (see Section 8.2.2.2). The total set of all subshapes you define is the "shape," which is the positioning pattern for the entire test.

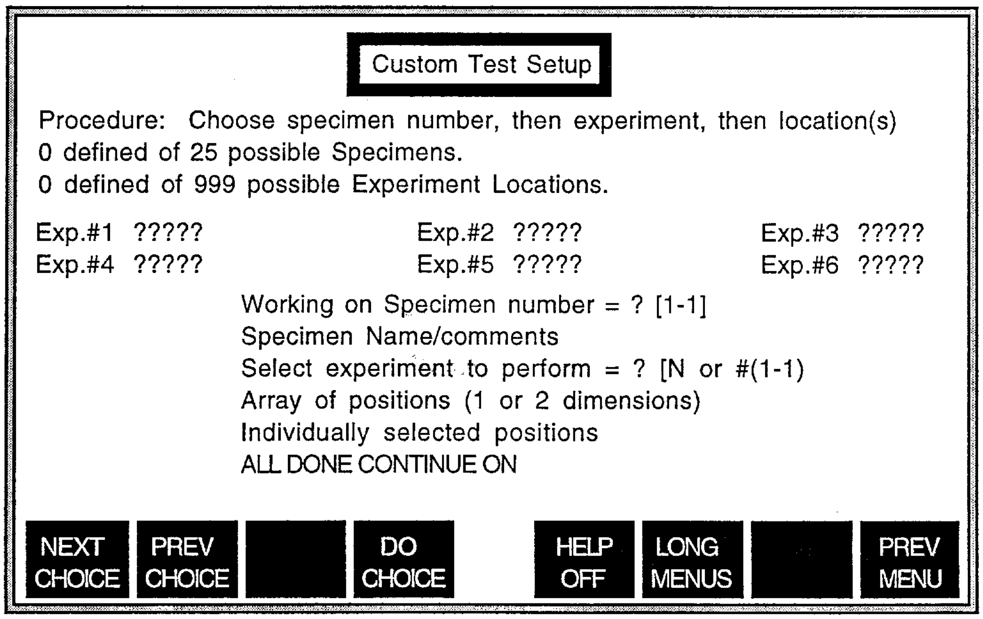

Figure 8.2 The Custom Test Setup Menu

8.2.1.2 Indentation Procedure

The indentation procedure governs the actual experiment to be performed. This procedure is made up of individual operations, such as "load," "unload," "hold," etc. It is not necessary for the user to define each of these segments. It is only necessary to define the desired "maximum" for the experiment. This maximum can be load or displacement controlled.

You can use up to six different indentation procedures in a single test. For example, if you define a max-load experiment and then later a max-depth experiment, you can assign these experiments to different positions. If you like, you can perform the first 25 indents with the max load, and the next 60 indents with the max depth, etc.

You can re-enter this menu option as many times as necessary to define the positions you need. Each time you enter this routine, you will be defining an "indent experiment." You can add as many indentation procedures as you like, up to the limit of six (see Section 8.2.2.3).

8.2.1.3 Data Filing Information

Data filing information does not actually affect the performance of the test. It does, however, help locate and recognize data. Thus, the ability to assign a specimen name to a given set of indent positions enables you to later recognize the sample on which those indents were performed. For example, if indents 1-50 were performed on a fused silica sample, and indents 51-75 were performed on aluminum, you could assign specimen names so that the report recognized that these positions referred to the samples you defined.

Since multiple samples can be tested in a single test, a sample number is used to reference the different sample definitions.

One important concept related to the data filing information is that of sample tracking. There is no way for the software to recognize whether you have set up positions on a new sample or not. Thus, the software must rely on your input in order to correctly format the output data. This is critical when it comes to defining the specimen number.

See Section 8.2.3.1 for more information about the specimen number definition.

8.2.2 Custom Test Menu Screen Displays

At the top of the menu, a running count of various menu parameters is displayed. These counters are updated as changes are made to the custom test definition.

8.2.2.1 Possible Specimens

Up to 25 specimens may be specifically defined in a single experiment. The term "specimens" in this case refers to the user input definitions of each specimen or sample. Thus, it is possible to define several specimens on a single sample, or several samples as a single specimen. It is only for your convenience in record sorting that the specimen names are requested.

Note that the limit of 25 specimens refers to the number of specimen names that can be input, not a physical limit on the number of different specimens that can be used with the instrument at one time.

8.2.2.2 Possible Experiment Locations

Up to 999 indentations may be made in a single test. The on-screen counter shows the current total of the selected indentation sites.

For example, if you have defined a 5x5 array for an experiment, when you return to the Custom Test Setup screen after the array definition, the counter will display:

25 defined of 999 possible Experiment Locations.

As more positions are defined, the counter will continuously update the displayed value.

8.2.2.3 Experiment Numbers

The experiment numbers displayed on the Custom Test Setup screen refer to the experiments defined in the current test setup, rather than permanently stored experiments. You can define up to six different experiments in a single run.

For example, if you define a "hardness" experiment under load control to a load of 1000 μN, and this is the first experiment you have defined, when you return to the Custom Test Setup screen, the display will read:

Exp.#1 HL 1000

This indicates the Hardness/Load experiment, with a desired maximum load of 1000 μN.

As the test setup proceeds, you can refer back to these experiment numbers. For example, if the first defined experiment is HL 1000, as shown above, and the second defined experiment is a Hardness/Depth experiment to a maximum depth of 400 nm, i.e.:

Exp.#2 HD 400

then in additional experiments to be added you can refer to either of these experiments when adding indents to the total run.

Refer to Sections 8.2.3 through 8.2.7 for more complete information about these topics.

8.2.3 Working On Specimen Number

The specimen number is the serialized number linked to the information entered in "Specimen Name and Contents." For example, Specimen 1 may have been defined as "Aluminum Sample", and Specimen 2 as "Fused Silica Sample." If you want indents performed on the Aluminum Sample, you would select "1" when asked for the specimen number. Note that this Specimen Number is for data keeping purposes only (see Section 8.2.1.3). When you select this option from the menu, the following text appears:

Working specimen number [1-1] ?

If you are preparing only one specimen, or working on the first specimen, the response should obviously be "1." Upon entering this value, you will be returned to the Custom Test Setup screen. You will notice, however, that the "Working On Specimen Number" option line on the screen now reads as:

Working on Specimen number = 1 [1-2]

The software is indicating that, having defined one specimen, you now have the option to define a second specimen, but that you are currently working on specimen #1. Any information you enter or change in the other Custom Test Setup options will pertain to the specimen number shown at this line.

8.2.3.1 Tracking Specimen Information

As mentioned in Section 8.2.1.3, it is very important that the specimen number is accurately used when setting up a test. For example, suppose you are setting up a test with three samples: aluminum, fused silica, and a silicon substrate. The aluminum sample is the first sample you have under focus, so you define this sample as Specimen 1, define an experiment to be performed, and define an array of 50 indents. Next, you change the specimen you are working on to Specimen 2 and name the specimen "fused silica." You then define an array of 30 indents using the same experiment defined for aluminum. Up to this point, all data has been entered correctly, and when the report prints it will correctly group the appropriate indents with the correct specimen information (that is, indents 1-50 will be reported with the aluminum specimen, and indents 51-80 will be reported with the fused silica specimen). However, if you then proceed to define a new experiment, and define an additional array of 25 indents on the silicon substrate without changing the Specimen Number to define a third specimen, then the report will include those 25 indents with the fused silica data (since Specimen 2 was the last specimen used), which could lead to confusion when viewing the results of the test. Although the indents will be performed on the silicon substrate, the software will have no way to know these positions are on an undefined specimen, and will group these indents with another specimen.

8.2.3.2 Specimen Number And Positions

The software includes the flexibility to "return" to a specimen to add more indents, but you should take care when using this ability to carefully track the specimens for the reasons described in Section 8.2.3.1. An example of this application is the case where you would like to run a standard sample before and after all other indentations.

Suppose you are testing an aluminum sample and you would like to run a fused silica sample as the standard. You plan to run the first 10 indents on fused silica, then run 50 indents on aluminum, and then another 10 indents on the same fused silica sample.

The proper method for performing this pattern would be to define the fused silica sample as Specimen 1, define the experiment to be performed, then select the positioning on the fused silica sample. Next you would define Specimen 2 as the aluminum sample, define the experiment (or use the experiment you have already defined), and select the positions on the aluminum sample. Finally, you would change the "Working On Specimen Number" value back to "1", define the experiment to be performed, and select new positions on the fused silica sample again.

When the report prints, the fused silica data will be properly grouped together, and the aluminum data will be separately reported.

Alternatively, you could define the fused silica sample as Specimen 3, so that you could compare the two groups of fused silica data as if they were two different specimens.

8.2.4 Specimen Name and Comments

Like the Specimen Number, the Specimen Name is for data keeping purposes only (see Section 8.2.1.3). The specimen name and comments can be up to 150 characters total, and can contain any text you wish to enter. When you select the "Specimen Name and Comments" option from the Custom Test Setup menu, you will be prompted:

```
Specimen Name/Comments [<150 characters] ?
```

Suggested specimen name and comments information are the material type, condition of the sample, your sample number, run number, or process information. Any information you would like to enter can be entered, of course. Once you have entered the information, you will be returned to the Custom Test Setup Menu.

8.2.5 Select Experiment To Perform

When you select the "Experiment To Perform" option, a new menu screen is displayed (Figure 8.3).

The options on this menu enable the definition of the type of experiment you would like to perform.

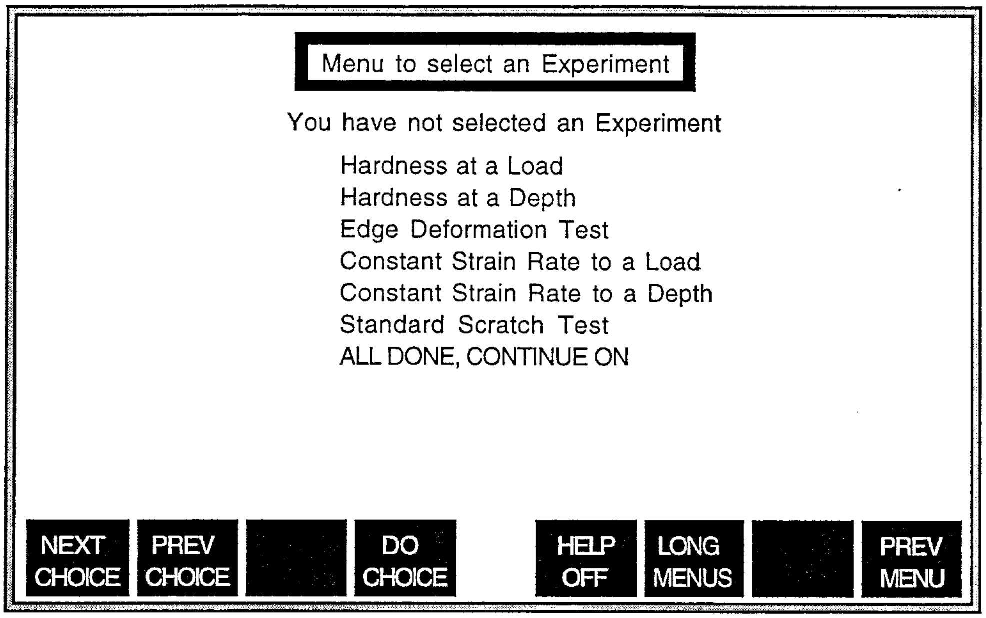

Figure 8.3 The Experiment Selection Menu

8.2.5.1 Hardness At A Load

When the Hardness At A Load option is selected, you will be prompted:

Target Load in microNewtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]

The target load refers to the maximum load you want to achieve with this experiment. The loading segment will terminate when you reach this load. The load is to be entered in microNewtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

8.2.5.2 Hardness At A Depth

When the Hardness At A Depth option is selected, you will be prompted:

Target Depth in nanometers (nm)?
1-6 digits numeric [nnnnnn or n.nExx]

The target depth refers to the maximum depth you want to achieve with this experiment. The loading segment will terminate when you reach this depth. The depth is to be entered in nanometers, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

8.2.5.3 Edge Deformation Test

When the Edge Deformation Test option is selected, you will be prompted:

```
(Pre/post)-Load in micro Newtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]
```

The Pre/post Load refers to the initial and final loads you want to achieve with this experiment. The loading segment will terminate when you reach this load, for both the initial "Pre-load" and the final "Post-load." The load is to be entered in microNewtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

Once you have entered the Pre-Load, you will be prompted:

```
Peak Load in micro Newtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]
```

The Peak Load refers to the maximum load you want to achieve with this experiment. The loading segment will terminate when you reach this load. The load is to be entered in microNewtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

The Edge Deformation Test is an experiment designed to test the durability of razor blade edges by examining the deformation of the edge of the blade as a load is applied. In the typical Edge Deformation Test, a pre-load will be applied to the edge of the razor, then a "Peak Load" will be applied. Finally, the indenter will return to the post-load, which is typically the same force as the pre-load. The difference in displacement between the pre-load, peak-load, and post-load cases is examined.

8.2.5.4 Constant Strain Rate To A Load

When the Constant Strain Rate to a Load option is selected, you will be prompted:

```
Final Load in micro Newtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]
```

The final load refers to the maximum load you want to achieve with this experiment. The loading segment will terminate when you reach this load. The load is to be entered in microNewtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

The Constant Strain Rate test is an experiment designed to achieve a constant indentation strain rate during loading. For more information on this method, refer to Reference J.3 in Appendix J.

8.2.5.5 Constant Strain Rate To A Depth

When the Constant Strain Rate to a Depth option is selected, you will be prompted:

```
Target Depth in nanometers (nm)?
 1-6 digits numeric [nnnnnn or n.nExx]
```

The target depth refers to the maximum depth you want to achieve with this experiment. The loading segment will terminate when you reach this depth. The depth is to be entered in nanometers, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

The Constant Strain Rate test is an experiment designed to achieve a constant indentation strain rate during loading. For more information on this method, refer to Reference J.3 in Appendix J.

8.2.5.6 Standard Scratch Test

Note that the Standard Scratch Test is only useful if your system is equipped with the Lateral Force Measurement (Scratch) Option. If your system is not equipped to perform scratch tests, do not attempt to use this option. **It is possible to damage the instrument by performing scratches without the proper hardware and software limitations!**

When the Standard Scratch Test option is selected, you will be prompted:

```
Starting Load in micro Newtons (uN)?
 1-6 digits numeric [nnnnnn or n.nExx]
```

The Starting Load refers to the initial load you want to achieve with this experiment. The initial loading segment will terminate when you reach this load. The load is to be entered in micro Newtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

When the standard scratch experiment is performed, this Starting Load will be the load applied to the sample during the initial profile, pre and post-scratch scan segments, and final profile segments. For more information on the Standard Scratch, refer to Section 18.

Once you have defined the Starting Load, you will be prompted:

```
Ending Load in micro Newtons (uN)?
 1-6 digits numeric [nnnnnn or n.nExx]
```

The Ending Load refers to the final load you want to achieve with this experiment. The "scratch" loading segment will terminate when you reach this load. The load is to be entered in micro Newtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

When the standard scratch experiment is performed, this Ending Load will be the final load reached during the scratch segment. During the scratch segment, the instrument will continuously ramp the load from the Starting Load to the Ending Load as the indenter travels across the surface over the scratch length. For more information on the Standard Scratch, refer to Section 18.

8.2.5.7 ALL DONE, CONTINUE ON

When you select this option, the experiment is recorded into the Test Set-Up, and you are returned to the Design Custom Test screen.

8.2.5.8 Defining Multiple Indentation Experiments

Once you have defined the first indentation experiment, you will need to proceed to define the positions for this experiment. Normally, you will proceed on to define either an Array of Positions or define Indvidual Positions.

If you re-enter the "Select Experiment To Perform" option, your choices will overwrite the previously defined experiment. Thus, the experiment displayed as "Exp.#1" at the top of the Custom Test Setup menu will be replaced by the new experiment.

If, however, you would like to use multiple indenation experiments in a single test run, then you will need to iterate between the Select Experiment To Perform option and the Array of Positions or Individually Selected Positions options.

8.2.5.8.1 An Example Of Multiple Indentation Experiments

As an example, suppose you have defined a Hardness at a Load experiment, and then proceeded to define a 4x4 array of indents. Upon completion of the array definition, you will be returned to the Custom Test Setup menu.

You now have the option to define more indentation experiments. If you re-enter the Select Experiment To Perform option, you can choose either "N" to indicate a new experiment, or "1" to indicate that you want to use the experiment you have already defined (the Hardness at a Load experiment mentioned above).

Suppose you want to define another experiment, and choose "N". Now you can add another Hardness at a Load, or a Constant Strain Rate to a Depth, or any of the other options. Upon completion of the definition of the experiment, when you are returned to the Custom Test Setup menu, the experiment you have defined will now be shown on the screen under "Exp.#2." You can then proceed to define the Array of Positions or Individual Positions for this experiment.

At the completion of the position definition, you can add a third experiment if you like, or proceed with the tests. You can continue with this method to add up to six different experiments, and you can re-use any of the six experiments to define more posiitions if you like.

8.2.6 Array Of Positions

The most common method of position selection is to define an array of positions. Such an array can be one or two dimensional, can have an angle associate with it's relative positioning, etc.

When this option is selected, the manual control screen is displayed.

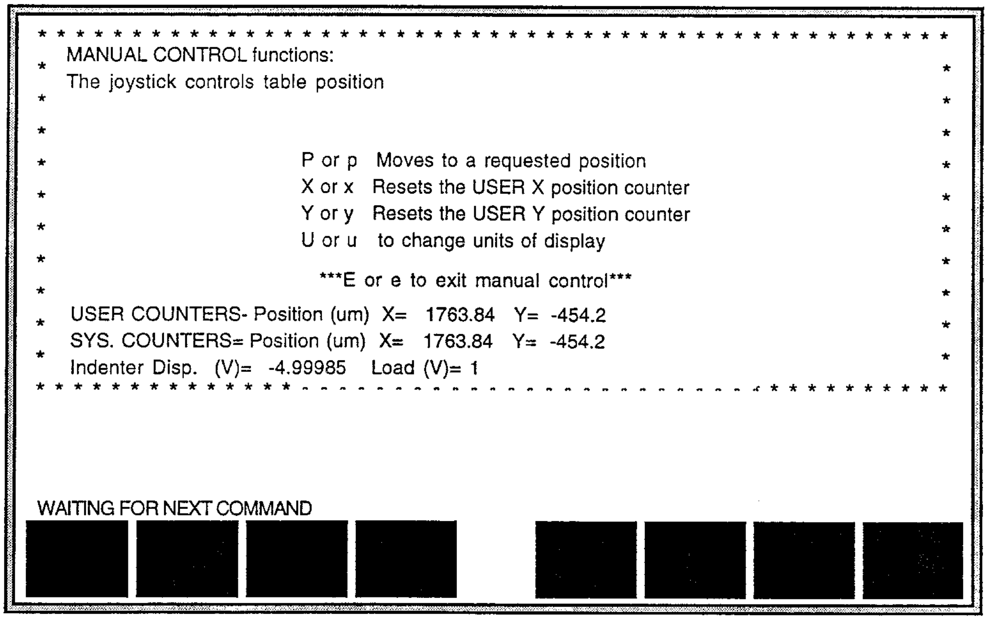

Figure 8.4 The Modified Manual Control Screen

For more information about the manual control function keys, see Section 10.0.

To define the array, use the joystick to move the sample under the microscope until the desired start point of the array is found. To select this point (positioned under the cross-hairs on the video monitor) simply exit from manual control. Once you have done so, you will be prompted:

No. of indents in the X dimension

Enter the number of indents for the X dimension of the array. After you answer this question, you will be asked:

No. of indents in the Y dimension

Again, the response to this question should be the number of indents in the Y dimension of the array.

If you enter "1" for both X and Y, you have defined an array of one indent. Thus, spacing between indents is not needed, and you will be returned to the Custom Test Set-Up Menu. If you enter a value greater than "1" for either X or Y, you will be prompted:

```
Input the angle (0-360) between the X axis of the tables
and the X-axis of the array
```

In most cases you will not desire an angle for the array to be rotated. If you do desire rotation of the array, simply enter the angle you would like the array rotated (in degrees). This angle affects only this array, and not any previous or subsequent arrays. After responding to the angle prompt, the computer requests information about the spacing between indents. You are only required to enter information when there is more than one indent in a specific direction. Thus, if you entered a number of indents greater than one for the X dimension, you will be prompted:

```
Spacing between indents in the X dimension (+ or -um)
```

The spacing between indents in the X dimension refers to the absolute spacing between indents in the X direction in micrometers, before any rotation of the array. If the number of indents in the Y dimension was specified as "1", no further information is needed to define the array, and you will be returned to the Custom Test Set-Up Menu. If the number of indents in the Y dimension was greater than "1," you will be prompted:

```
Spacing between indents in the Y dimension (+ or -um)
```

This desired spacing between Y dimension indents is the absolute spacing between indents in the Y direction in micrometers, before any rotation of the array.

When all of this input is provided to the software, the subshape you have defined will be displayed on the screen. You will be offered the opportunity to accept or re-define this subshape. Once you answer this last question, you will be returned to the Custom Test Set-Up Menu.

8.2.7 Individually Selected Positions

Upon selecting this option you will be prompted:

```
Select how many experiment positions [0=exit, 1-999]
```

Enter the number of individual positions you would like. Once this information is entered, the manual control screen will be displayed (see Section 10.3). Use the joystick to select the position you desire, and use the exit key to leave manual control (see Section 10.3.11). The software will prompt you to select the next point, and when you press the CONTINUE function key, the manual control screen will appear again. This pattern will continue until you have selected all of the desired positions. Once you have defined the last desired position, you will be offered the opportunity to accept or re-define this subshape. Once you answer this last question, you will be returned to the Custom Test Set-up Menu.

8.2.8 ALL DONE CONTINUE ON

After selecting all of the indent positions, experiments, and specimens, it's time to "continue on." Upon selecting the "ALL DONE, CONTINUE ON" option, a final menu screen will appear

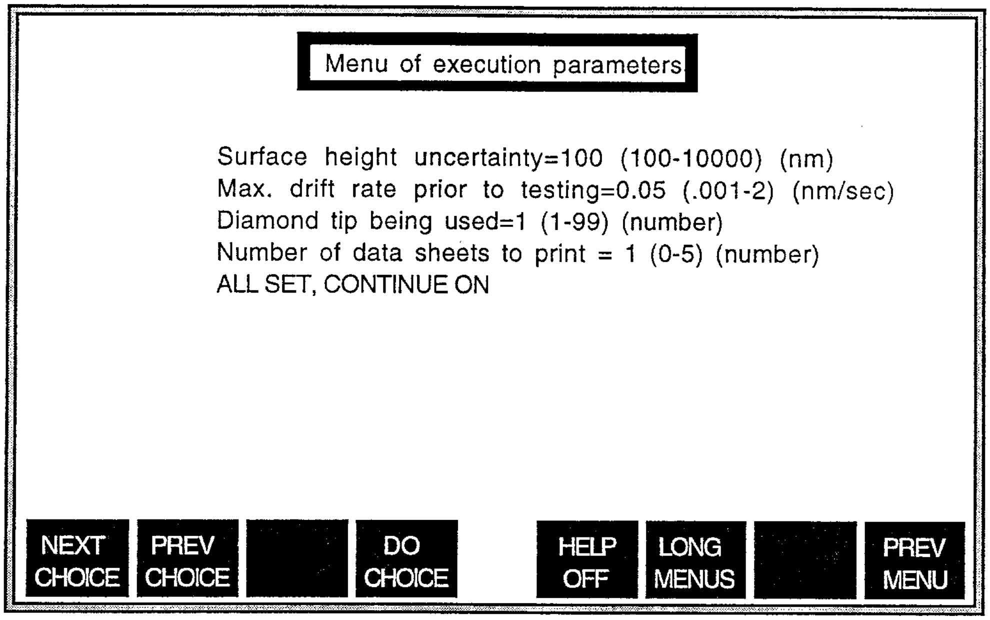

Figure 8.5 The Menu Of Execution Parameters

This menu contains the parameter options for the execution of the test as defined in the previous sections.

8.2.8.1 Surface Height Uncertainty

The surface height uncertainty refers to a "safety zone" above the surface of the sample. This input distance marks the estimated "roughness" to the best estimate of the user. Inputting an incorrect value will not affect the data, or damage the instrument, but it may cause the indenter to have more "false starts" or problems in surface recognition.

8.2.8.2 Maximum Drift Rate Prior To Testing

Setting the maximum drift rate allowable before the onset of testing allows you to define the beginning of the test based on the environmental conditions. The drift rate refers to the thermal instability of the environment. Setting the maximum drift rate instructs the system to begin testing when the drift rate has reached the desired value. Obviously, the more thermally unstable the environment, the higher you must set the drift rate in order to have the test begin. The default value is recommended.

8.2.8.3 Diamond Tip Being Used

Entering a value in response to this option allows you to define the coefficients of the diamond tip area in terms of a calibrated set of coefficients stored in the Nano Indenter® XP's calibration data array (see Section 11.0).

In most cases you will use the default value, unless you have changed the diamond tip and inserted new calibration data for the new diamond tip since the last test was performed.

8.2.8.4 Number Of Data Sheets To Print

As a default, only one copy of the data sheets is printed during a test. The data sheets contain information about the completion and performance of each indentation. If you would like more than one copy of these sheets, you can specify the number of copies by changing the value at this line.

If you would like no copies printed, you can change the number of desired copies to zero (0).

As a general rule, the data sheets are useful only to verify that there are no problems in the performance of the indentations. Thus, for a successful test run (no indentation failures), the data sheets are not particularly useful. When problems occur with the indentations, however, the data sheets are invaluable as a source for tracking the problem and discovering the source.

8.2.8.5 ALL SET, CONTINUE ON

Selecting this last option will end the required user input, and the test will run automatically.

It is still necessary, however, for you to enter the "operator name" at the prompt:

Who is the operator?

Generally, entering the initials of the operator is acceptable, although more characters can be used if desired (up to a limit of 100).

Once the test is finished, the raw data files will be automatically reduced and stored on the hard drive. The final report will be output from the printer. As it is possible to setup a test that will run over many hours, it is often advisable to setup such tests at the end of the day, since the instrument will operate entirely without user input while performing tests.

8.3 The Abort Run Function Key

Once a test begins to run, one of the function keys (f1) will display the option to abort the run. This is an extremely useful option, far preferable to performing a shut-down of the instrument (although the shut-down method is still prefered in the case of emergencies).

The Abort Run key must be pressed repeatedly until the key changes to "Remove Abort," as displayed on the computer screen. Once this change is made, the run will be aborted upon completion of whatever operation the instrument is executing.

Thus, if the Abort Run key is used during an indent, upon completion of theindent the computer will calculate the data and return the sample to its position under the microscope.

9.0 Review Data

9.1 Overview

The Review Data menu provides options for the manipulation and reduction of data. When a test is run with the instrument, the data files are stored to the hard drive (see Section 2.0). The Review Data options access these data files and perform various operations on the data.

9.2 Conventions For Selecting Data Files

A method of selecting data files is standard across all of the review data menu options (both "short menu" and "long menu.") This standard input format, called the "File Manipulation Menu," appears when any of the review data menu items are selected.

9.2.1 Basename For Input Data Files

The "basename" of a data file refers to the two digit prefix and the filename as selected during test set-up. Two examples of basename structure are shown in Table 9.1.

Basename Conventions			
File Type	**Test Type**	**Run Number**	**Base Name**
R	H	ABC	RHABC
D	T	8B8	DT8B8

Table 9.1 Basename Conventions

The Nano Indenter® XP uses a standard set of conventions for creating and manipulating the experiment data files.

As shown in Figure 9.1, there are many possible basenames. The basename structure is dependent upon the File Type, Test Type, and Run Number. Further extensions to the File Name include the Indent Number, and finally the Data Type.

9.2.1.1 File Type

The File Type refers to the level of manipulation of the data in a file. The various possible file types are:

R - Raw data (direct test output voltage data)
D - Load/Displacement/Time data (Raw Data converted & corrected)
S - Stiffness data

M - Hardness & Modulus data
B - Break Point data
A - Averaged data
C - Hardness - Plastic Only data
E - Hardness - Elastic Only data
Z - Delta Z Displacement data (Razor edge test)
H - Hardness at depth data
P - Profile data

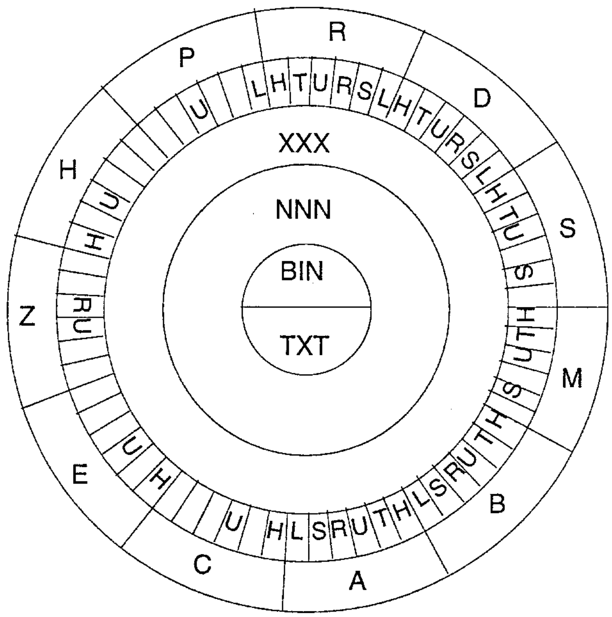

Figure 9.1 The File Name Convention[7]

9.2.1.2 *Test Type*

The Test Type refers to the type of test defined for the specific experiment run. There are six test types: Hardness, Tip Calibration, User Defined, Razor Edge Test., Constant Strain Rate Test, and Scratch Test.

H - Hardness Test: Typical loading to a depth or load to determine hardness and modulus.

T - Tip Calibration Test: Set of tests for the purpose of calibrating the diamond tip area function.

U - User Defined Test: Test set up as defined using the User Definition Utility[8].

---

[7]Note that data file types that are not possible are excluded on this diagram. For example, since it is not possible to generate Profile data from a Razor Edge Test, the "P" segment does not include the "R" secondary prefix.

[8]The User Definition Utility will not be enabled on all instruments. Contact Nano Instruments for more information about this option.

R - Razor Edge Test: A specific test set up for edge-on testing of razor blades.

S - Constant Strain Rate Test: Loading to a depth or load using a constant indentation strain rate.

L - Scratch Test: A test using the lateral force measurement option to apply lateral forces to the sample.

9.2.1.3 Run Number

The run number refers to the software assigned hexadecimal code that indicates the sequential order of a particular test. Thus, the first test run on your Nano Indenter® XP will have a run number of 001, while the tenth test run will have a run number of 00A.

9.2.1.4 Indent Number

The indent number makes up part of the File Name, as opposed to the Base File Name. The indent number simply refers to the data for a specific indent in a series of indents. Thus, an indentation experiment defined with a 5x2 array would have indent numbers 001 through 010.

9.2.1.5 Data Type

The data type refers to the actual format of the data file. BIN indicates a binary data file, and TXT indicates an ASCII text file.

9.2.2 Add a Contiguous Series of Files

In most cases, the series of files you wish to manipulate will be a contiguous series of files. For example, if you are manipulating data from a series of 20 indentations (for the purpose of this example, the series will have been named DH8B8), you will most commonly want to input all 20 data files when performing calculations or otherwise manipulating the data. Selecting the Contiguous Series of Files option calls up a series of menu options.

9.2.2.1 Start File Number of Series

The start file number is the first indent number in the series. In the example being used here, the first file would be DH8B8001. Thus, a "1" should be entered for the start file number.

9.2.2.2 End File Number of Series

The end file number is the last indent number in the series. In the example being used here, the last file would be DH8B8020. Thus, "20" should be entered for the end file number.

9.2.2.3 Display Previously Selected Files

When this option is selected, any files previously selected will be displayed. Because multiple series of files can be defined, this option provides the opportunity to review the files that have already been used in previous series. For example, if we had already defined a contiguous series of files from 1 to 6, and were in the process of defining a second contiguous series, selecting this option would show us that DH8B8001 through DH8B8006 had been selected.

9.2.2.4 Directory of Data Files On Disk

Selecting this option will cause the software to display the contents of the default data directory. This option is useful for verifying that the desired files actually exist, and how many of the desired files are available.

9.2.2.5 ACCEPT THIS SERIES

Accepting the series means that the file series you have specified is added to the list of accepted files. You are returned to the File Manipulation Menu once you have selected this option.

From the File Manipulation Menu, you can continue to enter series of files. For example, if you ran 200 indentations in a test (output file name RH8B8001 through RH8B8200), but you do not want to manipulate all of these files, you can define the different series of these indentations that you would like to manipulate by using multiple entries into the Contiguous Series Menu.

To further illustrate this point, suppose you only wanted to manipulate files RH8B8001 through RH8B8020, and RH8B8051 through RH8B8092. To accomplish this manipulation, you would enter the Contiguous Series Menu, define the contiguous series RH8B8 with files 1 through 20, use the "Accept this series" option to add these files to the list of desired files, and then once you are returned to the File Manipulation Menu, simply re-enter the Contiguous Series menu and define the second series of RH8B8 from file 51 to file 92. After accepting this second series, you are finished selecting files, and can proceed to define the parameters of the data manipulation.

9.3 Segments and Experiment Parameters

Each experiment, whether defined by the user or set up as a "library" experiment, is composed of discrete operations that control the indenter's behavior during the experiment. These discrete operations are called "Segments." Each Segment is itself defined by a set of "parameters" that control the behavior of the indenter during that segment.

9.3.1 Segments

As explained above, a segment is a discrete operation which the indenter performs during a test. Table 9.2 shows the standard segments (those used by the instrument when no optional methods are in place).

Segments & Segment Codes	
Segment	**Code**
Approach	A
Constant Loading Rate Loading	LL
Constant Displacement Rate Loading	LD
Hold	H
Step Load / Unload	S*
Draw (Scratch)	D*
Constant Displacement Rate Unload	UD*
Constant Loading Rate Unload	UL
Constant Strain Rate Loading	LS
*Indicates a segment only available with advanced user packages.	

Table 9.2 Segments and Segment Codes

Each of these segments performs a distinct operation. Experiments may be composed of some or all of these segments, but every experiment must begin with an Approach segment and end with an Unload segment.

An alternative term referring to the segment is "subfile." The two terms are used separately to describe the same part of the experiment data. The term "segment" is used to define the actual operation that the indenter performs, while the term "subfile" refers to a discrete set of data, usually the data for a specific segment. Thus, the software may ask you to select a subfile, and then display the segment codes as the subfiles to be picked.

9.3.1.1 The Approach Segment

The Approach segment defines the operation in which the indenter is moved in contact with the surface. Thus, this must be the first segment in any experiment, since the indenter must first be brought in contact with the sample before any known load or displacement can be applied.

9.3.1.2 The Constant Loading Rate Loading Segment

The Constant Loading Rate Loading segment uses a loading rate to achieve a desired value of load or displacement. In other words, when the experiment is "load-controlled," this segment is used to achieve that load control.

9.3.1.3 The Constant Displacement Rate Loading Segment

When a "displacement-controlled" experiment is desired, the Constant Displacement Rate Loading segment is used. When this segment is defined, the load is applied so that a constant rate of displacement is achieved.

9.3.1.4 The Hold Segment

The Hold segment defines an operation in which the indenter is kept at a constant load, and data is collected.

9.3.1.5 The Unload Segment

The Unload segment is simply an operation during which the load applied is decreased or removed. Because the indenter must be removed from contact with the sample before an experiment can be finished, the last segment in an experiment must be an unload.

9.3.1.6 The Constant Strain Rate Loading Segment

The Constant Strain Rate Loading segment uses a fixed ratio of load to loading rate to achieve a constant or near-constant indentation strain rate. For more information on this segment, see Reference J.3 in Appendix J.

9.3.2 Parameters

Parameters are individual descriptions for command of a segment. Each segment will have some common and some unique parameters. These parameters define the rates and terminating conditions for the segment. In standard operation, these parameters are set when the user defines the experiment (see Section 8.2.5).

9.4 The Review Data Menu

The Review Data menu provides options most useful when the standard indent experiment has been performed, and there is no need for further manipulation of the data. The options provided enable translation or viewing of reduced data, or manipulation of the data. All of the Review Data options are displayed on the main menu. Some options on the submenus, however, are not available as "short menus." For description of the "long menu" options, refer to Part Two of these instructions.

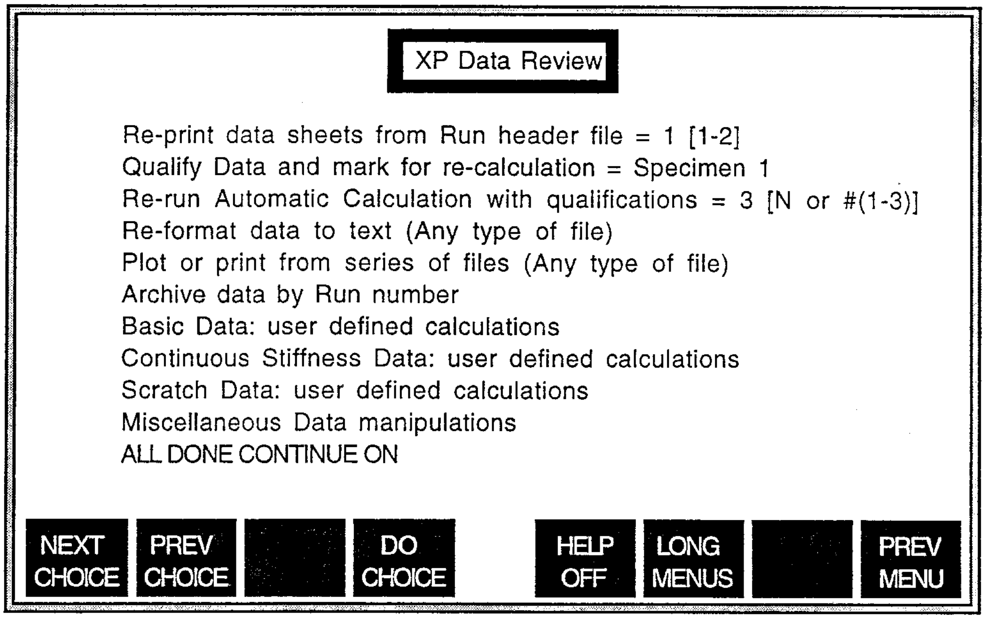

Figure 9.2 The Review Data Menu

9.4.1 Re-Print Data Sheets From Run Header File

The Re-Print Data Sheets From Run Header File option allows you to extract and view or print the data associated with the Header File for a series of indents. The "Header File" is a file created with each test run that describes the test "set-up." This file contains information about the specimens used, the indentation procedures, the positions, and any other information that was entered as part of the total test procedure.

9.4.1.1 Select Run Number

Enter the Run Number from which you want to extract the Header File information (see Section 9.2.1.3).

9.4.1.2 Display Data Sheet

If you would prefer to print a copy of the Header File data sheet, print this option. Refer to Appendix G of these instructions for a sample of the Data Sheet as printed.

9.4.1.3 ALL DONE, CONTINUE ON

When this option is selected, you will be returned to the Review Data menu.

9.4.2 Qualify Data And Mark For Re-Calculation

The "Qualify Data" routine allows you to review the calculated "D" files, and to select or mark files that you do not wish to be included in future calculations of this data.

This option is very useful if a data set contains one or more "bad indents" that can corrupt a set of averaged data. This routine can be used to exclude such "bad indents" from the data set, allowing the use of the calculation stream without review of the data at each step in the calculation process.

9.4.2.1 Select Run Number

Enter the Run Number of the data you want to review (see Section 9.2.1.3).

9.4.2.2 Select Variables To Display

During the Data Qualification routine, you can examine the "D" files in a number of ways, based upon the variables you want to have displayed during the qualification process. All of the variables associated with a "D" file will be available as plot axes, depending on the configuration of the system at the time the data was run. For example, if your system includes the continuuous stiffness measurement option, then you could review the plots of phase angle vs. displacement.

The most useful plot for qualification is a load vs. displacement plot.

When you select this option, you will be prompted:

```
Total number of variables= X
VARIABLES
No.        Name
1  ---  Disp. (nm)
2  ---  Load (mN)
3  ---  Time (sec)
4  ---  S (N/m)
5  ---  Dw (N/m)
6  ---  Tan Phi
```

```
Select X Axis variable number
```

Select the variable you want to use as the X axis of the resultant plot, and press enter. Upon entering the X axis variable, you will be prompted:

```
Select Y Axis variable number
```

Enter the variable you want to use as the Y axis of the resultant plot. Once you have entered the Y axis variable, you will be returned to the "Qualify Data" menu.

9.4.2.3 Examine (Accept/Reject) Data Files

Once the variables for the plot axes are selected, you can examine and accept or reject the data files in the run you are qualifying. Selecting the "Examine Data Files" option will cause the data files to be displayed in plot format, using the variables you have defined as the plot axes. A typical curve is shown in Figure 9.3.

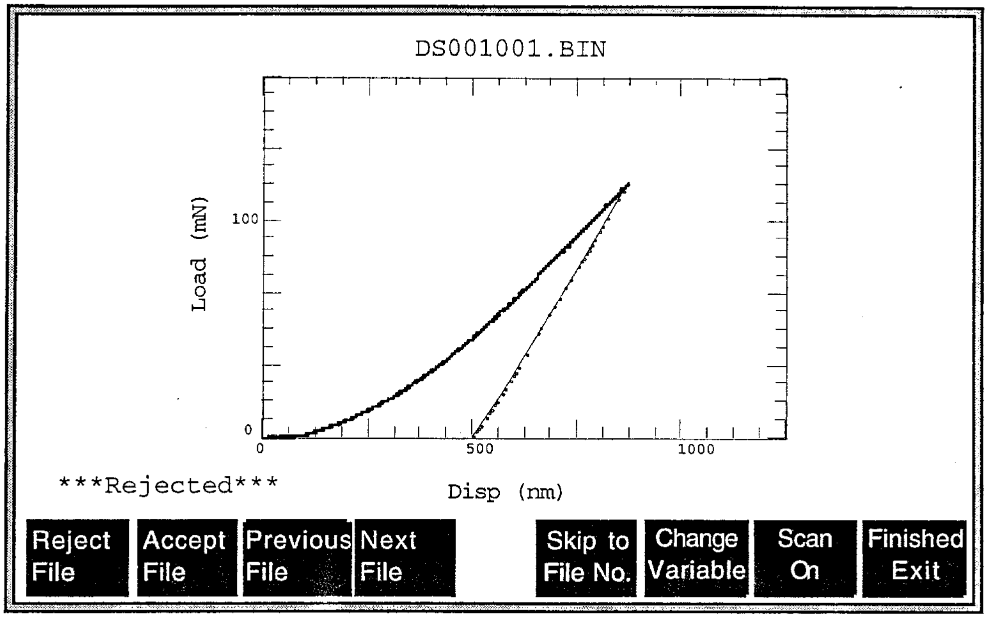

Figure 9.3 A Sample Plot Screen from the Qualification Option.

You can work with the data by using the function keys. The functions associated with keys 1 through 8 are displayed on the plot screen, as shown in Figure 9.3 above.

9.4.2.3.1 *f*1 Reject File

The Reject File key causes the currently displayed data file to be marked as "Rejected." This has no effect, other than the mark itself, until the "Mark Files As Selected" option is used (see Section 9.4.2.4).

9.4.2.3.2 *f*2 Accept File

The Accept File key causes a Rejected data file to be accepted. This has no effect, other than the "Accepted" mark, until the "Mark Files As Selected" option is used (see Section 9.4.2.4).

9.4.2.3.3 *f*3 Previous File

This key causes the Previous sequential file to be displayed on the screen. For example, if the currently displayed file is "DS00B005," when this key is pressed, the computer will display file "DS00B004," if there is such a file. Note that this option "wraps around" the file numbers. That is, if file "001" is displayed out of a set of 25 files, then the use of this option will cause "025" to be displayed.

9.4.2.3.4 *f*4 Next File

This key causes the Next sequential file to be displayed on the screen. For example, if the currently displayed file is "DS00B005," when this key is pressed, the computer will display file "DS00B006," if there is such a file. Note that this option "wraps around" the file numbers. That is, if file "025" is displayed out of a set of 25 files, then the use of this option will cause "001" to be displayed.

9.4.2.3.5 *f*5 Skip To File Number

You can use this key to skip directly to a file number, rather than "paging" through each file.

9.4.2.3.6 *f*6 Change Variable

If you want to alter the plot display so that different variables are used as the axes of the plot, use this option. When this option is selected, the Variable Selection screen will be displayed, and you will be prompted:

```
Select X Axis variable number
```

Select the variable you want to use as the X axis of the resultant plot, and press enter. Upon entering the X axis variable, you will be prompted:

```
Select Y Axis variable number
```

Enter the variable you want to use as the Y axis of the resultant plot. Once you have entered the Y axis variable, you will be returned to the plot display screen, where the currently chosen file will be displayed using the new variables as axes for the plot.

9.4.2.3.7 *f*7 Scan On / Scan Off

If you want to browse the data set without using the Previous or Next keys to change to a new file, you can use the "Scan On" option to cause the software to display each file automatically. The scan will continue until the end of the data file set is reached, or until you press the "Scan Off" Key. Note that performing any of the function key operations also causes the scan to interrupt. Thus, if you want to change variables during a scan, simply press the "Change Variable" key. The scan will be stopped, and the Variable Selection screen will be displayed as described in Section 9.4.2.3.6 above.

9.4.2.3.8 *f*8 Finished Exit

When you have finished examining or marking files, and are ready to proceed with the calculation of these files, press this function key. You will be returned to the "Qualification" screen. Note that until you use the"Mark Files As Selected" option, no changes are actually made to the data file set.

9.4.2.4 Mark Files As Selected During Examination

Once you have completed the examination and acceptance or rejection of data files, you must use this option to change the data set so that rejected files are not included in calculations. This option simply renames the rejected data files so that they do not appear to the software during calculation of a data set. No user input is necessary.

9.4.2.5 Start Over This Run Number

When this option is selected, any changes made to a data set will be eliminated, and the full data set will be restore, if the "Mark Files As Selected" option has not yet been executed. If you have already used "Mark Files," then the "Start Over" option will have no effect upon the data set.

9.4.2.6 ALL DONE, CONTINUE ON

When this option is selected, you will be returned to the Review Data menu. If you have marked files as "Rejected" during the examination of a data set, then these files remain "Rejected" when you select the "ALL DONE" option, and will not be included in subsequent operations on the data set.

9.4.3 Re-Run Automatic Calculation With Qualifications

The "Re-Run" option allows you to perform the entire "Calculation Stream" to provide the final Hardness and Modulus results for a data set. If you have rejected files from the data set, these files will not be included in the "Re-Run" of the Calculation Stream.

9.4.3.1 Select Run Number

Enter the Run Number of the data you want to review (see Section 9.2.1.3).

9.4.3.2 Re-Run Stream

When this option is selected, the data files associated with the Run Number you entered will be run through the automated "Calculation Stream." The final data report will be generated, as described in Section 8.2.8.4. If you are re-running the Calculation Stream for a data set that has been modified by "Qualifying" the data, then the rejected data files will be excluded from the final report.

9.4.3.3 ALL DONE, CONTINUE ON

Selecting this option will cause the software to return to the Review Data menu.

9.4.4 Re-Format Data To Text

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are selected, the Generic Text File Generator menu is displayed.

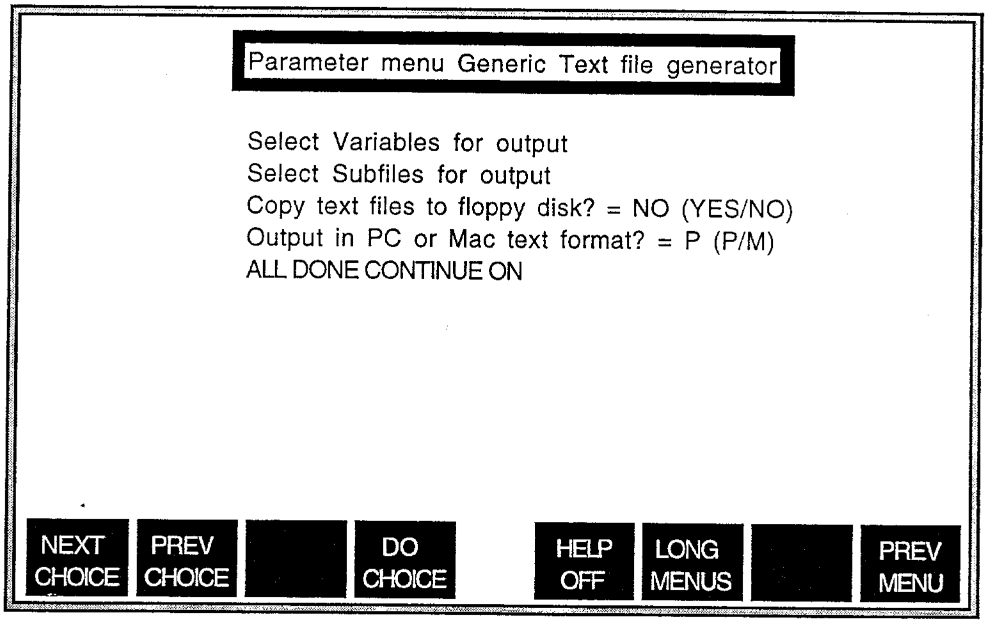

Figure 9.4 The Re-Format Data To Text Menu

Options are selected from this menu using the function keys.

9.4.4.1 Select Variables For Output

Before a file can be converted to text, you must tell the software which variables you want included in the output file.

When this option is selected, a list of the available variables will be displayed at the top of the screen.

VARIABLES	
No.	Name
1 ----	Disp (V)
2 ----	Load (V)
3 ----	Time (s)

Total number of variables = 3

At the bottom of the screen, the software prompt will be displayed:

Which Variables
(Enter Variable Number, 0 if done, or A for all) ?

Variables are selected by entering the number of the desired variable. Entering "A" selects all variables and returns to the previous menu. Selecting "0" ends the entry process and returns to the previous menu, using those variables you selected prior to entering "0." For example, if you only wanted to output the reformatted values of displacement and load, you would enter "1", then enter "2", and then enter "0" to finish selection.

The available variables will change depending on what type of file you are trying to reformat. In the monitor text displayed above, the variables were those for a "raw data" file from the basic configuration of the Nano Indenter® XP (no CSM, LFM, or High Load option installed). Had a "DH" file (raw data converted to appropriate units using the calibration values) been selected, the available variables would have displayed as:

No.		Name
1	----	Disp (nm)
2	----	Load (mN)
3	----	Time (s)

Additional variables may be displayed, depending on the input file.

Note that the order in which you select variables governs the order in which the variables are output to the resultant text file. Thus, if you plan to further reduce the data external to the Nano Software, be sure to output the data in the order that your external application requires.

9.4.4.2 Select Subfiles For Output

The term "Subfiles" is similar to the term "Segment" in this option (refer to Section 9.3.1 for a definition of Segments). Like the variables, you must specify which subfiles you want included in the output.

When this option is selected, a list of the available subfiles will be displayed at the top of the screen.

The number of subfiles = 6

No.		Name		No. of First Observation
1	--------	A	-------	1
1	--------	LL	-------	51
1	--------	H	-------	110
1	--------	UL	-------	139
1	--------	H	-------	197
1	--------	UL	-------	247

The subfile "code" is the one or two letter code displayed under "Name." This code refers to the type of segment corresponding to the subfile number. For more information about the subfile code, see Section 9.3.1).

At the bottom of the screen, the software prompt will be displayed:

```
Which subfile
(Enter Subfile number, 0 if done, or A for all) ?
```

Subfiles are selected by entering the number of the desired subfile. Entering "A" selects all subfiles and returns to the previous menu. Selecting "0" ends the entry process and returns to the previous menu, using those subfiles you selected prior to entering "0." For example, if you only wanted to output the reformatted values of the approach segment and the loading segment, you would enter "1", then enter "2", and then enter "0" to finish selection.

The available subfiles will change depending on what type of experiment was used to generate the file you are trying to reformat. In the monitor text displayed above, the subfiles were those for a "standard experiment" file from the basic configuration of the Nano Indenter® XP.

Note that the order in which you select subfiles governs the order in which the subfiles are output to the resultant text file. Thus, if you plan to further reduce the data external to the Nano Software, be sure to output the data in the order that your external application requires.

9.4.4.3 Copy Text Files To Floppy Disk

By default, the resultant reformatted text files will be copied to the internal hard disk in the PC. You can, however, copy these files to a floppy disk as well, if you so desire.

This option is simply a toggle line on the menu, so selecting "Do Choice" when this line is highlighted will switch back and forth from the "NO" response to the "YES" response.

9.4.4.4 Output In PC Or Mac Text Format

The Nano Software will format the data in ASCII text, readable on either the MS-DOS/Windows or Mac OS platform[9]. When this option is selected, you will be prompted:

```
New value in range ((P/M)) ?
Press enter to accept old value,
or new value to change

?
```

[9]MS-DOS is a registered trademark , and Windows is a trademark of Microsoft Corporation. Mac OS and Macintosh are trademarks of Apple Computer, Inc.

If you want the output file formatted for use with MS-DOS/Windows, select "P." If you want the output file formatted for use with the Mac OS[10],select "M." The default choice is "P."

9.4.4.5 ALL DONE, CONTINUE ON

Selecting this final option will initiate the file re-formatting. If you opted to save the files to a floppy disk as well as the hard drive, the following prompt will appear:

Please insert a blank floppy disk in drive A: and Press Continue

The floppy disk to be used does not need to be entirely blank. No disk formatting operation is performed unless the disk is unformatted. Inserting a floppy disk that already contains data will not cause problems.

Once this last prompt has been resolved, the file translation will begin. Each file's name will be displayed on the screen as it is translated. The files will be saved with the same name, but a different extension. For example, a file translated from "DH8B8001.BIN" will be saved as "DH8B8001.TXT."

9.4.5 Plot Or Print From Series Of Files

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are selected, the Printing or Plotting Parameter menu is displayed.

---

[10]If files are transferred directly over a network from the Nano PC to a Macintosh, the resource fork and data fork information may not be fully prepared, and thus the files may not be readable in the Mac OS. Macintosh platforms using System 7.X, however, automatically add the appropriate forks when opening PC data from a floppy disk. Thus it may be preferable to transfer data files to the Macintosh via floppy disk, rather than transferring over a network. Installing the appropriate conversion software should allow network transfer with both forks.

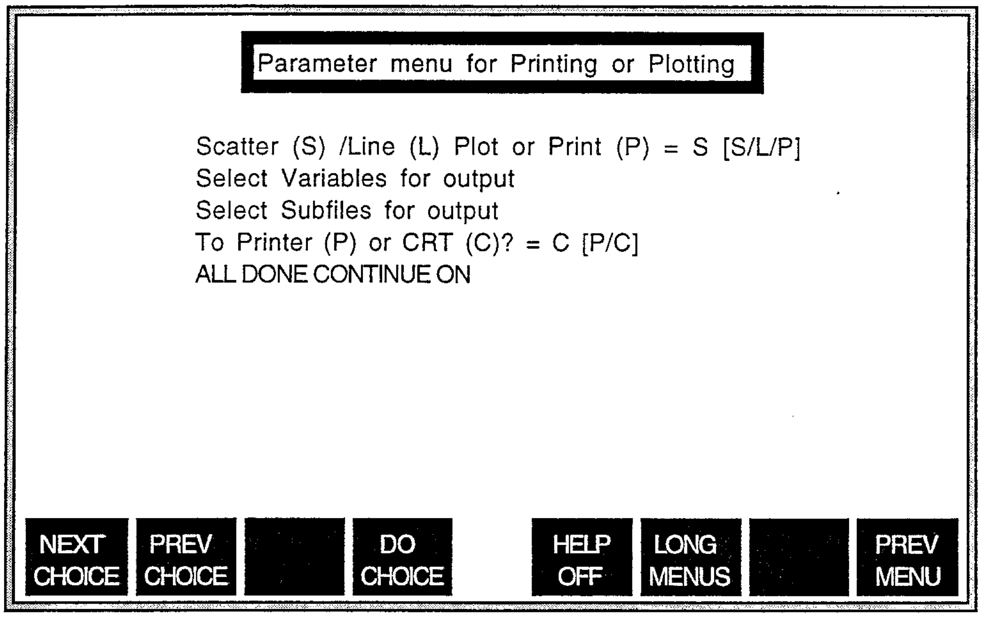

Figure 9.5 The Print or Plot Parameter Menu

Options are selected from this menu using the function keys.

9.4.5.1 Scatter/Line Plot Or Print

There are three options for the Plot or Print routine: Scatter Plotting, Line Plotting, or Printing.

9.4.5.1.1 Scatter Plots

Scatter Plots display the data in a plot format, with the data shown as individual points.

9.4.5.1.2 Line Plots

Line Plots display the data in a plot format, with the data connected by a line running from the first point to the last point using a linear interpolation between successive points.

9.4.5.1.3 Printing

If the Printing option is selected, the data will be displayed as text rather than as a plot.

To select the appropriate method of displaying the data, simply execute the "Scatter/Line Plot or Print" option, and enter the appropriate letter to indicate the desired method. "S" indicates a Scatter Plot, "L" indicates a Line Plot, and "P" indicates a Printed output.

9.4.5.2 Select Variables For Output

The input for this option is dependent upon the selection chosen in the previous menu option, as defined in Section 9.4.5.1. If a Plot was chosen (either Scatter or Line), then this option is used to select the axes for that plot. If a Print was selected, then this option can be used to specify all of the variables desired in the printed output. Regardless of the desired output format, however, when this option is selected, the available variables are displayed at the top of the screen:

```
VARIABLES                 Total number of variables = 3
No.       Name
1   ----  Disp (V)
2   ----  Load (V)
3   ----  Time (s)
```

If a Print was previously selected, you will be asked to input the variables you want included in the printed text (see Section 9.4.4.1 for the specific nature of this prompt). If a Plot was selected, you will be prompted:

```
Select X Axis variable number
```

and then:

```
Select Y Axis variable number
```

Simply enter the desired variable number for each axis, and you will be returned to the previous menu.

9.4.5.3 Select Subfiles For Output

The term "Subfiles" is similar to the term "Segment" in this option (refer to Section 9.3.1 for a definition of Segments). Like the variables, you must specify which subfiles you want included in the output.

When this option is selected, a list of the available subfiles will be displayed at the top of the screen.

```
The number of subfiles = 6
No.          Name        No. of First Observation
1   -------- A  -------  1
1   -------- LL -------  51
1   -------- H  -------  110
1   -------- UL -------  139
1   -------- H  -------  197
1   -------- UL -------  247
```

The subfile "code" is the one or two letter code displayed under "Name." This code refers to the type of segment corresponding to the subfile number. For more information about the subfile code, see Section 9.3.1).

At the bottom of the screen, the software prompt will be displayed:

```
Which subfile
(Enter Subfile number, 0 if done, or A for all) ?
```

Subfiles are selected by entering the number of the desired subfile. Entering "A" selects all subfiles and returns to the previous menu. Selecting "0" ends the entry process and returns to the previous menu, using those subfiles you selected prior to entering "0." For example, if you only wanted to output the reformatted values of the approach segment and the loading segment, you would enter "1", then enter "2", and then enter "0" to finish selection.

The available subfiles will change depending on what type of experiment was used to generate the file you are trying to reformat. In the monitor text displayed above, the subfiles were those for a "standard experiment" file from the basic configuration of the Nano Indenter® XP.

Note that the order in which you select subfiles governs the order in which the subfiles are output to the Print or Plot. For a Print, you can enter the subfiles in any order you desire, although ascending is recommended. For a Plot, however, the subfiles should always be entered in ascending order. While entering the subfiles out of order will have no effect on a Scatter Plot, in a Line Plot, the line running through the data points may be affected by the order the subfiles are entered, so that rather than running continuously through the data, the line "jumps" from segment to segment.

9.4.5.4 To Printer Or CRT

The output from this routine can be sent either to the printer or to the computer monitor (CRT). The default choice is the computer monitor. To change this choice, select this option from the menu, and simply enter "P" to select the printer as the output device.

9.4.5.5 ALL DONE, CONTINUE ON

Selecting this final option will initiate the printing or plotting. If you opted to output data to the computer monitor, then each plot or print will appear on the screen, and you will need to press the CONTINUE function key between each plot or print to continue the display of all files. If you selected the printer as the output device, then all data will be automatically sent to the printer.

Figure 9.6 shows an example of a Scatter Plot as it appears on the computer monitor.

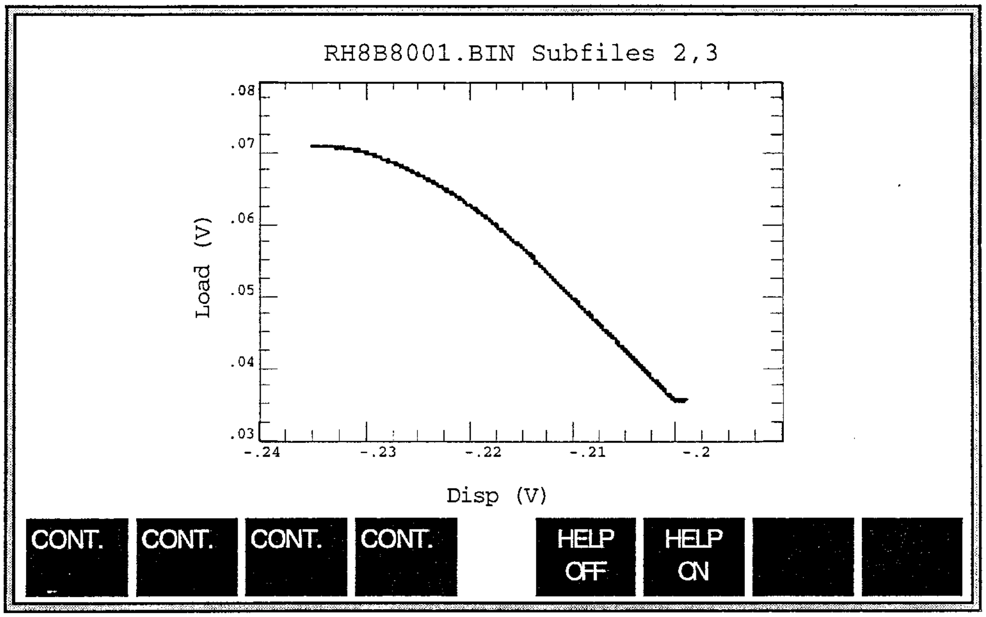

Figure 9.6 A Sample Scatter Plot on the CRT Screen

9.4.6 *Archive Data By Run Number*

As of the writing of these instructions, this routine was not yet operational. Future versions of these instructions will include the procedure for using this routine. If your software is enabled to allow operation of this option, please contact Nano Instruments for an update to these instructions.

9.4.7 *Basic Data: User Defined Calculations*

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

9.4.7.1 Load/Displacement/Time

All data sets should be run through this program. It converts raw voltage data from the data acquisition system into values of load and displacement, and allows the user to use one of several methods to determine the exact point of surface contact. When the file numbers have been defined for this option, the menu of data manipulation parameters is displayed:

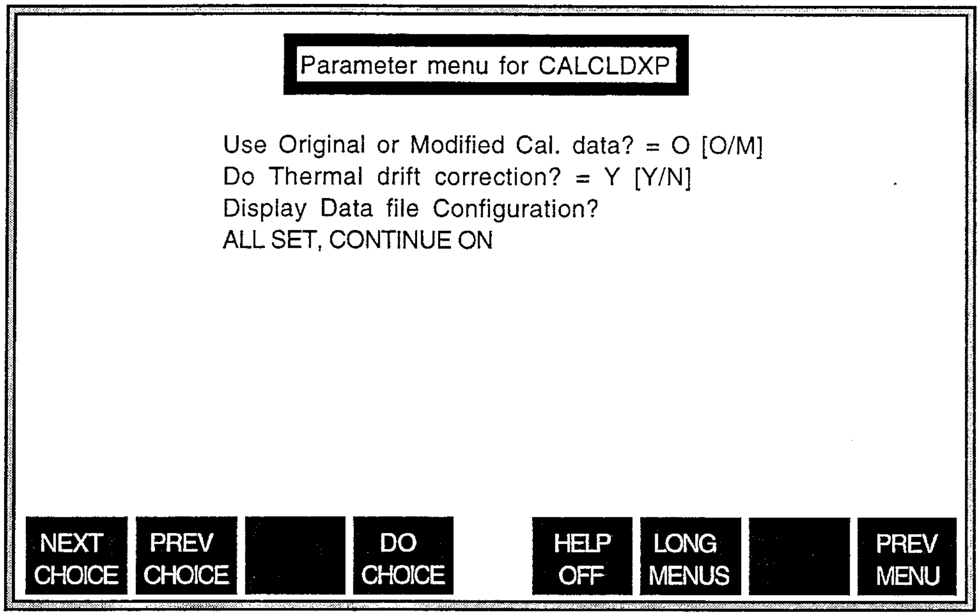

Figure 9.7 The Load/Displacement/Time Parameter Menu

9.4.7.1.1 Use Original or Modified Calibration Data

Certain information related to the calibrations of the Nano Indenter® XP is required in order to generate the load/displacement/time data from the raw voltage data. These required values include the load calibration values (µN/Volt), the displacement calibration values (nm/Volt), and the stiffness calibration values (N/m). The calibration values which were in place on the instrument when tests were run are stored in that test's header file. These are the "Original" calibration values. If the calibration values have been changed and you wish to use the values currently stored on the instrument, then you can select "New" calibration values. When this option is selected, you will be prompted:

New value in range ([O/M]) ?
Press enter to accept old value, or new value enter to change.

The default value is "O" indicating "original" calibration values. To use this default value, simply press the enter key. To change to "New" values, enter "N" and press the enter key.

9.4.7.1.2 Do Thermal Drift Correction

It is possible to use the data collected during a hold segment to correct the load-displacement data for thermal drift. The default choice is "Y" for drift correction. To skip drift correction, select "N". There is one method of correction.

This method utilizes the change in displacement measured during a hold period to estimate a drift rate. The selection of drift correction leads to a prompt regarding the "subfile" to be used for making the drift correction. "Subfile" in this instance means the "segment" of the indent experiment, and that segment must always be a hold segment of sufficient duration to accurately measure the thermal drift rate, and significantly small load to avoid the inclusion of creep effects. Thus, the proper hold segment to be selected will be a hold segment that immediately follows a 80% or 90% unload segment, and immediately precedes the final unloading segment.

When this option is selected, you will be prompted:

```
New value in range ([Y/N]) ?
Press enter to accept old value, or new value
enter to change.
```

If you do not want to perform a drift correction, enter "N", and you will be returned to the previous menu. If you want to perform a drift correction, enter "Y." If "Y" is entered, you will be prompted:

```
Calculate drift rate from which subfile [X-X] ?
```

Enter the subfile number for the desired segment. For the standard experiment, the subfile number "5" refers to the appropriate Hold segment that should be used.

Once you have entered the subfile number, you will be returned to the preceding menu.

9.4.7.1.3 Display Data File Configuration

When this option is selected, the variables (e.g. load, time, etc.) and the subfiles (e.g., Approach, Hold, etc.) that are contained in a set of experiment data are displayed on the screen. You are given the option of printing this information:

```
The file type = XXXXX
VARIABLES              Total number of variables
= 3
No.         Name
1   ----  Disp (V)
2   ----  Load (V)
3   ----  Time (s)
```

The number of subfiles = 6

No.	Name	No. of First Observation
1	-------- A -------	1
1	-------- LL -------	51
1	-------- H -------	110
1	-------- UL -------	139
1	-------- H -------	197
1	-------- UL -------	247

Do you want a print out of the file structure
(Y/N)

To print this display, enter "Y", or enter "N" to return to the previous menu without printing.

9.4.7.1.4 ALL SET, CONTINUE ON

When this final line is executed, the software will proceed with the conversion of raw data into load/displacement/time data using the information you have entered in the preceding routine.

9.4.7.2 Stiffness From Unloading

This program calculates the stiffness of a contact using data from the unloading segments in the load vs displacement curves (the "D" files) and stores the results in "S" files. One value of stiffness is entered into a stiffness file for each unloading segment in the "D" file. The corresponding displacement listed for each stiffness value is the total displacement at the beginning of the unloading segment used in the calculation (see Figure 9.8).

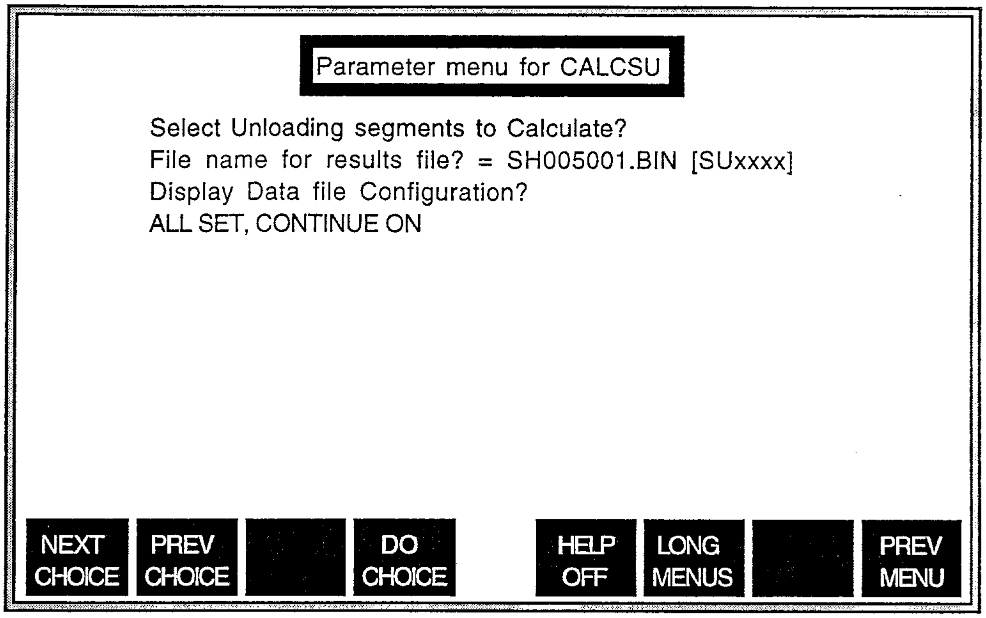

Figure 9.8 The Stiffness Calculation Menu

9.4.7.2.1 Select Unloading Segments To Calculate

You will be required to list the unloading "subfiles" to be used in the stiffness calculations. The unloading subfiles contain data acquired during unloading segments of the indent experiment and have the same number as the corresponding unloading segment. For example, in the indent experiment shown in Table 14.3, the unloading segments are 3, 5, 7, and 10. Therefore, in response to the prompt:

Which subfile (Enter Subfile number, 0 if done, or A for all) ?

type 3 and press ENTER, type 5 (ENTER), type 7 (ENTER), type 10 (ENTER), and then type 0 (ENTER) to indicate that the numbers of all unloading segments have been entered. You will notice that when you type 3 and press ENTER, the cursor moves one space to the left so that it directly under the "3" that you just typed. Thus when you type successively 5, 7, and 10, you will type over the previously entered number. That will not be the case when you type "0"; now the cursor will be under the "0" of the "10." Just be careful to delete the "1" before pressing ENTER for the final time!

9.4.7.2.2 Filename For Results File

The default choice for this option is the prefix "SU" or "SH" plus the user-defined base filename, the run number, and the ".BIN" suffix. It is sometimes useful, however, to change the results filename so that "S" filesnames are not duplicated. This may occur if you are running several different varieties of the same basic data, that is, for example, if you are changing the fraction of the segment use in the calculations and generating new "S" data, but wish to keep the old data as well. This option is also useful for record keeping in such cases.

9.4.7.2.3 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

9.4.7.2.4 ALL SET, CONTINUE ON

When you are finished defining the options for "S" file calculation, the software will calculate the appropriate variables. The most important information displayed on the screen will be the plot of stiffness (N/m) versus displacement (nm).

A subfile is defined as the data contained in the unloading segments of "D" files for a series of indents. In this example, a subfile is made up of data for five indents, each of which contains four unloading segments.

9.4.7.3 Hardness/Modulus/Depth

This program uses displacement/load/time/stiffness data (the "S" files) to calculate hardness and modulus values (see Figure 9.9).

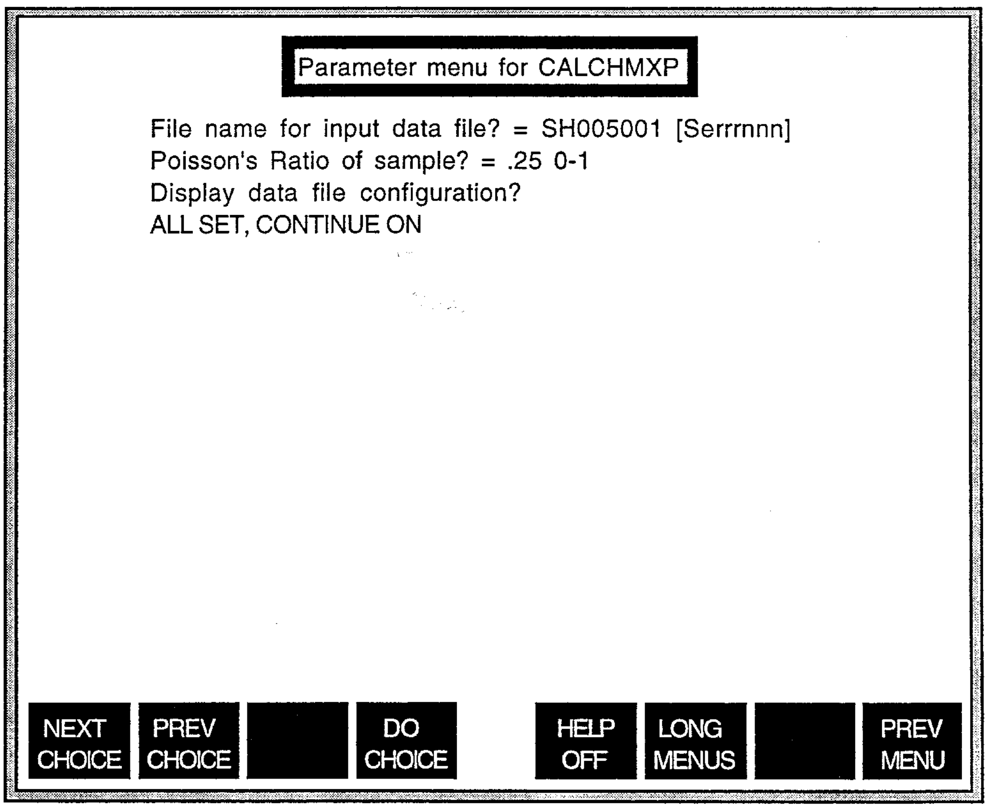

Figure 9.9 The Hardness/Modulus Calculation Parameter Menu

9.4.7.3.1 File Name For Input Data File

The file name for the input data will generally be the base file name (rrr) with a prefix "SU" or "SH" and the run number suffix. This will not be true, of course, if you have named the SU or SH file something other than the default name. Thus, the format for the input data file should be:

Serrrxxx

Where "S" is the file type indicator, "e" is the experiment type code (U or H), "rrr" is the base file name, and "xxx" is the run number. A typical file name might appear as:

SH005001.

9.4.7.3.2 Poisson's Ratio Of Sample

The default value, "0.25," represents a reasonable 'midrange' estimate. If the Poisson's ratio of a sample is known, it should be entered in place of the default value.

9.4.7.3.3 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

9.4.7.3.4 ALL SET, CONTINUE ON

As specified above, when tip radius "T" is selected rather than the experimental area function, immediately upon executing "ALL SET, CONTINUE ON," you will be required to input an estimated tip radius in nanometers. Once this is done (and additional questions related to data output have been answered as described later in this section) the software will begin calculations of hardness and modulus.

9.4.7.4 ALL DONE, CONTINUE ON

Selecting this option will cause the software to return to the Review Data menu.

9.4.8 Continuous Stiffness Data: User Defined Calculations

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

9.4.8.1 Load/Displacement/Time

This option behaves as described in Section 9.4.7.1.

9.4.8.2 Hardness/Modulus From Lock-In

This option behaves as described in Section 17.2.1. This option for data reduction is useful only if your Nano Indenter® XP is equipped for the Continuous Stiffness Measurement option.

9.4.8.3 Average Data

This program takes any data files and returns average values of two of the variables over discrete ranges of a third variable. The data for an indent are divided into discrete "windows," and the averages of the discretized variable and of the values of a maximum of two other user-specified variables in each "window" are determined. Input data into this program can be contained in any type file. At the beginning of the program, you are shown the familiar file manipulation menu. Once the desired files have been selected, the parameter menu for CALCAV is displayed (see Figure 9.10).

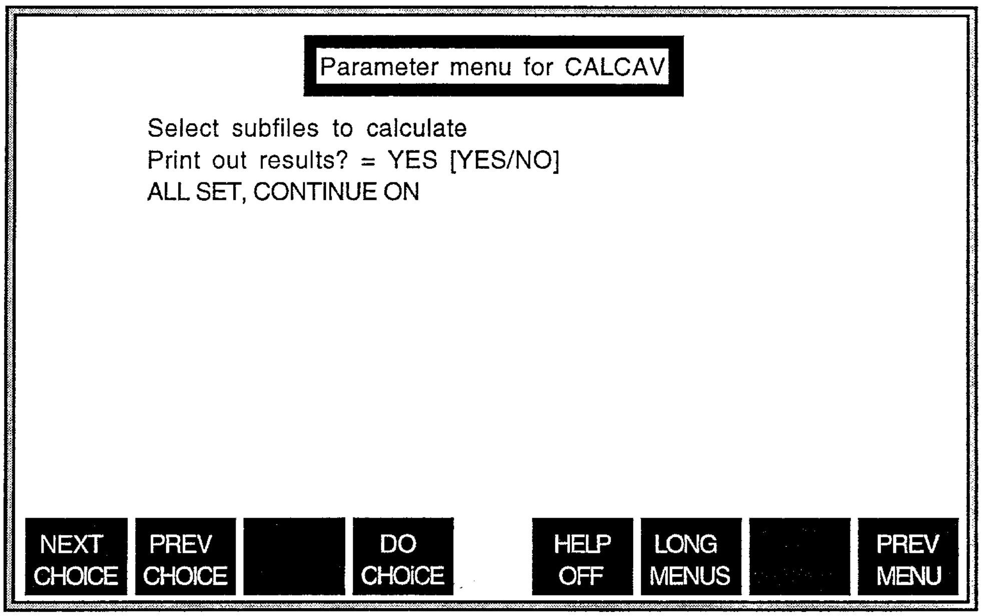

Figure 9.10 The Calculate Average Parameter Menu

9.4.8.3.1 Select Subfiles To Calculate

Selection of this option will cause the subfile selection screen to be displayed. Select the variables you want included in the averaged output file. The first variable you select will be the “base” variable. The data for this variable will be discretized into “windows”, and an average value calculated for that window. The other variables you select will be averaged within that window as well. Refer to the extended menu instructions (Section 14.4.8.3) for more information about the specific operations that occur when averaging data.

9.4.8.3.2 Print Out Results

This option can be used to toggle the printing function on and off. If "YES" is selected, a copy of the results of this routine will be sent to the printer, and the result file will be stored on the hard drive. If "NO" is selected, the file will be stored on the hard drive, but no copy will be printed.

9.4.8.3.3 ALL SET, CONTINUE ON

Once all information has been input, the software will perform the averaging as defined by the parameters of the routine. The output data will be stored on the hard drive.

9.4.8.4 ALL DONE, CONTINUE ON

Selecting this option will cause the software to return to the Review Data menu.

9.4.9 Scratch Data: User Defined Calculations

Refer to Section 18.0 for more information on the Scratch Data calculations.

9.4.10 Miscellaneous Data Manipulations

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

9.4.10.1 Average Data

This option behaves in the same manner as the option described in Section 9.4.8.3.

9.4.10.2 Break Points

The "break points" routine allows you to automatically search data for 'breaks' in the data. These 'breaks' could indicate cracking or other phenomenon that indicate a rapid change in the response of a material. The routine flags or records the first discontinuity in the load displacement data, according to the user-specified definition of a 'break point (see Figure 9.11).

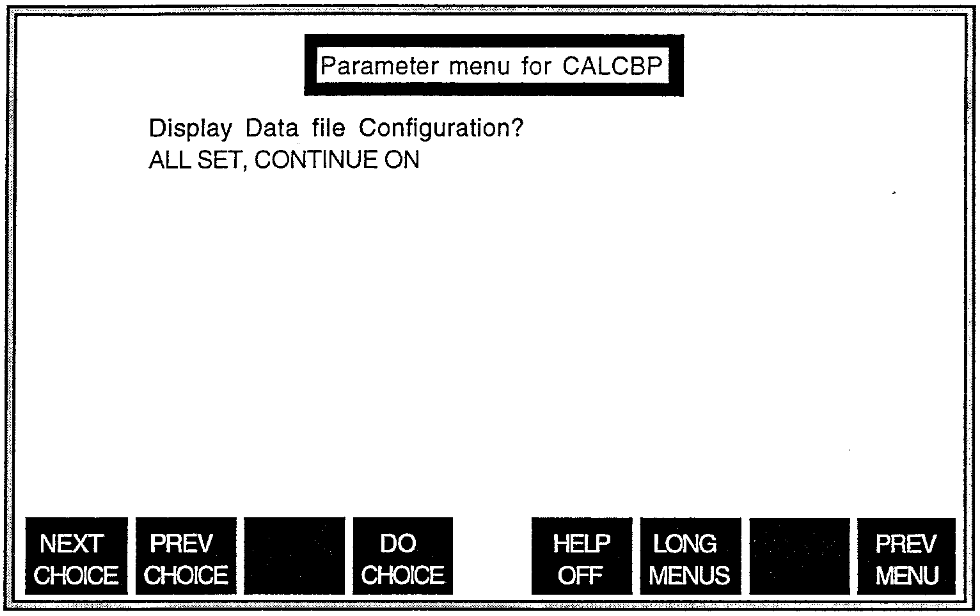

Figure 9.11 The Break Points Parameter Menu

9.4.10.2.1 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

9.4.10.2.2 ALL SET, CONTINUE ON

Once all information has been input, the software will search the input data for discontinuity as defined by the parameters of the routine. The output data will be stored on the hard drive.

9.4.10.3 Calculate Delta & Average Displacement From Subfiles

As of the writing of these instructions, this routine was not yet operational. Future versions of these instructions will include the procedure for using this routine. If your software is enabled to allow operation of this option, please contact Nano Instruments for an update to these instructions.

9.4.11 ALL DONE CONTINUE ON

Selecting this final option will return you to the Master Menu.

10.0 Load Specimen/Manual Control

10.1 Overview

The Load Specimen / Manual Control routine simply provides control of the instrument into your hands. There are two modes of input: control through the joystick and manual control keyboard functions.

10.2 Joystick Operation

Operation of the Joystick is done through the control stick, the trigger, the focus speed knob, and the focus buttons (see Figure 10.1).

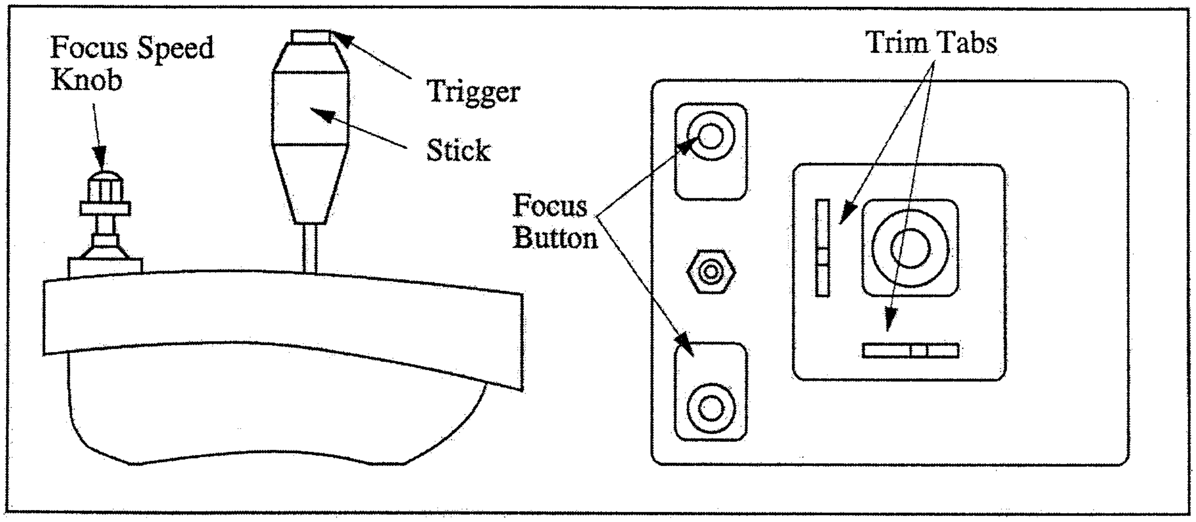

Figure 10.1 The Joystick

10.2.1 Moving the X-Y Tables

Control of the X-Y tables is accomplished by moving the stick on the joystick and using the trigger button to move from high speed mode to precision mode. The movement of the tables corresponds to the direction of the joystick (see Figure 10.2).

The speed of table motion is also governed by the degree of deflection of the joystick, in both high speed mode and precision mode.

In terms of X-Y coordinate conventions, movement of the joystick to the "east" position causes the tables to move to the "east," the counters to move toward negative, and an appearance of negative motion on the video monitor (see Figure 10.2). Likewise, movement of the joystick toward the "north" position causes the tables to move toward the "north," the counters to move toward negative, and an appearance of negative motion on the video monitor.

The trigger button is used to alter the speed at which the tables move. Depressing the button and moving the stick causes the tables to move at the maximum allowable rate. Leaving the button un-depressed and moving the stick causes the table to move at the minimum rate. Only these two states (max rate and min rate) exist. That is, partially depressing the button will not achieve a "medium" speed. To achieve variable speed, change the degree of deflection of the joystick.

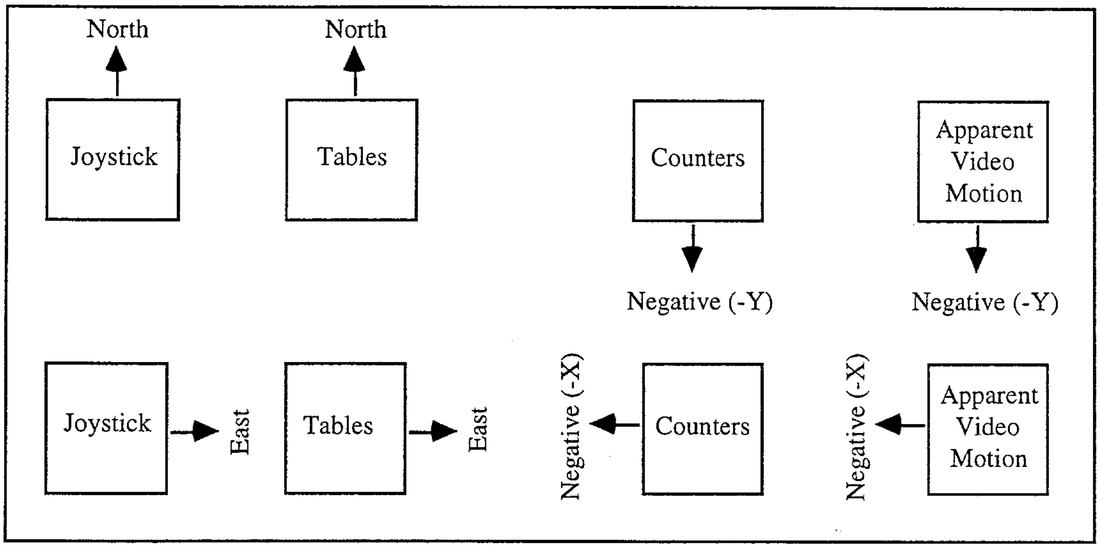

Figure 10.2 The Motion System Direction Conventions

The limit switches on the X-Y tables are active while you are in the manual control screen. For more information about the limit switch behavior refer to Section 3.0.

10.2.2 Controlling the Optic Focus

The focussing of the optic system is controlled through the two focus buttons and the focus speed knob. The optic system is focussed by an eccentric bearing coupling so that rotation of the optic focus motor results in vertical movement of the microscope. Thus, either of the two focus buttons can result in upward or downward movement of the microscope, depending on which side of the eccentricity curve the button is operating on. In other words, by continuously depressing one of the buttons, you can move the microscope through its entire range of motion and back again.

The speed of the optic focussing can be altered by using the focus speed knob. Turning this knob clockwise decreases the speed of the focussing, while turning the knob in the opposite direction will increase the focussing speed.

For more information about the optic system, refer to Section 4.0.

10.2.3 Adjusting The Joystick

The ability to adjust the joystick's "center" has been provided. The centering adjustment can be performed by turning the "Trim Tabs" on the joystick (see Figure 10.1). This adjustment should be performed while the Manual Control screen is displayed. If the tables "drift" in one direction when the joystick is not deflected, then you can eliminate this "drift" by adjusting the Trim Tab for the axis displaying the movement. Remove the sample tray before performing this correction, so that there is no possibility of damage to the indenter system if there is a sudden movement of the motion system.

10.3 Manual Control Functions

The manual control functions are available through the keyboard, and are described on the manual control screen. These controls are not case-sensitive. For example, to activate the load specimen command, simply press "S" or "s" on the keyboard. In the following descriptions, only the capital letters are used, although the lower case letter can be used in operation as well.

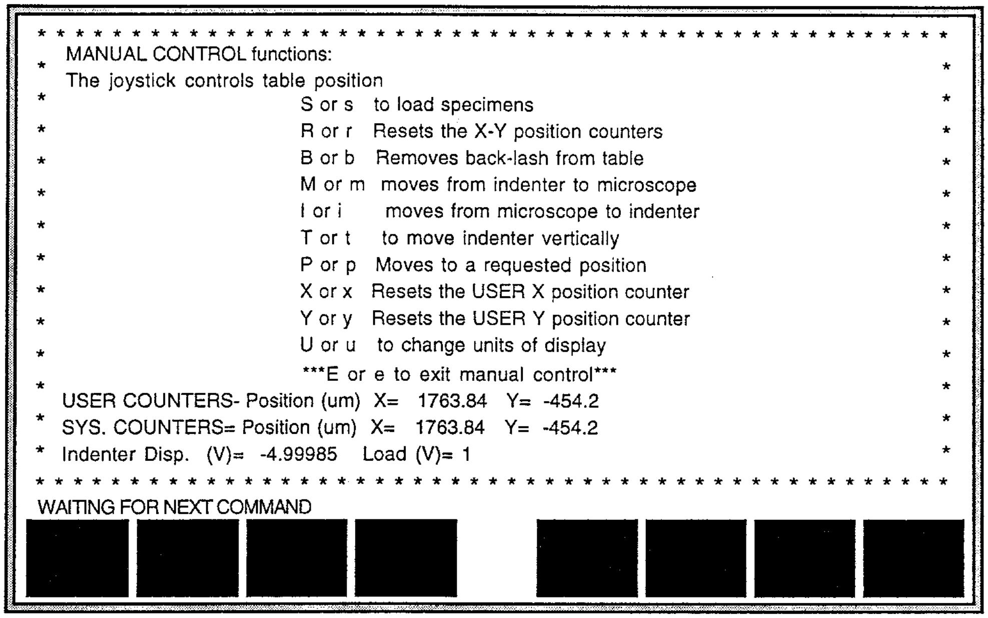

Figure 10.3 The Manual Control Screen

10.3.1 Load Specimens

The load specimen key enables an automated routine which moves the sample tray to the front of the cabinet. In the "loading" position, it is easier to turn the sample wedge screw and remove or load the sample tray. When this routine is activated, the software will assume control of the positioning system and move the sample mount to the front of the gantry. Once the sample mount is in its proper position, you will be prompted:

Please load your specimens and hit continue.

You should remove the sample tray and load your samples, and then lock the sample tray in the positioning system again. Once you have done this, simply press continue, and the sample mount will be moved back to its original position (probably under the microscope, since this is the most likely location for the sample mount prior to pressing the "S" key).

10.3.2 Reset The X-Y Position Counters

To activate this option, press the "R" key. This command causes the X-Y position counters to reset to zero. The counters affected by this command are the System Counters. This command has no effect on the actual position of the table.

10.3.3 Remove Backlash From Table

The Remove Backash routine is activated by pressing "B" on the keyboard. This routine carries out a specific series of moves of the X-Y tables in order to remove accumulated backlash from the motion system.

10.3.4 Move From Indenter To Microscope

The Move From Indenter To Microscope command is activated by pressing the "M" key on the keyboard, and simply causes the X-Y tables to move the calibrated distance from the indenter to the microscope. Since the X-Y table system does not have an "absolute home position," there is no way for the software to know exactly where the tables are relative to the physical location of the indenter. You should not select this command unless the sample table is actually underneath the indenter, and the indenter is raised out of contact with the sample itself.

Once this automated move begins, it will continue until the calibrated microscope to indenter distance has been achieved. Thus, if this move is begun while the sample stage is under the microscope, the system will try to move the sample stage to the "east," and will continue to run in this direction until the limit switches are activated and the motion is interrupted.

10.3.5 Move From Microscope To Indenter

The Move From Microscope to Indenter command is activated by pressing the "I" key on the keyboard, and simply causes the X-Y tables to move the calibrated distance from the microscope to the indenter. Since the X-Y table system does not have an "absolute home position," there is no way for the software to know exactly where the tables are relative to the physical location of the indenter. You should not select this command unless the sample table is actually underneath the microscope, and the indenter is raised into its protective housing.

Once this automated move begins, it will continue until the calibrated microscope to indenter distance has been achieved. Thus, if this move is begun while the sample stage is under the indenter, the system will try to move the sample stage to the "west," and will continue to run in this direction until the limit switches are activated and the motion is interrupted.

10.3.6 Move Indenter Vertically

If you would like to move the indenter to a specific position, you can use the "T" or "t" key to achieve this move while in manual control. Upon using this key, you will be prompted:

```
What displacement voltage do you want (-5<X>5) ?
```

Once a voltage value has been entered, the software will display the text:

```
Moving tip, please wait.
```

When the operation is completed, and the indenter tip has been positioned, the software will display:

```
All Done.
```

You can now continue to use Manual Control.

Note that it is strongly advised that you do not attempt to use this function to make indentations on a sample. The instrument is not recording data during this procedure, and there is a possibility of damage to the indenter if the indenter comes in contact with a material during this operation.

10.3.7 Move To A Requested Position

When the "P" key is pressed, the software will prompt:

```
Please input the X and Y coordinates of the target (X,Y) ?
```

The X and Y coordinates you wish to move to should be input in micrometers. Note that the coordinates are relative to the zero position on the system counters. Thus, if the counters display X = 50 and Y = 50, and you input "25,25", the system will move to a position of 75,75. Once you have entered the desired coordinates, you will be prompted:

```
You want to move to the position XX YY
Is this correct ? (Y/N)
```

If the position is correct, answer "Y." One item of note, be sure that the indenter is at a safe voltage position before you perform these moves. Obviously, it is not desirable to strike the indenter from the side with the sample tray. Inputting a large desired move when the indenter is exposed could result in damage to the system.

10.3.8 Reset The USER X Position Counter

Pressing the "X" key on the keyboard activates the Reset User X Position Counter command. This command resets the X User Counter to zero.

10.3.9 Reset The USER Y Position Counter

Pressing the "Y" key on the keyboard activates the Reset User Y Position Counter command. This command resets the Y User Counter to zero.

10.3.10 Change Units Of Display

The default units for the manual control screen display are micrometers (μm) and Voltage (V). Pressing the "U" key on the keyboard will activate the Change Units of Display command, which will toggle the units to micrometers (μm) and the corresponding units for load (microNewtons μN) and displacement (nanometers nm). To change the units back to Voltage, simply press the "U" key a second time.

10.3.11 Exit

To leave the manual control screen, simply press the Exit key ("E" or "e"). You will be returned to the menu from which you entered Manual Control. That is, if you selected Manual Control from the Master Menu, you will be returned to the Master Menu upon using the Exit function. If you entered Manual Control from other points in the software (such as test set-up), then you will proceed with the expected software path upon using the Exit function.

11.0 Calibrations

11.1 Overview

Several of the calibrations accessible under the Calibration "long" Menu are not intended for general use. These calibrations may require tools or special procedures, and are thus performed only at Nano Instruments or by a representative of Nano Instruments. From a "daily use" perspective, only the calibrations available on the "short" menu are of particular interest.

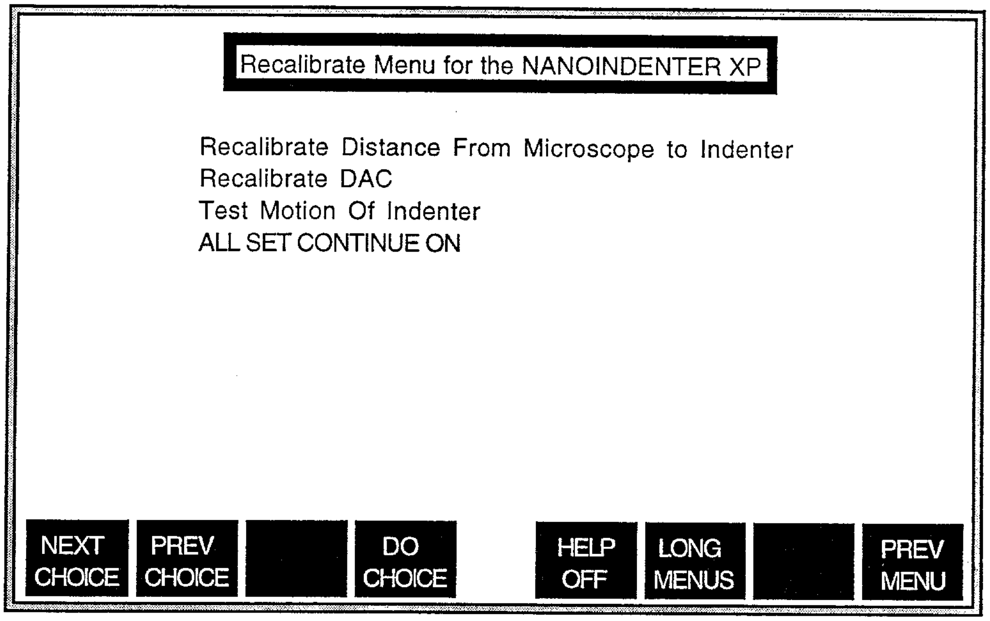

Figure 11.1 The Calibrations Menu

11.2 The Calibration Menu

The Calibration Menu (or "short menu") lists those calibrations of most interest to the user of the Nano Indenter® XP. None of these calibrations requires additional hardware or expertise. The possible exception to this rule is the Microscope To Indenter routine, which requires a sample of soft material (single crystal aluminum is preferred).

11.2.1 Recalibrate Distance From Microscope To Indenter

Because the indenter and the microscope are in different "X-Y" locations, the distance between them must be accurately established if indents are to be precisely positioned. This routine establishes that distance. Upon entry into this routine, you will be prompted:

Hit CONTINUE to proceed to manual control menu.

Selecting the CONTINUE button will bring up the manual control screen and enable the joystick. Operation of the joystick and optic system focussing is as described in Section 10.0.

The proper method for performing the microscope-to-indenter calibration is to select a clear and highly polished area on a soft material. Indents will be made in this material, and you will use the joystick to move these indents under the crosshairs on the screen. Upon exiting the routine, you will be offered the option to save this "correction" distance into the calibration data array.

The first step is to select the indentation site. The objective in use will govern the indentation site The standard objective (40X) is ideal for the microscope-to-indenter calibration, as it provides the ability to resolve small indentations (in a soft material) with a wide field of view (for locating the indentations). The indentation site should be clear of surface defects, and, most importantly, clear of other indentations that could lead to confusing the results.

Once the initial point is selected under the crosshairs of the video monitor, use the "E" key to exit the manual control routine. You will be prompted:

Hit CONTINUE when ready to move to indenter.

Once you press the CONTINUE button, the X-Y tables will begin moving the sample the calibrated distance to the expected indenter site. When the tables stop, the indenter will make three indentations. After the last indentation, the indenter will lift to it's safe position, and the X-Y tables will move the sample back under the microscope. After an automated backlash correction you will be prompted:

Position the furthest right of the three
indents directly under the cross-hairs.
hit CONTINUE when ready

Once you select the CONTINUE button, the manual control screen will return, and it will be necessary to locate the three indents. Once you have found them, follow the instructions and position the furthest right indent under the cross-hairs. Use the "E" key to exit the manual control routine. You will be prompted:

Do you wish to check the calibration again (Y/N)

If you wish to execute another iteration of the routine to refine the calibration, you can do so at this point, and the procedure will repeat. If you do not wish to, choose "Y." Once you have chosen to exit, you will be prompted:

Store these results (Y/N)

In most cases you will want to store the results. Once you have done so, the microscope-to-indenter calibration is finished. It is a good idea to double-check the routine, since the positioning of the indents is often critical.

11.2.2 Recalibrate DAC

The Recalibrate DAC (digital analog converter) option instructs the instrument to perform a series of indenter moves in free-air. The response of the indenter is analyzed, and the values of the DAC are recorded for specific indenter displacement voltage positions. This information is stored on the hard drive, and is used when the indenter must obtain a specific position.

Note that the values of the DAC calibration do not affect the data recorded by the instrument. The DAC calibration values will affect the instrument performance only to the extent that the indenter will more quickly achieve desired positions if the DAC calibration has recently been performed.

The DAC calibration also serves as a good indicator of the Nano Indenter® XP's operating status. If significant damage has been done to the system, the DAC calibration will commonly fail. Thus, the DAC calibration is an excellent method for verifying the operation of the Nano Indenter® XP.

Upon selecting the "Recalibrate DAC" item from the Calibration Menu, the software will immediately begin performing the DAC calibration. Once the procedure is complete, you will be prompted:

Do you wish to check the calibration again (Y/N)

In most cases there is no need to run the calibration twice. If you choose to do so, the procedure will simply repeat and offer the prompt again. Once you have chosen to continue by selecting not to perform the calibration again, you will be prompted:

Store these results (Y/N)

Unless there is some unusual indenter response, you should choose to store the results. The DAC calibration values will be automatically stored on the hard drive. The software will return to the Calibration Menu.

11.2.3 Test Motion Of Indenter

The Test Motion Of Indenter routine is a powerful option for observing and verifying the behavior of the instrument. Unlike the DAC calibration, this routine does not store any values or results. Rather, the user is given control of the indenter's movement, which is shown graphically on the monitor. There are a number of control options that alter the way the indenter's motion is displayed.

Upon entering the Test Motion routine, you are prompted:

Plot Disp. vs. Load- (1), Disp. vs Time-(2), Load vs Time-(3)?

These three options define how the indenter's motion is graphically displayed.

11.2.3.1 Displacement Vs. Load

Displacement vs. Load is most useful for examining the response of the indenter to changes in load, and thus can be used to examine hysteresis or linearity in the load displacement trace. Upon choosing this option, you will be prompted:

DISP. minimum? [-5.5]

This prompt indicates a request for the minimum displacement to be plotted and provides a default value. The minimum displacement value selected will serve as the Y axis lower limit on the graphical display[11]. Following entry in response to this prompt, you will be prompted, in order:

DISP. maximum? [5.5]

LOAD minimum? [-2.1]

LOAD maximum? [2.1]

As described above, these prompts indicate requests for the limits of the graphical display. The default values indicate the reasonable extreme limits of the indenter's travel in terms of load and displacement. The unit for these limits is Voltage (V). Once the limits have been defined, you are asked to input:

Displacement range (0-7)

Response to this prompt will indicate the desired range of the displacement signal. The displacement range is a specific subset of the entire displacement range. There are eight different ranges available. The lowest range will have the highest displacement resolution. During operation, the indenter control software will use the smallest displacement range (highest resolution) that it can during an experiment. You should use the default value of the displacement range, unless you have good reason to do otherwise. Once this final piece of information is entered, you will be prompted:

Hit CONTINUE when you are ready to start plotting data.

Upon selection of the CONTINUE button, you will be transferred to the graphical screen, with the limits of the plot as selected above. Section 11.2.3.4 describes the operation of the graphical screen and the various control options available.

---

[11]Since indenter motion is vertical, and this plot represents the free motion of the indenter, displacement is represented on the Y axis.

11.2.3.2 Displacement Vs. Time

The Displacement vs. Time option is useful for examining the level of environmental or electronic displacement noise in the system. Upon choosing this option, you will be prompted:

DISP. minimum? [-5.5]

This prompt indicates a request for the minimum displacement to be plotted and provides a default value. The minimum displacement value selected will serve as the Y axis lower limit on the graphical display. Following entry in response to this prompt, you will be prompted, in order:

DISP. maximum? [5.5]

Screen sweep time (sec)? [60]

As described above, these prompts indicate requests for the limits of the graphical display. The default values indicate the reasonable extreme limits of the indenter's travel in terms of displacement and time. The unit for the displacement limits is Voltage (V), and the unit for the sweep time is seconds (sec). While the displacement minimum and maximum limits describe the static "window" of displacement data to be displayed, the sweep time limit describes the "length" of the X axis in terms of time. Since this unit is specified as a sweep, the screen will automatically refresh when the time has reached the end of a sweep of the defined number of seconds.

Once the limits have been defined, you are asked to input:

Displacement range (0-7)

Response to this prompt will indicate the desired range of the displacement signal. The displacement range is a specific subset of the entire displacement range. There are eight different ranges available. The lowest range will have the highest displacement resolution. During operation, the indenter control software will use the smallest displacement range (highest resolution) that it can during an experiment. Once this final piece of information is entered, you will be prompted:

Hit CONTINUE when you are ready to start plotting data.

Upon selection of the CONTINUE button, you will be transferred to the graphical screen, with the limits of the plot as selected above. Section 11.2.3.4 describes the operation of the graphical screen and the various control options available.

11.2.3.3 Load Vs. Time

The Load vs. Time option is useful for examining the level of load signal noise in the system. Upon choosing this option, you will be prompted:

```
LOAD minimum? [-2.1]
```

This prompt indicates a request for the minimum load to be plotted and provides a default value. The minimum load value selected will serve as the Y axis lower limit on the graphical display. Following entry in response to this prompt, you will be prompted, in order:

```
LOAD. maximum? [2.1]
```

```
Screen sweep time (sec)? [60]
```

As described above, these prompts indicate requests for the limits of the graphical display. The default values indicate the reasonable extreme limits of the indenter's travel in terms of load and time. The unit for the load limits is Voltage (V), and the unit for the sweep time is seconds (sec). While the load minimum and maximum limits describe the static "window" of load data to be displayed, the sweep time limit describes the "length" of the X axis in terms of time. Since this unit is specified as a sweep, the screen will automatically refresh when the time has reached the end of a sweep of the defined number of seconds.

Once the limits have been defined, you are asked to input:

```
Displacement range (0-7)
```

Response to this prompt will indicate the desired range of the displacement signal. The displacement range is a specific subset of the entire displacement range. There are eight different ranges available. The lowest range will have the highest displacement resolution. During operation, the indenter control software will use the smallest displacement range (highest resolution) that it can during an experiment. Once this final piece of information is entered, you will be prompted:

```
Hit CONTINUE when you are ready to start plotting
data.
```

Upon selection of the CONTINUE button, you will be transferred to the graphical screen, with the limits of the plot as selected above. Section 11.2.3.4 describes the operation of the graphical screen and the various control options available.

11.2.3.4 The Graphic Test Motion Screen

The graphic test motion screen will appear differently depending on the type of plot selected (as described above in sections 11.2.3.1 through

11.2.3.3). Figure 11.2 shows the test motion screen as displayed when Displacement Vs. Load is selected and all default options are chosen.

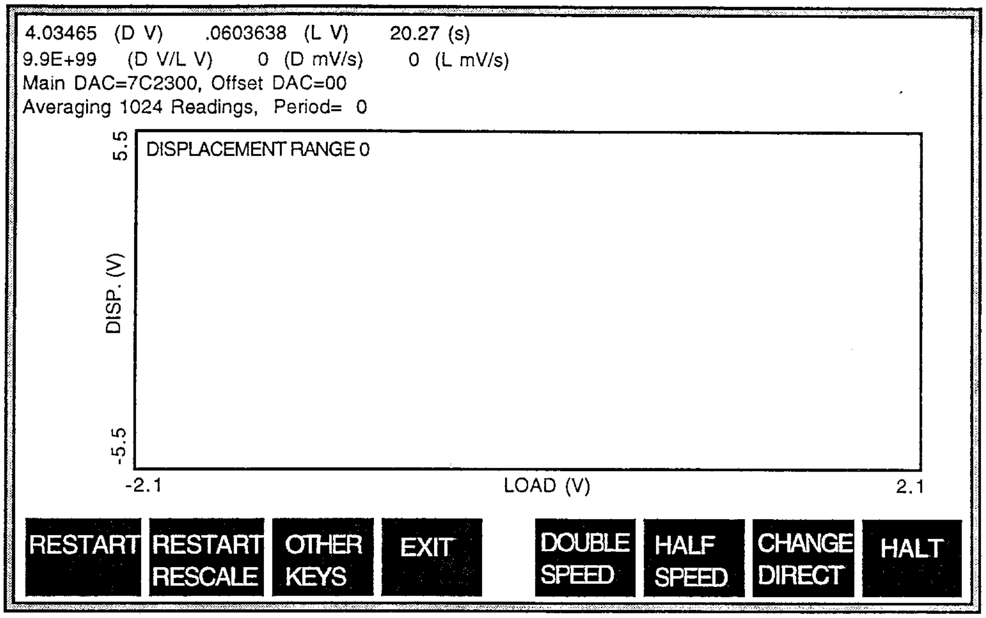

Figure 11.2 The Test Motion Screen

The top portion of the screen displays the relative current position of the indenter. Reading across the first row:

4.03465 (D V)	.0603638 (L V)	32.78 (s)

These values indicate the current indenter position in Displacement, Load, and Time. The units of this display can be altered, as explained in Section 11.2.3.17. Reading across the second row:

0 (L V/D V)	-98.3179 (D mV/s)	0 (L mV/s)

These values indicate additional information about the indenter's behavior. The first value indicates the load/displacement ratio (in free air this should be the spring constant), the second value indicates the active displacement rate, and the third value indicates the active load rate.

Reading across the third row:

Main DAC=7C2300, Offset DAC=00

These displayed values refer to the actual DAC "word" describing the DAC position of the indenter. In almost all cases, these values are of no interest to the user.

Reading across the fourth row:

Averaging 1024 Readings, Period = 0

Averaging refers to the number of data points that are averaged for each point displayed on the screen. This is related to the Time Constant (see Section 11.2.3.13). The Period refers to operation of the DAC. There should be no need for the user to observe or note this data item.

The limits of the display are shown at the corners of the plot axes, as is the variable being plotted. The displacement range is displayed in the upper left corner of the plot. Finally, below the plot are the function keys used to control operation of the test motion routine.

11.2.3.5 The Restart Key

The Restart Key simply refreshes the screen, eliminating all previously plotted points.

11.2.3.6 The Rescale/Restart Key

The Rescale/Restart Key allows you to reset the scale and limits of the plot area, or to switch to a different set of variables to display. Upon using this key, you will see the familiar prompt:

Plot Disp. vs. Load- (1), Disp. vs Time-(2), Load vs Time-(3)?

Resetting the plot is identical to the method described in sections 11.2.3.1 through 11.2.3.4, with some minor differences. First, upon selecting one of the three plot types, you will be prompted:

Reset limits to extreme values? (Y/N)

In this case, the term "extreme values" refers to the minimum and maximum values offered, which were originally defined when you first selected the plot limits (the default values). If you choose "N", the software will use the minimum and maximum points from the resident acquired data to determine the limits.

For example, if you had defined a Displacement vs. Load plot using the default values for the limits, and then manually moved the indenter from a displacement of 1.2V to a displacement of -1.6V, chosen the Rescale/Restart key, and then chosen not to reset to the extreme values, the resultant plot would have a Y-axis (displacement) scale from -1.6V to 1.2V, and the X-axis (load) would carry whatever limits of whatever loads corresponded to these displacements on the previous plot. In the case of a plot of displacement or load vs. time, the X-axis (time) is set to whatever sweep time was originally defined. If you choose to reset the limits to extreme values, of course, you can set whatever limits you like (including the sweep time).

A second different behavior is that the indenter position in the new "Rescaled" plot is dependent upon the state the indenter was in when the Rescale/Restart key was pressed.

For example, if the indenter was moving at a rate of 10nm/s, and a Rescale/Restart was performed, when you re-enter the plot screen, the indenter location will not be the same as when you pressed the Rescale/Restart key. This is because the indenter will continue to move while you are re-entering the plot information, etc. For this reason, if you want the indenter to remain at a constant position while you rescale the plot, you should use the Halt key (Section 11.2.3.12) before pressing the Rescale/Restart key.

11.2.3.7 Other Keys

The use of this key will cause the function key display to switch to the secondary keys (Sections 11.2.3.13 through 11.2.3.20).

11.2.3.8 Exit

The Exit key simply leaves the Test Motion routine, and returns you to the Calibration Menu. There is no need to be concerned about what state the indenter is in before Exiting.

11.2.3.9 Double Speed

The Double Speed key literally doubles the rate of change of the load signal. Thus, if the rate change in load voltage is 0.5 V/s, using this key will double the rate to 1.0V/s. Note that there are maximum and minimum rates which the software will sustain. If you attempt to double the speed beyond the maximum rate, the software will notify you that you have reached the maximum.

It is the double speed key, along with half speed, change direction, and halt, that controls the indenter movement on the plot screen.

11.2.3.10 Half Speed

The Half Speed key halves the rate of change of the Primary variable. Thus, if the rate change in load voltage is 0.5 V/s, using this key will halve the rate to 0.25V/s. Note that there are maximum and minimum rates which the software will sustain. If you attempt to half the speed beyond the minimum rate, the software will notify you that you have reached the minimum.

11.2.3.11 Change Direction

The Change Direction key simply changes the direction of motion of the indenter. Thus, if the indenter is moving from negative to positive displacement voltage, the change direction key will reverse that process so that displacement is becoming more negative.

11.2.3.12 Halt

The Halt key stops the indenter motion at whatever point the indenter occupies when the key is used. This is particularly useful when Displacement or Load Vs. Time is being plotted.

11.2.3.13 Double Tc

The Double Tc key doubles the time constant over which data is averaged for display on the plot screen. This is most useful when plotting against Time, but the key can be used for Displacement Vs. Load plots as well. When this key is used, the data density on the plot will decrease and data points will appear less often.

11.2.3.14 Half Tc

The Half Tc key halves the time constant over which data is averaged for display on the plot screen. Like the Double Tc key, this is most useful when plotting against Time, but the key can be used for Displacement Vs. Load plots as well. When this key is used, the data density on the plot will increase, and data points will appear more often.

11.2.3.15 Other Keys

The use of this key will cause the function key display to switch to the secondary keys (sections 11.2.3.5 through 11.2.3.12).

11.2.3.16 Exit

The Exit key simply leaves the Test Motion routine, and returns you to the Calibration Menu. There is no need to be concerned about what state the indenter is in before Exiting.

11.2.3.17 Units

The Units key changes the units of the displayed data at the top of the plot screen.

Units Of Displayed Data

Variable	*Unit 1*	*Unit 2*
Displacement	V	nm
Load	V	μN
Time	seconds	seconds
Load/Displacement	V/V	N/m
Displacement Rate	mV/s	nm/s
Load Rate	mV/s	μN/s

Table 11.1 Units Of Displayed Data

11.2.3.18 Freeze/Cont.

The Freeze key causes the plot screen to stop refreshing once the data has reached the end of the sweep time. No further data is displayed until the Freeze is stopped by pressing the alternative key, "Cont." The Freeze key is only useful in the Displacement and Load Vs. Time plots.

11.2.3.19 Overlay/No Overlay

Like the Freeze Key, the Overlay Key is only useful in Displacement and Load Vs. Time plots. The Overlay key causes the data to superimpose rather than refreshing at the end of each sweep. The alternative key, No Overlay, returns the screen to autorefresh mode.

11.2.3.20 Print Screen

This command key will print a copy of the test motion screen to the printer.

11.2.4 ALL SET CONTINUE ON

Selecting this option causes the software to return to the Master Menu.

12.0 Perform Standard Test

12.1 Overview

There are no "advanced functions" currently enabled for the Perform Standard Test menu option. Refer to Section 7.0 for a complete description of the standard operation of this option.

13.0 Design Custom Test

13.1 Overview

The Custom Test Extended Menu adds several powerful options to test set-up procedures. These options are accessed by pressing the "Long Menus" function key ($f6$). All of the functions available in the short menu are available in the long menu as well. Refer to Section 8.0 for complete instructions on operating the standard Custom Test Menu options.

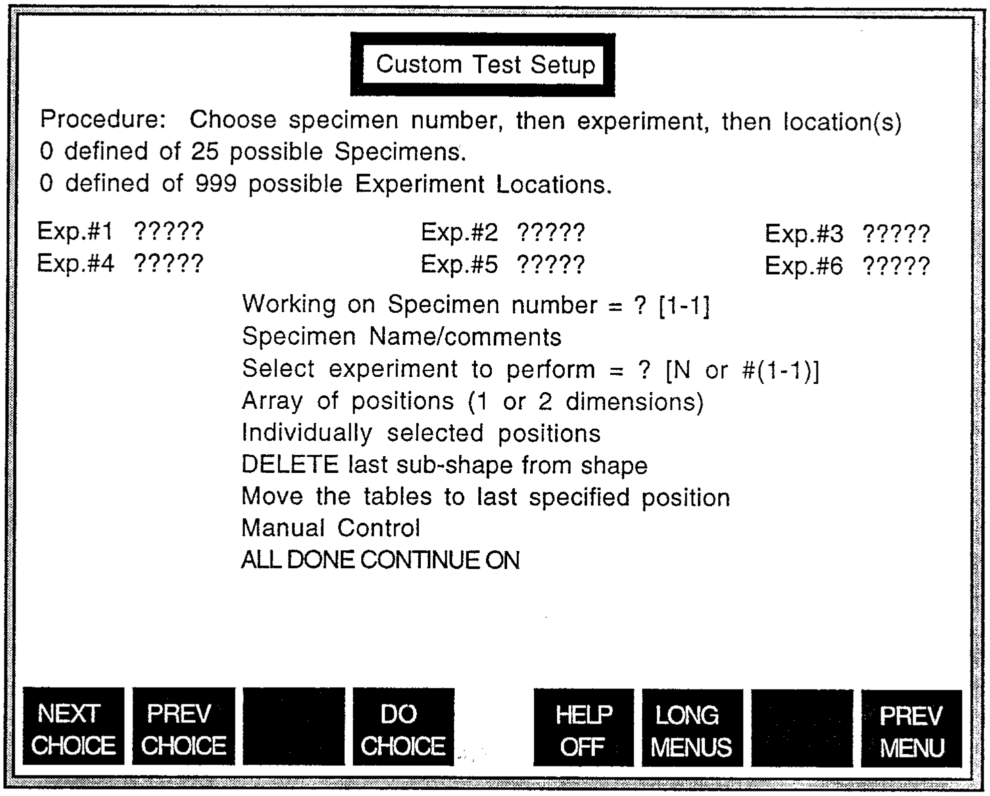

Figure 13.1 The Custom Test Setup Menu

13.2 The Custom Test Extended Menu

As described above, the Custom Test Extended menu displays both the "short" and "long" menu options.

13.2.1 Working On Specimen number

Refer to Section 8.2.3 for more information about the Working On Specimen Number option.

13.2.2 Specimen Name and Comments

Refer to Section 8.2.4 for more information about the Specimen Name and Comments option.

13.2.3 Select Experiment To Perform

The only significant change enable by the use of the long menus is the ability to include user defined experiments as part of the Custom Test. With the long menu enabled, the Select Experiments Menu will appear as:

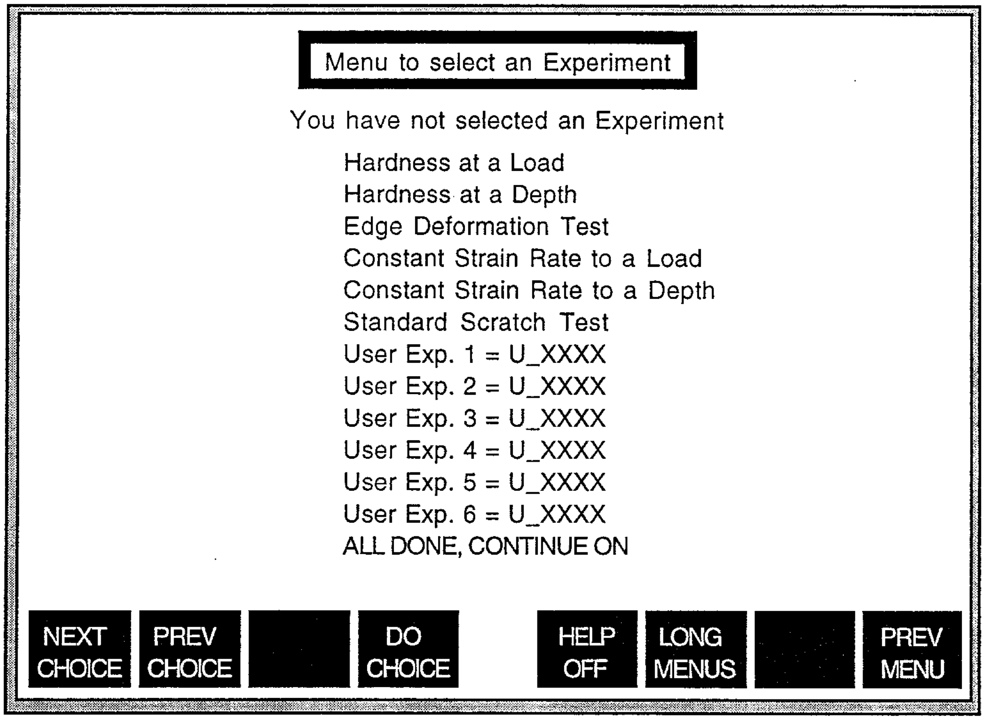

Figure 13.2 The Experiment Selection Menu

The options on this menu enable the definition of the type of experiment you would like to perform.

13.2.3.1 Hardness At A Load

Refer to Section 8.2.5.1 for more information about this option.

13.2.3.2 Hardness At A Depth

Refer to Section 8.2.5.2 for more information about this option.

13.2.3.3 Edge Deformation Test

Refer to Section 8.2.5.3 for more information about this option.

13.2.3.4 Constant Strain Rate To A Load

Refer to Section 8.2.5.4 for more information about this option.

13.2.3.5 Constant Strain Rate To A Depth

Refer to Section 8.2.5.5 for more information about this option.

13.2.3.6 Standard Scratch Test

Refer to Section 8.2.5.6 for more information about this option.

13.2.3.7 User Experiment

There is no need to enter additional information when the User Experiment is selected.

The User Experiments will only be useful if your software includes the User Experiment Definition Utility, which is an additional option for the Nano Indenter® XP. If you have this option installed in your software, refer to Appendix D for further instructions in the definition of these experiments.

13.2.3.8 ALL DONE, CONTINUE ON

When you select this option, the experiment is recorded into the Test Set-Up, and you are returned to the Design Custom Test screen.

13.2.4 Array Of Positions

Refer to Section 8.2.6 for more information about the Array Of Positions menu option.

13.2.5 Individually Selected Positions

Refer to Section 8.2.7 for more information about the Individually Selected Positions option.

13.2.6 Delete Last Sub-Shape From Shape

The difference between "Shapes" and "Subshapes" is briefly explained in Section 8.2.1.1. However, a more detailed description is useful for the purposes of operating in the "advanced mode."

13.2.6.1 Shapes & Subshapes

There is a specific distinction between the terms shape and subshape which it is very helpful to understand. The term shape refers to the entire pattern of indent locations over all specimens and all positions. This is the final pattern which is used during the execution of an experiment. A shape can be made up of subshapes, or groups of indent positions, usually a single matrix or pattern of individual indent locations on one specimen (although this is not a requirement). For example, if a 4 x 5 matrix is defined as an array of positions, and no more positions are defined, then it is a shape. If more positions are defined, on different specimens, loaded from memory, selected individually, etc., then the 4 x 5 matrix originally defined is a subshape, as are all other groups of defined positions. The entire pattern then becomes the shape (see Figure 13.3).

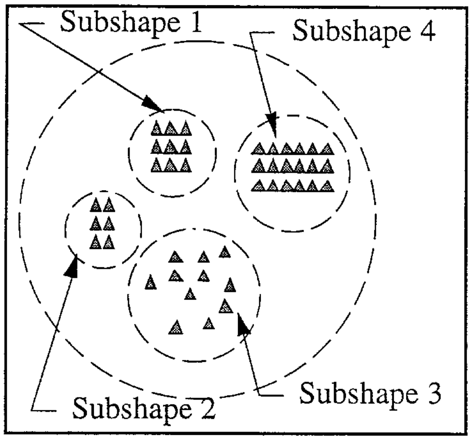

Figure 13.3 Each subshape is a group of indents at a different site. Subshape 1 is a 3x3 array. Subshape 2 is a 2x3 array. Subshape 3 is a group of individually selected positions. Subshape 4 is a 6x3 array. The entire set is a shape.

13.2.6.2 Deleting A Sub-Shape

The routine "Delete Last Sub-shape from Shape simply allows you to eliminate a shape that you have defined. For example, if you have defined a 3x5 array of indents, and then defined a 3x3 array of indents, but decide you do not want to use the 3x3 array of indents, then you can use this routine to delete the 3x3 array, even though you have "accepted" the shape when defining positions. Note that this routine only operates on the last sub-shape. That is, you cannot delete the second sub-shape from a series of five sub-shapes. When this option is selected, you will be prompted:

Delete last sub-shape [Y or N]

Answering "Y" will cause the last sub-shape to be removed from the shape.

13.2.6.3 The Effect On Experiment Procedures

Another important point is how the deletion of a sub-shape affects an entire test run. If you have defined multiple indentation procedures, and have specified that one procedure be used on a specific sub-shape, but then delete that sub-shape, then the experiment procedure used on that sub-shape will be deleted as well.

13.2.6.4 Deleting The Only Sub-Shape

Finally, if you have only defined a single sub-shape, and you perform the "Delete Last Sub-shape" option, then you will be prompted:

```
Only one sub-shape defined, delete this sub-shape
[Y/N]?
```

If you choose to delete this last sub-shape, then you will have eliminated all selected positions for the test, and will need to reselect positions before the test can be run.

13.2.6.5 Deleting Multiple Sub-Shapes

It is not possible to iterate back through a test set-up to delete multiple sub-shapes. That is, only the last sub-shape can be deleted. If you have defined five sub-shapes, only the fifth can be deleted. It is not possible to re-enter the "Delete Last Sub-Shape" option to delete the fourth, third, second, or first sub-shapes, for example.

13.2.7 Move the Tables To Last Specified Position

When this option is selected, the positioning system will move to the last specified position. For example, if you have specified a 4x5 array of indents, and then specified a 3x3 array, then using this option will cause the positioning system to move to the position of the last indent in the 3x3 array. In most cases there is no need to use this option, since the system is automatically positioned at the last indent site when a new array is specified. However, if you have used the Manual Control option (Section 13.2.8) to move the tables, then this option is useful for returning to the last indent site before beginning a new array or selecting new positions.

13.2.8 Manual Control

The use of this option simply provides a manual control screen and enables joystick control of the positioning system. This option is useful if you want to examine the specimens prior to defining an array of indents, or if you are searching for specific features on the samples to indent, and do not want to perform this search while defining indentation locations. The operation of the manual control screen is the same as that described in Section 8.2.6. Note that there is no "reset counters" option available on this screen, so it is not possible to "accidentally" affect the positioning of indents already selected.

13.2.9 ALL DONE CONTINUE ON

After selecting all of the indent positions, experiments, and specimens, it's time to "continue on." Upon selecting the "ALL DONE, CONTINUE ON" option, a final menu screen will appear (see Figure 13.4).

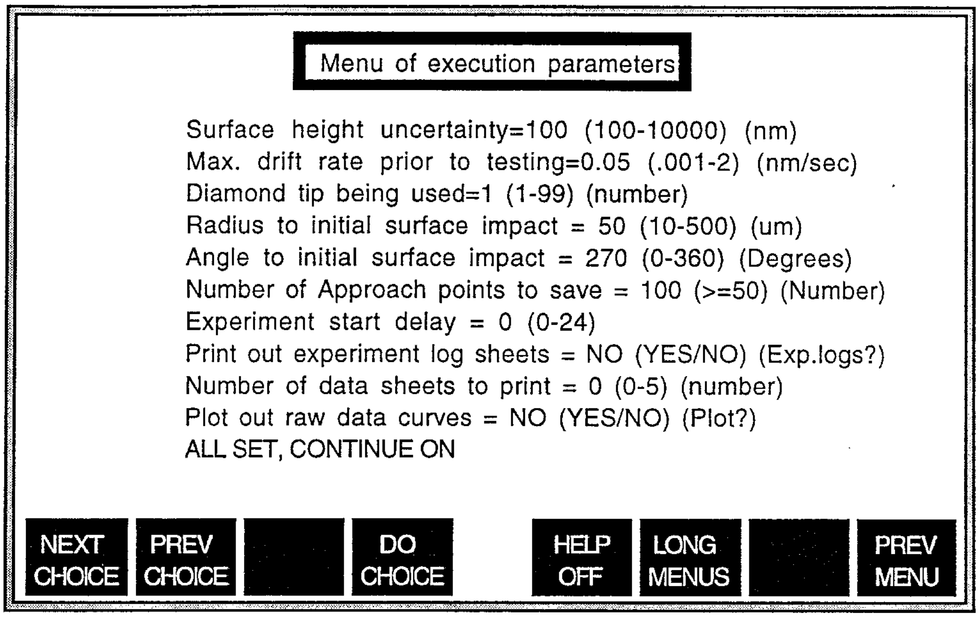

Figure 13.4 The Menu Of Execution Parameters

This menu contains the parameter options for the execution of the test as defined in the previous sections.

13.2.9.1 Surface Height Uncertainty

Refer to Section 8.2.8.1 for more information about the Surface Height Uncertainty option.

13.2.9.2 Maximum Drift Rate Prior To Testing

Refer to Section 8.2.8.2 for more information about the Maximum Drift Rate Prior To Testing option.

13.2.9.3 Diamond Tip Being Used

Refer to Section 8.2.8.3 for more information about the Diamond Tip Being Used option.

13.2.9.4 Radius To Initial Surface Impact

When the computer first begins a series of indents, it must first "locate" the sample surface. To do so the Table moves the specimen a distance to be specified in this choice (default value is 50 μm) from the position chosen for the first indent (see Figure 13.5). An indent is made at this point. A second indent is made half the distance between the first and the position chosen for the first indent of the shape that is to be made. The making of these two indents produce first a coarse and then a refined estimate of the relative heights of the specimen surface and the indenter, in addition to providing the information needed by the computer to establish with great accuracy the "zero" of the displacement measurement for the first indent of the shape.

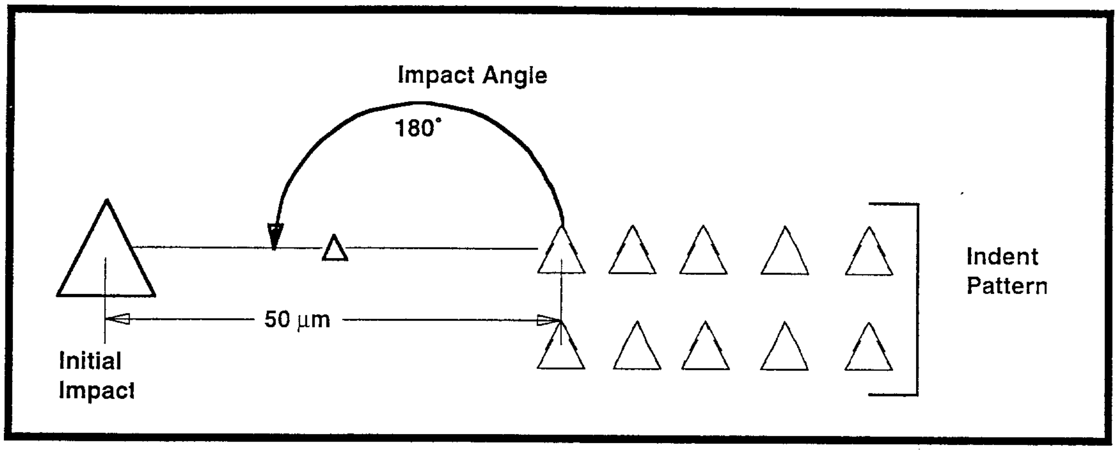

Fig. 13.5 Schematic drawing showing position of initial impact site relative to the first indent of a pattern. The distances and angles shown are modified values. The small indent halfway between the initial impact site and the pattern is the indent used to sense surface height precisely.

You can change the default value, but the site of the initial "rough" indent obviously ought to be relatively close to the first indent if a meaningful measurement of the surface location is to be made. Nor should it be close enough for this initial indent to affect the first indent of the shape. The default value is generally a good choice.

13.2.9.5 Angle To Initial Surface Impact

The angle to the point of initial surface impact is the angle, measured counterclockwise, between a horizontal line on the monitor passing through the position chosen for the first indent of an experiment and a line connecting that point with the desired point of initial impact. The default value is 270°, but any angle between 0° and 360° may be specified. See Figure 13.6 for an example of the default angle to initial surface impact.

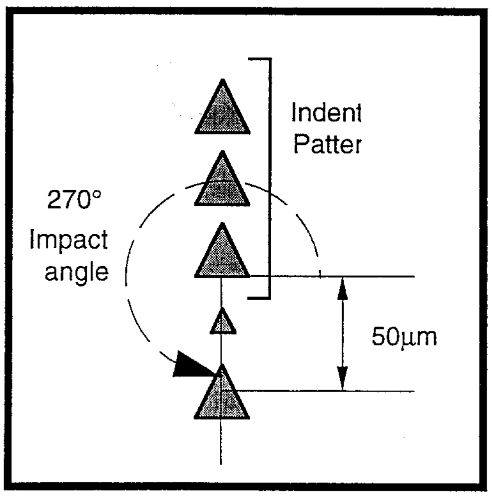

Figure 13.6 Specifying an initial impact angle of 270° for a 1x3 array will place the initial surface finding indents in line with the array. This is the default condition used in all tests unless otherwise specified.

13.2.9.6 Number Of Approach Points To Save

The Number of Approach Points refers to a "buffer" size used during the indenter's approach to the surface of the sample. These parameters of the approach are defined when the Approach Segment is specified (see Section 9.3.1). However, the number of approach points can be altered outside of the experiment procedure definition by using this function.

When the indenter approaches the surface of the sample, the software monitors the stiffness of the indenter motion. When the stiffness of the indenter motion reaches a given value (defined in the Approach segment) contact with the sample is recognized. As the stiffness is monitored, the software contains this stiffness data in the approach buffer. When the buffer is filled, the "oldest" data point is discarded so that a new data point can be accepted into the buffer. When contact is recognized, the load and displacement data points in this buffer are stored in the Approach data segment in the resultant data file for the indent. This data is later used to further refine the accuracy of contact recognition (see Section 14.4.3.4). The accuracy of contact recognition can be improved in some cases by increasing the number of data points that may be stored in the Approach buffer. The default value of 50 points, however, is an excellent value for most indentation procedures.

13.2.9.7 Experiment Start Delay

The Experiment Start Delay option allows you to set a delay from the time of test definition to the time of the test run. This option is useful if your Nano Indenter® XP is in an environment that improves in terms of stability at certain times of the day, or if you would like to allow time for the samples to reach thermal equilibrium with the indentation system. For example, if you find that the thermal stability of the environment greatly improves at night, you can use this option to delay the start of a test run until that time.

13.2.9.8 Print Out Experiment Log Sheets

If you would prefer not to have printed output from a test run, you can use this option to toggle the printing off or on. It is advised that you print at least one copy of the output report when running tests.

13.2.9.9 Number Of Data Sheets To Print

The data sheets identify the specimens and describe the shapes and indent experiments used. The default value, 1, causes one set to be printed out. Usually only one set is needed as an archival copy, but you may occasionally want two or more copies. Note that if you have turned printing off using the option described in Section 13.2.9.8, then you will receive no printed output, regardless of the number of copies you have requested with this option. Likewise, if you select "0" copies with this option, then you will receive no printed output, even if you set the "Print Out Log Sheets" option to "YES".

Refer to Section 8.2.8.4 for more information about this option.

13.2.9.10 Plot Out Raw Data Curves

Selecting this choice toggles the default value of "No" to "Yes." No entry by the user is required. The raw data curves [Load (in volts) vs. Displacement (in volts)] are plotted automatically on the monitor at the end of each indent. If you are monitoring the instrument while a test is running, then it is sometimes useful to see the raw data curves as each indent is performed. If you are not observing the instrument as it runs, then you should leave this option set to the default value.

13.2.9.11 ALL SET, CONTINUE ON

Selecting this last option will end the required user input, and the test will run automatically.

It is still necessary, however, for you to enter the "operator name" at the prompt:

Who is the operator?

Generally, entering the initials of the operator is acceptable, although more characters can be used if desired (up to a limit of 100).

Once the test is finished, the raw data files will be automatically reduced and stored on the hard drive. The final report will be output from the printer. As it is possible to setup a test that will run over many hours, it is often advisable to setup such tests at the end of the day, since the instrument will operate entirely without user input while performing tests.

13.3 The Abort Run Function Key

Once a test begins to run, one of the function keys (f1) will display the option to abort the run. This is an extremely useful option, far preferable to performing a shut-down of the instrument (although the shut-down method is still prefered in the case of emergencies).

The Abort Run key must be pressed repeatedly until the key changes to "Remove Abort," as displayed on the computer screen. Once this change is made, the run will be aborted upon completion of whatever operation the instrument is executing.

Thus, if the Abort Run key is used during an indent, upon completion of theindent the computer will calculate the data and return the sample to its position under the microscope.

14.0 Review Data

14.1 Overview

The Review Data menu provides options for the manipulation and reduction of data. When a test is run with the instrument, the data files are stored to the hard drive (see Section 2.0). The Review Data options access these data files and perform various operations on the data.

14.2 Conventions For Selecting Data Files

A method of selecting data files is standard across all of the review data menu options (both "short menu" and "long menu.") This standard input format, called the "File Manipulation Menu," appears when any of the review data menu items is selected.

14.2.1 Basename For Input Data Files

The "basename" of a data file refers to the two digit prefix and the filename as selected during test set-up. Two examples of basename structure are shown in Table 14.1.

Basename Conventions			
File Type	**Test Type**	**Run Number**	**Base Name**
R	H	ABC	RHABC
D	T	8B8	DT8B8

Table 14.1 Basename Conventions

The Nano Indenter® XP uses a standard set of conventions for creating and manipulating the experiment data files.

As shown in Figure 14.1, there are many possible basenames. The basename structure is dependent upon the File Type, Test Type, and Run Number. Further extensions to the File Name include the Indent Number, and finally the Data Type.

14.2.1.1 File Type

The File Type refers to the level of manipulation of the data in a file. The various possible file types are:

R - Raw data (direct test output voltage data)
D - Load/Displacement/Time data (Raw Data converted & corrected)
S - Stiffness data

M - Hardness & Modulus data
B - Break Point data
A - Averaged data
C - Hardness - Plastic Only data
E - Hardness - Elastic Only data
Z - Delta Z Displacement data (Razor edge test)
H - Hardness at depth data
P - Profile data

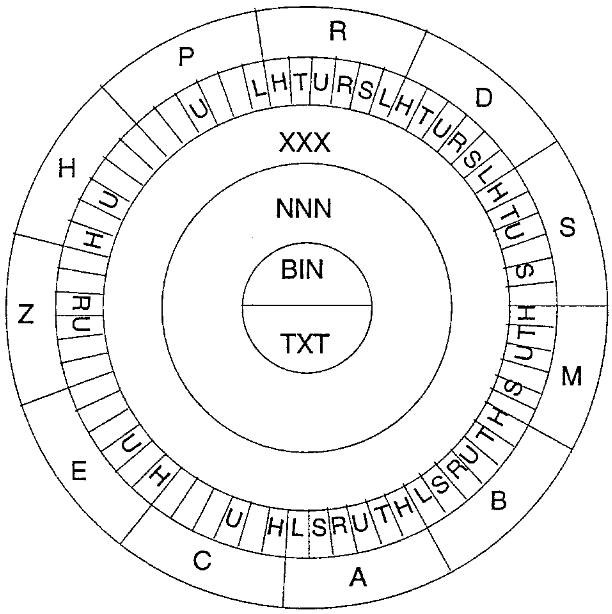

Figure 14.1 The File Name Convention[12]

14.2.1.2 Test Type

The Test Type refers to the type of test defined for the specific experiment run. There are six test types: Hardness, Tip Calibration, User Defined, Razor Edge Test., Constant Strain Rate Test, and Scratch Test.

H - Hardness Test: Typical loading to a depth or load to determine hardness and modulus.

T - Tip Calibration Test: Set of tests for the purpose of calibrating the diamond tip area function.

U - User Defined Test: Test set up as defined using the User Definition Utility[13].

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[12]Note that data file types that are not possible are excluded on this diagram. For example, since it is not possible to generate Profile data from a Razor Edge Test, the "P" segment does not include the "R" secondary prefix.

[13]The User Definition Utility will not be enabled on all instruments. Contact Nano Instruments for more information about this option.

R - Razor Edge Test: A specific test set up for edge-on testing of razor blades.

S - Constant Strain Rate Test: Loading to a depth or load using a constant indentation strain rate.

L - Scratch Test: A test using the lateral force measurement option to apply lateral forces to the sample.

14.2.1.3 Run Number

The run number refers to the software assigned hexadecimal code that indicates the sequential order of a particular test. Thus, the first test run on your Nano Indenter® XP will have a run number of 001, while the tenth test run will have a run number of 00A.

14.2.1.4 Indent Number

The indent number makes up part of the File Name, as opposed to the Base File Name. The indent number simply refers to the data for a specific indent in a series of indents. Thus, an indentation experiment defined with a 5x2 array would have indent numbers 001 through 010.

14.2.1.5 Data Type

The data type refers to the actual format of the data file. BIN indicates a binary data file, and TXT indicates an ASCII text file.

14.2.2 Add a Contiguous Series Of Files

Refer to Section 9.2.2 for more information about the Continuous Series of Files menu option.

14.2.3 Display Previously Selected Files

This option behaves in the same manner as the option described in Section 9.2.2.3. The only difference is in the placement of this option on the File Manipulation Menu.

14.2.4 Add A Discontinuous List Of Files

There are occasions when you will want to select only specific indents from a series of indents that has been stored. For example, if you had run the DH8B8 series as described above, but only wanted to look at every other indent, then you could use this option to select every other indent, rather than specifying the entire contiguous series.

When the "Discontinuous List" option is selected, the Add List Menu is displayed.

14.2.4.1 Build List Of File Numbers

The Build List option allows you to enter the list of files you would like added to the series for manipulation. Once this option is selected from the menu, the following text is displayed:

Enter a file number.

To define the list of files, simply enter the file numbers you want to use. For example, if you wanted only files "1", "56", and "75", you would simply type in "1" and then enter, type in "56" and then enter, and type in "75" and enter. To indicate that you have finished entering files, type in "0" (the numeral) and enter. To discard the list you are working on and begin again, enter "-1." Up to 999 file numbers may be entered (depending, of course, on whether you have already entered files with the Contiguous Series or Discontinuous List methods).

14.2.4.2 Display Previously Selected Files

This option behaves in the same manner as the option described in Section 9.2.2.3.

14.2.4.3 Directory Of Data Files On Disk

This option behaves in the same manner as the option described in Section 9.2.2.4.

14.2.4.4 ACCEPT THIS LIST

This option behaves in the same manner as the option described in Section 9.2.2.5.

14.2.5 Edit Previously Selected Files

There are occasions when you will want to remove or replace specific indents from a series of indents that has been selected. For example, if you had run the DH8B8 series as described above, and selected indents 1-50 using the Contiguous Series option, but you did not want to look at indent #34, then you could use this option to remove indent #34 from the selected list. Alternatively, if you wanted to look at indent #45 instead of indent #34, you could use this method to replace #34 in the list with #45.

When the "Edit Previously Selected Files" option is selected, the Edit Selected Files Menu is displayed.

14.2.5.1 Build Edit List

The Build Edit List option allows you to remove the files you would like from the series for manipulation, or to replace a file in a series with another file. Once this option is selected from the menu, the following text is displayed:

Enter selected file index, new file number.

To remove a file from the list to be input, you would enter the index number, followed by a comma, and then "-1" to remove the file. The input would appear as:

34,-1

if you wanted to remove indent #34. To replace a file from the list with another file, you would enter the index number, followed by a comma, and then the new file number. The input would appear as:

34, 45

if you wanted to replace indent #34 with indent #45. To cancel any changes you have made, and start over, you can enter:

-1, -1

and to finish making changes and accept the changes that you have made, enter:

0,0

To properly use this method, it is necessary to understand the concept of "index numbers" and how the index number relates to the file number.

The Index Number is the number that the computer assigns to a file number in a list of files to be manipulated. Normally, the index number equals the file number. For example, if you enter a contiguous series of files from file number 1 through file number 50, the index numbers 1 through 50 will correspond to files number 1 through 50.

When entering multiple contiguous series, the index number and file number may not always match. For example, if you enter a contiguous series from 1 through 50, and then enter an additional contiguous series from 75 through 100, then the index numbers 1 through 50 will correspond to files 1 through 50, but index numbers 51 through 75 will correspond to indents 75 through 100.

The Index Number is always sequential, while the file number will appear as entered. For example, if you enter a discontinuous list of files 3, 7, 19, 23, and 45, then the index number 1 will correspond to file number 3, and index number 5 will correspond to file number 45.

This is mostly transparent to the user, except in the case of building an Edit List. When removing or replacing files, it is important to keep track of the index number. For example, if you have input a contiguous series from 1 through 50, and want to remove file number 34 from the list, then the index number to be modified is number 34. If you have entered a series from 1 through 50, and then another series from 75 to 100, and want to remove file number 76, then the index number to be modified is number 52.

Since you can enter the Build Edit List as many times as you like, it is important to keep track of previous changes to the list. For example, if you have entered a series from 1 through 50, and then removed indent 45, and accepted the new list, and then decide you want to remove indent 46 as well, you should be aware that index numbers 1 through 44 still correspond to files number 1 through 44, but now index numbers 45 through 49 correspond to files 46 through 50. So to remove indent 46, you would edit index number 45.

The best way to understand the behavior of the indexing function is to practice using this option.

It should be emphasized that when you work with the list of files for manipulation, you are not actually affecting the files stored on the hard drive. You are only modifying the list for the current manipulation process. Thus, you cannot accidentally permanently remove files from the hard disk while using this method.

14.2.5.2 Display Previously Selected Files

This option behaves in the same manner as the option described in Section 9.2.2.3.

14.2.5.3 Directory Of Data Files On Disk

This option behaves in the same manner as the option described in Section 9.2.2.4.

14.2.5.4 ACCEPT THIS LIST

This option behaves in the same manner as the option described in Section 9.2.2.5.

14.2.6 Directory Of Data Files On Disk

This option behaves in the same manner as the option described in Section 9.2.2.4.

14.3 Segments and Experiment Parameters

Each experiment, whether defined by the user or set up as a "library" experiment, is composed of discrete operations that control the indenter's behavior during the experiment. These discrete operations are called "Segments." Each Segment is itself defined by a set of "parameters" that control the behavior of the indenter during that segment.

Refer to Section 9.3 for more information about Segments and Subfiles.

14.4 The Review Data Extended Menu

There are no "long menu" options displayed on the Review Data menu that are not displayed on the "short menu." When stepping through the submenus, however, some long menu options can be enabled. The following sections describe these additional options as they are available.

14.4.1 Re-Print Data Sheets From Run Header File

This option behaves as described in Section 9.4.1.

14.4.2 Qualify Data And Mark For Re-Calculation

The "Qualify Data" routine allows you to review the calculated "D" files, and to select or mark files that you do not wish to be included in future calculations of this data.

This option is very useful if a data set contains one or more "bad indents" that can corrupt a set of averaged data. This routine can be used to exclude such "bad indents" from the data set, allowing the use of the calculation stream without review of the data at each step in the calculation process.

14.4.2.1 Select Run Number

This option behaves as described in Section 9.4.2.1.

14.4.2.2 Select Variables To Display

This option behaves as described in Section 9.4.2.2.

14.4.2.3 Examine (Accept/Reject) Data Files

This option behaves as described in Section 9.4.2.3.

14.4.2.4 Mark Files As Selected During Examination

This option behaves as described in Section 9.4.2.4.

14.4.2.5 Start Over This Run Number

This option behaves as described in Section 9.4.2.5.

14.4.2.6 Accept ALL data files.

This option is only available on the "long menu." Selecting this option will cause all files in a data set to be accepted. This option is useful if you have rejected many data files, and want to re-accept them all rather than accepting each individually.

14.4.2.7 ALL DONE, CONTINUE ON

When this option is selected, you will be returned to the Review Data menu. If you have marked files as "Rejected" during the examination of a data set, then these files remain "Rejected" when you select the "ALL DONE" option, and will not be included in subsequent operations on the data set (see Section 9.4.2.6).

14.4.3 Re-Run Automatic Calculation With Qualifications

This option behaves as described in Section 9.4.3.

14.4.4 Re-Format Data To Text

This option behaves as described in Section 9.4.4.

14.4.5 Plot Or Print From Series Of Files

This option behaves as described in Section 9.4.5.

14.4.6 Archive Data By Run Number

As of the writing of these instructions, this routine was not yet operational. Future versions of these instructions will include the procedure for using this routine. If your software is enabled to allow operation of this option, please contact Nano Instruments for an update to these instructions.

14.4.7 Basic Data: User Defined Calculations

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

14.4.7.1 Load/Displacement/Time

All data sets should be run through this program. It converts raw voltage data from the data acquisition system into values of load and displacement, and allows the user to use one of several methods to determine the exact point of surface contact. When the file numbers have been defined for this option, the menu of data manipulation parameters is displayed:

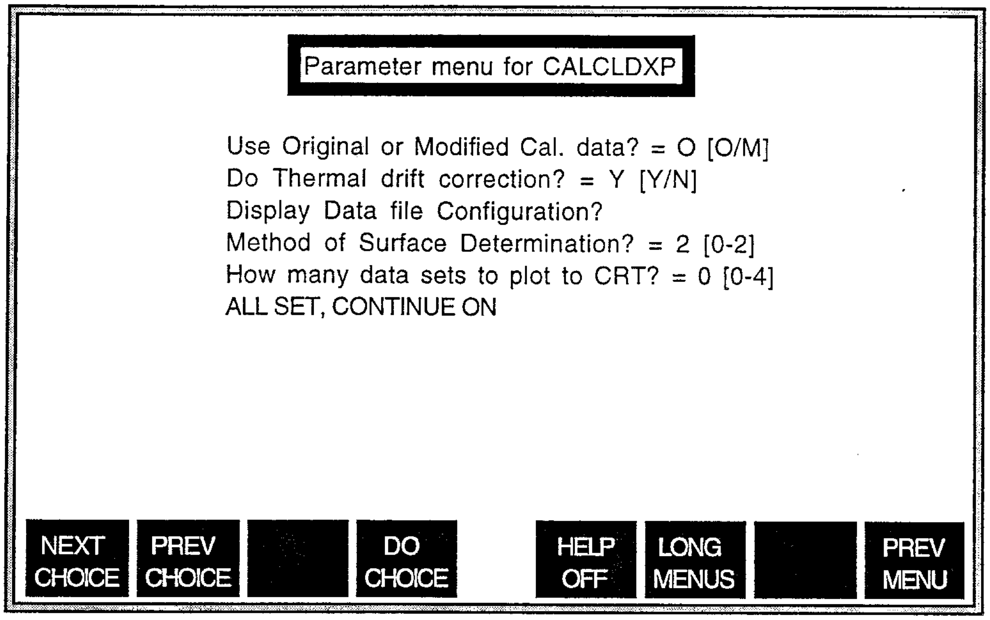


Figure 14.2 The Load/Displacement/Time Parameter Menu

14.4.7.1.1 Use Original or Modified Calibration Data

This option behaves as described in Section 9.4.7.1.1.

14.4.7.1.2 Do Thermal Drift Correction

This option behaves as described in Section 9.4.7.1.2.

14.4.7.1.3 Display Data File Configuration

This option behaves as described in Section 9.4.7.1.3.

14.4.7.1.4 Method Of Surface Determination

During the actual indentation, the surface of a sample is detected with a rough method that signals the software that contact with the sample has been achieved. For more precise determination of the exact point of surface contact, it is necessary to use a post-indentation method to choose the initial contact with the surface of the sample.

The number of points back to the surface is the number of data points of the approach segment during which the indenter can be assumed to be on the surface of the specimen. By examining the raw data approach curves only , using "Plot or print from a series of files" (see Section 9.4.5), the number of points to be used can be evaluated.

In many cases, the number of points back to the surface will be constant over an entire set of indents. For certain materials, an algorithm may be able to adequately select this point of contact without user input. For other materials, the algorithm may not apply, and the user may need to identify the point of contact for each indent. To allow for these separate possibilities, there are three methods of surface determination:

Method "0" asks you to input a number of points back to the surface. This number is assumed to be constant over the entire set of indents. Upon selecting Method "0", you will be asked:

How many data points back to the surface ?

Enter the desired number of points back. You will be returned to the previous menu.

Method "1" enables you to graphically determine the point of contact for each indent by moving a cursor on a plot of the approach data. If Method "1" is selected, no further information is required until you choose ALL SET, CONTINUE ON (14.4.7.1.6, below). When the software begins to calculate the data, it will display the plot of the approach segment:

Method "2" will automatically use the algorithm to select the point of surface contact. No further user input will be required. As described above, the algorithm will operate on the load and displacement if standard indentation data is input. If continuous stiffness data files are input, then the algorithm will operate on the phase and displacement.

The function keys displayed on the CRT describe the various operations that can be used to determine the point of surface contact.

Graphical Method Cursors		
	Indicator Type	**Indicator Color**
Cursor	Crosshair	Green
Current Selected Point	Angled Line	Red
Algorithm Point	Angled Line & "A"	Yellow
Constant Number Point	Angled Line & "C"	Purple
Graphical Selected Point	Angled Line & "G"	Light Blue

Table 14.2 Cursors on the Graphical Method Screen

14.4.7.1.4.1 f1 Next Point

This function key moves the cursor (the green crosshairs on the screen) to the next point in the approach data.

14.4.7.1.4.2 f2 Previous Point

This function key moves the cursor (the green crosshairs on the screen) to the previous point in the approach data.

14.4.7.1.4.3 f3 Select Cursor

This function key moves the current selected point (shown by the angled red line) to the point currently occupied by the cursor. It is worth noting here that the Graphical Point marker (shown as an angled blue line with a "G" next to it) indicates the last point selected using the graphical point selection routine.

14.4.7.1.4.4 f4 Select Algorithm

This function key moves the current selected point (shown by the angled red line) to the point currently selected by the algorithm (shown by the angled white line and the letter "A").

14.4.7.1.4.5 f5 Select Constant Number

This function key moves the current selected point (shown by the angled red line) to the point currently selected by the constant number of points back to the surface (shown by the angled blue line and the letter "C"). The constant number of points is calculated as an average of all the points selected for that load range. Thus, in a series of indents from 1 to 10, if you chose the 42nd point on the first two indents, and the 44th point on the second two indents, the "C" marker would appear at the 43rd indent on the fifth plot of the series.

14.4.7.1.4.6 f6 Dump Plot

This function key dumps a copy of the plot to the printer.

14.4.7.1.4.7 f7 Exit

This function key exits the plot screen for the displayed indent, selects the point marked with the red line (the current point), then performs the calculations to convert the data.

14.4.7.1.4.8 f8 Other Keys

This function key provides access to the alternate functions described below.

14.4.7.1.4.9 f1 Hide Cursor

The Hide Cursor key will change the plot so that only the Constant Points Back, Cursor, and Current Point markers are displayed. When Hide Cursor is selected, the other markers are hidden, and you are returned to the main set of function keys. If you re-enter "Other Keys," the Hide Cursor key will be changed to "Disp. Cursor," which, of course, returns the hidden markers to view.

14.4.7.1.4.10 f2 Replot Right

This function key alters the plot screen so that only the points to the right of the cursor's location (including the point the cursor is on) are shown.

14.4.7.1.4.11 f3 Replot Left

This function key alters the plot screen so that only the points to the left of the cursor's location (including the point the cursor is on) are shown.

14.4.7.1.4.12 f4 Full Set

This function key eliminates the effect of the Replot Right and Replot Left keys so that the entire data set is again displayed on the plot screen.

14.4.7.1.4.13 f5 Finish Algorithm

Selecting "Finish Algorithm" will instruct the software to use the algorithm method to select the surface contact for all ensuing indents in this indentation set. This key will not take effect until you choose "Exit" from the main set of function keys.

14.4.7.1.4.14 f6 Finish Constant Number

Selecting "Finish Constant Number" will instruct the software to use a constant number of points back to the surface to select the surface contact for all ensuing indents in this indentation set. This key will not take effect until you choose "Exit" from the main set of function keys. The value used will be the current value of the running average, as explained in section 14.4.7.1.4.5 above.

14.4.7.1.4.15 f7 Help

The help key displays information about the methods than can be used in the graphical point selection.

14.4.7.1.4.16 f8 Other Keys

Selecting "Other Keys" on the alternate Key screen will return the main keys to view.

14.4.7.1.4.17 A Note About Continuous Stiffness Data

A standard indent data set will cause the software to display the function keys as indicated above, at the base of a plot of Load vs. Displacement. If a continuous stiffness file is input into the routine, however, the software will generate a default plot showing the Phase vs. Displacement. When this latter case is in effect, the algorithm will operate on the Phase & Displacement, rather than the Load & Displacement, as is the case when standard files are input.

If there is a desire to view the plot as Load vs. Displacement rather than Phase vs. Displacement, a function key which is specific to continuous stiffness manipulation can be used, as described in the following section.

14.4.7.1.4.18 Show L vs. D f4

When a continuous stiffness file is selected for this routine, the default plot is shown with Phase as the Y axis, rather than Load as the Y axis. In addition, the "Select Algorithm" function key is replaced on the main key screen by this key, "Show L vs. D." Selecting this key will return the plot to a Load vs. Displacement plot. This key will then change to "Show P vs. D," which, of course, shows the Phase vs. Displacement. Regardless of which plot is displayed, whenever continuous stiffness data is input, the algorithm operates from the Phase.

14.4.7.1.5 How Many Data Sets To Plot To CRT

While the "D" files are being calculated, certain information is reported on the screen, including the drift rate and the number of "bad points" in each data file. You also have the option of having the Load vs. Displacement curves plotted on the screen as the "D" files are generated. The number displayed to the right of the line "How many data sets..." is the number of data files that will be plotted to the screen. The maximum for this number is equal to the number of raw data files you have selected for conversion to "D" files.

14.4.7.1.6 ALL SET, CONTINUE ON

When this final line is executed, the software will proceed with the conversion of raw data into load/displacement/time data using the information you have entered in the preceding routine. As described before, certain information will be plotted on the screen. In addition to creating the "D" files, the software will also print out a report of information for permanent storage. This information includes the appropriate new or original calibrations, the number of points back by file, the drift rate at that indent, and the original raw data name under which each indent is stored . If Method "1" was selected, then the software will behave as described in Section 14.4.7.1.4. Otherwise, no further user input is required.

14.4.7.2 Stiffness From Unloading

This program calculates the stiffness of a contact using data from the unloading segments in the load vs displacement curves (the "D" files) and stores the results in "S" files. One value of stiffness is entered into a stiffness file for each unloading segment in the "D" file. The corresponding displacement listed for each stiffness value is the total displacement at the beginning of the unloading segment used in the calculation (see Figure 14.3).

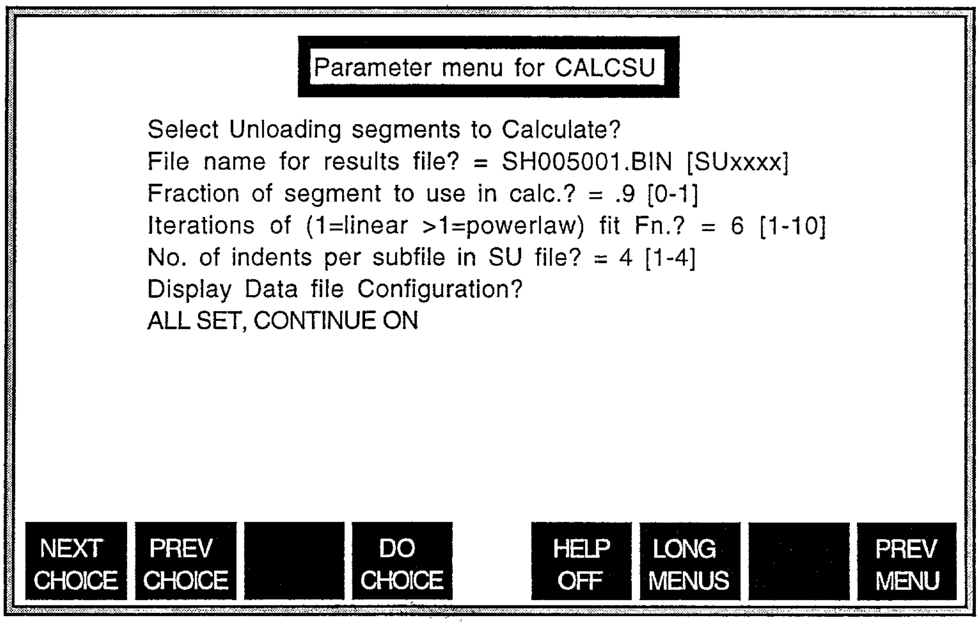


Figure 14.3 The Stiffness Calculation Menu

14.4.7.2.1 Select Unloading Segments To Calculate

This option behaves as described in Section 9.4.7.2.1.

14.4.7.2.2 Filename For Results File

This option behaves as described in Section 9.4.7.2.2.

14.4.7.2.3 Fraction Of Segment To Use In Calculation

The fraction of the unloading curve to be used in calculating stiffness is the amount of the curve that can be linearly fit, beginning with the first point of the unloading segment (the 'top' of the unloading curve). As the unloading curve is always nonlinear, there is an optimum point at which a linear fit will best describe the unloading curve, and thus, the stiffness of contact. The powerlaw fit, however, is relatively independent of the fraction of the curve that is used.

14.4.7.2.4 Iterations Of Linear Or Powerlaw Fit

This option allows you to specify the fitting method to be used to obtain stiffness data from the unloading segment. The linear fit, as described above, is dependent on the percentage of the unloading curve which is used in the fit. The number of iterations of the linear fit can be specified.

14.4.7.2.5 Number Of Indents Per Subfile In SU File

As the computer stores stiffness data, it can simply put all the data into one file or it can divide the data within that file into subfiles. In general, data from all indents in a given sample should be collected in a single subfile to facilitate the averaging and other statistical treatment. Thus the answer depends on the number of different samples indented. If 20 indents are made in a single, homogeneous sample, the default value that will appear under this prompt is 20. Since all 20 indents are on the same sample and are presumed to be identical, the default answer of 20 is correct. On the other hand, suppose that the shape shown in Figure 14.4 is used to make nine indents in each of five different but homogeneous samples (Figure 14.5). The default value that will appear under the prompt is 45, but the proper answer is nine. In response to an input of nine, the computer will create five subfiles, each containing data for nine indents, and the data will be ordered so that data from all indents of a given sample are in a single subfile.

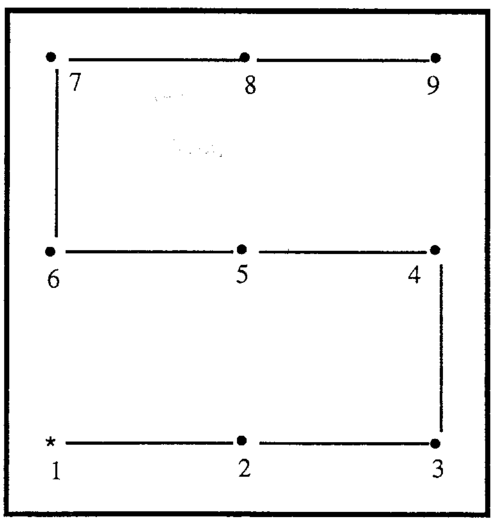


Figure 14.4 Schematic Representation of a Nine Indent Shape

Multiphase specimens provide a fairly obvious exception to the rule of putting all indents from a given sample into a single subfile. Suppose 20 indents are made in a two-phase sample, the first ten in Phase A and the second ten in Phase B. In this case it would make sense to put the first ten indents in one subfile and the next ten in a second. Thus the response to the prompt would be 10 and not 20.

A Complex Indent Experiment					
1	**2**	**3**	**4**	**5**	**6**
A	LD	UL	LD	UL	LD
	50Dep	100%Ra	300Dep	100%Ra	450Dep
		80%Un		80%Un	
7	**8**	**9**	**10**	**11**	**12**
UL	LD	H	UL	H	UL
100%Ra	600Dep	1Log	100%Ra	1Log	100%Ra
80%		20Poi	80%un	60Poi	

Table 14.3 Example of a relatively complex indent experiment. The experiment incorporates an approach segment (1), four loading segments (2, 4, 6, and 8), four partial unload segments (3, 5, 7, and 10), two hold segments (9 and 11), and a final complete unloading segment (12). The use of multiple unloading segments allows sample stiffness to be calculated at several different displacement values.

The number of indents assigned to a given subfile does not necessarily indicate the number of stiffness values that will appear in that subfile. Consider the indent experiment shown in Table 14.3, which contains four unloading curves not counting the unloading curve in Segment 12. (This latter unloading segment does not yield data suitable for stiffness calculations because it begins after 80% of the load has been removed, and thus under conditions where almost all of the elastic displacement has already been recovered.). Thus for each indent we obtain four values of stiffness corresponding to the stiffness of the sample at four different displacements. Therefore, in the example above where nine indents were made in five different samples, each of the five resulting subfiles will contain 36 stiffness values. How these different values are sorted out is discussed in the following section on Hardness/Modulus calculations.

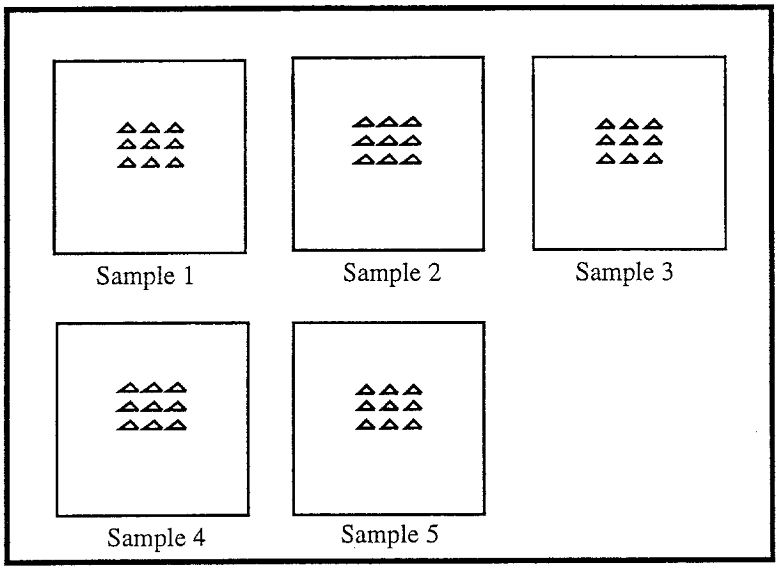


Figure 14.5 Schematic Representation of a Nine-IndentShape on each of Five different specimens

14.4.7.2.6 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.7.2.7 ALL SET, CONTINUE ON

When you are finished defining the options for "S" file calculation, the software will calculate the appropriate variables. The most important information displayed on the screen will be the plot of stiffness (N/m) versus displacement (nm).

A subfile is defined as the data contained in the unloading segments of "D" files for a series of indents. In this example, a subfile is made up of data for five indents, each of which contains four unloading segments.

14.4.7.3 Hardness/Modulus/Depth

This program uses displacement/load/time/stiffness data (the "S" files) to calculate hardness and modulus values (see Figure 14.6).

Parameter menu for CALCHMXP

File name for input data file? = SH005001 [Serrrnnn]
Poisson's Ratio of sample? = .25 0-1
Display data file configuration?
Print out calculations? = NO [YES/NO]
Use Original or Modified Cal. data? = O [O/M]
Use Area function or Tip radius model? = A [A/T]
Reorder data and average each subfile? = NO [YES/NO]
Number of indents to calc. for A(E) X? = 1
Do Diamond calibration calculation? = NO [YES/NO]
Do Iterative compliance correction? = NO [YES/NO]
Value for additional Compliance corr.? = 0 nM/mN
Value of Epsilon to use? = .75 [.72-1]
Value of Beta to use? = 1.034
Poisson's ratio of indenter? = .07 [0-1]
Modulus of indenter? = 1141 GPa
ALL SET, CONTINUE ON

NEXT CHOICE	PREV CHOICE		DO CHOICE	HELP OFF	LONG MENUS		PREV MENU

Figure 14.6 The Hardness/Modulus Calculation Parameter Menu

14.4.7.3.1 File Name For Input Data File

This option behaves as described in Section 9.4.7.3.1.

14.4.7.3.2 Poisson's Ratio Of Sample

This option behaves as described in Section 9.4.7.3.2.

14.4.7.3.3 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.7.3.4 Print Out Calculations

The Print Out Calculations option allows you to send the results of this manipulation to the printer as well as to the resultant data file. This option acts as an "ON/OFF" toggle switch. If you turn the toggle "ON" by choosing the "YES" option, you will be asked:

Print all calculations (A) or only final results (F) ?

If you do want to print the results of the manipulation, it is recommended that you use the default choice "F" to print only the final results. Otherwise, a large amount of paper can be used printing the full set of calculations and results.

14.4.7.3.5 Use Original or Modified Calibration Data

This option behaves in the same manner as the option described in Section 9.4.7.1.1.

14.4.7.3.6 Use Area Function or Tip Radius Model

This option allows the user to model the shape of the indenter tip in order to make corrections to the calculations of the projected area of an indent. The simplest and recommended approach is to select the default value, A, indicating that an internal area function is to be used to convert plastic indent depth values into projected area. This method depends on values that have been determined through a tip-area calibration. Thus, if no such calibration has been performed, you will have to assume a perfect tip, or rely on the alternative answer to the prompt, using a tip model (T), which will require an estimate of the radius of the indenter tip. When this option is selected, you will be prompted:

```
New value in range ([A/T]) ?
Press enter to accept old value, or new value
enter
to change.
```

If "T" is selected, you will not be immediately prompted for an estimated tip radius. You do not have to input this information until you select "ALL SET, CONTINUE ON." If "A" is selected, the software will use the diamond area function coefficients for the diamond number used when the experiment run was first described (see Section 8.2.8.3).

14.4.7.3.7 Reorder Data And Average Each Subfile

A response of "N" ("No") to this prompt causes the computer to calculate and store hardness and modulus values from each stiffness in the order that the stiffnesses were measured. For the indent experiment in Table 14.3, the first four lines of the data would, therefore, contain hardness and modulus values calculated from the unloading curves of segments 3, 5, 7, and 10, respectively, of the first indent. The next four lines would contain similar data for indent No. 2, and so on. Thus it is possible to relate each hardness and modulus value to a particular indent whose location on the specimen is known. Therefore, a "No" answer to the prompt is most appropriate in situations where there is some reason to feel that not all indents will yield the same H and E values, e.g., a two-phase sample. In such situations the M file could be transferred to an appropriate spreadsheet and the data manually separated and averaged. At the least, a "No" answer offers one an opportunity to inspect the data and to eliminate any obvious out-lying points before averaging.

An answer of "Y" to the prompt causes the computer to reorder the data in a given subfile and store the results in the order of decreasing total displacement. For the indent experiment in Table 14.3 the data for the first sample would consist of the nine H and E values calculated from Seg. 10 (which presumably produces the deepest indents), followed by data from Segs. 7, 5, and 3. The last entries in such a data table are the averages of the nine H and E values from each segment. This approach is good when representative data for a homogeneous sample is desired, but the reordering process means that data for individual segments can no longer be associated with a particular indent in the shape. The advantage of this procedure, obviously, is that averaged data are obtained immediately without recourse to spreadsheet or other calculations. The disadvantage is that the averages will contain not only the good data, but also the results of any bad indents that might have been produced.

14.4.7.3.8 Number Of Indents To Calculate For A(E)

The Nano Software can calculate the hardness and modulus in a number of different ways. In most cases, you will be interested in only the H(F) and E(F) results, indicating the hardness and modulus calculated using the diamond area function. An alternative method of calculating the hardness and modulus is to use the constant modulus assumption. When this option is selected, an assumption is made that the first “X” sets of data are identical experiments, that these first “X” indents are the deepest indents in the total data set, and that the area function is accurate for these depths. The areas at other depths are then calculated by assuming that the stiffness is proportional to the square root of the area.

The calculations for area based on constant modulus should be based on the deepest indents where the deviations in tip geometry have the least effect. Because the "number of indents" are counted from the first indent, you should always design your indent experiments with the deepest indents performed first.

If you intend to use this method, you should reorder the data, as described in Section 14.4.7.3.7.

14.4.7.3.9 Do Diamond Calibration Calculation

The diamond area coefficients must be generated from a specific set of indentation data. See Appendix C for detailed information regarding the diamond calibration.

However, it is also possible to perform a diamond calibration by inputting a desired modulus and allowing the software to calculate coefficients of the diamond area function based on the resultant area and contact depth. This is not a recommended method for diamond area function determination.

14.4.7.3.10 Do Iterative Compliance Correction

The procedure for determining the load frame stiffness of the indenter system requires that an iterative procedure be used to converge on a 'correct' stiffness value. See Appendix C for more detailed information regarding the procedure for correcting load frame stiffness.

This option will only be useful during a manual load frame stiffness / tip calibration manipulation.

14.4.7.3.11 Value For Additional Compliance Correction

The default answer is zero ("0"), and "0" should be entered unless you have an indication that the machine compliance should be corrected. The compliance of the instrument is accounted for automatically with the load frame stiffness value in the calibration constants array. This additional compliance correction is to be used only if you believe that further correction is required.

14.4.7.3.12 Value Of Epsilon To Use

Epsilon is a geometric constant needed in the estimation of the plastic depth for each indent. The constant, ε, may have values ranging from 0.72 to 1 depending on whether the indenter is assumed to behave as a flat punch ($\varepsilon = 1$), a parabola of rotation ($\varepsilon = 0.75$), or a conical punch ($\varepsilon = 0.72$). The recommended value is the default value, "0.75."

14.4.7.3.13 Value Of Beta To Use

The correction factor, Beta, is used in the calculations of hardness and modulus to correct for the use of a triangular indenter. The default value of 1.034 was experimentally determined. If a value of "1" is input, then no correction is made.

14.4.7.3.14 Poisson's Ratio Of Indenter

The default value, 0.07, corresponds to the Poisson's ratio for diamond. In most cases this default value should not be changed.

14.4.7.3.15 Modulus Of Indenter

As with the Poisson's ratio for the indenter, the modulus default value should not be changed unless you are sure of what you are doing.

14.4.7.3.16 ALL SET, CONTINUE ON

As specified above, when tip radius "T" is selected rather than the experimental area function, immediately upon executing "ALL SET, CONTINUE ON," you will be required to input an estimated tip radius in nanometers. Once this is done (and additional questions related to data output have been answered as described later in this section) the software will begin calculations of hardness and modulus.

14.4.7.4 Hardness/Displacement/Plastic

This subroutine calculates hardness from Load/Displacement/Time data ('D' files), assuming that the entire displacement is plastic depth. No attempt is made to correct for the elastic response of the specimen. The routine requires that a minimum depth is entered and that you select either the experimental tip area function or the spherical cap tip model (see Figure 14.7). The output of the routine is a hardness-displacement curve for each indent stored in an "XP" file. In addition, the routine assumes that the surface "zero point" was accurately determined, that is, the approach data was not preserved. Approach data is only preserved if the number of points back to the surface is set to 50, in which case the entire approach will be included in the first load subfile.

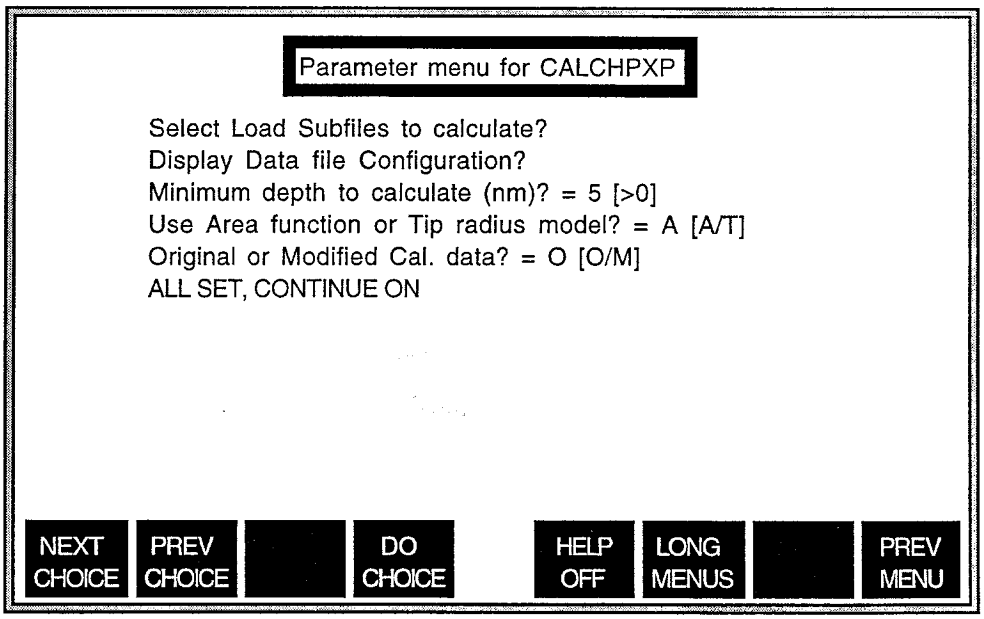


Figure 14.7 The Hardness/Displacement/Plastic Parameter Menu

14.4.7.4.1 Select Load Subfiles To Calculate

This routine calculates hardness from the loading segments. Use whichever load segment in which you have the most confidence. When this option is selected, you will be prompted to select the desired loading segments from the list of available subfiles:

```
Which subfile (Enter Subfile number, 0 if done,
or A for all) ?
```

Note that the order in which you select the subfiles determines the order in which the subfiles are processed. Select subfiles in ascending order unless you have a good reason to do otherwise.

14.4.7.4.2 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.7.4.3 Minimum Depth To Calculate

The first data points (or the shallowest data points) are often outside the limits of the theoretical depth resolution. If spurious data points from extremely shallow depths are used, the area calculated from these data points can be either extremely small or extremely large, thus skewing the results of the entire routine. The minimum depth to calculate, therefore, is given an arbitrary default value of 5 nm to exclude any such data from the calculations.

14.4.7.4.4 Use Area Function Or Tip Radius Model

This option behaves in the same manner as the option described in Section 14.4.7.3.6.

14.4.7.4.5 Use Original Or Modified Calibration Data

This option behaves in the same manner as the option described in Section 14.4.7.3.5.

14.4.7.4.6 ALL SET, CONTINUE ON

As specified above, when tip radius "T" is selected rather than the experimental area function, immediately upon executing "ALL SET, CONTINUE ON," you will be required to input an estimated tip radius in nanometers. Once this is done (and additional questions related to data output have been answered as described later in this section) the software will begin calculations of hardness and modulus.

The plastic depth, hi, is the direct equivalent of the displacement of the indenter corrected for surface position (i.e., each displacement point during the load segments is assumed to be plastic only).

14.4.7.5 Hardness/Displacement/Elastic

This subroutine calculates the hardness from Load/Displacement/Time data, correcting for the elastic response of the specimen. The method assumes that the ratio of elastic to plastic depth is constant with depth and that the elastic modulus is also constant with depth. This routine requires you to enter a minimum depth to calculate, the unloading segment to use for the elastic correction, the fraction of the unloading segment to use in the calculation, either a powerlaw fit (# of iterations) or a linear fit, and either the experimental tip area function or the tip model.

The essence of this procedure is the correction of displacement data for elastic effects. This correction is not particularly important for highly plastic metals such as annealed copper or aluminum, but it should be applied for more elastic materials, both metals and ceramics. Data from an unloading segment is used for this calculation (see Figure 14.8).

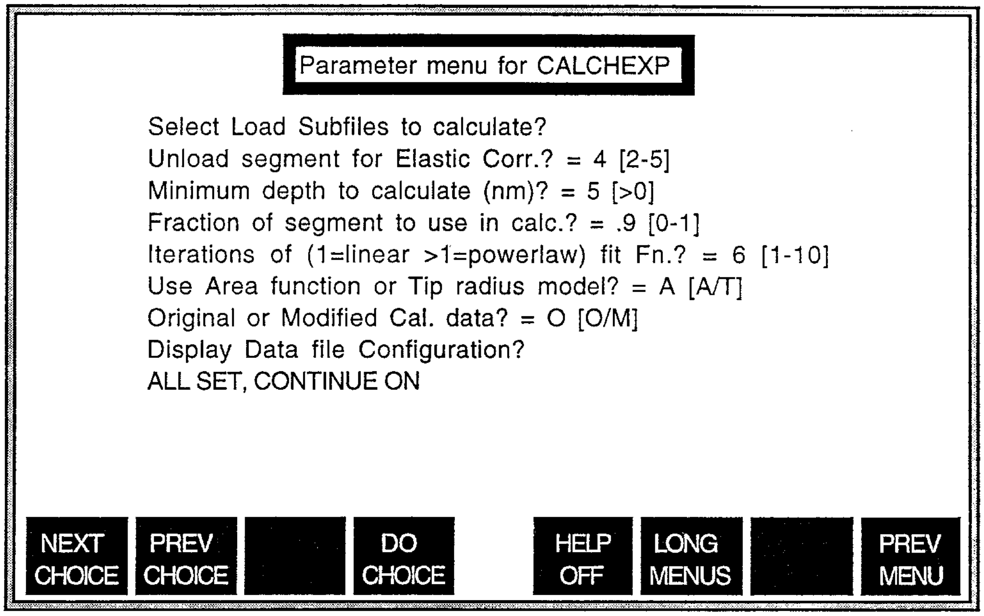


Figure 14.8 The Hardness/Displacement/Elastic Parameter Menu

14.4.7.5.1 Select Load Subfiles To Calculate

This option behaves in the same manner as the option described in Section 14.4.7.4.1.

14.4.7.5.2 Unload Segment For Elastic Correction

The unloading segment to be used for the elastic component of the hardness should be the same unloading segment that would be used to calculate stiffness from unloading. See Section 9.4.7.2.1 for more details.

14.4.7.5.3 Minimum Depth To Calculate

This option behaves in the same manner as the option described in Section 14.4.7.4.3.

14.4.7.5.4 Fraction Of Segment To Use In Calculation

This is a decimal fraction which determines the amount of the unloading curve used in the curve fit calculation. The function is fit to the first, highest load part of the curve, down to the percentage selected. See Section 14.4.7.2.3 for a more complete description of this process.

14.4.7.5.5 Iterations Of Linear Or Powerlaw Fit

The type of fit function to use may be selected. Choosing '1' will result in a least squares linear fit. Picking 2-6 will result in a multipass power law fit of the curve. This multipass fit will terminate early if the best correlation is found. See Section 14.4.7.2.4 for more information about the fitting techniques.

14.4.7.5.6 Use Area Function Or Tip Radius Model

This option behaves in the same manner as the option described in Section 14.4.7.3.6.

14.4.7.5.7 Use Original Or Modified Calibration Data

This option behaves in the same manner as the option described in Section 9.4.7.1.1.

14.4.7.5.8 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.7.5.9 ALL SET, CONTINUE ON

As specified above, when tip radius "T" is selected rather than the experimental area function, immediately upon executing "ALL SET, CONTINUE ON," you will be required to input an estimated tip radius in nanometers. Once this is done (and additional questions related to data output have been answered as described later in this section) the software will begin calculations of hardness and modulus.

14.4.7.6 ALL DONE, CONTINUE ON

Selecting this option will cause the software to return to the Review Data menu.

14.4.8 Continuous Stiffness Data: User Defined Calculations

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

14.4.8.1 Load/Displacement/Time

This option behaves as described in Section 9.4.7.1.

14.4.8.2 Hardness/Modulus From Lock-In

This option behaves as described in Section 17.2.1. This option for data reduction is useful only if your Nano Indenter® XP is equipped for the Continuous Stiffness Measurement option.

14.4.8.3 Average Data

This option behaves as described in Section 9.4.8.3, with the additional of several menu options.

This program takes any data files and returns average values of two of the variables over discrete ranges of a third variable. The data for an indent are divided into discrete "windows," and the averages of the discretized variable and of the values of a maximum of two other user-specified variables in each "window" are determined. Input data into this program can be contained in any type file. At the beginning of the program, you are shown the familiar file manipulation menu. Once the desired files have been selected, the parameter menu for CALCAV is displayed (see Figure 14.9).

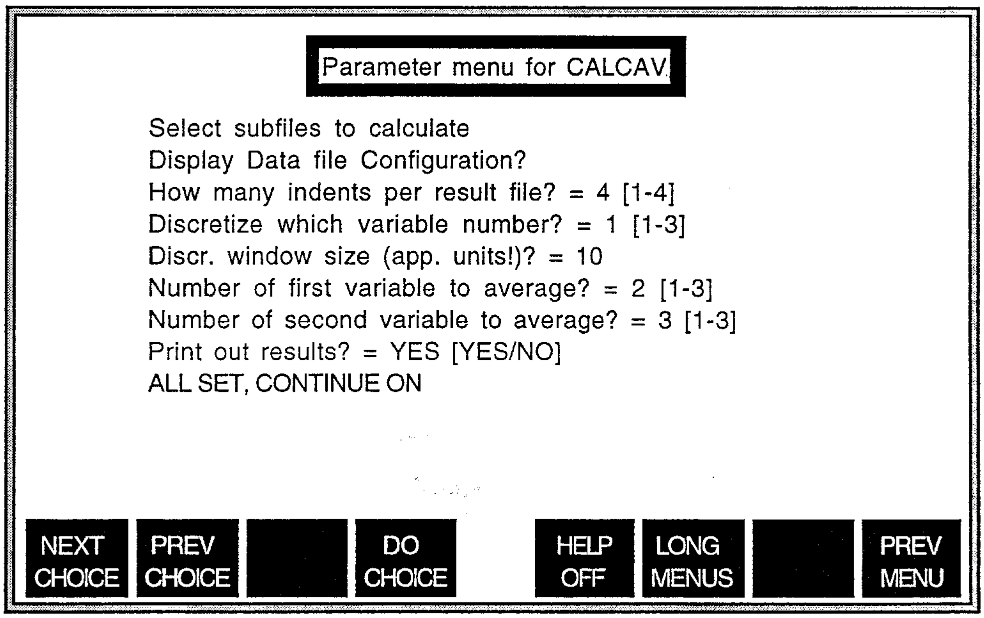


Figure 14.9 The Calculate Average Parameter Menu

14.4.8.3.1 Select Subfiles To Calculate

This option behaves in the same manner as the option described in Section 9.4.8.3.1.

14.4.8.3.2 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.8.3.3 How Many Indents Per Result File

If you so desire, you can divide the indent data into different results files. Essentially, the number of results files will be equal to the number of original indent files divided by the number of indents per result file. Thus, if you have ten original files, and you select two indents per result file, you will obtain five result files. These will be numbered 1, 3, 5, 7, and 9, and each will contain data for two indents.

14.4.8.3.4 Discretize Which Variable Number

Enter the number of the variable you want discretized. If you wish to divide depth into discrete windows, as described above in Section 14.4.8.3.3, you would enter "1" for variable number 1.

14.4.8.3.5 Discrete Window Size

Enter the range of the discretized variable, e.g., "10" if displacement is chosen as the discretized variable and a window width of 10 nm is desired. Be sure that the width is specified in the units used in the Nano Indenter® XP software for that variable (see list at end of prompt).

Note that the "window size" determines the spacing between the data points for the discretized variable in the final array. For example, if displacement is chosen as the discretized variable in a "M" file with hardness as one of the variables to be averaged and a window size of 10 nm is specified, all hardness values in each 10 nm segment of the displacement values will be averaged and will be reported as the hardness at the midpoint of that displacement window. If the hardness varies from 20 and 22 GPa with an average value of 21 GPa between displacements of 80 and 90 nm, the program produces a table containing an entry of 21 GPa for the hardness at a displacement of 85 nm. The next entry will show the hardness at a displacement of 95 nm, and so on.

14.4.8.3.6 Number Of First Variable To Average

The variables to be averaged are chosen next: Any two of the remaining variables in the Table of Variables may be chosen. Enter the number for the first variable (in ascending order).

14.4.8.3.7 Number Of Second Variable To Average

As above, enter the second variable number here.

14.4.8.3.8 Print Out Results

This option behaves in the same manner as the option described in Section 9.4.8.3.2.

14.4.8.3.9 ALL SET, CONTINUE ON

Once all information has been input, the software will perform the averaging as defined by the parameters of the routine. The output data will be stored on the hard drive.

14.4.8.4 Stiffness From Holds Lock-In

As of the writing of these instructions, this routine was not yet operational. Future versions of these instructions will include the procedure for using this routine. If your software is enabled to allow operation of this option, please contact Nano Instruments for an update to these instructions.

14.4.8.5 ALL DONE, CONTINUE ON

This option will cause the software to return to the Review Data menu.

14.4.9 Scratch Data: User Defined Calculations

Refer to Section 18.0 for more information on the Scratch Data manipulation routines.

14.4.10 Miscellaneous Data Manipulations

When this option is selected, you are first required to input information about the files to be re-formatted, as described in Section 9.2. Once the desired files are

14.4.10.1 Average Data

This option behaves in the same manner as the option described in Section 14.4.8.3.

14.4.10.2 Break Points

The "break points" routine allows you to automatically search data for 'breaks' in the data. These 'breaks' could indicate cracking or other phenomenon that indicate a rapid change in the response of a material. The routine flags or records the first discontinuity in the load displacement data, according to the user-specified definition of a 'break point (see Figure 14.10).

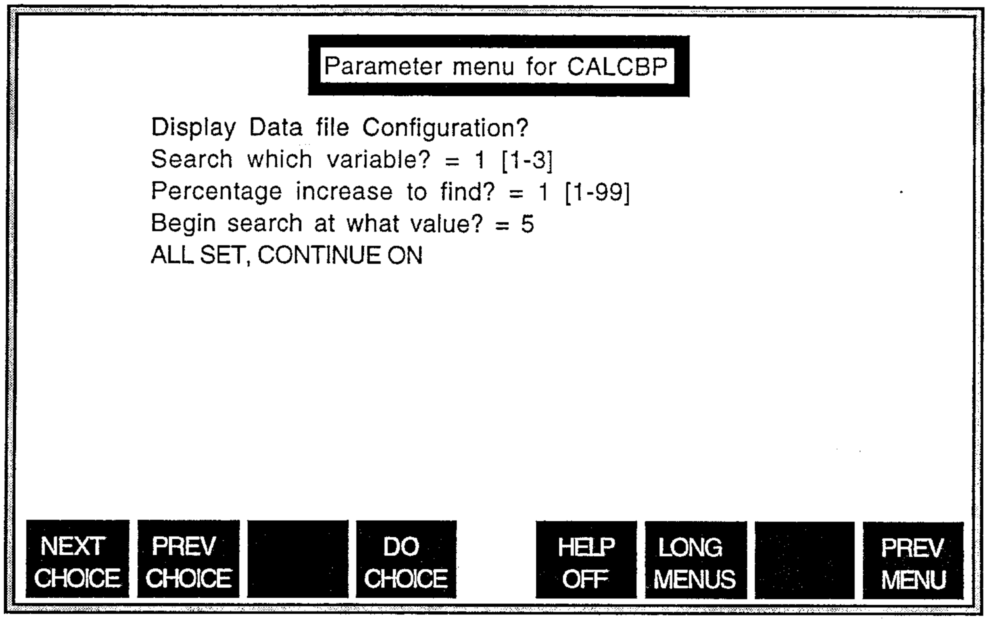


Figure 14.10 The Break Points Parameter Menu

14.4.10.2.1 Display Data File Configuration

This option behaves in the same manner as the option described in Section 9.4.7.1.3.

14.4.10.2.2 Search What Variable Number

This option allows you to specify which of the available variables you wish to search for break points. Any of the available variables can be searched, although you will most commonly use load or displacement. If a constant loading rate has been specified, you may want to search the displacement data for sudden 'jumps' in response to the constant load. On the other hand, if a constant displacement rate loading segment was specified, you may want to search the load variable for sudden increases in the load data.

14.4.10.2.3 Percentage Increase To Find

You can specify how large of a percentage increase in the variable you are searching defines that variable as a 'break.' The default value of "1" can be altered to any arbitrary percentage you wish to use.

14.4.10.2.4 Begin Search At What Value

The search for break points will begin at whatever data point you specify. The default value of "5" indicates that the search will begin from the fifth data point of the specified variable to be searched.

14.4.10.2.5 ALL SET, CONTINUE ON

Once all information has been input, the software will search the input data for discontinuity as defined by the parameters of the routine. The output data will be stored on the hard drive.

14.4.10.3 Calculate Delta & Average Displacement From Subfiles

As of the writing of these instructions, this routine was not yet operational. Future versions of these instructions will include the procedure for using this routine. If your software is enabled to allow operation of this option, please contact Nano Instruments for an update to these instructions.

14.4.11 ALL DONE CONTINUE ON

Selecting this final option will return you to the Master Menu.

15.0 Load Specimen/Manual Control

15.1 Overview

There are no "advanced functions" currently enabled for the Load Specimen/Manual Control menu option. Refer to Section 10.0 for a complete description of the standard operation of this option.

16.0 Calibrations

16.1 Overview

Several of the Calibrations accessible under the Calibration "long" Menu are not intended for general use. These calibrations may require tools or special procedures, and are thus performed only at Nano Instruments or by a representative of Nano Instruments. From a "daily use" perspective, only the calibrations available on the "short" menu are of particular interest. However, advanced operation of the instrument requires the use of some of the additional extended options (see Figure 16.1).

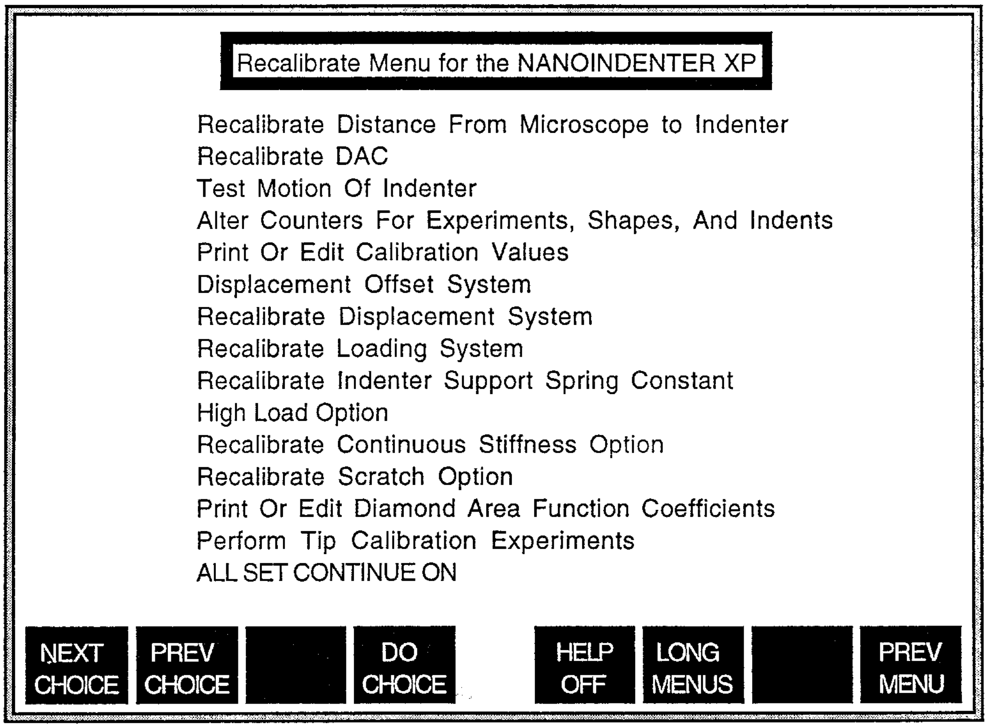


Figure 16.1 The Recalibrate Extended Menu

16.2 The Calibration Extended Menu

You will rarely need to access the extended options on the calibration menu. Of the following options, 16.2.4, 16.2.5, 16.2.13 and 16.2.14 are of most interest in general use of the instrument.

16.2.1 Recalibrate Distance From Microscope To Indenter

Refer to Section 11.2.1 for more information about this option.

16.2.2 Recalibrate DAC

Refer to Section 11.2.2 for more information about this option.

16.2.3 Test Motion Of Indenter

Refer to Section 11.2.3 for more information about this option.

16.2.4 Alter Counters For Experiments, Shapes, And Indents

After sustained use of the instrument, the serial counters which set the experiment, shape, and indent numbers may reach or approach the maximum counter size. This option allows you to reset the counters.

16.2.4.1 Run Number

The Run Number refers to the overall experiment number. Choosing this option simply allows you to input a new number.

16.2.4.2 Shape Number

The Shape Number refers to the counter value of the stored indent position shapes. Choosing this option simply allows you to input a new number.

16.2.4.3 Indent Number

The Indent Number is the counter representing the stored indent experiments. Choosing this option simply allows you to input a new number.

16.2.4.4 Hardness Experiment Number

The Hardness Experiment Number refers to the counter value for the stored hardness experiments. Choosing this option simply allows you to input a new number.

16.2.4.5 Print Stored Value Of Counters

This command simply prints a copy of the values of these four counters.

16.2.4.6 START OVER

The Start Over command allows for correction of mistakes. As long as you have used the "ALL SET CONTINUE ON" command, using the Start Over command will return the counter values to the numbers that appeared when you first entered this submenu.

16.2.4.7 ALL SET, CONTINUE ON

This final command saves the new values of the counters (if they have been changed) and returns you to the Calibration Menu.

16.2.5 Print Or Edit Calibration Values

The Print Or Edit Calibration Values routine allows you to manually alter the stored calibration values. Obviously, care should be exercised in the use of this routine, as arbitrary alteration of the calibration data array can result in incorrect calculation of data or incorrect instrument operation.

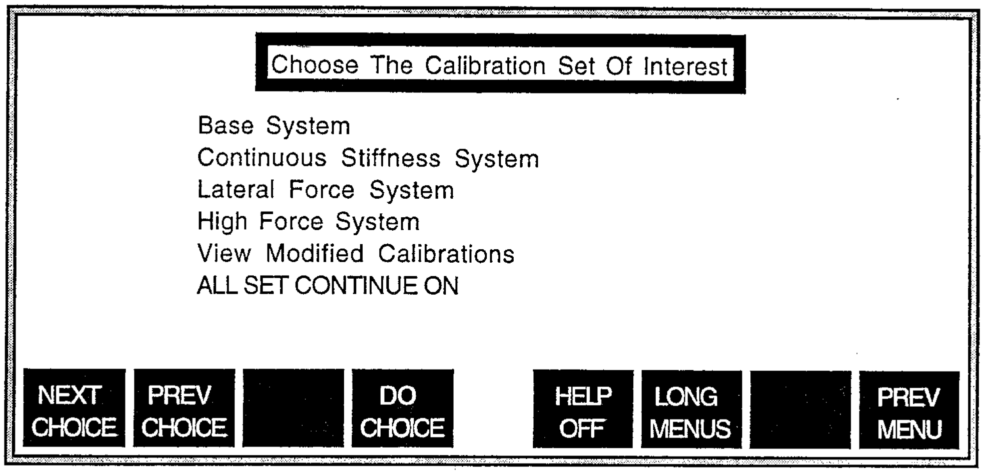


Figure 16.2 The Calibration Set Menu

16.2.5.1 Base System

The Base System calibrations are those stored for the standard instrument without additional attachments or options. These are the "core" calibrations that affect all aspects of the instrument's operation.

Base System Calibrations	
Calibration	**Units**
Load Frame Stiffness	N/m
Microscope To Indenter X	µm
Microscope To Indenter Y	µm
Spring Stiffness	N/m
Displacement	nm/V
Force	µN/V

Table 16.1 System Calibrations and Associated Units

Subfunctions allow you to print a copy of these calibrations. Selecting the individual calibration and using the "Do Choice" key will allow you to change the calibration value. Once "ALL SET, CONTINUE ON" is selected, any changes in the calibration values are saved.

16.2.5.1.1 Load Frame Stiffness

The Load Frame Stiffness Calibration is a measure of the compliance of the load frame that surrounds the indenter head.

This calibration is usually performed in conjunction with the diamond tip area calibration. Refer to Appendix C for more information about this value.

16.2.5.1.2 Microscope To Indenter X & Y

The Microscope To Indenter calibration is the stored distance between the microscope focal point (in the X-Y plane) and the indenter's impact site. For more information on this calibration, refer to Section 11.2.1.

16.2.5.1.3 Displacement Coefficients C, B, A

The Displacement Coefficients are the coefficients of the second order polynomial fit that describes the displacement response of the indentation system. These should never be changed except during a re-calibration of the indenter system.

16.2.5.1.4 Force Coefficients C, B, A

The Force Coefficients are the coefficients of the second order polynomial fit that describes the load response of the indentation system. These should never be changed except during a re-calibration of the indenter system.

16.2.5.1.5 Print Out Calibrations

Selection of this option will cause a copy of the values displayed on screen to be printed.

16.2.5.1.6 START OVER

If you have made changes to the calibration set, and want to negate those changes and return to the previously stored values, select this option. Note that this will only be effective on changes that you have made before leaving the routine. That is, if you changed and stored a value previously, the use of the START OVER option will not be able to restore the value you replaced.

16.2.5.1.7 ALL SET, CONTINUE ON

Once you select this option you will be returned to the previous menu. If you have made changes to the calibration set, you will be offered the opportunity to store those changes.

16.2.5.2 Continuous Stiffness System

Refer to Section 17.0 describing the continuous stiffness measurement option for further details on this routine. If your system is not equipped with the CSM option, you will not be able to enter this routine.

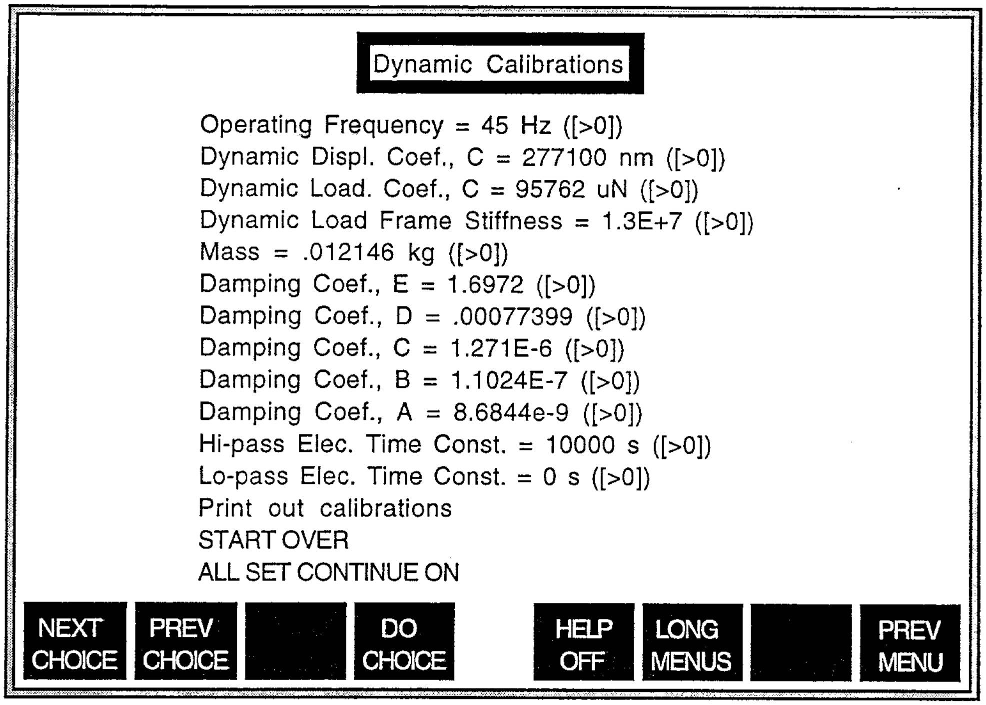


Figure 16.3 The Continuous Stiffness Calibrations

16.2.5.2.1 Operating Frequency

The operating frequency refers to the frequency of oscillation used with the CSM option. If you would like to change this value, you should do so by following the procedure described in Section 17.3.

16.2.5.2.2 Dynamic Displacement Coefficient

The Dynamic Displacement Coefficient is the first term of the second order dynamic displacement calibration that describes the displacement response of the indentation system. This value should never be changed except during a re-calibration of the indenter system.

16.2.5.2.3 Dynamic Load Coefficient

The Dynamic Load Coefficient is the first term of the second order dynamic load calibration that describes the force response of the indentation system. This value should never be changed except during a re-calibration of the indenter system.

16.2.5.2.4 Dynamic Load Frame Stiffness

The Dynamic Load Frame Stiffness calibration is a measure of the compliance of the load frame that surrounds the indenter head under an oscillating load. This calibration is usually performed in conjunction with the diamond tip area calibration. Refer to Appendix C for more information about this value.

16.2.5.2.5 Mass

The mass of the indenter is determined during the Dynamic Calibration of the instrument. This value should never be changed except during a re-calibration of the indenter system.

16.2.5.2.6 Damping Coefficients E, D, C, B, A

The Damping Coefficients are the coefficients of the fourth order polynomial fit that describes the damping response of the indentation system. These should never be changed except during a re-calibration of the indenter system.

16.2.5.2.7 Hi-pass and Lo-pass Electronic Time Constants

The Hi-pass and Lo-pass Time Constants correspond to the electronic interface between the phase lock amplifier and the data acquisition and control system. These values should never be changed.

16.2.5.3 Lateral Force System

Refer to Section 18.0 describing the lateral force option for more information about these calibrations. If your system is not equipped with the lateral force option, you will not be able to enter this routine.

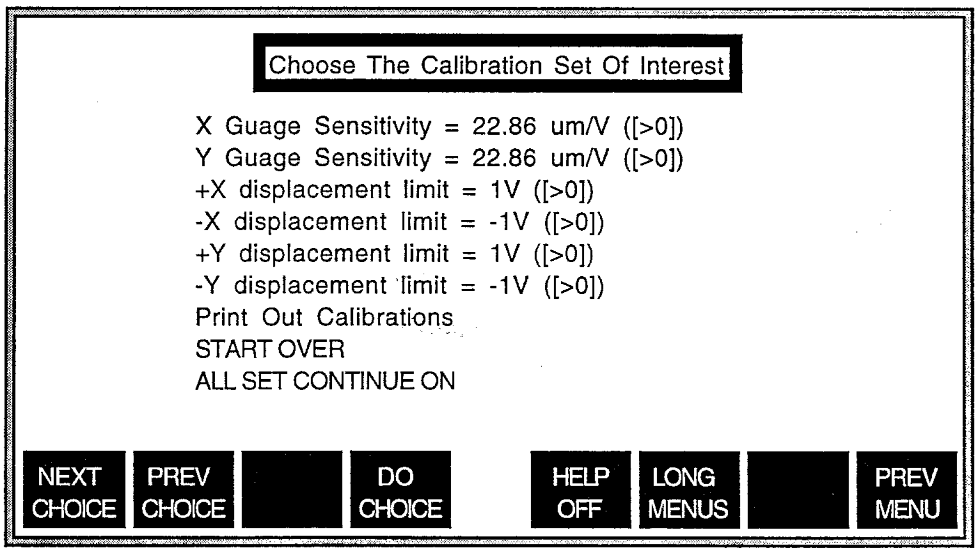


Figure 16.4 Lateral Force Calibrations

16.2.5.3.1 X & Y Gauge Sensitivity

The X & Y Gauge Sensitivities refer to the displacement/voltage response of the proximity probes used with the Scratch Option. These values are determined during production of the instrument and should not be changed.

16.2.5.3.2 X & Y Displacement Limit

The X & Y Displacement Limits refer to the maximum allowable deflections of the indenter shaft during a scratch test. These values are determined during production of the instrument and should not be changed.

16.2.5.4 High Force System

Refer to Section 19.0 describing the high force option for more information about these calibrations. If your system is not equipped with the high force option, you will not be able to enter this routine.

16.2.5.4.1 Force Calibration

The High Load Force Coefficient describes the force response of the high load option for the indentation system. This value should never be changed except during a re-calibration of the indenter system.

16.2.5.4.2 Spring Constant

The High Load Spring Constant describes the stiffness response of the indentation system with the high load option engaged. This value should never be changed except during a re-calibration of the indenter system.

16.2.5.5 View Modified Calibrations

Selecting the "View Modified Calibration" option will display the most recent set of stored modified calibrations. This option is useful for tracking changes to the calibration data array.

16.2.6 Displacement Offset System

Calibration of the displacement offset system of the Nano Indenter® XP is performed during production at Nano Instruments. This calibration is a determination of the displacement ranges of the displacement sensing electronics, and should not change over time. Performance of this calibration will require that the Displacement Calibration be performed again (see Section 16.2.7). It is not recommended that the user perform this calibration.

16.2.7 Recalibrate Displacement System

Calibration of the displacement sensing system of the Nano Indenter® XP is performed during production at Nano Instruments. This calibration is a measurement of the response of the displacement sensing electronics, and should not change over time. In any case, the tools for performing this calibration are not available to the user.

16.2.8 Recalibrate Loading System

Like the displacement calibration, the loading system calibration is performed during the production of your instrument. In general operation, there is no need to perform this calibration in the field. Like the displacement calibration, the load calibration procedure requires tools and equipment that are not supplied with the instrument.

16.2.9 Recalibrate Indenter Support Spring Constant

Although the indenter support spring calibration does not require any tools or equipment, there should rarely be a need to perform this procedure. Thus, instructions are omitted. If you have been advised by Nano Instruments to perform this option, please request instructions from your contact at Nano.

16.2.10 High Load Option

If your Nano Indenter® XP is equipped with the High Load Option, an additional calibration, the High Load calibration is performed during the production of your instrument. In general operation, there is no need to perform this calibration in the field. The High Load calibration procedure requires tools and equipment that are not supplied with the instrument. Refer to Section 19.0 for more information on the High Load option.

16.2.11 Recalibrate Continuous Stiffness Option

If your Nano Indenter® XP is equipped with the continuous stiffness measurement (CSM) option, you may occasionally need to recalibrate the dynamic response of the CSM hardware. See the CSM instructions later in this manual (Section 17.0) for a description of this procedure.

16.2.12 Recalibrate Scratch Option

If your Nano Indenter® XP is equipped with the lateral force measurement (LFM) option, then the Scratch Option calibrations will have been performed during the production of your instrument. The Scratch Option calibration procedure requires tools and equipment that are not supplied with the instrument. See the LFM instructions later in this manual (Section 18.0) for a description of this scratch option..

16.2.13 Print Or Edit Diamond Area Function Coefficients

Like the Print Or Edit Calibration Values routine, the Print or Edit Diamond Area Function Coefficients routine allows you to manually alter the stored calibration values. As described above, care should be exercised in the use of this routine, as arbitrary alteration of the calibration data array can result in incorrect calculation of data. This routine differs from the "General Calibration" editing procedures in that multiple data arrays can be stored, and referenced by "Diamond Number." This allows you to have multiple diamond tips, to store the area function coefficients for each diamond, and to automatically retrieve those coefficients depending on which diamond you are using. Refer to Section 8.2.8.3 for instructions on using these coefficients.

There are nine coefficients in each diamond's data array. The first coefficient is almost always "24.5" (the coefficient for a perfect tip). The remaining eight coefficients are determined in the diamond area function calibration, described in Appendix C.

Upon selecting the Print or Edit Diamond Area Function Coefficients option from the Calibration Menu, the Coefficient Manipulation Menu will be displayed.

16.2.13.1 Diamond Number

The diamond number refers to the sequential number assigned to the diamond for which a set of calibration coefficients is valid. For example, if you have multiple users, each with a different diamond tip, you can assign each user's tip a different diamond number, and store the coefficients for that diamond with the diamond number. When you select the "Diamond Number" option, and enter a specific number, the coefficients for that diamond will be displayed.

16.2.13.2 Print Out Stored Coefficients

When this option is selected, you will be prompted:

```
Print coefficients of Selected diamond or All
diamonds [S/A]
```

If you only want to print the coefficients for the diamond number you entered (Section 16.2.13.1 above) then choose the "S" option. If you want a printout of all diamond's coefficients, then choose the "A" option.

16.2.13.3 Coefficient of h^#

The coefficients for the diamond as selected by number are displayed on the nine coefficient lines. To change or enter a coefficient, select the line the coefficient is displayed on, and use the "DO CHOICE" key to activate the input line.

16.2.13.4 Start Over, This Diamond

If you have made changes to a diamond's coefficients, and want to erase those changes and return to the original diamond coefficients, use the "Start Over" option to reload the original coefficients.

16.2.13.5 ALL SET, CONTINUE ON

Selection of this final option will cause any changes you have made to take effect, and return you to the previous menu.

16.2.14 Perform Tip Calibration Experiments

When a new diamond tip is to be used, or an old diamond tip has become worn, you may want to perform a new tip area calibration. The tip area calibration is performed automatically by the software, using a pre-defined experiment to run a number of indentations on a standard material (fused silica). To perform this calibration, load the fused silica sample into the sample tray. Then select this option from the Recalibrate menu. Once this option is selected, you will be prompted:

What is the number of the currently installed diamond ? [1-99]

Diamonds are identified by a sequential number. This number can be used in any way you desire. For example, each new diamond tip can be assigned a number, so that in multi-user facilities diamond tips can be exchanged when a new user needs the instrument. Alternatively, multiple numbers can be assigned to single diamonds, so that all sets of diamond area function coefficients are retained in the software.

Once you enter the desired diamond number, you will be prompted:

**Please select the start point for this experiment run.
Hit CONTINUE when ready.**

Select the start point for the experiment array, just as you would when using the Standard Test option (see Section 7.0). Once you have selected the start point, the software will assume control of the instrument, and the test will be run automatically. The data will be automatically manipulated following the completion of the test, and an output file will be written to the hard drive of the computer.

Refer to Appendix C for instructions on manipulation of this output file.

16.2.15 ALL SET CONTINUE ON

Selecting this option will return the software to the master menu.

17.0 The Continuous Stiffness Measurement Option

17.1 Overview

The Continuous Stiffness Measurement Option adds Nano Instruments' patented dynamic oscillation method to the Nano Indenter® XP. Briefly described, the CSM technique adds a small oscillatory force to the load applied by the indenter during testing. The output oscillation can be compared to the applied oscillation to continuously provide the stiffness of contact during the indentation process.

The functionality added to the system does not add any additional set-up requirements. CSM operation is transparent to the user. Data reduction does add one optional step, the "Hardness/Modulus From Lock-In" option.

The term "Lock-In" refers to the additional hardware included with the CSM option: the phase lock amplifier (PLA). There is no need for the user to directly access this hardware. The only direct interface with the PLA is through the CSM recalibration routines, briefly discussed at the end of this section.

For additional information about the Continuous Stiffness Measurement method, see Reference J.1 in Appendix J.

17.2 Data Reduction

When data is reduced using the automated calculation stream, there will be no need to execute this additional data manipulation step. If you desire to change the parameters of manipulation of the CSM data, however, the Hardness & Modulus From Lock-In routine provides the reduction functionality to convert the "D" files to hardness and modulus data.

17.2.1 Hardness/Modulus From Lock-In

This program uses displacement/load/time/lock-in data (the "DS" files) to calculate hardness and modulus values (see Figure 7.1). Files are selected using the standard conventions for selecting data files (see Section 14.2).

17.2.1.1 Poisson's Ratio of Sample

The default value, "0.25," represents a reasonable 'midrange' estimate. If the Poisson's ratio of a sample is known, it should be entered in place of the default value.

17.2.1.2 Display Data File Configuration

This option behaves in the same manner as the option described in Section 14.4.3.3.

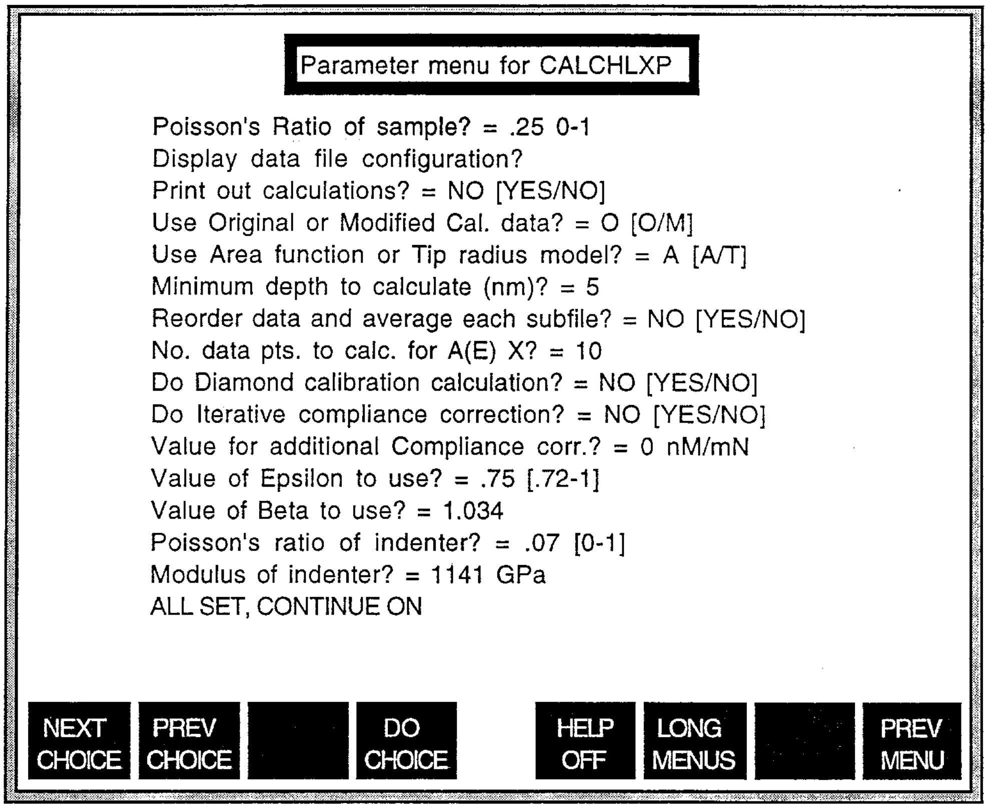


Figure 17.1 The Hardness/Modulus from Lock-In Calculation Parameter Menu

17.2.1.3 Print Out Calculations

The Print Out Calculations option allows you to send the results of this manipulation to the printer, as well as to the resultant data file. This option acts as an "ON/OFF" toggle switch. If you turn the toggle "ON" by choosing the "YES" option, you will be asked:

Print all calculations (A) or only final results (F) ?

If you do want to print the results of the manipulation, it is recommended that you use the default choice "F" to print only the final results. Otherwise, a large amount of paper can be used printing the full set of calculations and results.

17.2.1.4 Use Original or Modified Calibration Data

This option behaves in the same manner as the option described in Section 14.4.3.1.

17.2.1.5 Use Area Function or Tip Radius Model

This option allows the user to model the shape of the indenter tip in order to make corrections to the calculations of the projected area of an indent. The simplest and recommended approach is to select the default value, A, indicating that an internal area function is to be used to convert plastic indent depth values into projected area. This method depends on values that have been determined through a tip-area calibration. Thus, if no such calibration has been performed, you will have to assume a perfect tip, or rely on the alternative answer to the prompt, using a tip model (T), which will require an estimate of the radius of the indenter tip. When this option is selected, you will be prompted:

```
New value in range ([A/T]) ?
Press enter to accept old value, or new value enter
to change.
```

If "T" is selected, you will not be immediately prompted for an estimated tip radius. You do not have to input this information until you select "ALL SET, CONTINUE ON." If "A" is selected, the software will use the diamond area function coefficients for the diamond number used when the experiment run was first described (see Section 8.2.8.3).

17.2.1.6 Minimum Depth To Calculate

The first data points (or the shallowest data points) are often outside the limits of the theoretical depth resolution. If spurious data points from extremely shallow depths are used, the area calculated from these data points can be either extremely small or extremely large, thus skewing the results of the entire routine. The minimum depth to calculate, therefore, is given an arbitrary default value of 5 nm to exclude any such data from the calculations.

17.2.1.7 Reorder Data and Average Each Subfile

A response of "N" ("No") to this prompt causes the computer to calculate and store hardness and modulus values from each stiffness in the order that the stiffnesses were measured. For the indent experiment in Table 14.3, the first four lines of the data would, therefore, contain hardness and modulus values calculated from the unloading curves of segments 3, 5, 7, and 10, respectively, of the first indent. The next four lines would contain similar data for indent No. 2, and so on. Thus, it is possible to relate each hardness and modulus value to a particular indent whose location on the specimen is known. Therefore, a "No" answer to the prompt is most appropriate in situations where there is some reason to feel that not all indents will yield the same H and E values, e.g., a two-phase sample. In such situations the M file could be transferred to an appropriate spreadsheet and the data manually separated and averaged. At the least, a "No" answer offers one an opportunity to inspect the data and to eliminate any obvious outliers before averaging.

An answer of "Y" to the prompt causes the computer to reorder the data in a given subfile and store the results in the order of decreasing total displacement. For the indent experiment in Table 14.3, the data for the first sample would consist of the nine H and E values calculated from Segment 10 (which presumably produces the deepest indents, followed by data from Segments 7, 5, and 3. The last entries in such a data table are the averages of the nine H and E values from each segment. This approach is good when representative data for a homogeneous sample is desired, but the reordering process means that data for individual segments can no longer be associated with a particular indent in the shape. The advantage of this procedure, obviously, is that averaged data are obtained immediately without recourse to spreadsheet or other calculations. The disadvantage is that the averages will contain not only the good data but also the results of any bad indents that might have been produced.

17.2.1.8 Number of Data Points to Calculate for A(E)

The Nano Software can calculate the hardness and modulus in a number of different ways. In most cases, you will be interested in only the H(F) and E(F) results, indicating the hardness and modulus calculated using the diamond area function. An alternative method of calculating the hardness and modulus is to use the constant modulus assumption. When this option is selected, an assumption is made that the deepest "X" data points are identical, and that the area function is accurate for these depths. The areas at other depths are then calculated by assuming that the stiffness is proportional to the square root of the area.

17.2.1.9 Do Diamond Calibration Calculation

The diamond area coefficients must be generated from a specific set of indentation data. See Appendix C for detailed information regarding the diamond calibration.

However, it is also possible to perform a diamond calibration by inputting a desired modulus and allowing the software to calculate coefficients of the diamond area function based on the resultant area and contact depth. This is not a recommended method for diamond area function determination.

17.2.1.10 Do Iterative Compliance Correction

The procedure for determining the load frame stiffness of the indenter system requires that an iterative procedure be used to converge on a 'correct' stiffness value. See Appendix C for more detailed information regarding the procedure for correcting load frame stiffness.

This option will only be useful during a manual load frame stiffness / tip calibration manipulation.

17.2.1.11 Value For Additional AC Compliance Correction

The default answer is zero ("0"), and "0" should be entered unless you have an indication that the machine compliance should be corrected. The compliance of the instrument is accounted for automatically with the load frame stiffness value in the calibration constants array. This additional compliance correction is to be used only if you believe that further correction is required in the dynamic load frame stiffness.

17.2.1.12 Value of Epsilon To Use

Epsilon is a geometric constant needed in the estimation of the plastic depth for each indent. The constant, ε, may have values ranging from 0.72 to 1 depending on whether the indenter is assumed to behave as a flat punch ($\varepsilon = 1$), a parabola of rotation ($\varepsilon = 0.75$), or a conical punch ($\varepsilon = 0.72$). The recommended value is the default value, "0.75."

17.2.1.13 Value of Beta To Use

The correction factor, Beta, is used in the calculations of hardness and modulus to correct for the use of a triangular indenter. The default value of 1.034 was experimentally determined. If a value of "1" is input, then no correction is made.

17.2.1.14 Poisson's Ratio of Indenter

The default value, 0.07, corresponds to the Poisson's ratio for diamond. In most cases this default value should not be changed.

17.2.1.15 Modulus of Indenter

As with the Poisson's ratio for the indenter, the modulus default value should not be changed unless you are sure of what you are doing.

17.2.1.16 ALL SET, CONTINUE ON

As specified above, when tip radius "T" is selected rather than the experimental area function, immediately upon executing "ALL SET, CONTINUE ON," you will be required to input an estimated tip radius in nanometers. Once this is done (and additional questions related to data output have been answered as described later in this section) the software will begin calculations of hardness and modulus.

17.2.2 Stiffness From Holds Lock-In

Stiffness can be generated in one of two ways if the continuous stiffness option was in use when the data was taken. The standard method for calculating stiffness is to select one or more unloading segments from the data set, and to calculate stiffness based on the slope of the unloading segments. If the continuous stiffness option is in use, the stiffness can also be calculated from hold segments (Stiffness from holds Lock-in). It is generally recommended that the hold segment selected for this procedure be a relatively long (i.e., greater than 10 seconds) segment directly following the maximum load segment. As with stiffness from unloading segments, however, multiple hold segments can be used to calculate stiffness from holds Lock-in.

Continuous stiffness measurements are made by monitoring the frequency and amplitudes of indenter vibrations induced through the superposition of a small AC current on the DC current of the force coil. Thus, it is possible to obtain stiffness values during a HOLD segment when both gross load and gross displacement are constant. This program uses data from "D" files to create files that can be fed into the Hardness/Modulus calculational program in the same manner as "S" files (i.e., files of stiffness values obtained from unloading curves.)

Upon entering the routine, the data files for manipulation are selected as usual, in the File Manipulation menu. As specified above, only "D" files can be accepted. Once the files have been selected, the parameter menu appears:

17.2.2.1 Display Data File Configuration

This option enables you to view the variables and segments contained in the data files you have selected to work with. See Section 9.2 for further information on its operation.

17.2.2.2 Indents Per Subfile in Results File

As with the "S" files, this option is essentially a data filing tool which can be used to separate the data into 'subfiles' within the actual output file. See Section 9.4.7.2 for more detailed information.

17.2.2.3 Select Subfiles to Calculate

As described above, the stiffness from holds Lock-in routine calculates the stiffness from a hold segment. The recommended hold segment to be used is a relatively long segment directly following the maximum load segment. As with the stiffness from unloading calculations, multiple hold segments can be chosen.

17.2.2.4 File Name for Results File

The output data will appear in a “S” file on the hard disk. The default value is the prefix, “S” with the secondary suffix and the base file name appended. See Section 9.2.1 for more information about the Base File Name.

17.3 Calibration & Configuration

There should be no need for the user to recalibrate the continuous stiffness measurement option, although there is no specific hardware required. One step of the calibration procedure, however, is useful in changing the configuration of the CSM option. This step is naturally called the “Configure” step.

17.3.1 The Configure Step

The primary use of the configure step is to set the operating frequency of the phase lock amplifier. For most users this step will not be necessary.

To change the frequency of operation, proceed to the Recalibrate option on the Master Menu. Choose “Recalibrate Continuous Stiffness Option” from the Recalibrate Menu. You will be prompted:

```
Exit now to run other calibrations [Y/N]?
```

Choose "N" to indicate that you do not wish to exit. Next, you will be prompted:

Do you wish to run all parts of the calibration automatically (Y/N)?

Choose "N" to indicate that you do not want to run all parts of the calibration. Finally, you will be prompted:

Skip calibration steps and perform configuration only (Y/N)?

Select "Y" to instruct the indenter to proceed to the configuration step. Once this is done, the software will position the indenter at the appropriate displacement voltage, and the configuration will begin. You will be asked to enter the frequency. The default frequency of 45Hz is an excellent choice for almost all environments and applications.

Once you have entered the frequency, the configuration step will complete and you will be returned to the Recalibrate Menu.

17.3.2 The Calibrations

If you would like to recalibrate the continuous stiffness option, please contact Nano Instruments, or your local representative.

18.0 Lateral Force Measurement Option

18.1 Overview

The lateral force option (or "Scratch option") allows the Nano Indenter® XP to monitor and withstand lateral forces which would otherwise endanger the instrument.

The additional hardware for the lateral force option includes a set of proximity probes which mount around the indenter shaft.

In effect, the Nano Indenter® XP is physically capable of scratch testing without the lateral force option in place (although the software is not designed to allow you to use the Nano Indenter® XP to perform scratching when the hardware is disengaged). The danger to the instrument, however, discourages this application. If, however, laterally applied forces are held within strict limits, the indenter assembly is capable of withstanding these forces without damage to the instrument.

Thus, the lateral force option consists primarily of hardware designed to monitor the lateral deflection of the indenter shaft away from its 'centered' or 'home' position. A proximity probe is mounted in each lateral direction (x and y). These probes are calibrated and then centered facing a reference surface on the indenter shaft.

Once the appropriate scratch testing hardware has been installed and the desired scratch tip is in place on the indenter shaft, the lateral force option is ready to run.

18.2 Performing A Scratch As The Standard Test

If your Nano Indenter® XP software has been configured so that a scratch test is the "Standard Test," then there is nothing further to be done to perform scratch tests. The Standard Test will run automatically, and the reduced data will be generated at the end of the test. Refer to Section 7.0 for information on performing the Standard Test, and Section 18.8 for information on interpreting the reduced data.

In almost all cases, the scratch used in the Standard Test will be a Y-direction scratch of 700µm length ("upward" from the first indent site). The number of such scratches will vary depending on the application. Contact Nano Instruments or your local representative for more information if your Standard Test is set to a scratch test.

18.3 Performing A Scratch Using "Design Custom Test"

If your Nano Indenter® XP is equipped with the Lateral Force Measurement option, the "Standard Scratch" test will appear in the Select Indentation Test menu (see Section 8.2.5.6). If you select the Standard Scratch as the desired indentation experiment, you will be required to enter information about the initial and final loads for the scratch.

18.3.1 Defining The Standard Scratch

When the Standard Scratch Test option is selected, you will be prompted:

```
Starting Load in micro Newtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]
```

The Starting Load refers to the initial load you want to achieve with this experiment. The initial loading segment will terminate when you reach this load. The load is to be entered in micro Newtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

When the standard scratch experiment is performed, this Starting Load will be the load applied to the sample during the initial profile, pre and post-scratch scan segments, and final profile segments.

Once you have defined the Starting Load, you will be prompted:

```
Ending Load in micro Newtons (uN)?
1-6 digits numeric [nnnnnn or n.nExx]
```

The Ending Load refers to the final load you want to achieve with this experiment. The "scratch" loading segment will terminate when you reach this load. The load is to be entered in micro Newtons, and can be entered in either "general format" or "scientific notation." The maximum number of characters in either format is six.

When the standard scratch experiment is performed, this Ending Load will be the final load reached during the scratch segment. During the scratch segment, the instrument will continuously ramp the load from the Starting Load to the Ending Load as the indenter travels across the surface over the scratch length.

18.3.2 The Mechanics Of The Standard Scratch Experiment

18.3.2.1 Segment Codes For The Standard Scratch

In terms of the Segment Codes used to define an experiment, (see Section 9.3.1) the Standard Scratch can be written as:

Standard Scratch Experiment		
Segment	**Segment Code**	**Parameters**
1	A	Defaults
2	LL	2 Rat, 20 For
3	D	0 Rat, 1 Shp
4	D	0 Rat, 2 Shp
5	D	0 Rat, 3 Shp
6	LL	2 Rat, 20 For
7	D	3999.6 Rat, 4 Shp, 100 Dac
8	UL	50% Ra, 20 For
9	D	0 Rat, 3 Shp
10	D	0 Rat, 2 Shp
11	D	0 Rat, 1 Shp
12	UL	300 Rat

Table 18.1 The Standard Scratch Experiment

18.3.2.2 The Standard Scratch Operations

In terms of the actual operations the experiment is performing, the Standard Scratch is described as:

Approach to the surface using the default conditions, then apply a load at a rate of 2µN/sec until a load of 20µN is reached. Then scratch without changing the load using Shape 1 as the pattern. Next, scratch using Shape 2, without changing the load. Scratch using Shape 3, again leaving the load constant. Apply load again (in this case since the load is the same as that applied in segment 2, no further load will be applied). Finally, begin the ramp-load scratch, scratching using Shape 4 at the default speed, with an acceleration of 100 µm/s^2, while applying a load of 3999.6 µN/sec. Upon completion of this scratch segment, remove the load at half of the rate used during the scratch (50% of 3999.6 µN/sec) until only 20µN is applied to the sample. Next, repeat the scratches of Shapes 3, 2, applying no further load. At the completion of these segments, remove the indenter from the surface of the sample.

18.3.2.3 Physical Behavior During The Standard Scratch

Physically, the actions of the Standard Scratch can be described as:

Approach the surface, apply a small load, then profile the surface over the total scratch length. Reverse direction and profile back to the start point. Then profile over a smaller distance and apply the preload. Next, apply the ramp load while scratching over the majority of the remaining profiled length, until reaching the desired maximum load. At the end of the ramp load scratch, remove most of the load, profile to the end of the profile length, and repeat the initial profiles. Finally, remove the indenter from the surface.

18.3.2.4 The Purpose For Each Scratch Segment

These segments can also be described by their purposes. Segments 3 and 4 represent the Initial Profile, which is used to obtain a baseline for surface roughness and slope. Segment 5 represents the Pre-scratch scan, which will be used for levelling the data during manipulation. Segment 7 is the scratch itself, during which deformation of the sample is achieved and coefficient of friction data is obtained. Segment 9 is the Post-scratch Scan, which will be used with the Pre-scratch Scan to level the data. Finally, Segments 9 and 10 are the Final Profile segments.

18.3.2.5 The Shape Parameter

The Shape Parameter is the method by which the scratch vector is specified. As mentioned in Section 18.4, the direction and length of the scratch segment are defined as the line between two indentations. This is for positional reference only; that is, the indentation sites that describe the endpoints of the scratch segment are sites only, no "indentations" will necessarily be made at these sites.

To input the direction and length of the scratch into the experiment design, a "Shape" is used. The Shape is simply a recorded set of coordinates that can represent an indent pattern or a scratch (see Section 18.4.2). If your Nano Indenter® XP includes the Lateral Force Measurement option, the standard shapes will be #'s 1,2,3, and 4. By default, these shapes are:

Standard Scratch Shapes		
Shape	**Length**	**Direction**
1	700 µm	Positive Y direction from initial point
2	700 µm	Negative Y direction
3	100 µm	Positive Y direction
4	500 µm	Positive Y direction

Table 18.2 The Standard Scratch Experiment Shape Definitions

If you have requested other geometries during the production of your Nano Indenter® XP, contact Nano Instruments or your local representative for a definition of Shapes 1 through 4 on your instrument.

18.3.3 The Reduced Data

Upon completion of the Standard Scratch test, the data will be automatically reduced. Reduction of the scratch data includes the conversion of the raw data through the use of the calibrations. Refer to Appendix K for a list of the variables associated with the scratch option.

Once the raw data is converted, a profile can be generated. The profile is created by assuming that the regions associated with the Pre-scratch scan and Post-scratch scan are unaffected by the deformation achieved during the scratch itself, at least over the majority of their lengths. The data from this unaffected region is used to generate the slope of the sample for the Initial profile, Scratch, and Final Profile segments. The slope of the sample is then removed so that the entire scratch can be viewed with the surface of the sample as the baseline for deformation.

In addition, the lateral deflection data acquired during the Scratch itself is used to generate coefficient of friction information.

The output of the Standard Scratch test is thus best represented as a plot. Two plots will be generated for each scratch. The first plot shows the profile data, and the second shows the coefficient of friction response for the scratch segment only.

Refer to Appendix G for representative plots and result files.

18.4 Understanding The Scratch Procedure

The nature of the indentation system used with the Nano Indenter® XP encourages the treatment of scratches as lines drawn between indentation sites. Constant forces can be applied during these "draw segments", or the load can be ramped as the scratch is performed. In addition, the speed and acceleration of the draw segment can be modified.

18.4.1 Varying The Scratch Experiment Pattern

The Standard Scratch experiment is described above. This pattern must be maintained if the Profile Manipulation is to be performed (see Section 18.8 for instructions on manually manipulating the scratch data). It is possible, however, to perform different patterns of scratches. Reference J.6 in Appendix J illustrates a different type of scratch designed as a wear test.

The wear test scratch experiment is a good example of the use of the draw segment to profile across another scratch. The flexibility allowed by the method of defining scratch shapes allows you to set up reciprocating scratches or more complicated scratch patterns that scan across a feature over a large distance of the sample surface (see Section 18.4.2).

In the case of scratches such as these, the Profile Manipulation routine is not capable of anticipating the pattern of scratches in such a way that the surface levelling could be performed.

It is important to note that you will not be able to construct custom scratch experiments unless your instrument is equipped with the Enhanced Software Option, which includes the User Defined Experiment Definition Utility. If your

Nano Indenter® XP includes this option, refer to Appendix D for more information on custom scratch design.

18.4.2 Shapes And Draw Segments.

The ability to define different scratch experiments will only be useful if your Nano Indenter® XP is equipped with the User Defined Experiment Definition Utility (see Appendix D).

If you do have this additional software option, you can design scratch experiments however you like. It is first necessary, however, to understand how shapes are used in conjunction with draw segments to determine the scratch vector.

18.4.2.1 Drawing The Scratch Shape

The scratch shape is determined by defining the start and end points of the scratch. This is typically done by defining and saving an array of indents that represents the scratch shape, as illustrated in Figure 18.1.

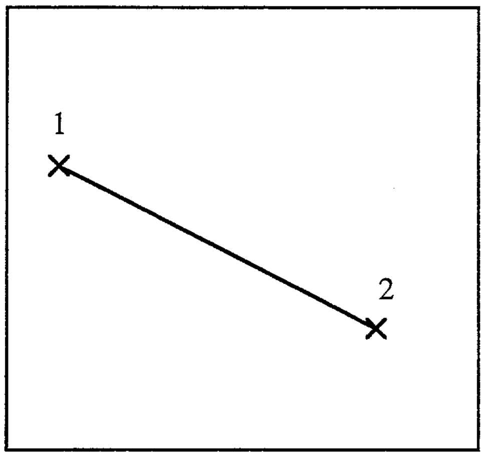


Figure 18.1 A Scratch as a Line Between Two Indent Sites

The scratch shape does not have to be limited to a line between two endpoints. A scratch might have a complicated shape, such as a line drawn between the multiple points of an array.

18.4.2.2 The Relation Between Scratches And Indent Sites

When the scratch shape has been defined, it can be used in a Draw Segment as a part of a Scratch Experiment. When the sites for the experiment are selected during test set-up (see Sections 8.2.6 and 8.2.7), the scratches begin from the indent sites, as shown in Figure 18.2.

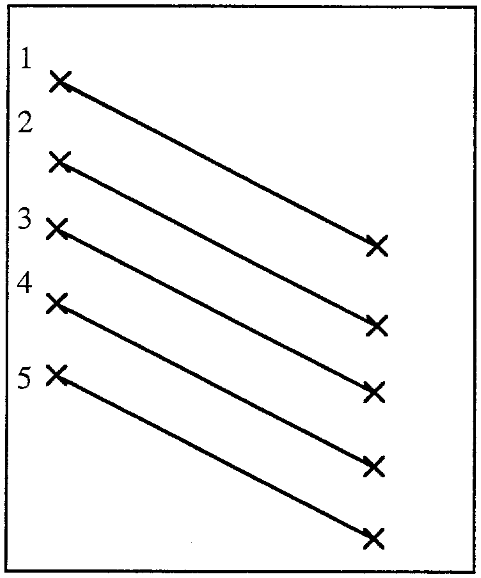


Figure 18.2 Five Scratches Starting at the Indent Sites in a 1x5 Array

It is important when defining scratch start points are selected to be aware of the direction and length of the scratches, so as to avoid overlapping previous deformed areas, or scratching over the initial surface find (see Section 13.2.9.4).

18.5 Diamond Tips And Scratch Testing

The standard tip used in scratch testing on the Nano Indenter® XP is the same Berkovich geometry used for indentation. There are two primary concerns related to the diamond tip: tip wear and tip orientation.

18.5.1 Diamond Tip Wear

The sliding of the diamond tip against the sample during a scratch results in a rapid wear of the diamond tip. Thus is it important that a calibrated diamond tip used for indentation should not be used for scratching. Performing scratches with such a tip will invalidate the area function. In addition, as a diamond tip becomes blunter, the tip of the diamond begins to resemble a sphere rather than a sharp corner. Thus the behavior of the diamond tip is altered, especially at very shallow depths.

For these reasons, it is recommended that a diamond tip be set aside as a scratch tip, and that you change tips between indentation and scratching modes.

18.5.2 Diamond Tip Orientation

Another issue in scratch testing is the orientation of a three sided Berkovich tip relative to the direction of scratching. If the scratch tip is an asymmetric geometry (i.e., a Berkovich diamond) then the alignment of the scratch tip becomes important. As the indentation shape of the Berkovich diamond is that of a triangle, you can obtain different results during scratches depending on whether your diamond is aligned "point-on" or "face-on" to the direction of the scratch (see Figure 18.3).

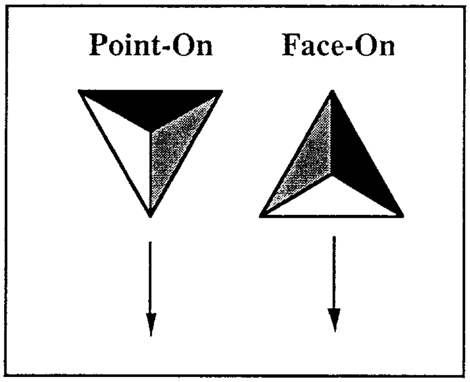


Figure 18.3 Point-On vs. Face-On Scratching

To determine the diamond tip's current orientation, run an indent experiment in a soft material (i.e., Aluminum) with load or displacement great enough to leave a large indentation. One point of the diamond will be oriented in either the positive Y or negative Y direction.

By convention, the Standard Scratch test will be performed in the positive Y direction from the selected start point. While it is possible to perform scratches in the X direction, or at an XY vector, is generally preferred to scratch in the Y direction only due to the diamond tip orientation.

18.6 Scratch Tests And Continuous Stiffness Measurement

If your system is equipped with both the Continuous Stiffness Measurement and Lateral Force Measurement options, then the Continuous Stiffness Measurement option will be active during scratching. Currently there is no support for utilizing or analyzing the Continuous Stiffness Measurement data from a scratch test.

18.7 Scratch Tests And The High Force Option

As the High Force option is transparent to the user, there should be no concern over its interaction with the Lateral Force Measurement option. Generally there should be no need to scratch at loads requiring the High Force option.

18.8 Manipulating Scratch Data

As described above, performance of the Standard Scratch test will generate the profile and coefficient of friction data automatically upon completion. If you have defined a different scratch using the User Defined Experiment Definition Utility, however, it will be necessary to manually manipulate the scratch data.

Regardless of whether your Nano Indenter® XP is equipped with the User Defined Experiment Definition Utility, the capability is in place to manually manipulated scratch data.

These manipulation routines are accessed from the Review Data Menu (see Section 9.4). All of the scratch routines are accessed from the menu option “Scratch Data: User Defined Calculations”.

18.8.1 Scratch Data: User Defined Calculations

When this option is selected, the XP Scratch Data Menu will be displayed:

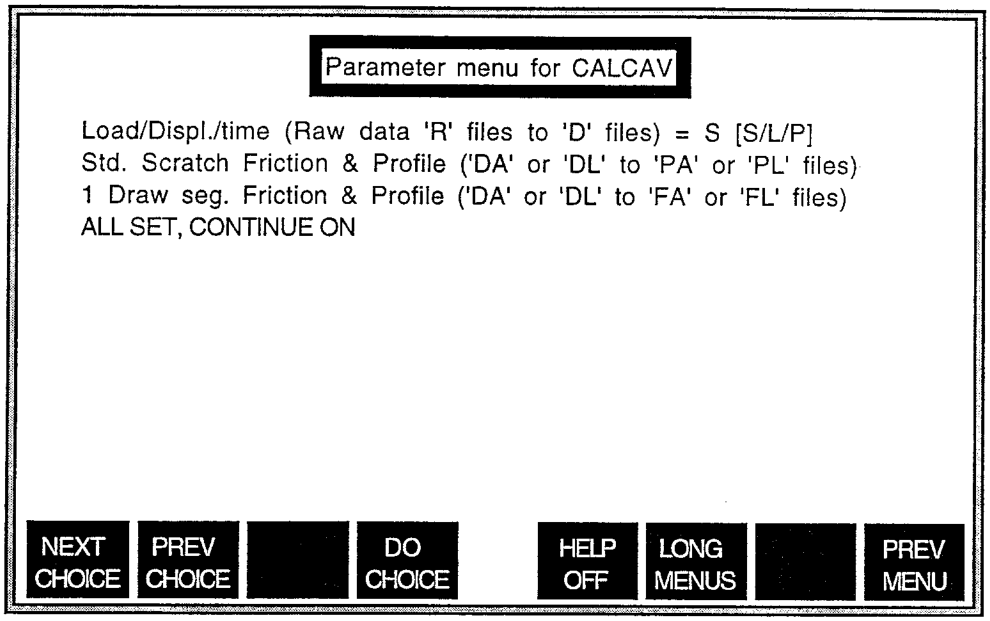


Figure 18.4 The Scratch Data User Defined Calculation Menu

18.8.1.1 Load/Displacement/Time

This option behaves as described in Section 9.4.7.1.

18.8.1.2 Standard Scratch Friction & Profile

When a scratch experiment is designed correctly, the slope of the sample surface can be determined and eliminated from the data, thus "leveling" the data and showing only that displacement into the sample as opposed to the total displacement. The routine also calculates the contact friction from the draw segment data. The file manipulation menu appears at the beginning of routine (see Section 9.2) and the acceptable data files are limited to those prefixed "DL" or "DA" (converted data from tests including a draw segment).

The routine will only operate with a certain type of scratch experiment, which must have the form:

Acceptable Profile Experiment		
Segment	**Segment Code**	**Purpose**
1	A	Approach
2	LX	Profile Loading Segment
3	D	First Pass, Initial Profile
4	D	Second Pass, Initial Profile
5	D	Pre-Scratch Scan
6	LX	Scratch Segment Preload
7	D	Scratch Segment
8	UX	Unloading Segment
9	D	Post-Scratch Scan
10	D	First Pass, Final Profile
11	D	Second Pass, Final Profile
12	UL	Complete Unloading

Table 18.3 Acceptable Scratch Experiment Design

For an explanation of the various segments referenced in Table 18.3, refer to Section 18.3.2.4 above.

Once the appropriate files containing this scratch data have been selected, you are taken to the Standard Scratch Friction & Profile menu:

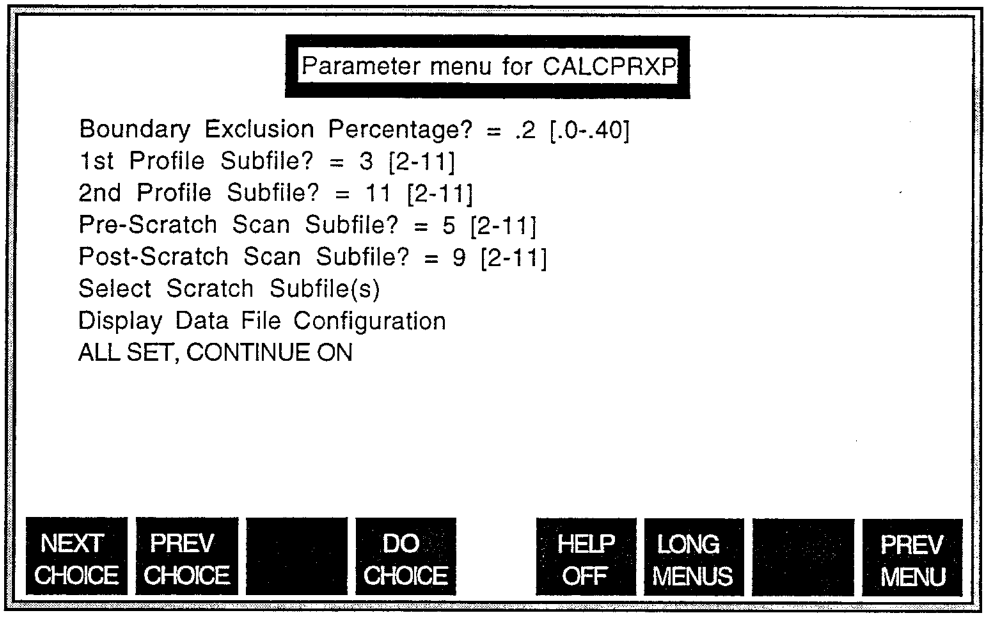


Figure 18.5 The Standard Scratch Friction & Profile Menu

18.8.1.2.1 Boundary Exclusion Percentage

The boundary exclusion zone is a small percentage (maximum 40%) at the beginning and end points of the pre and post-scratch scan segments. Ideally, the transition at the beginning and end of each scratch segment is smooth and provides an accurate measurement of the surface of the sample. Because of tip deflection during direction changes, however, data can be recorded that does not reflect the actual displacements produced by the surface of the specimen. Thus, you are given the opportunity to eliminate the data at the beginning or end of the pre and post-scratch scans. The default percentage (20%) is a good choice for an exclusion zone.

This boundary exclusion percentage is also applied during the leveling of the initial and final profile scans. That is, leveling data is taken only from the regions in the initial and final profile scans that corresponds to the pre and post-scratch scans. The boundary exclusion zone is applied in the same way for all of these segments.

18.8.1.2.2 1st Profile Subfile

As described in Section 18.3.2.4 above, the 1st Profile Subfile corresponds to the first segment of the Initial Profile. You should always choose the Initial Profile segment during which the indenter is moving in the same direction as the Scratch segment.

18.8.1.2.3 2nd Profile Subfile

As described in Section 18.3.2.4 above, the 2nd Profile Subfile corresponds to the first segment of the Final Profile. You should always choose the Final Profile segment during which the indenter is moving in the same direction as the Scratch segment.

18.8.1.2.4 Pre-Scratch Scan Subfile

As described in Section 18.3.2.4 above, this subfile is the short (usually about 10% of the total scratch length) scratch which provides part of the surface slope that will be used to level the actual ramping load scratch.

18.8.1.2.5 Post-Scratch Scan Subfile

As described in Section 18.3.2.4 above, this subfile is performed at the same load as the pre-scratch scan, and covers the same smaller length as a percentage of the total scratch length. Used with the pre-scratch scan, it provides the surface slope for leveling the ramping load scratch.

18.8.1.2.6 Select Scratch Subfile(s)

As described in Section 18.3.2.4 above, the scratch subfile is the actual ramping load segment, or other scratch experiment, during which penetration into the surface of the specimen is measured. It is possible to select more than one scratch subfile, but in most cases there will only be one segment to be selected.

18.8.1.2.7 Display Data File Configuration

This option behaves as described in Section 9.4.7.1.3. Refer to Appendix K for more information on the variables used in the Lateral Force Measurement option.

18.8.1.2.8 ALL SET, CONTINUE ON

Once the necessary information has been entered, ALL SET, CONTINUE ON will execute the calculation routines.

The software uses the ends of the scratch to calculate the slope of the sample surface. That is, the beginning and end of the total scratch length, where the ramp load is not applied, are used to provide the slope of the sample.

In addition, the slope is calculated independently for the initial profile, scratch, and final profile. Thus, there are three slopes calculated. The first slope is determined from beginning and end of the initial 1st Profile scan, the second slope is determined from the pre and post-scratch scans, and the third slope is determined from the 2nd Profile scan. These slopes are used to level only the scratches in the same direction. That is, the first slope is used to level the 1st Profile scan, the second slope is used to level the pre-scratch, scratch, and post-scratch segments, and the third slope is used to level the 2nd Profile scan.

With the leveling data subtracted from the draw segments, the remaining data should represent the displacement relative to the sample surface. Thus, displacements during the ramping load scratch provide actual depths into the sample, and can easily be compared with film thickness, etc.

One other operation of note is the vector calculation performed on the coefficient of friction and the actual X and Y positions. The software calculates a position vector (magnitude and angle relative to +X axis) from the X and Y position data. The lateral forces in the X and Y directions are resolved into forces in the scratch direction perpendicular to the scratch direction. The values for load and lateral force in the scratch direction are used to calculate a value for the coefficient of friction. Thus, scratches performed at angles relative to the X axis can be treated based on the length of the scratch rather than X and Y components of the vector.

The final data appears in the output file PXXXXX.NNN The prefix of this file will either appear as "PL" or "PA" depending on whether "DL" or "DA" data is entered. Thus, if DL01E001.BIN through DL01E010.BIN are selected for calculation, the output files will appear as PL01E001.BIN through PL01E010.BIN.

These final results can then be plotted or printed to provide the profile, vector position, lateral force in the vector direction and perpendicular to the vector, or calculated coefficient of friction.

18.8.1.3 1 Draw Segment Friction & Profile

As of the writing of these instructions, this routine was not yet activated.

18.8.1.4 ALL DONE, CONTINUE ON

Selecting this option will cause the software to return to the Review Data menu.

19.0 The High Force Option

19.1 Overview

The High Force Option extends the load capability of the Nano Indenter® XP well into the microhardness range. When this option is in place, you can set the maximum load up to 1 kg. Implementation of this method is transparent; there are no alterations in the user interface in terms of setting up tests or manipulating resultant data.

20.0 The Dynamic Contact Module

20.1 Overview

The Dynamic Contact Module (DCM) adds additional dynamic capability and performance to the Nano Indenter® XP system. Section 1.2 of these operating instructions describes the Nano Indenter® XP system as a collection of "subsystems." The DCM adds another subsystem to the Nano Indenter® XP, which can be interchanged with the indenter head when the DCM's functionality is required.

20.1.1 CAUTIONARY NOTES ABOUT THE DCM

The DCM was designed with optimization of performance as the primary objective. One of the primary requirements for improved dynamic performance was the reduction of the mass of the system. Coupled with the overall miniaturization of the system, this requirement resulted in very small, and very lightweight structural components. Thus, the system is not as robust as the Nano Indenter® XP standard indenter head. Care should be taken while working with the head. Be especially careful not to strike the DCM indenter tip laterally.

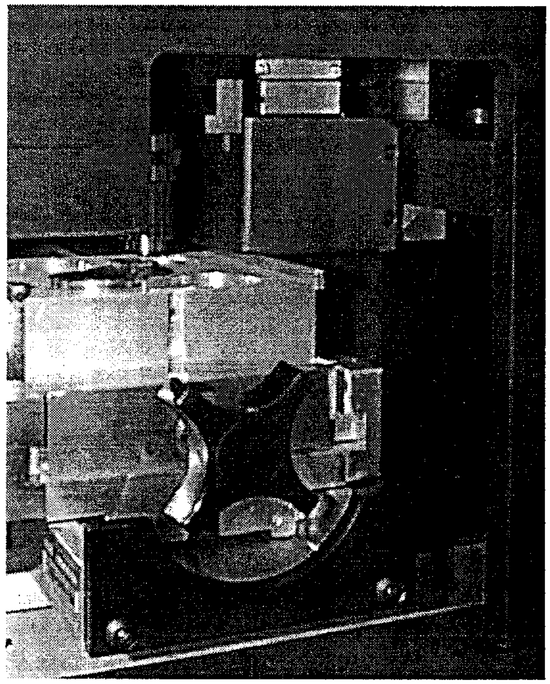


Figure 20.1 The DCM Mounted In The Gantry

20.2 Installing the DCM

The DCM Installation procedure will only be required when you are first installing the DCM, or if you are removing the DCM for a tip change operation (see Section 20.9 below).

The DCM is mounted on the right hand side of the Nano Indenter® XP gantry. The DCM mounts so that the diamond tip is in line with the Nano Indenter® XP head's diamond tip and the microscope focal point. The DCM is held in place by a single screw that can be accessed through the right side panel of the Nano Indenter® XP gantry. The electronic connection to the DCM is made through a single multi-pin connector that is suspended within the Nano Indenter® XP gantry.

20.2.1 Remove Sample Tray

Use the Load Specimen/Manual Control routine to move the sample tray to the sample loading position, and remove the sample tray from the Nano Indenter® XP. Then move the XY stages to provide maximum clearance under the microscope and DCM mounting area.

20.2.2 Shut off Power to the Instrument

Shut off power to the instrument, either by shutting off the power strip that powers the entire system, or by shutting off power at the PC.

20.2.3 Remove Microscope Objective

Remove the microscope objective to provide clearance to the left of the DCM mounting area.

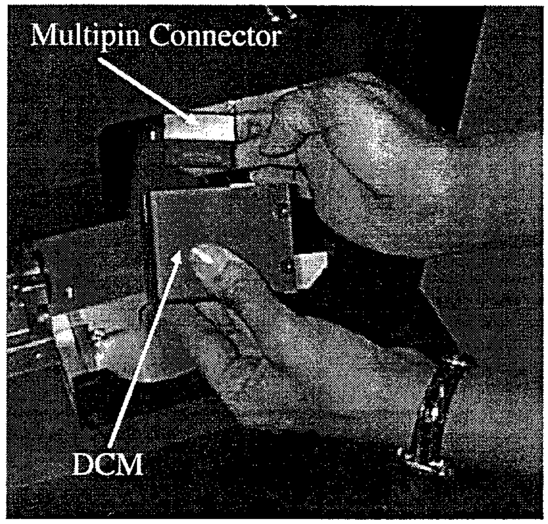


Figure 20.2 Attaching the Multipin Connector to the DCM

20.2.4 Attach Multipin Connector to DCM

Attach the DCM cable that is suspended inside the Nano Indenter® XP gantry to the DCM connector socket on the DCM itself (see Figure 20.2).

BE EXTREMELY CAREFUL WHILE EXECUTING THIS PROCEDURE THAT YOU DO NOT TOUCH OR PUSH AGAINST THE EXPOSED DCM TIP!

20.2.5 Insert DCM into the Gantry

The Nano Indenter® XP adapter on the right hand side of the DCM slides upward and to the left into the mating adapter piece inside the Nano Indenter® XP gantry (see Figure 20.3). To fit the DCM onto this adapter piece, it will be necessary to move the microscope upward while simultaneously moving the DCM into position.

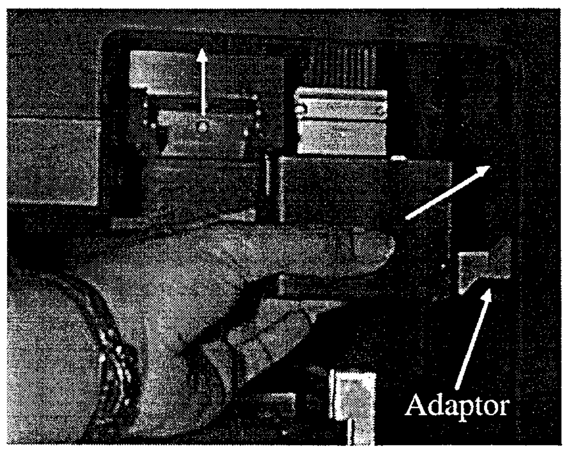


Figure 20.3 Inserting the DCM into the Gantry Adaptor

BE EXTREMELY CAREFUL WHILE EXECUTING THIS PROCEDURE THAT YOU DO NOT TOUCH OR PUSH AGAINST THE EXPOSED DCM TIP!

20.2.6 Lock DCM to the Gantry

Lock the DCM in place in the Nano Indenter® XP gantry by inserting a Metric 3mm allen wrench through the access hole on the right side panel of the gantry (see Figure 20.4). This opening in the gantry provides access to the M4 socket head cap screw that locks the DCM/Nano Indenter® XP adapter in place. Once you have fully tightened this screw in place, the DCM is fully attached to the Nano Indenter® XP gantry.

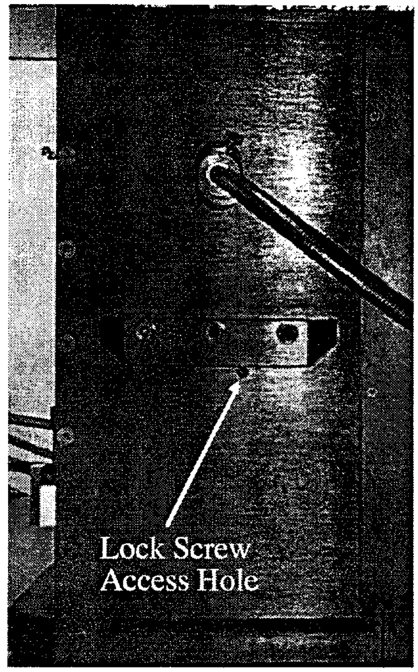


Figure 20.4 The DCM Locking Screw

20.3 Switching From the Nano Indenter® XP Indenter Head to the DCM

Once the DCM is physically installed into the Nano Indenter® XP gantry, it is available for use. It is first necessary, however, to switch functionality between the standard Nano Indenter® XP indenter head and the DCM.

20.3.1 Lock the Nano Indenter® XP Indenter Head

Use the procedure for locking the standard indenter head, as described in Appendix A of the Nano Indenter® XP Operating Instructions.

20.3.1.1 CAUTIONARY NOTES

During normal operation of the Nano Indenter® XP without the DCM engaged, the power supplied to the standard indenter head is used to raise the indenter head out of the plane of the sample tray. When the DCM is active, there is no power supplied to the standard indenter head. Thus it is crucial that the standard indenter head be locked in its retracted position. If it is not, either the diamond tip or the standard indenter head itself can be damaged.

20.3.2 Shut the Instrument Power Off

Once the indenter head is locked in place, shut off power to the instrument.

20.3.3 Switch the Head Cable

The head cable is attached to the upper left side connector panel on the Nano Indenter® XP gantry (see Figure F.4 in Appendix F of the Nano Indenter® XP Operating Instructions). The lower position is used for the standard indenter head. The upper position is used for the DCM mode. Unscrew and unplug the head cable from the lower position and move it to the upper position to enable the DCM (see Figure 20.5 below).

20.3.4 Turn the Instrument Power On

Once the head cable has been switched, restore power to the instrument.

20.3.5 Verify DCM Operation

Enter the software as described in Section 20.4 (below) and verify the operation of the DCM as described in Section 20.5 (below). Once you are certain that the DCM is connected properly and is functioning, perform a DAC calibration (see Section 11.2.2). If you are also using the Continuous Stiffness Measurement Option, you should also perform a Configuration of the frequency of oscillation (see Section 20.3.6 below).

Once the DAC calibration has been performed, the DCM should be ready for operation.

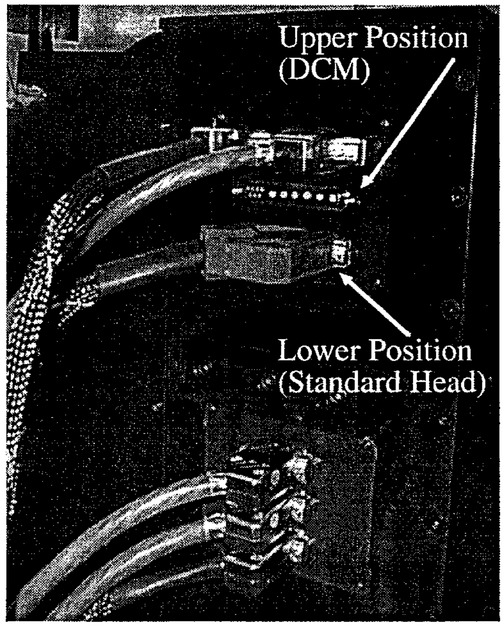


Figure 20.5 The Head Cable Positions

20.3.6 Reconfigure The Operating Frequency

If your Nano Indenter® XP includes the Continuous Stiffness Measurement Option, you should perform a Configuration of the frequency of oscillation. As the Nano Indenter® XP standard indenter head and the DCM have very different dynamic characteristics, the normal frequency of oscillation is very different for the two heads. Thus, when you switch between the standard indenter head and the DCM, the phase lock amplifier that provides functionality to the Continuous Stiffness Measurement option must be reconfigured for the appropriate operating frequency.

The standard frequency for the DCM is 120Hz, although it is generally advisable to use the default frequency that appears on the screen when you enter the Configure Step. This default frequency is the last frequency set, and unless this has been changed after installation of the Nano Indenter® XP at your site, this is the frequency used during production of the instrument. As the Dynamic Load Frame Stiffness (LFS) is frequency dependent, you should not change the frequency away from this default value unless you plan to redetermine the Dynamic LFS.

For detailed instructions on how to reconfigure the frequency of the phase lock amplifier oscillation, refer to Section 17.3 of the Nano Indenter® XP Operating Instructions.

20.4 DCM/Software Interaction

The most salient difference in the operation of the Nano Indenter® XP software with the DCM installed is the initial prompt that requests input as to the system type. This prompt appears whenever the Nano Indenter® XP software is started:

```
What is the head type [ 1 char from 12]?

1) Standard XP Head

2) DCM Head

?
1
```

If the DCM head is currently the active head (see Section 20.3 above), then you should enter “2” to indicate this.

Other software changes include the motor status and exercise motor functions, as described in Section 20.5.2 below.

20.5 Verifying DCM Operation

Once the DCM is physically installed into the Nano Indenter® XP gantry, it is available for use. It is first necessary, however, to switch functionality between the standard Nano Indenter® XP indenter head and the DCM.

20.5.1 Test Motion / Indenter Performance

The Test Motion of Indenter routine operates for the DCM in the same manner as the standard indentation head (see Section 11.2.3). There are a couple of differences between the behavior of the DCM and the standard indenter head that are worth describing.

20.5.1.1 Range of Motion

The typical DCM will not be capable of the full range of motion that the Nano Indenter® XP standard indenter head can achieve. Typical DCM displacement voltage ranges are ±1.5V of displacement in displacement range 0, although some DCM units have ranges as large as ±3V. It is worth noting that the range of motion for the DCM affects the damping and the displacement resolution. The calibrations for the DCM account for these differences.

20.5.1.2 Maximum Load

While the Nano Indenter® XP standard indenter head can achieve loads on the order of 60g, the DCM is only capable of achieving loads of 1g to 1.5g.

20.5.2 The DCM Motion System

The Nano Indenter® XP uses the full 2mm range of the standard indentation head to accomplish all Z-direction motion. Because the DCM only has a range of motion of 100µm, an additional Z-motion module is built into the DCM unit to achieve larger Z-motion moves, such as the initial surface contact or backing the head away to a "safe clearance."

20.5.2.1 Principles of the Motion System

The motion system is built into the DCM outer housing, and consists of a high precision gearmotor, springs, and bearings. This motion system is activated during all "head repositioning" operations; the surface contact routines that occur before each array of indentations (see Section 13.2.9.4). The motion system has physical limits that cause the motion system to stall at the extremity of its travel, which is a little more than 0.15" (about 4mm). In addition, limit switches are built into the motion system. These limit switches activate before the motion system reaches either end of its travel. Note that the limit swtiches are not active during direct command moves (see Section 20.8.4 below).

20.5.2.2 Accessing the Function Keys

In most cases, the motion system operates by direct command of the software, requiring no user input. If you want to test the motion system, or require information about the status of the motion system, this information and control is available through the Test Motion of Indenter routine (see Section 11.2.3). When your system is equipped with the DCM, additional functions are added to the Test Motion routine. By pressing the "Other Keys" function key twice in the Test Motion routine, a set of motion control keys are displayed. The useful keys are the Motor Status key (f1) and the Exercise Motor key (f2).

20.5.2.3 Motor Status

Pressing the Motor Status key will display a binary code that represents various states of the motor. The bits correspond to:

Motor Status Bits		
Bit	**0 Condition**	**1 Condition**
0	Motor Up	Motor Down
1	Slow Speed	Fast Speed
2	Motor Stopped	Motor Running
3	Limit Switch On	Limit Switch Off
4	Fast Latch Off	Fast Latch On
5	Total Latch Off	Total Latch On
6	Fast Lockout Off	Fast Lockout On
7	Total Lockout Off	Total Lockout On

Table 20.1. Motor Status Bits

20.5.2.4 Exercise Motor

The Exercise Motor function commands the motor to begin moving downward until it hits a limit switch, then to reverse direction and move upward until it hits a limit switch, and then to continue to loop. The function will continue to run until either the power to the instrument is cut off, or until the software is restarted and the communication links are re-established.

The Exercise Motor function will not allow the motor to start if either the Fast or Total Lockouts are set on (see Section 20.5.2.3 above).

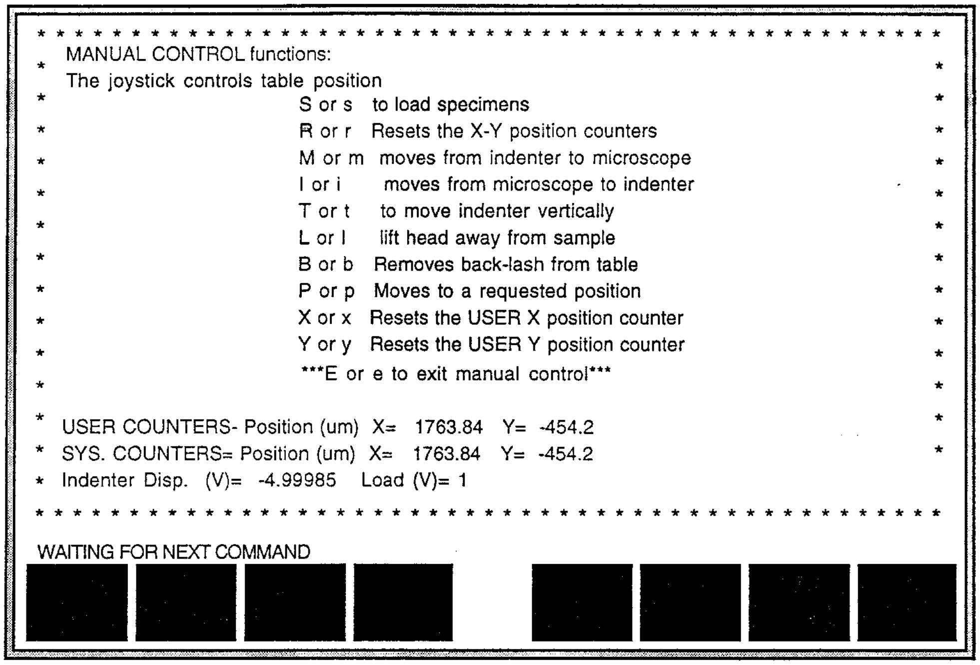
Once you press the Exercise Motor key, you will be prompted:

```
What speed (F/S/Q) (enter Q to quit) ?
```

Entering “F” will cause the motor to move in “Fast speed,” entering “S” will cause the motor to move in “Slow speed,” and entering “Q” will abort the exercise motor command.

20.5.3 Manual Control of the DCM Motion System

The DCM motion system operates on principles similar to the focus motor in the Nano Indenter® XP optic system (see Section 4.2). Rather than providing direct control of the DCM motor via the joystick, as is done with the Nano Indenter® XP optic system, a single command has been added to the Load Specimen/Manual Control screen (see Figure 20.6).



```
* * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * *
*  MANUAL CONTROL functions:                                                                          *
   The joystick controls table position
*                                S or s   to load specimens                                           *
*                                R or r   Resets the X-Y position counters                            *
*                                M or m   moves from indenter to microscope                           *
*                                I or i    moves from microscope to indenter                          *
*                                T or t   to move indenter vertically                                 *
*                                L or l   lift head away from sample                                  *
*                                B or b   Removes back-lash from table                                *
*                                P or p   Moves to a requested position                               *
*                                X or x   Resets the USER X position counter                          *
*                                Y or y   Resets the USER Y position counter                          *
*                                ***E or e to exit manual control***                                  *

*  USER COUNTERS- Position (um) X=  1763.84  Y= -454.2                                                *
*  SYS. COUNTERS= Position (um) X=  1763.84  Y= -454.2                                                *
*  Indenter Disp.  (V)=  -4.99985   Load (V)= 1
* * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * * *
WAITING FOR NEXT COMMAND
```

Figure 20.6 The Load Specimen/Manual Control Screen

Pressing "L" or "l" on the keyboard will cause the "Lift Head" command to activate. Note that this command is only useful if the DCM is the active head.

When the Lift Head command is issued, the software will command the DCM motor to raise the head away from the sample for 20 seconds, providing adequate distance between the DCM head and the sample.

Note that you should only issue this command once, as the DCM motion system limit switches are not active while this function is operating. Thus, it is possible to cause the DCM motion system to get "stuck" in its upper position by issuing multiple Lift Head commands.

20.6 DCM/CSM Interaction

The DCM is optimized for performance when used with the Continuuous Stiffness Measurement (CSM) option. The DCM has a very low mass and very low damping. Thus, the range of frequencies at which the DCM can operate effectively is significantly different than that of the higher mass/higher damping Nano Indenter® XP.

20.6.1 The Configure Step

As described in Section 17.3.1 and Section 20.3.6 above, the Configure Step of the Dynamic Calibrations is most useful in setting the frequency of oscillation for the indenter head. With the DCM in place on the Nano Indenter® XP system, frequencies up to 130Hz can be used. During production of the instrument, the most appropriate frequency is chosen, based on the performance of the instrument. Do not change the frequency from the preset value unless you are sure you know what you are doing.

20.6.2 Dynamic Calibration

Contact Nano Instruments if you are interested in performing a full dynamic calibration with the DCM.

20.7 Performing Indentations with the DCM

The DCM operates in exactly the same manner as the Nano Indenter® XP indentation head. There are two notable exceptions.

20.7.1 The Accessible Sample Area

The position of the DCM in the Nano Indenter® XP gantry results in an accessible indentation area on the sample tray that is significantly smaller than the area that can be reached by the Nano Indenter® XP standard indenter head. Roughly speaking, the DCM can reach about half of the area of the two rightmost sample disks.

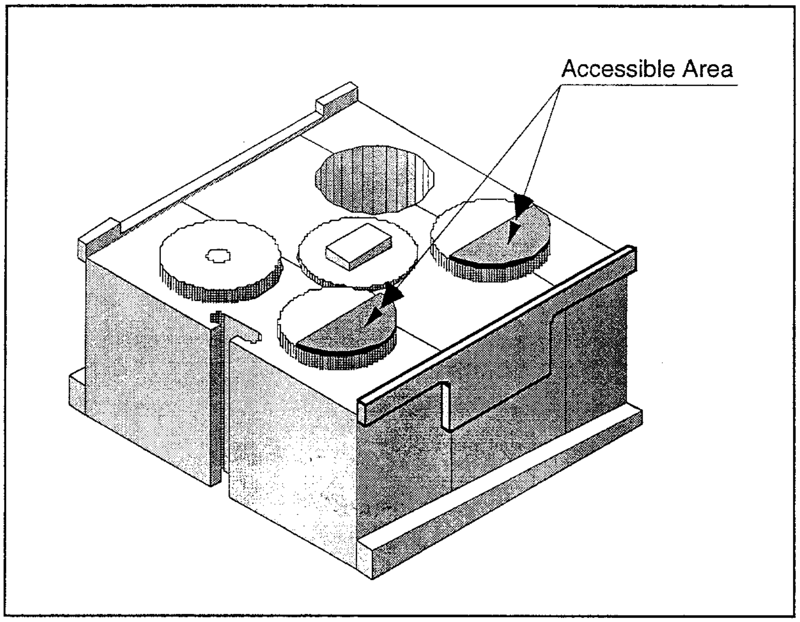


Figure 20.7 The accessible area for the DCM

20.7.2 The Indenter Range

The DCM maximum load and maximum displacement capabilities are significantly less than those of the Nano Indenter® XP indentation head. The maximum suggested load for the DCM head is 1g (10mN), while the maximum displacement is on the order of 20μm. While the DCM can actually achieve greater loads and greater displacements, these are good limits for performance criterea.

20.8 Recovering from an Indentation Crash

If the DCM tip is stuck in contact with the sample due to a software, computer, or power failure, a specific procedure must be followed to remove the tip from contact and restore functionality to the system. It is critical that you do not move the X-Y tables until you have removed the DCM from contact with the sample. If you move the X-Y tables while the DCM tip is in contact, you will destroy the DCM unit.

The following procedure describes the steps to be taken to remove the DCM from contact with the sample.

20.8.1 Restart the Software

If you have not already done so, restart the software either by shutting off power to the instrument and then turning the power back on, or by performing a BASIC-RESET operation (pressing the CONTROL and RESET keys together, and then typing "RUN" and pressing ENTER). Once the software comes up, select the DCM head, and proceed to the calibrations menu.

20.8.2 Enter Manual Control

Enter the Load Specimen/Manual Control routine, and execute the Lift Head command as described in Section 20.5.3 above.

20.8.3 Move Back to the Microscope

Once you are sure that the DCM is out of contact with the sample, and that it will clear the sample tray arms, use the Indenter to Microscope function to move back under the microscope (see Section 10.3.4). Watch the sample as it moves to ensure that nothing comes close to hitting the DCM tip. If you think either the sample or the sample tray arm is going to hit the DCM tip, shut off power to the instrument immediately.

Once you have moved the sample back under the microscope, you should be ready to proceed with operating the instrument as usual. It is probably a good idea to perform a new microscope to indenter calibration (see Section 11.2.1) to ensure that the DCM tip will clear the sample, and that the system is operating normally.

20.9 Tip Change

Performing the tip change operation on the DCM is a very delicate procedure, requiring special tools that are not currently available for customer use. As a result, if a new tip is desired, the DCM must be sent back to Nano Instruments for the tip replacement. Contact Nano Instruments for more information about tip replacement.

20.10 Switching from the DCM to the Nano Indenter® XP Indenter Head

To switch back to the standard Nano Indenter® XP indenter head after using the DCM head, it is necessary to execute a few standard procedures in order to ensure that the Nano Indenter® XP indenter head is fully functional, and that the DCM head is safely secured.

20.2.1 Ensure that the DCM Tip is Fully Retracted

Examine the DCM to ensure that the indenter shaft is fully retracted inside the outer housing of the DCM unit. This operation should normally be automatically performed at the completion of a test or upon entering any routine that communicates with the electronics (Calibrations, Load Specimen/Manual Control, etc.).

If the DCM is not fully retracted, enter the Calibrations menu. At the start of this menu, the indenter head should be lifted to the limit of its vertical movement. If there are any problems, contact Nano Instruments for help.

20.10.2 Shut the Instrument Power Off

Once the DCM indenter shaft position is verified, shut off power to the instrument.

20.10.3 Switch the Head Cable

The head cable is attached to the upper left side connector panel on the Nano Indenter® XP gantry (see Figure F.4 in Appendix F of the Nano Indenter® XP Operating Instructions). The lower position is used for the standard indenter head. The upper position is used for the DCM mode. Unscrew and unplug the head cable from the upper position and move it to the lower position to enable the Nano Indenter® XP indenter head.

20.10.4 Turn the Instrument Power On

Once the head cable has been switched, restore power to the instrument.

20.10.5 Unlock the Nano Indenter® XP Indenter Head

Use the procedure for unlocking the standard indenter head, as described in Appendix A of the Nano Indenter® XP Operating Instructions.

20.10.5.1 CAUTIONARY NOTES

Ensure that the pins are out of the Nano Indenter® XP standard indenter head before attempting to run a test.

20.10.6 Verify Indenter Head Operation

Enter the software and verify the operation of the Nano Indenter® XP standard indenter head by performing a DAC calibration (see Section 11.2.2). If you are also using the Continuous Stiffness Measurement Option, you should also perform a Configuration of the frequency of oscillation (see Section 20.10.7 below).

Once the DAC calibration has been performed, the Nano Indenter® XP indenter head should be ready for operation.

20.10.7 Reconfigure the Operating Frequency

If your Nano Indenter® XP includes the Continuous Stiffness Measurement Option, you should perform a Configuration of the frequency of oscillation. As the Nano Indenter® XP standard indenter head and the DCM have very different dynamic characteristics, the normal frequency of oscillation is very different for the two heads. Thus, when you switch between the DCM and the standard indenter head, the phase lock amplifier that provides functionality to the Continuous Stiffness Measurement option must be reconfigured for the appropriate operating frequency.

The standard frequency for the Nano Indenter® XP standard indenter head is 45Hz, although it is generally advisable to use the default frequency that appears on the screen when you enter the Configure Step. This default frequency is the last frequency set, and unless this has been changed after installation of the Nano Indenter® XP at your site, this is the frequency used during production of the instrument. As the Dynamic Load Frame Stiffness (LFS) is frequency dependent, you should not change the frequency away from this default value unless you plan to redetermine the Dynamic LFS.

For detailed instructions on how to reconfigure the frequency of the phase lock amplifier oscillation, refer to Section 17.3 of the Nano Indenter® XP Operating Instructions.

Appendix A: Tip Change Routines

A.1 Overview

The tip change operation is used for installation or removal of the diamond tip, although part of the routine is also useful for locking the indenter shaft in place. Tip changing is performed when a diamond tip is worn, a different geometry is needed, or the tip needs to be cleaned.

A.2 Locking The Indenter Shaft

Before a tip changing operation can be performed, the indenter shaft must be locked in place to prevent possible damage to the indenter system when the tip is inserted or removed. The indenter shaft is locked in place with two pins that are inserted in the X and Y directions into the indenter's protective housing, and through the indenter shaft itself.

These pins are generally described as the upper and lower locking pins.

To lock the indenter shaft in place:

A.2.1 Enter Manual Control

Enter the Load Sample/Manual Control routine (see Section 10.0). Use the "Load Samples" manual control function to move the sample tray out from under the indenter shaft (Section 10.3.1). Once the sample tray is moved to the "loading position," you can begin the procedure to lock the indenter shaft. Do not press the "CONTINUE" button until you have completed the tip change operation.

A.2.2 Insert The Upper Pin

Locate the pin holes on the indenter's protective housing. Insert one of the pins into the upper pin hole. Push steadily and rotate the pin until you feel the pin penetrating the indenter shaft. There will be slight resistance to the motion of the pin. Continue to smoothly rotate and insert the pin through the indenter shaft until the pin has emerged from the opposite side of the indenter's protective housing. Do not abruptly insert the pin into the indenter housing.

A.2.3 Insert The Lower Pin

Locate the lower pin hole on the indenter's protective housing. Insert the second pin into the lower pin hole. Push steadily and rotate the pin until you feel the pin penetrating the indenter shaft. There will be slight resistance to the motion of the pin. Continue to smoothly rotate and insert the pin through the indenter shaft until the pin has emerged from the opposite side of the indenter's protective housing. Do not abruptly insert the pin into the indenter housing.

A.3 Removing The Indenter Tip

Once the indenter shaft is locked in place with the two pins, locate the tip change tool. The tip change tool has three segments: the base, the tip shaft, and the collar remover. The tip shaft is permanently inserted into the base, so that rotating the base rotates the tip shaft. The collar remover fits around the base. The base and the collar remover are knurled so that they can be easily rotated (see Figure A.1).

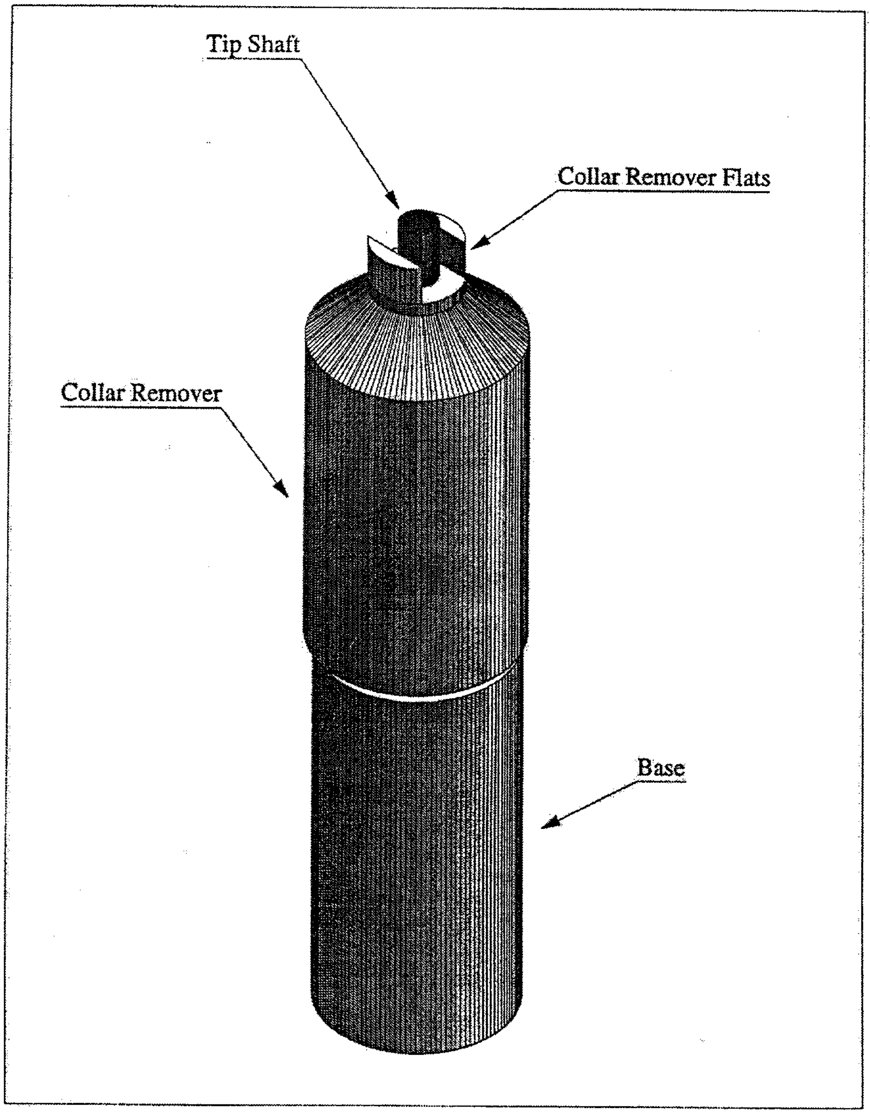


Figure A.1 The Tip Change Tool

A.3.1 Contacting the Indenter Tip

With the collar remover in its bottom-most position on the base, raise the tip change tool so that the tip shaft is moving toward the indenter tip (see Figure A.2).

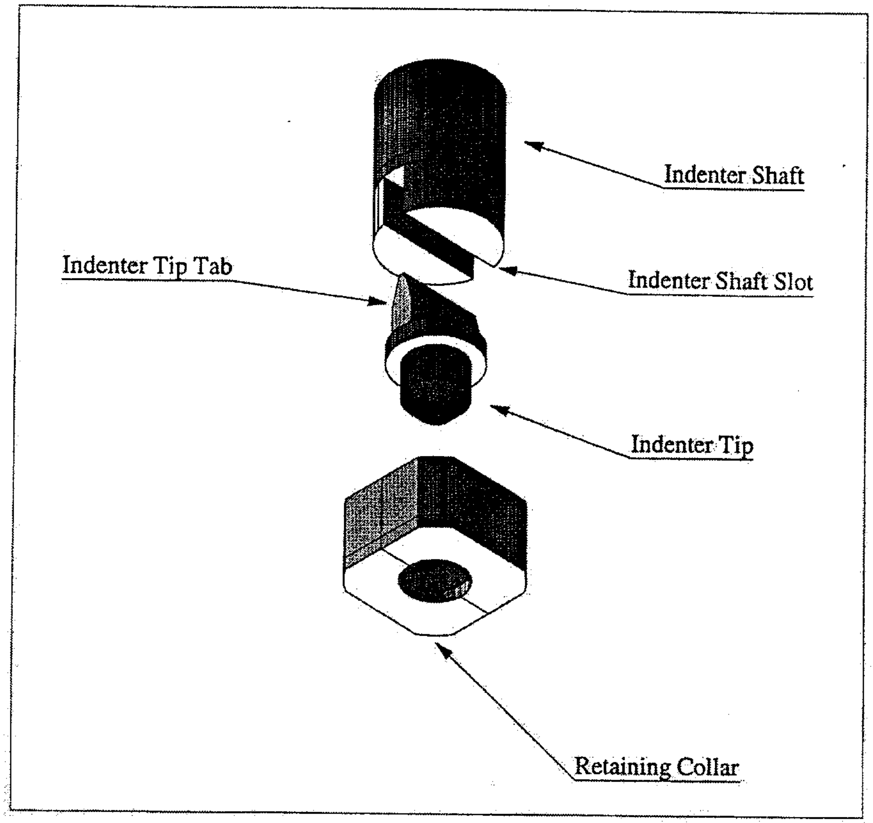


Figure A.2 The Tip Assembly

Be careful not to contact the diamond portion of the indenter tip with the tip shaft. Move the tip shaft upward until you have fully "captured" the indenter tip (see Figure A.3).

A.3.2 Loosening the Retaining Collar

Once you have captured the indenter tip with the tip shaft, lift the collar remover until the flats on the collar mover are aligned with the flats on the retaining collar on the indenter shaft. The retaining collar is held to the indenter shaft with standard right hand threads. Turn the collar remover counter-clockwise, until you have fully loosened the retaining collar.

A.3.3 Removing the Indenter Tip

Lower the tip change tool away from the indenter shaft. The indenter tip should remain in the tip shaft. If it does not, rotate the base very slightly while holding the collar remover in place. The tip should come out of contact with the indenter shaft.

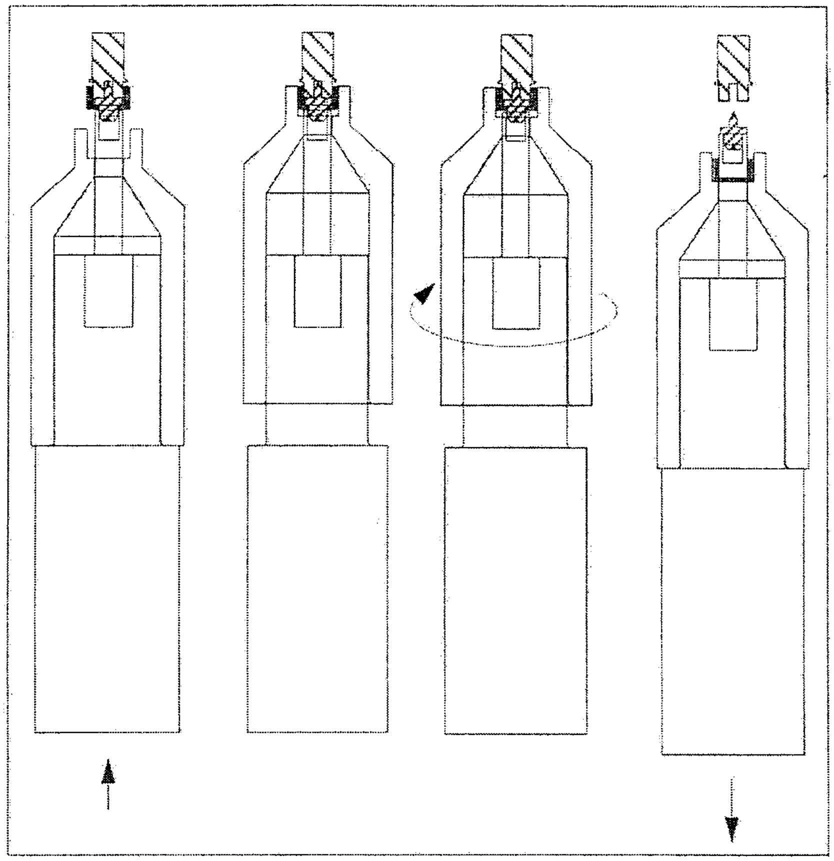


Figure A.3 The Tip Change Procedure

A.4 Inserting The Indenter Tip

Obviously, the procedure for inserting the indenter tip is very similar to the procedure for removing it. There is one additional step, however, which involves aligning the tab on the back of the indenter tip with the slot in the indenter shaft.

A.4.1 Inserting the Indenter Tip

Raise the tip change tool toward the indenter shaft, with the indenter tip in place on the tip shaft. Make very gentle contact with the indenter tip touching the indenter shaft. Rotate the base so that the indenter tip is rotated until the tab on the back of the indenter tip is aligned with the slot in the indenter shaft. When the alignment is done, press upward slightly until the indenter tip fits fully into the slot in the indenter shaft.

A.4.2 Tightening the Retaining Collar

Once the indenter tip is seated in the indenter shaft, gently raise the collar remover and rotate clockwise until the retaining collar begins to thread onto the indenter shaft. Continue to thread the retaining collar on until you feel resistance. Tighten the retaining collar until it is "snug" on the indenter shaft. Do not overtighten or undertighten. The indenter tip should not be loose.

A.4.3 Removing the Tip Change Tool.

Once you have tightened the retaining collar, lower the tip change tool away from the indenter shaft, being careful not to contact the diamond portion of the indenter tip.

A.5 Unlocking The Indenter Shaft

If you have inserted a diamond into the indenter shaft, you must unlock the indenter shaft before you can continue to use the indenter.

A.5.1 Remove the Lower Pin

Slide the lower pin out of the protective indenter shaft housing in the direction that you inserted it. Pull steadily on the pin until you feel the pin sliding out of the indenter shaft. There will be slight resistance to the motion of the pin. Continue to smoothly remove the pin from the indenter shaft until the pin is fully removed from the indenter's protective housing. Do not abruptly remove the pin from the indenter housing.

A.5.2 Remove the Upper Pin

Slide the upper pin out of the protective indenter shaft housing in the direction that you inserted it. Pull steadily on the pin until you feel the pin sliding out of the indenter shaft. There will be slight resistance to the motion of the pin. Continue to smoothly remove the pin from the indenter shaft until the pin is fully removed from the indenter's protective housing. Do not abruptly remove the pin from the indenter housing.

A.5.3 Enter Manual Control

The tip change operation should always be performed in the Load Specimen/Manual Control routine. When locking the indenter shaft in place, you will most likely first move the sample tray away from the indenter using the "Load Specimen" manual control function. The last step in the unlocking of the indenter shaft will then be to press the "CONTINUE" key to cause the sample tray to move back to its position under the indenter. Once this is done, you can exit manual control using the "Exit" key. See Section 10.0 for more information about manual control and the manual control functions.

A.6 Post-Tip Change Operations

Once a tip change has been performed you should always perform the following diagnostic / calibration procedures:

A.6.1 Run a DAC Calibration

The DAC calibration is an excellent diagnostic routine to verify that the indenter is working properly. In addition, changing the tip will certainly result in different DAC behavior, so for optimum operation of the indenter, the DAC calibration should always be performed following a tip change. See Section 11.2.2 for more information about this operation.

A.6.2 Perform a Microscope-To-Indenter Calibration

After a tip change, the microscope-to-indenter calibration distance will almost certainly change. Thus, before samples are run, it is important that this distance be redetermined, if accurate positioning of indents is to be achieved. See Section 11.2.1 for more information about this option.

A.6.3 Determine the New Tip's Area Function

If you are inserting a new diamond tip, you will most likely want to determine the load frame stiffness and area function for the new diamond. Refer to Appendix C for more information about these calibrations.

Appendix B: Step By Step Test Setup Instructions

B.1 Overview

The following instructions are provided as a quick reference to test setup using the "Perform Standard Test" method. Refer to Section 7.0 for more complete instructions.

B.2 Test Setup Instructions

B.2.1 Select "Load Specimen/Manual Control" from the Master Menu

See Section 10.0.

B.2.2 Press the "S" Key to Load Specimens

See Section 10.3.1.

B.2.3 Load the Sample Tray

See Section 3.3.1.

B.2.4 Press the CONTINUE key

B.2.5 Press the "E" key to Exit Manual Control

B.2.6 Select "Perform Standard Test" from the Master Menu

B.2.7 Press the CONTINUE Key

B.2.8 Move the Sample Under the Microscope Using the Joystick

See Section 10.2.

B.2.9 Focus on the Sample

See Section 10.2.2.

B.2.10 Use the Joystick to Select the Start Point for the Experiment

B.2.11 Press the "E" Key to Exit the Manual Control Screen

Appendix C: Diamond Tip Area Calibration

C.1 Overview

The majority of the Tip Area Calibration is performed automatically by the Nano Software, using a fused silica (Si02) sample (see Section 16.2.11). The last step, however, must be performed by the user.

C.2 Manipulating The Tip Area Calibration Data

The following procedure assumes that you have a copy of Kaleidagraph 3.0 installed on the PC, and that you have the Tip Calibration Templates for Kaleidagraph. If you do not have this software, contact Nano Instruments.

C.2.1 Identify The Output Data Filename

The output data filename will be of the format:

MTXXX001.TXT

where "XXX" is the run number. The run number is identified on the output data file sheets, printed upon completion of the tip calibration experiments.

C.2.2 Use "Return To Windows" To Exit the Nano Software

The "Return To Windows" command is located on the Master Menu of the Nano Software (see Section 2.7.2.3).

C.2.3 Open Kaleidagraph

The Kaleidagraph application should be located in the Program Manager screen in Windows (under the File Manager, the Kaleidagraph folder will be titled "KGRAPH").

C.2.4 Use Kaleidagraph To Open MTXXX001.TXT

The MTXXX001.TXT file resides in the directory:

C:\XPCODE\XPDATA\MOD\TXT

The data is tab delimited. Use the "Read Titles" option, and do not skip any rows of data.

C.2.5 Insert a Row at the Top of MTXXX001.TXT

Insert the row so that there is an empty row of data at the top of the MT file.

C.2.6 Enter "0" Into Each Cell In This New Row

There will be three columns of data. Enter "0" (zero) into each cell in the new empty row.

C.2.7 Use Kaleidagraph To Open the Template File

The Template File is named:

TIPCAL.qpc

C:\ > kgrph > template

and is ~~located on the floppy disk:~~

TIP CALIBRATION TEMPLATES

C.2.8 Select "Template" from the Gallery Menu

C.2.9 Use "Cont. D (nm)" for the X axis

C.2.10 Use "A (E) nm^2" for the Y axis

C.2.11 Wait For Curve Fit Completion

The curve fit may take some time to complete.

C.2.12 Save The Resultant Plot

Once the curve fit is complete, you can change the title or format of the resultant plot. If you choose to save this plot, you can then use this new plot as a template, if you desire to do so.

C.2.13 Copy the Coefficients or Print the Plot

When the curve fit is complete, the coefficients of the general curve fit function will be displayed on the plot.

C.2.14 Exit Kaliedagraph

C.2.15 Use the Nano Icon to Return To Nano Software

C.2.16 Enter The Coefficients Into The Calibration Data Array

Proceed to the menu "~~Recalibrate~~ Calibration" and open the option "Print Or Edit Diamond Area Function Coefficients" from the "Long Menu" of options. Enter the appropriate diamond number (see Section 16.2.11). Use the "Long Menu" option to display the coefficients for this diamond. Enter the new coefficients from the plot, making sure that you enter the absolute value of the coefficients (do not enter the numbers as negative).

C.2.17 Locate The Output Data Sheet

At the completion of the tip calibration experiment, data sheets are printed. These data sheets contain the load frame stiffness value used to calculate the diamond area function. This load frame stiffness value should also be entered into the calibration data array.

C.2.18 Enter The Load Frame Stiffness Calibration Data

Calibration

Proceed to the menu “~~Recalibrate~~” and open the option “Print Or Edit Calibration Values” from the “Long Menu” of options. Select the “Base System” calibrations. Enter the new Load Frame Stiffness value.

C.3 The Tip Calibration Procedure

For more information on the procedure used to calibrate the diamond tip, refer to “An Improved Technique For Determining Hardness And Elastic Modulus Using Load And Displacement Sensing Indentation Experiments,” W.C. Oliver, G.M. Pharr, Journal Of Materials Research, Volume 7, Number 6, June 1992.

Appendix D: User Experiment Definition Utility

D.1 Overview

The User Experiment Definition Utility is an optional routine for use with the Nano Indenter® XP, provided at additional cost on some instruments. This utility allows additional operations for defining experiment procedures.

D.2 The User Experiment Definition Utility Menu

There are three available options in the User Experiment Definition Utility; Create and Store Custom Indent Files, Examine and Edit User Experiment Set, and Create and Store Custom Shape Files. There is a specific difference between the terms "File" and "Set" when referring to the use of experiment definitions. Experiment Files are the individual group of instructions that define the procedure used to make an indent (or a group of indents). An Experiment Set is a collection of Experiment Files that is made available for use in a specific test run. Finally, a Shape File is a set of coordinates that defines an array or Scratch Shape to be recalled when necessary.

D.2.1 Create And Store Custom Indent Files

This portion of the program is used to select or construct the procedure or procedures to be followed in the making of the desired indents. The descriptions of how indents are to be made are called "indent experiments."

All indent experiments consist of several segments. The first is always the "Approach Segment" in which first contact is made with the surface. A "Load Segment" is generally next and may be performed under either displacement control or load control, i.e., at a constant displacement rate until a specified indent depth (displacement) or load is achieved, or at a constant loading rate until a specified load or a specified displacement is reached. A short Hold segment is sometimes inserted at the end of a load segment to allow time for the system to equilibrate fully before proceeding to an "Unloading Segment" (which should almost always be carried out under load control). If no additional load segments are planned, the specimen should be unloaded until 80 to 90% of the load has been removed. If a Hold Segment is now specified, the indenter can sense any displacement caused by thermal drift, and a correction for thermal drift can be made. The last segment of an indent must always involve a complete unloading of the indenter, thus allowing the tip to rise above the specimen surface.

The format in which indent experiments are represented is illustrated in Table D.1.

Examples Of Typical Indent Experiments	
Indent Experiment No. 1	**Indent Experiment No. 2**
1. A	1. A
2. LL 2500Rat, 50000For	2. LD 10Rat, 150Dep
3. UL 100%Ra, 80%Un	3. UL 100%Ra, 80%Un
4. H 1Log, 20Poi	4. LD 10Rat, 300Dep
5. UL 100%Ra	5. UL 100%Ra, 80%Un
	6. H 1Log, 20Poi
	7. 100%Ra

Table D.1 Examples Of Typical Indent Experiments

Although the two indent experiments shown in Table D.2 are different, they share in common the sequence of their segments. Both begin with an Approach Segment followed by a Load Segment. In Experiment No. 1 the indent is made at a constant loading rate of 2500 µN/s (i.e., under load control) with the load increasing until a total load of 50,000 µN is applied to the indenter. In Experiment 2, however, the first load segment is performed at a constant displacement rate of 10 nm/s (i.e., under displacement control), and the load is increased until a displacement depth of 150 nm is achieved. A load limit rather than a displacement limit could have been specified if desired in this case. The third segment in each experiment is an Unload Segment that is load controlled with an unloading rate equal to 100% of the loading rate at the end of the previous load segment. In both cases the unloading continues until 80% of the previous load has been removed from the indenter. In Experiment No. 2, which is the more complex experiment, the load and unload pattern of segments is repeated in Segments 4 and 5, although the displacement limit in Segment 4 is increased to 300 nm. Both experiments end with a Hold Segment and a final Unload Segment in which 100% (the default value) of the load is removed.

Table D.2 shows but two of the many variations in indent design that are possible in the indentation procedures available in the Nano Indenter® XP program. The various indent parameters are discussed in greater detail below, but for the moment it will suffice to emphasize the general pattern of an indent experiment: an Approach Segment, repeating Load and Unload Segment pairs, and finally a Hold Segment followed by a complete Unload Segment. Of course, extra Hold Segments may also be added before or after any Load or Unload Segment except the last Unload Segment, which must the last segment of the experiment.

D.2.1.1 Load Indent File From Library To Memory

This allows you the option of re-using previously stored indentation experiments. As with shapes, indent experiments may be stored in memory and recalled by the user. You will be asked to provide a number for the indent experiment. The proper format is:

I+(1-7Char)

where "I" is the indentation experiment prefix, and "1-7Char" indicates that the name of the experiment can be up to seven alphanumeric characters. For example, an indentation experiment might be named "I75DEP" or "ICSM0011."

Make this choice when you want to use a stored indentation pattern as the basis for constructing a new pattern. A listing of the indent experiments stored in memory should be accumulated in an appropriate notebook as recommended in connection with the storing of indent shapes.

D.2.1.2 Modify Indent Currently In Memory

This option allows you the opportunity to use previously stored experiments as a basis for new experiments. For example, load segments may be altered while keeping a previously defined hold/unload structure.

When this option is selected, the experiment currently in memory will be displayed, followed by the prompt:

```
Do you want to change a segment [Y/N]?
```

If you choose "Y", you are prompted:

```
Which segment do you wish to change? [1-12]
```

Select the segment you want to modify. Upon entering the segment number, the Menu of Segments will be displayed, allowing you to select a different segment type. The Menu of Parameters will be displayed when the Segment is selected. After the Segment is re-defined, choose "ALL DONE, CONTINUE ON" from the Menu of Segments. You will be prompted:

```
Is everything right [Y/N]?
```

If the experiment is defined as desired, choose "Y." Upon confirming that the experiment is correct, you will be returned once more to the option:

```
Do you want to change a segment [Y/N]?
```

If everything is correct, choose "N." After confirming that the experiment is correct, you will be returned to the "Create And Store Custom Indent Files" menu.

D.2.1.3 Build a New Indent In Memory

This routine enables you to build new indentation experiments without having to modify a previously stored experiment.

While building a new experiment you will toggle back and forth between two menus: "Menu of Segment Types" and "Menu of Parameters." The former is a list of the segments, e.g., approach, loading, unloading, etc., that must be specified to set up an indentation experiment (see Section 9.3 and Section D.3). The latter is a list of the various parameters required to describe individual segments (see Section D.3).

D.2.1.4 Display Indent Currently In Memory

When this option is selected, whatever experiment is currently being defined is displayed. Press the CONTINUE function key to return to the previous menu.

D.2.1.5 Store Indent In Memory To Library File

If you would like to save the experiment you have defined so that it can be recalled for future use, then you must select this option. When this option is selected, you will be prompted:

Store this indent as [I+1-7 Char.Alphanumeric]?

As described in Section D.2.1.1, the filename should be entered in the format:

IXXXXXXX.

Once the filename is entered, the file will be stored in the appropriate directory on the hard drive, where it can be recalled for future use.

D.2.1.6 ALL DONE, EXIT

Once you have finished defining or modifying experiments, select this option to return to the User Experiment Definition Utility menu.

D.2.2 Examine And Edit User Experiment Set

The second option available on the User Experiment Definition Utility menu allows you to edit the Experiment Set. As explained above, the Experiment Set is the collection of Experiment Files that is made available for use in a specific test run. You can have up to six different Experiment Files in a single Experiment Set. Typically, the Experiment Set will be comprised of those Experiment Files used most often. On entry to this menu, the currently defined Experiment Set will be displayed.

D.2.2.1 Load Indent File to Memory, Name =?

This allows you to load a previously stored indentation experiment. This option behaves in the same manner as described in Section D.2.1.1.

D.2.2.2 Display Indent Currently In Memory

When this option is selected, whatever experiment is currently loaded is displayed. This option behaves in the same manner as described in Section D.2.1.4.

D.2.2.3 Add Indent to Set, As Experiment No.?

As described above, the Experiment Set can comprise up to six Experiment Files. Once you have loaded an Experiment File to memory, you must add it to the Experiment Set before it can be used in a test. When the "Add Indent To Set" option is selected, simply choose the index number in the Experiment Set which will be used for the new Experiment File. The previous Experiment File occupying that index number will be eliminated and replaced with the new Experiment File.

When this option is selected, you will be prompted:

```
New value in range ([0,1-6])?
Press enter to accept old value, or new value enter
to change
```

Select the index number in the Experiment Set that you wish to replace with the Experiment File currently in memory. The old Experiment File will be overwritten in the Experiment Set currently in memory.

D.2.2.4 Delete Experiment No? From Set

To delete an Experiment File from the Experiment Set, choose this option. You will be asked to enter the index number for the Experiment File to be deleted:

```
New value in range ([0,1-6])?
Press enter to accept old value, or new value enter
to change
```

Enter the desired index number. Once you have done so, there will no longer be an Experiment File defined at that index number in the currently loaded Experiment Set.

D.2.2.5 Display The Experiment Set

When this option is selected, the current Experiment Set is displayed. Each Experiment File in the Experiment Set will be displayed, with the name of the saved Experiment File displayed at the top of the screen, along with the index number for the Experiment File in the current Experiment Set. If an Experiment File has been deleted from the Experiment Set, there will be no information displayed for that index number. Press CONTINUE to return to the Examine And Edit User Experiment Set menu.

D.2.2.6 START OVER: Load The Experiment Set From Disk

Select this option if you want to reload the last saved Experiment Set without saving any changes you have made in the currently loaded Experiment Set.

D.2.2.7 SAVE: Store The Experiment Set On Disk

When this option is selected the Experiment Set currently in memory will be saved to the hard drive, and the previous Experiment Set will be overwritten. Note that any Experiment Files associated with the previous Experiment Set will still reside on the hard drive, and can be used by recalling the files from memory.

D.2.2.8 ALL DONE, EXIT

Once you have finished examining, defining or modifying the Experiment Set, select this option to return to the User Experiment Definition Utility menu.

D.2.3 Create And Store Custom Shape Files

This portion of the program is used to select or construct shapes or patterns of indentations. The only use of this routine is to generate custom Scratch Shapes (see Section 18.0). The set of coordinates saved by this routine is called a "Shape File"

The specific distinction between the terms shape and subshape is defined is Section 13.2.6.1. The term shape refers to the entire pattern of indent locations over all specimens and all positions. The "Shape Files" created in this routine are more accurately called "Draw Shape Files," as the pattern of indentation sites saved with this routine is only used to define a Draw Shape

Typically, Draw Shapes are simply 2x1 or 1x2 arrays, in which the spacing between indents represents the length of the desired scratch, and the array or "move between indents" represents the direction of the scratch.

All of the options for defining indent sites are available, however. The result is that the Shape Files you create can have any pattern you desire.

Note that because of this flexibility, the capability is in place to create high complicated patterns for scratch shapes. In almost all cases, you will never use this capability. Most scratch shapes are simply 1x2 or 2x1 arrays. Do not save complicated arrays of indentation sites unless you have a very good reason to do so. Contact Nano Instruments or your local representative if you have questions about this operation of this option.

D.2.3.1 Array Of Positions

This subprogram offers the user the option of specifying the azimuth of a line of indents relative to the x axis of the indenter table. The x axis of the Table is an imaginary *horizontal* line on the monitor screen, and azimuth angles are measured *counterclockwise* from that line. You will first be asked to select a starting position on the specimen using the microscope. Follow the on-screen instructions, and the computer will automatically shift control of the system to the joystick. Use the joystick to move the Table until the desired position on the specimen lies directly under the cross hairs on the monitor screen (see Section 8.2.6). Now exit manual control by pressing the "E" key of the keyboard. This exiting action identifies the starting position of the indent for the computer[1] and brings a new question to the screen.

```
Do you wish to input spacings, dimensions, and
rotation (S), OR
define the entire array (other corners) using the
microscope
and then select dimensions (P)?
```

If you type "S" and RETURN, you will then be asked to input the desired azimuth of the initial line of indents, and you are prompted with an additional series of questions to specify the spacing and number of indents in the x and y directions.

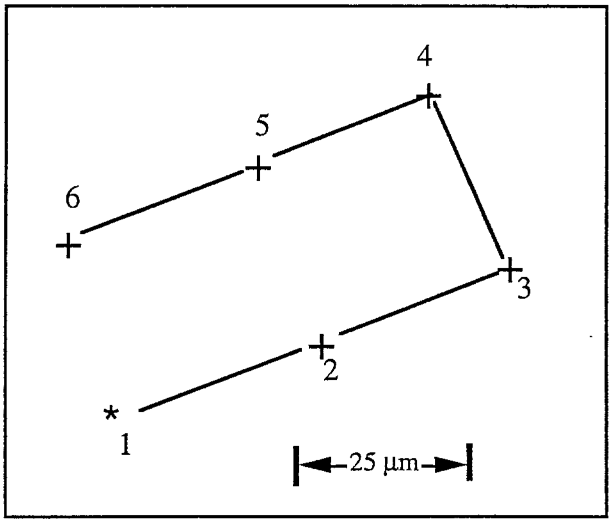


Figure D.1 A Typical Indent Shape

If you type "P" and RETURN, you will be taken back into manual control where you will be asked to select with the microscope the "corners" of the shape that you are building. To select a corner you must position the desired spot on the specimen under the cross hairs and exit manual control. "Corners" in this case means those points at which a straight line of indents changes direction. After the corners have been selected, you then specify the number and spacing of the indents.

Using either method, after the completion of the selection routine, a drawing of the shape will appear on the monitor screen, and you will be asked whether the shape suits you. If you say no (type "N"), you will be given an opportunity to modify the scale or rotation of the shape. A "yes" ("Y") takes you to the part of the program dealing with "head repositioning during a shape" (see below).

[1]Note: This action also automatically sets the x and y coordinates of starting indent to (0,0) as may be seen at the bottom of the screen. The computer references all future indent positions of the series to this (0,0). Thus once the initial indent position is chosen, the "R" key, which manually resets the x and y position counters to zero (see p. 8-2), will not execute its function.

D.2.3.1.1 Head Repositioning

When you have completed the specification of a shape, you will be presented with the following prompt:

Add/subt. a head positioning (Z direction) procedure prior to a specified indent position? (A/S/N)

The purpose of this option is to permit the indenter to make allowances for small variations in height on the specimen surface. The positioning procedure is the same as the one followed prior to the making of the first indent of any subshape, and the details of this process are discussed in Section 13.2.9. This procedure provides the computer with an accurate update of the relative positions of the specimen surface and the indenter in the vertical direction, and the next indent can, therefore, be made without the danger of the indenter being dragged across the sample surface as it approaches that indent position. If any of the indents of a shape are to be located on a region of a specimen significantly higher (or lower) than the area where the preceding indents are to be made, it is a good idea to add one or more head repositioning to the shape before these indents. As described above, the effect of this action is to cause to indenter to go through its surface-finding routine just prior to making the specified indent.

It should be noted that this procedure is limited as to the variation in height that can be accommodated. Under no circumstances should a specimen be used in which the variation in surface height exceeds 1 mm.

Entering an "A" (for "ADD") results in a request for the number of the indent before which the additional head positioning procedure is to be carried out. Suppose, for example, that a small ledge exists on a sample and that in a shape containing a total of 10 indents, the first five are made on one side of the ledge and the other five on the ledge itself. A head positioning should then be carried out before the sixth indent is made; thus, a "6" should be entered in response to the request for the number of the indent before which head positioning occurs.

The entry of an "S" for "SUBTRACT" in response to the above prompt has the effect of canceling a head repositioning procedure. As will be described in the following section, a given shape can be programmed to repeat automatically as many times as desired on the same specimen or on different specimens. Each time it is repeated, a head repositioning procedure is carried out just prior to the making of the first indent of the shape. This provision is intended as a safeguard against unexpected changes in the height of a specimen surface. However, if you are certain that the specimen surface is really flat and not tilted in holder, these additional head positioning can be eliminated by the "SUBTRACT" command. The use of SUBTRACT is not generally recommended, and at a minimum, before issuing the SUBTRACT command, the user should ascertain that all of the surface area that will pass under the indenter as the shapes are made stays in focus in the microscope. The advantage of the SUBTRACT command is the saving of the time (usually about 10 minutes) required for the completion of a head repositioning.

An entry of "N" for "NO" in response to the above prompt means that no additional head positioning procedures will be added to the chosen pattern of indents.

Keep in mind, however, that a head repositioning procedure is automatically added prior to the first indent of a new shape, and to the first in a series of individually selected positions.

Once the initial set of indent positions is defined, you are done with this routine. If, however, you wish to specify additional positions, simply enter the method you wish to use, as if you were beginning your first set of indent positions. There is no restriction on the type of methods you can use. An array of positions can be defined, followed by several individual positions, followed by a selected shape from memory, etc. You should only exit "Select positioning of indents" when you have fully defined all indent positions that you will be using during a test.

D.2.3.2 Individually Selected Positions

As the name suggests, this choice allows the user to create any desired shape by using the microscope to select the position of each indent. You are asked to specify the number of indents you wish to make, and then prior to the choice of each indent location, the system is returned to "manual control" so that you can use the joystick to move to the desired location on the specimen. As always, exiting manual control selects that indent position. The prompts which are displayed while using this routine are:

```
You can now select the positions of each indent of a
series
independently.  Simply exit the manual control mode
(E) when
you are satisfied with the position selected.
```

```
Select how many indent positions [0=exit,1-XXX]?
```

Once all indent positions have been selected, the shape defined by those indent positions will be displayed, and you will be asked:

```
Is this the shape you wanted [Y/N]?
```

Enter "Y" if the shape is correct. If you enter "N" you will be returned to the Menu of Positioning Procedures. All indent locations chosen while selecting individual positions will be ignored.

D.2.3.3 Select A Shape From Memory

This choice allows the user to recall from the computer memory previously used shapes that have been stored on the hard disk. You will be asked to enter the number of the shape, and you must know the number if you are to use this choice. (It is a good idea to keep a record of new shapes as you create them so that you may check their numbers.)

```
Number of shapes to be entered from memory
[0=exit,1-999]?
```

The correct format for entering the shape number is:

xxx

where xxx is the shape number.

There are two methods for positioning the shape:

```
Do you wish to position the first point in this
configuration using the microscope (M), or by
inputting its numerical position (I) ?
```

Selecting "M" will send you to "Manual Control" where the initial point of the shape is selected as usual. Choosing "I" will give you the opportunity to define the first point of the shape by inputting the desired X and Y coordinates. Needless to say, selecting the position using the coordinates requires that you use caution. Ensure that the location you select by defining it's coordinates is actually on the specimen. It is easier to make large errors when defining an indent's location by position than when 'physically' choosing the location.

D.2.3.4 Alter The Scale Or Rotation Of Last Sub-Shape

This choice permits the scale or orientation of a shape already stored in memory to be modified. Simply follow the on-screen instructions.

D.2.3.5 Delete Selected Indents From Shape

The operation of this routine is identical to the description in Section 13.2.6, with the exception that rather than allowing the deletion of Subshapes, this routine allows the deletion of specific indent sites.

D.2.3.6 Move The Tables To Last Specified Position

This routine operates as described in Section 13.2.7.

D.2.3.7 ALL DONE, CONTINUE ON

When you have finished selected positions, choose "ALL DONE CONTINUE ON" to exit the routine. You will be asked:

```
Do you wish to store this shape for future use? (Y/N)
```

A "YES" answer causes the entire pattern of indents that you have defined (i.e., the "shape") to be stored on the hard disk. It will be assigned a "Shape Number" with the format

Sxx,

and that number can be used in the future to recall the shape from memory. The shape will also be automatically printed out by the printer, and a copy of the new shape should be stored in the Indent/Shape Library[2]. Shapes that are likely to be helpful to future users should always be saved. For example, every Indent/Shape Library probably has in it standard shapes such as those shown in Figure D.1. Shapes that are highly specific to a particular specimen, for example, a shape in which the indents are individually placed on one or more phases in a multiphase sample, should not be saved. Such a shape is unique to that specimen and can never be used again. To save it would simply clutter up the hard disk.

You are given the opportunity to enter comments to be stored with the shape. This information is for record keeping purposes only, so enter whatever information is helpful. The comments are limited to 150 characters.

D.2.4 ALL DONE, EXIT

Once you have finished defining or modifying Experiment Files, the Experiment Set, or Custom Shapes, select this option to return to the Master Menu.

D.3 Segments And Parameters

In order to use the User Experiment Definition Utility to its full potential, it is necessary to understand the many options available for experiment definition. These options include the segment definitions, the parameters that define the segments, and the use of segments in defining the experiment.

D.3.1 Segments

As explained in Section 9.3, segments are individual operations that take place during an indentation. There are several options for the segments to be used, although each test must begin with an Approach Segment and end with an

[2] Every laboratory should have a special notebook, the Indent/Shape Library, in which copies of indent shapes and indent experiments are recorded and identified by their Sxx and Ixx numbers. A great deal of time can frequently be saved by using indent experiments and shapes that have been defined by previous users.)

Unloading Segment. Table D.2 displays the various segments available for use in the User Experiment Definition Utility.

Segments & Segment Codes	
Segment	**Code**
Approach	A
Constant Loading Rate Loading	LL
Constant Displacement Rate Loading	LD
Constant (Load rate/Load) Loading	LS
Hold	H
Step Load / Unload	S*
Draw (Scratch)	D*
Constant Displacement Rate Unload	UD*
Constant Loading Rate Unload	UL
*Indicates a segment only available with advanced user packages.	

Table D.2 Segments and Segment Codes

D.3.1.1 The Approach Segment (A)

This segment incorporates instructions that allow the computer to locate the relative positions of the indenter and the sample surface in the vertical direction, i.e., the "zero" point of the displacement and load data. This must always be the first segment in an experiment, and can be used only once (re-approaching the surface during an experiment has little value, and is not allowed).

D.3.1.2 Constant Loading Rate Loading Segment (LL)

The Nano Indenter® XP is designed as a load controlled instrument, thus load controlled loading segments and step loading segments are the most precisely defined load segments that can be performed with the indenter. Both a loading rate and a load and/or displacement limit can be assigned by the user.

D.3.1.3 Constant Displacement Rate Loading Segment (LD)

Through the use of feedback loops, displacement rate control is also possible. A displacement rate and a displacement and/or load limit are specified by the user. The user should be aware that because of inevitable limitations on the speed of response in a feedback loop, departures from strict constancy of the displacement rate can occur in this type of indent experiment. In any case, displacement rates should be limited to rates above 1.5 nm/s.

Segment & Parameter Matrix

Segment Type	Segment Code	Rate	Data Logging Delay	Factor Change In Stiffness	Depth Limit	Force Limit	Force Rate Max	Displace-ment Rate Min	Percent Unload-ing	Number Of Hold Points	Time Limit	Draw Shape Number	Draw Accel-eration	Draw Velocity
Parameter Code		Ra	Log	Con	Dep	For	Fra	Dra	Un	Poi	Tim	Shp	Dac	Dvl
Parameter Units		nm/s µN/s 1/s	sec	*	nm	µN	µN/s	nm/s	%	*	sec	*	µm/s^2	µm/s
Approach Segment	A	√	√	√	√X	√X					√			
Constant Loading Rate Loading Segment	LL	√	√		√X	√X	√	√			√			
Constant Displacement Rate Loading Segment	LD	√	√		√X	√X	√				√			
Constant (Load Rate/Load) Loading Segment	LS	√	√		√X	√X	√				√			
Hold Segment	H		√		√X	√X		√			√			
Step Loading / Unloading Segment	S	√								√	√			
Constant Unloading Rate Unloading Segment	UL	√	√		√N	√N	√	√	√		√			
Constant Displacement Rate Unloading Segment	UD	√	√		√N	√N	√		√		√			
Draw Segment	D	√	√			√X					√	√	√	√

Table D.3 Matrix showing the relationship between the segments that constitute an indentation experiment and the parameters required to describe each segment. The units in which the various parameters are expressed are shown in the row directly beneath the parameters themselves. The "Codes", appearing in the row above the parameter units and in the column immediately to the right of the list of segments, are the abbreviations used for the segments and the parameters in on-screen and hard-copy listings of the segments of an indent experiment. The letters "X" or "N" beside the check mark on some options indicate a maximum or minimum limit, respectively. For example, "X" indicates that the segment will terminate when the force or displacement increases to the limit value. "N" indicates that the segment will terminate when the force or displacement decreases to the limit value.

D.3.1.3 Constant (Load Rate/Load) Loading Segment (LS)

Refer to Reference J.3 in Appendix J for more information on the Constant Load Rate/Load Loading Segment (more commonly referred to as the Constant Strain Rate Segment).

D.3.1.4 Hold Segment (H)

It is good practice to include a hold segment for making corrections for thermal drift. A Hold Segment can be inserted before or after either a loading or a partial unloading segment, the latter being the more common practice when checking thermal drift. Typically a Hold Segment follows an unloading segment in which 80 to 90% of the load is removed. Hold Segments can also be inserted immediately after a load segment to monitor possible creep in the specimen or to permit the system to come to equilibrium before initiating the next segment.

D.3.1.5 Step Loading/Unloading Segment (S)

A step-loading segment (+S) is available in which a load step is specified, and the load on the indenter is increased virtually instantaneously by this amount. Step unloading segments (-S) are also possible in which a load step is also specified, and the load is reduced by that amount. The direction of the load change is signified by the sign of the step parameter ("+" for loading and "-" for unloading).

D.3.1.6 Constant Unloading Rate Unloading Segment (UL).

Unloading segment types that are the reverse of the loading segments described above are available. Unloading at a constant unloading rate is again the most fundamental type of unloading segment because of the load control design of the Nano Indenter® XP. The user specifies the desired unloading rate and the percent of the load that is to be removed. Unloading may be either complete or partial, and the unloading rate specified may be absolute or a percentage of the loading rate at the end of the preceding loading segment.

D.3.1.7 Constant Displacement Rate Unloading Segments (UD)

Feedback loops also permit unloading at a constant displacement rate. The user can specify the unloading rate either in absolute terms or as a percentage of the loading rate at the end of the preceding loading segment. Unloading may be complete (100%) or partial, the percentage of the load to be removed being specified by the user. While displacement control is possible (>1.5 nm/s), in general, the user will obtain better results by specifying load-controlled unloading sequences. **Use of this option is not recommended.**

D.3.1.8 Draw a Shape Segment (D)

This choice relates to the use of the Nano Indenter® XP in performing scratch tests. Using this segment without the optional scratch hardware and software may cause damage to the system. The optional equipment is designed to provide for safe operation of the hardware, as well as providing tangential force and position data. When "Draw a Shape" is selected, the parameter menu is displayed which allows you to define the parameters that describe the scratch. See Section 18, "The Tangential Force Measurement Option" if your Nano Indenter® XP is equipped to perform scratch tests.

It is worthwhile to note that after a Draw Segment is performed, Displacement Limits (see Section D.3.2.4) should not be used to control the experiment, as the extremely large relative displacements that can be achieved, due to the sample slope, render the control of the experiment through displacement limits somewhat unreasonable.

D.3.2 Parameters

This menu presents the variety of parameters needed to characterize indent segments. The choice of any segment from the "Menu of Segments" will cause the "Menu of Parameters" to appear on the monitor. Default values are assigned to each parameter, and these default values can be modified by the user through the selection in turn of each parameter for which changes are desired. The meaning of these parameters is summarized below.

D.3.2.1 The Rate Parameter

Unlike most other entries in the "Menu of Parameters," different labels appear for the Rate parameter depending on the Segment choice. The label shown in the Menu of Parameters reproduced above is used with the Approach Segment and defines the rate at which the indenter approaches the specimen surface during its surface-finding routine. The default value is 10 nm/s, which is the rate at which the indenter moves, once it reaches a position 1000 nm above the plane the computer has provisionally identified as the sample surface (this position, default of 100 nm, is the "surface height uncertainty"; see Section 8.2.8.1 for information concerning this value).

Other labels used for the Rate Parameter include "Displacement Rate" (for the Constant Displacement Rate Loading Segment) and "Unloading Displacement Rate" (for an unloading segment to be performed at a constant displacement rate). Rate in these instances refers to the rate at which the indenter is to be moved into or out of a specimen. Constant loading rate segments have still another label, but in all instances, the Rate parameter is easily identifiable, and its meaning is obvious except, perhaps, in the case of a Step Loading/Unloading Segment when the Rate Parameter becomes the Step Parameter (the step size.). During a draw (scratch) segment, the Rate Parameter can be used to ramp the load upward (or downward if the sign of the rate is negative) as the scratch is performed. If a constant load scratch is desired, the Rate Parameter should be set to zero.

The default values for the different rates are good choices for many indent experiments, but they may be adjusted downward for small indents to ensure the acquisition of an adequate number of data points, or upward for deep indents to keep within bounds the time required to make an indent.

Finally, the Rate Parameter can be specified as either "Absolute," "Percentage of Last Maximum Rate," or "Percentage of Limit per Second," depending on what type of segment the rate parameter is set for:

D.3.2.1.1 Absolute Rate

If an absolute rate is selected, then the units of the Rate Parameter correspond to the type of segment (i.e., 10 μN/s for load control, 10 nm/s for displacement control).

D.3.2.1.2 Percentage Of Maximum Rate

If the rate is specified as a percentage of the last maximum, then the Rate Parameter used in the current segment is the specified percentage of the maximum loading rate achieved in the preceding segment.

For example, if a load segment of 100 µN/s rate is followed by an unloading segment that has a specified rate of 50%, then the rate used in the unloading will be 50 µN/s. If a second unloading segment follows with a specified rate of 25%, then the rate used in the second unloading will be 12.5 µN/s.

If a percentage rate is specified and the preceding segment is displacement controlled, then the final load voltage rate required to achieve the specified displacement is used as the basis for the percentage rate. (During displacement controlled experiments, a feedback loop monitors displacement and adjusts the ramping of the load in order to achieve a constant displacement rate. While the loading rate has not been specified by the user, the software stores the effective loading rate and a following percentage rate will refer to this value).

D.3.2.1.3 Percentage Of Limit Per Second

If the rate is specified as a percentage of the limit per second, then the Rate Parameter used in the current segment is the specified percentage of the active segment termination limit.

For example, if a constant loading rate loading segment is specified with a load limit of 5000 µN, and the Rate is specified as 10% of the Limit per second, then the loading rate used in the experiment will be 500µN/sec. Likewise, if a constant displacement rate loading segment is specified with a displacement limit of 5000 nm, and the Rate is specified as 20% of the Limit per second, then the displacement rate used in the experiment will be 1000nm/sec.

Note that the limit used as the basis for the rate parameter will depend on the type of segment. For example, in a Constant Displacement Rate Loading segment, only the Displacement Limit will be used as a basis for the Rate Parameter, regardless of the value of the Load Limit. Likewise, in a Constant Loading Rate Loading Segment, the Load Limit will be the active limit for the purpose of the Rate parameter.

Note that because Draw Segments are restricted to Constant Loading Rate Loading, any percent of limit per second rates used will reference the Load Limit.

D.3.2.1.4 Dialog Associated with Absolute & Percentage Limits

For a Constant Loading Rate Loading Segment, or for a Draw Segment:

Upon entering the Rate parameter value you will be prompted:

```
(A)bsolute, or (P)ercent of Max Rate, or percent
of
(L)imit per sec. [A/P/L]
```

Select the type of rate you want by entering the letter associated with the desired type.

For a Constant Displacement Rate Loading Segment:
Upon entering the Rate parameter value you will be prompted:

```
(A)bsolute, or percent of (L)imit per sec. [A/L]
```

Select the type of rate you want by entering the letter associated with the desired type. Note that the Percent Of Max Rate option is not allowed for Constant Displacement Rate Loading Segments.

For a Constant Loading Rate Unloading Segment:
Upon entering the Rate parameter value you will be prompted:

```
(A)bsolute, or (P)ercent of Max Rate  [A/P]
```

Select the type of rate you want by entering the letter associated with the desired type. Note that the Percent Of Limit per Second option is not allowed for Constant Loading Rate Unloading Segments.

D.3.2.2 Delay Between Logging Data Points

During the making of an indent, the computer continually records data triplets corresponding to the indenter load voltage, displacement voltage, and time. The maximum rate at which these data can be acquired is approximately three readings per second, which corresponds to the default value of "0" (i.e., no delay). An entry of "1" instead of "0" would mean that a point is to be logged every second instead of every 1/3 s. For indenters equipped with the continuous stiffness measurement option, the maximum rate is slightly slower — about 2 readings per second, but specification of 1, 2, 3, ... as the delay between logging still results in a logging rate of one point every 1s, 2s, 3s, ... just as in the case of the system without the constant stiffness measurement option or one with that option turned off.

D.3.2.3 Factor Change in Stiffness to Determine Surface

During the approach to the surface, the indenter senses stiffness in terms of the stiffness of the delicate springs that support the center assembly to which the indenter is attached. As soon as the indenter begins to make contact with the surface, it begins to sense the stiffness of the specimen, and the reported stiffness readings increase sharply. The computer defines the surface as that point where the stiffness reaches a magnitude four times greater than the stiffness of the springs. For a great many samples (e.g., metals and ceramics) this factor of four is a good choice. However, for very soft materials (e.g., certain polymers) it is useful to specify a smaller factor to make sure that the indenter does not penetrate a significant distance into the specimen before tripping the start of the first load segment. Inspection of plots of the raw Approach data (see Section 14.4.3.4) for a representative number of indents is a useful exercise. Make sure that the "knee" in the Approach curve corresponding to surface contact is well defined. Adjustments in approach rate and/or the factor change in stiffness can be used to insure proper definition. However, altering the stiffness change factor and/or the approach rate will almost certainly produce a change in the number of the Approach Segment data point at which the surface contact is made. Thus, if such changes are made, it is imperative that the approach data be examined and appropriate changes be made to the number of points back to the surface during the load / displacement calculations. In most cases, this change can be performed automatically by allowing the algorithm to select this surface contact point, as is done during the automatic calculation following the completion of a standard test.

D.3.2.4 Depth Limit

This parameter specifies the depth limit for loading and unloading segments. It is applicable to both load controlled and displacement controlled loading segments where it represents a maximum allowable indenter penetration depth. The default value of 25,000 nm shown in the "Menu of Parameters" above is for loading segments. For unloading segments, this entry in the "Menu of Parameters" changes and shows a default value of 0 nm. Thus, the "Depth Limit" parameter can be used to specify a minimum or a maximum limit depending on the nature of the segment.

This default value of 25,000 nm (25 µm) should be sufficient for most tests. When using the high force option (see Section 19), however, this value should be increased when very high loads are specified.

D.3.2.5 Force Limit

This parameter specifies the maximum load to be applied in a loading segment. If the maximum load proves insufficient to reach a specified load limit or to create an indent of the specified depth, the indent process is stopped, and "Maximum Load Exceeded" is noted both on the monitor screen and on the hard copy record of the experiment. When "Maximum Load Exceeded" occurs, the indenter aborts that particular load segment and goes on to the next segment of the indent. As in the case of the Depth Limit parameter, the default value of the Force Limit switches to zero in the "Menu of Parameters" for unloading segments, thus becoming a minimum force limit.

D.3.2.6 Force Rate Maximum

This parameter specifies the maximum rate at which force can be applied to the indenter in a loading experiment. This parameter should be changed only if the user needs to limit the maximum force rate to which his specimen is subjected. **Alteration of the default value is not recommended.**

D.3.2.7 Displacement Rate Minimum

This parameter provides a means for limiting the temporal length of segments. For example, consider a Hold segment that is being used to monitor creep in a sample. Once the displacement rate approaches the previously measured thermal drift rate, further continuation of the hold segment is useless, and in the interest of saving time it should be terminated. If the displacement rate drops below the limit set by the Displacement Rate Minimum, the computer advances the program to the next segment of the indent.

D.3.2.8 Percent Unloading

This parameter specifies the percentage of the load being applied at the end of the preceding segment that is to be removed in an unloading segment. Thus 80% means that at the end of the unloading segment, the load on the indenter will be 20% of the previous maximum.

D.3.2.9 Number of Hold Points

This parameter refers to the number of "load-displacement" pairs to be recorded by the computer during a hold segment or after the load change in a step segment. A value of 50 for the number of Hold Points in conjunction with Data Logging Delay of 1 s means that the Hold Segment will last for 50 s and will result in the accumulation of 50 load-displacement data pairs.

D.3.2.10 Time Limit

The Time Limit parameter simply establishes the maximum length of time any segment can last. The default value is 3600 s. If a segment for whatever reason lasts for more than 3600 s, the computer terminates the segment and moves on to the next step in the program. Note that a Time Limit could be used instead of a Number of Hold Points to control the length of a Hold.

D.3.2.11 Draw Shape Number

If your Nano Indenter® XP is equipped with the Tangential Force Measurement Option, then you can use the "Draw" Parameters to define the specifics of a scratch. The Draw Shape Number refers to the saved shape that defines the length and vector position of the scratch. Refer to Section 18 for more information on defining Draw Shapes. The physical length of the scratch remains as specified in the scratch shape, and cannot be altered in this menu, except by imposing a time limit upon the segment. The scratch shape is specified by selecting "Draw shape number" from the menu and entering the desired scratch shape number.

D.3.2.12 Draw Acceleration

If your Nano Indenter® XP is equipped with the Tangential Force Measurement Option, then you can use the "Draw" Parameters to define the specifics of a scratch. A parameter which can be used to control the scratch is the "Draw Acceleration" (µm/second/second). This parameter controls the acceleration to the speed with which the scratch is performed. The Draw Acceleration should normally be set to a very high value so that the desired scratch speed is achieved as soon as possible. A default value of 100µm/s^2 is recommended. Note that if you set the Draw Acceleration lower than the physical limit of the system, errors can be introduced in the total time of the scratch, as well as in the desired loading rate.

D.3.2.13 Draw Velocity

If your Nano Indenter® XP is equipped with the Tangential Force Measurement Option, then you can use the "Draw Parameters" to define the specifics of a scratch. A parameter which can be used to control the scratch is the "Draw Velocity" (µm/second). This parameter controls the speed at which the scratch is performed. The default value of 10µm/s is a very "safe" rate at which to perform the scratch. The Draw Velocity is limited by the physical minimum speed of the positioning system, as well as the maximum rate of data acquisition.

D.3.3 Experiments

The set of segments is defined as an "experiment" or sometimes "indent file." There is a great amount of flexibility in the definition process, with only the following limitations:

The first segment must always be an Approach Segment.
The last segment must always be a Constant Unloading Rate Unloading Segment
There can be no more than 12 segments in an experiment.

Appendix E: Troubleshooting

E.1 Overview

For proper operation of the Nano Indenter® XP, both the software and the hardware must operate correctly. While the Nano Software that runs your Nano Indenter® XP has been rigorously tested, it is possible that an occasional error might interrupt normal operation. Likewise, though the hardware has been designed for long-term stability and reliability, all of the components have a finite service life. The vast majority of any problems you might encounter with your Nano Indenter® XP system can be easily solved with the proper adjustments to either the software or the hardware.

If you are unable to locate the answer to a problem or question in these operating instructions, or if you suspect there is a technical problem with the instrument, feel free to contact either Nano Instruments or your local representative for further assistance.

E.2 Contacting Nano Instruments

If you need to contact Nano Instruments regarding the operation of your Nano Indenter® XP system, you can reach in the following ways:

Address:	Nano Instruments, Inc. 1001 Larson Drive Oak Ridge, TN 37830 USA
	865
Phone:	~~423~~-481-8453
Fax:	~~423~~-481-8455
e-mail:	nano_service@nanoinst.com

E.3 Dealing With Software Problems & Error Messages

In order to correctly diagnose a problem with the operation of the Nano Indenter® XP, we may need specific information about the problem. In almost every case, we will request a “screen shot” of the error message or unusual behavior, a screen shot of the lines in the software that pertain to an error message, and information about the particular subroutine in which the error occurred.

E.3.1 Getting a Screen Shot of the Error

When a software problem occurs, you will most likely find an “Error” statement at the bottom of the screen. The Error Statement is very useful for diagnosis of the problem. Thus, before seeking help, you should get a copy of the Error Statement, as well as the displayed text on the screen, by using the SHIFT-PRINT SCREEN command. A screen shot will be sent to the printer when this command is used.

E.3.2 Printing the Error Line

If the "Error" statement is at the bottom of the screen, and you have gotten a screen shot of this line, the next step is to enter the software to display the lines of code pertaining to the error statement. The Error Statement will contain a line number that refers to a line in the code. To view this line of the code, type in:

Edit XXXX <enter>

where XXXX is the line of code displayed in the Error Statement. Once you have entered this text, the code will be displayed. You should get a copy of the displayed text on the screen, by using the SHIFT-PRINT SCREEN command. A screen shot will be sent to the printer when this command is used.

E.3.3 Determining the Subroutine

Once you have printed the screen shot of the line in the code, use the Page Up/Page Down keys to move upward in the code until you find the Subroutine name in which the code is located. Use the SHIFT-PRINT SCREEN command to print the code containing the Subroutine name.

E.3.4 What To Do With This Information

Once you have gathered this information, fax it to Nano Instruments or your local representative with a description of the problem and the circumstances that led to the problem.

E.4 Dealing With Hardware Problems

Some possibilities for correcting problems with data reduction are explored in Section E.6. In most cases, simple measures such as re-starting the instrument will correct hardware (electronics) problems. If there is mechanical failure of a system component, or if you cannot diagnose a problem with the instrument, contact Nano Instruments or your local representative.

Under no circumstances should the indenter gantry be opened. There are no user-serviceable parts inside, and opening the gantry could result in damage to the indenter (see Section 5.0).

E.5 Data Reduction Or Results Analysis Questions

Some possibilities for correcting problems with data reduction are explored in Section E.6.8. As a general rule, the better understood the process of indentation, the easier it is to track problems with data reduction. Thus, we recommend reviewing the available literature on nanoindentation as a source for answers about the indentation process. A list of suggested technical papers is provided in Appendix J.

E.6 Suggestions For Resolving Problems

There are a number of measures that can be taken to resolve or further characterize problems with the instrument. If the instrument is not operating correctly, try these routines before seeking further help.

E.6.1 Unusual Behavior During Automated Operation

If the instrument is not behaving properly during automated operation, it is possible that the DAC values may be incorrect. This is most likely if the DAC calibration has not been performed for an extended period of time, or if aberrant values were stored in the DAC calibration data array.

If you are able to access the Recalibrate menu, try running a DAC Calibration (see Section 11.2.2). While the DAC Calibration does not affect the final data, it can improve the performance of the instrument in terms of time between indents, and it serves as an excellent diagnostic routine (if the DAC Calibration successfully completes, there is very little chance that the instrument is seriously damaged or that there is a serious problem).

E.6.2 Positioning Problems

If the instrument is not properly achieving the desired indentation positions, or if there are other problems with the X-Y table operation, try running a Microscope-To-Indenter Calibration (Section 11.2.1). Running the M to I calibration can correct mis-positioning, as well as serve as a diagnostic for the operation of the motion system.

E.6.2.1 Microscope To Indenter Calibration

When setting up a test run, always perform the microscope to indenter calibration on the specimen of interest and as near the specific region of interest as possible. Depending on whether your Nano Indenter® XP is equipped with the standard motion system or the high performance tables, the lead screws that you have may not be precision ground lead screws and can vary from position to position along the screw. It is not just a problem associated with introducing backlash in moving long distances. By performing the microscope to indenter calibration near the location for the indentations you insure the highest degree of uniformity in the motion system.

E.6.2.2 The Backlash Routine

In performing the microscope to indenter calibration, be sure that the last task that you have the machine perform is a backlash routine and that this leaves the indent of interest underneath the cross-hairs. Another important aspect of this calibration is to continue to move the indentation toward the cross-hairs only in the final direction that the backlash routine approaches it from. Remember that the backlash routine moves the specimen down and to the right for its final positioning.

E.6.2.3 Selecting Indent Sites

When placing the indentations, it would be best to have the indent location left underneath the cross-hairs by the backlash routine. As of the writing of this manual, the capability to perform a backlash routine in the manual control menu that is used for selecting indent locations is not yet in place. This is a functionality that should exist and will be added to the XP software.

One way you can achieve this in the interim is by using the "P" command to move to a specific position and using the X and Y coordinates of the SYSTEM counters as the position to go to. This feature has an anti-backlash routine added to the end of it. So by telling the motion system to go to the current position you are telling it to perform a backlash routine.

The next best thing, if possible, is to place the microscope to indenter calibration indentation in a location that allows you to move to the first indentation site again in the same direction as the final moves of the backlash routine.

E.6.3 Computer Lock-Up or Software Stalling

If the computer seems “frozen” or the software is “locked-up,” check the printer to verify that there is an adequate paper supply and that the printer is not in an error state. Printer problems can sometimes cause such computer freezes.

E.6.4 Computer/Hardware Communication Problems

If you receive an error message that indicates that the computer is no longer able to communicate with any of the instrument subsystems, try shutting down power to the entire instrument (including the PC), allowing a five to ten second wait period, and then restarting. Performing such a “clean restart” of the system can reset the data buffers and clear hardware errors from the system.

Such problems are normally rare. If you find that you must frequently restart the system in order to clear such errors, contact Nano Instruments or your local representative for help.

E.6.5 Unusual Behavior in the Indentation System

If the Indentation System does not seem to be behaving properly, try running the “Test Motion Of Indenter” subroutine (Section 11.2.3). Test Motion is the most flexible diagnostic option available to the user.

E.6.5.1 The Standard Test Motion Screen Plot

The Standard Test Motion Screen Plot is used to verify that the indenter will move over its full range of motion, and that the motion in the center of the indenter's travel is linear and free of hysteresis. Following is a set of instructions for preparing this plot. These instructions assume that you are familiar with the operation of the Test Motion subroutine (Section 11.2.3).

E.6.5.1.1 Enter Test Motion
E.6.5.1.2 Select Plot Displacement Vs. Load (1)
E.6.5.1.3 Select Displacement Minimum -5.5
E.6.5.1.4 Select Displacement Maximum 5.5
E.6.5.1.5 Select Load Minimum -2.1
E.6.5.1.6 Select Load Maximum 2.1
E.6.5.1.7 Select Displacement Range 0
E.6.5.1.8 Press the CONTINUE key
E.6.5.1.9 Press the DOUBLE SPEED key 14 times
E.6.5.1.10 When Displacement Voltage is at 0V, press the HALT key
E.6.5.1.11 Press the RESTART key
E.6.5.1.12 Press the HALF SPEED key
E.6.5.1.13 When Displacement Voltage is at -2V, press the CHANGE DIRECTION key
E.6.5.1.14 When Displacement Voltage is at 2V, press the CHANGE DIRECTION key
E.6.5.1.15 When Displacement Voltage is at 0V, press the HALT key
E.6.5.1.16 Press the RESTART RESCALE key
E.6.5.1.17 Select Plot Displacement Vs. Load (1)
E.6.5.1.18 Select Reset Limits To Extreme Values (No)
E.6.5.1.19 Select Displacement Minimum (Default)
E.6.5.1.20 Select Displacement Maximum (Default)
E.6.5.1.21 Select Load Minimum (Default)
E.6.5.1.22 Select Load Maximum (Default)
E.6.5.1.23 Select Displacement Range 0
E.6.5.1.24 Press the CONTINUE key
E.6.5.1.25 Press the HALF SPEED key 2 times
E.6.5.1.26 When Displacement Voltage is at -2V, press the CHANGE DIRECTION key
E.6.5.1.27 When Displacement Voltage is at 2V, press the CHANGE DIRECTION key
E.6.5.1.28 When Displacement Voltage is at 0V, press the HALT key
E.6.5.1.29 Press the OTHER KEYS key
E.6.5.1.30 Press the PRINT SCREEN key (*f*8)
E.6.5.1.31 Press the OTHER KEYS key
E.6.5.1.32 Press the EXIT key

You have now generated and printed the Standard Test Motion Screen Plot. Look for discontinuity in the curve, or for hysteresis in the curve. If the curve is smooth, runs in a rough diagonal across the screen, and changing direction does not generate a different path (i.e., there is no hysteresis in the curve), then the indenter is most likely functioning correctly. For verification of this fact, fax a copy of this resultant plot to Nano Instruments, or your local representative.

E.6.5.2 Example Test Motion Plots

Three plots of the motion of the Nano Indenter® XP indentation system are provided here as references. The first two plots illustrate unacceptable behavior of the indentation system, and the third plot illustrates proper operation.

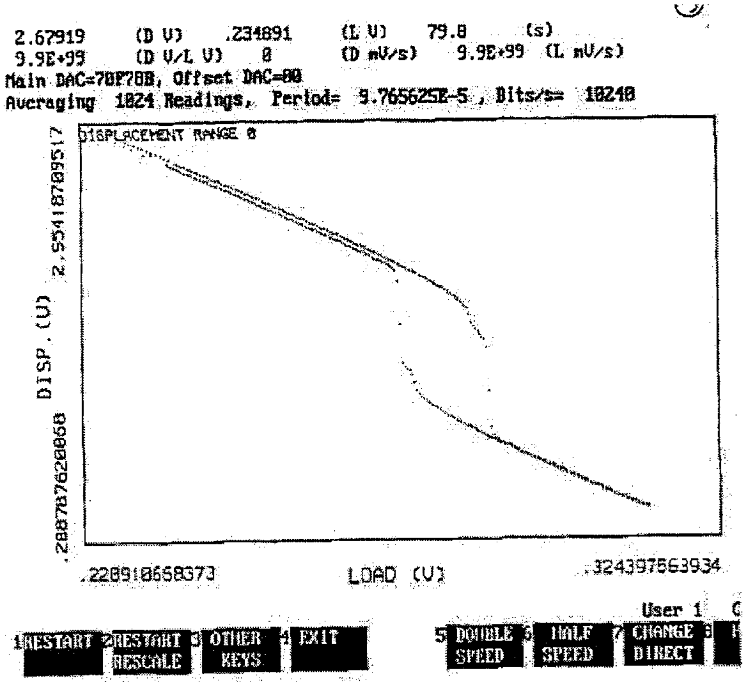


Figure E.1
A Test Motion Plot illustrating discontinuity in the curve. Data is plotted from 0.2V to 3V Displacement.

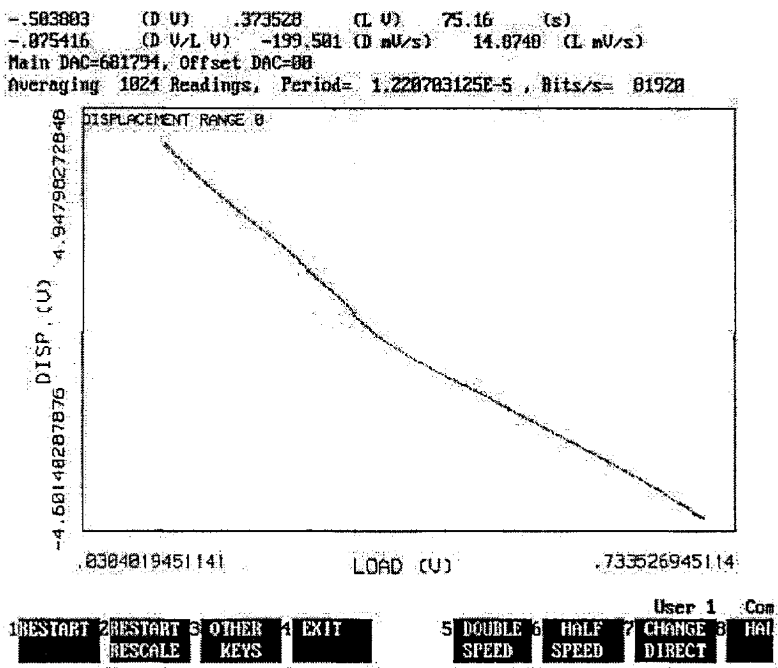


Figure E.2
A Test Motion Plot illustrating an unacceptable bend in the curve. Data is plotted from -4V to 4V Displacement.

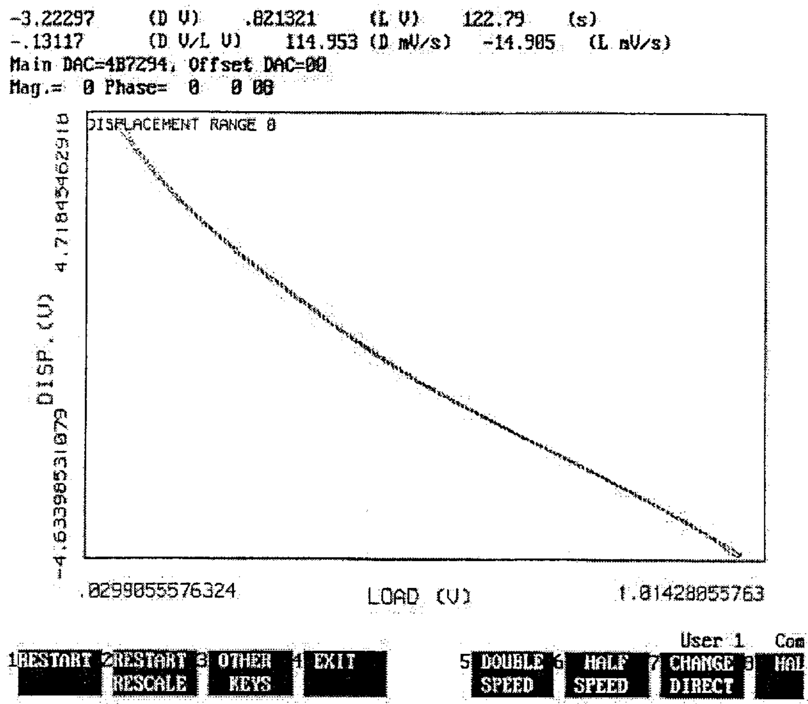


Figure E.3 An example of an acceptable Test Motion Plot. Data is plotted from -4V to 4V Displacement

E.6.6 Noise in the Data

The most common cause of noise in the results data is the vibration table. Check to make sure that the vibration table is properly supplied with air and that the legs of the table are levelled.

A second place to check is the physical connections to the system. Verify that nothing is in contact with the vibration table top or the instrument itself that could be transmitting vibration.

E.6.7 Unique Mechanical Problems

If any of the subsystems are behaving in unusual ways, a good place to start trying to diagnose the problem is to verify that all cables are properly connected between the instrument and the computer.

E.6.8 Problems With Test Results

If you believe there is a problem with the indenter that is resulting in incorrectly reduced data, there are several items that should be verified prior to seeking help. First, verify that the problem is in fact a part of the manipulation of the data by examining the raw data files (see Section 9.4.3). Once it is determined that the raw data files are correct, compare the test results to tests run on a standard material (we recommend SiO_2). It is a good practice to run a standard material with each test, so that this data is available if questions about the manipulation procedures arise. If the standard material data is behaving as expected, examine the specific characteristics of the experimental procedure used to generate the data.

Some of the more probable causes of problems with test results are:

Incorrect tip calibration (see Appendix C).
Incorrect Load Frame Stiffness calibration (see Appendix C).
Normal tip wear has invalidated the tip calibration.
There is debris on the tip that is affecting the test.
There is debris on the sample that is affecting the test.
Test set up using the incorrect diamond number (see Section 8.2.8.3).

E.7 Interrupted Operation

The Nano Indenter® XP was tested by Underwriters Laboratories, Inc. per the Electromagnetic Compatibility procedures as required by the EMC Directive to obtain the "CE" mark. The Nano Indenter® XP meets acceptable criteria in all situations evaluated in that in no case did the instrument become dangerous or unsafe due to any test conditions. In most cases, Performance Criteria A (Normal performance) was obtained, however, in two situations Performance Criteria C was observed. Performance Criteria C is temporary loss of function during and after the test, requiring operator intervention to restore operation, but operating normally once it is recovered (restarted by operator).

In one situation the computer "locks up" when exposed to a 4kV static discharge either on the joystick knob or a computer key. This problem was not encountered at 23 other locations on the instrument and not on these locations when exposed to a 2kV static discharge. Since the operator only touches these areas during test set up and not during operation, the impact to productivity should be minimal. Additionally, if this is a chronic problem, it would be easily resolved by providing a method to ground the operator before contact with the keyboard or joystick occurs. (Possibly a conductive mat in front of the computer in conjunction with ESD shoes.)

The other situation would be an electrical power fast transient/burst of 1000v. This situation also caused the computer to "lock up." No problems occurred with a burst of 500v. This problem could be eliviated with a good quality power line conditioner if these power line situations are probable at any given user site.

Appendix F: Assembly Instructions

F.1 Overview

The Nano Indenter® XP ships in a partially assembled condition. It is necessary for you to complete the assembly at your site.

The required tools are listed below:

A metric Allen wrench kit is included with the Nano Indenter® XP. You will need some of these wrenches for assembly of the instrument. You will also need:

Small Phillip's head screwdriver
Small flat-head screwdriver
Large Phillip's head screwdriver or a Phillip's head bit for a drill or power screwdriver.
Adjustable wrench for attaching the vibration table hose to the air supply.
Vibration Table Levelling Spanner (provided with the vibration table)

F.2 Assembly Instructions

Following is a step-by-step set of instructions for assembly of the Nano Indenter® XP. At times in this procedure you will be referred to the documentation for a specific component (such as the printer). If you have any questions at all while performing this procedure, contact Nano Instruments or your local representative prior to continuing.

F.2.1 Remove All Components From Packaging

Following is a list of the components included in the shipment of the Nano Indenter® XP:

Vibration Table (Base, Legs, Levelling Pads, Hoses, and Tools)
Isolation Cabinet (Left, Right, Rear, Front, and Top Panels)
PC
PC Door Key
PC Keyboard
HTBasic Key
PC Monitor
Printer
Video Monitor
Gantry[1]
Fiber Optic Illuminator
Fiber Optic Cable
Electronic Cables

[1] When removing the gantry from the shipping crate, use the handles on the sides of the gantry to lift. As the gantry is very heavy, it is recommended that at least two people lift the gantry out of the shipping crate.

Joystick
Microscope Objective and Slide
Sample Mount
Sample Mount Disks
Sample Clamp Mount & Clamp Screw
Microscope Plug Screw
Metric Allen-Wrenches
Single Crystal Aluminum Sample
Fused Silica Sample
CSM/LFM Module[2]

A Note About System Configurations

There are three primary options for the Nano Indenter® XP that can affect how the instrument is assembled. These options are: Continuous Stiffness Measurement (CSM), Lateral Force Measurement (LFM), and the High Force Option. A Nano Indenter® XP can be provided with some or all of these options. If either the CSM or LFM option is included with your system, an additional module is present (the CSM/LFM module) which requires additional connections to the PC. If the High Force option is included, additional connections are required to the PC and the gantry. Be sure to pay close attention to the assembly instructions to note which connections are pertinent to your system.

F.2.2 Set Up the Vibration Table

Carefully select the site for the vibration table. Refer to the documentation provided by the manufacturer of the vibration table system. Make sure that there is room around the vibration table for the installation of the isolation cabinet.

F.2.3 Place the Gantry On the Vibration Table

Place the Gantry in the center of the vibration table top, so that the long sides of the indenter are aligned with the long sides of the table top, and the Nano Indenter® XP logo is facing toward the front of the vibration table top. The bulk of the weight of the gantry is located toward the front of the gantry, rather than under the sheet metal cover on the rear of the gantry. You should try to align this "center of mass" of the gantry with the center of the vibration table top. Make sure that no part of the gantry extends off of the table top.

F.2.4 Check the PC Voltage Setting

Check the voltage setting on the rear of the PC to verify that the voltage is set correctly for your site. The voltage can be set to 115V or 230V (see Figure F.1).

F.2.5 Place the PC On A Desk Or Table

Locate a suitable table or desk that will serve as the Operator's Table. Put the PC on top of this table (see Figure F.2).

[2]The CSM/LFM Module will only be present if your Nano Indenter® XP is equipped with the Continuous Stiffness and/or Lateral Force Measurement options.

F.2.5.1 Place the CSM/LFM Module on the PC

If your Nano Indenter® XP is equipped with the Continuous Stiffness Option and/or the Lateral Force Option, an additional module will be present with the instrument. This module attaches to the PC through an HPIB cable, and three coaxial/BNC cables. These cables are color coded as described in Section F.2.12. Additional cables connect the CSM/LFM Module to the gantry when the Lateral Force Measurement option is included with your system.

F.2.6 Place the Monitor On the PC and Connect

Place the Monitor on top of the PC. Connect the Monitor cable to the video card port on the PC (see Figure F.1). Note that if your Nano Indenter® XP is equipped with the Continuous Stiffness Option and/or the Lateral Force Option, the Monitor should be placed on top of the CSM/LFM Module.

F.2.7 Place the Printer On the Operator's Table and Connect

Place the Printer on the operator's table. Refer to the manufacturer's documentation for assembly of the printer. Load paper into the printer's paper tray, and insert a toner cartridge into the printer. Attach the HTBasic key to the parallel port of the PC. Attach the parallel cable from the HTBasic key to the Printer. There is no need to install additional software to use the printer (see Figure F.1).

A Note About the HTBasic Key:

If you receive an "ID Error" during initial start up of the computer, then the HTBasic Key is not in place. The HTBasic key must be in place, or HTBasic will not operate properly.

F.2.8 Place the Video Monitor On the Operator's Table

Place the video monitor on the operator's table.

F.2.9 Place the Illuminator On the Operator's Table

Place the fiber optic illuminator on the operator's table with the light guide port and the intensity control facing toward the front of the operator's table (see Figure F.2).

F.2.10 Assemble the Cabinet Sides

Partially assemble the cabinet, so that the Left, Right, and Rear panels are assembled around the vibration table. Locate the cable port closest to the operator's desk. Push through the acoustic isolation lining and push the cable port cover out of the cabinet panel (see Figure F.3).

F.2.11 Connect the Fiber Optic Light Guide

Insert the fiber optic light guide through the cable port and into the isolation cabinet. Remove the four screws attaching the fiber optic panel to the gantry. Insert the fiber optic light guide through the fiber optic panel. Insert the fiber optic light guide into the microscope's fiber optic port and lock in place using the thumbscrew. Reattach the fiber optic panel to the gantry. Insert the other end of the fiber optic light guide into the fiber optic illuminator's light guide port. Lock in place using the thumbscrew (see Figure F.4).

F.2.12 Connect the Color Coded Cables

Separate all of the electronic cables. Note the color coded dots on the ends of the cables. Note that some of the cables may have multiple color coded dots on one end. Attach the cables to the gantry using the color & number coded dots as a guide (see Figure F.5). Once all cables are attached to the gantry, thread the cables through the cable port to the outside of the isolation cabinet. Attach the other ends of the cables to the PC and video monitor, using the color & number coded dots as a guide (see Figure F.1).

The cables should be attached as:

X motor cable from the gantry X motor port to the PC X motor port.
Y motor cable from the gantry Y motor port to the PC Y motor port.
Head cable from the gantry Head cable port to the PC Head cable displacement port, load port, joystick port, and the video monitor video input BNC port.
HPIB Cable from the CSM/LFM Module to the PC IEEE interface port.[3]
BNC Cable from the CSM/LFM OSC Out and "A" ports to the PC displacement and load card coaxial ports.[4]
LFM Cable from the gantry LFM port to the CSM/LFM Module Power, ADC1, and ADC2 ports.[5]
POWER SUPPLY cable from the small power supply unit to the load card power plug, and from the power supply unit to the ISOBAR or main power strip (see Figure F.9).

F.2.13 Attach the Joystick to the PC

Attach the Joystick to the PC joystick port (see Figure F.1).

F.2.14 Attach the Keyboard to the PC

Attach the Keyboard to either the front or back PC keyboard port (see Figure F.1). Plug the mouse cable (from the keyboard trackball) into the PC mouse port.

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[3]Only necessary when the CSM/LFM module is present.

[4]A third BNC Cable is present in this BNC bundle, and would normally connect to the "B/I" port on the CSM/LFM module. This cable is only necessary when calibrating the CSM option. Do not attach the BNC end of this cable to the "B/I" port on the CSM/LFM module. You should, however, attach the coaxial end of this cable to the appropriate coaxial port on the PC card, as indicated by the color code.

[5]Only necessary when the Lateral Force Measurement option is included with your Nano Indenter® XP.

F.2.15 Inflate and Level the Vibration Table

Thread the vibration table hose through the cabinet port and connect one end to the vibration table and the other end to an air supply. Refer to the manufacturer's instructions for the proper procedure for attaching the hose. Inflate and level the vibration table per the manufacturer's instructions.

F.2.16 Remove the Microscope Lock Screw

Use the metric Allen wrenches to remove the microscope lock screw from the top of the gantry. Replace the microscope lock screw with the microscope plug screw to seal the gantry (see Figure F.6).

F.2.17 Attach the Cabinet Top

Attach the top of the isolation cabinet (see Figure F.7).

F.2.18 Insert Microscope Objective

Insert the Microscope Objective into the Microscope. Refer to Section 4.2.1 of this manual for instructions on inserting microscope objectives and adjusting the parcentricity with multiple objectives.

F.2.19 Plug In Power Cords

Attach the power cords to the PC, Monitor (this may be a jumper cable from the PC power output to the monitor), and Printer. Attach the power cords from the PC, Monitor, Printer, Video Monitor, and Fiber Optic Illuminator to the power strip or to a power supply.

F.2.20 Unlock PC Door and Power Up System

The key for the PC door should be in the lock on the PC door. Use this key to unlock the PC door. Press the PC power button to turn on the instrument. Turn on power to the Monitor, Video Monitor, Printer, and Fiber Optic Illuminator.

F.2.21 Remove Indenter Head Lock Pins

Refer to Appendix A of this manual for instructions on removing the pins from the indenter head. Remove the bottom pin from the indenter head. Then remove the top pin from the indenter head.

F.2.22 Enter Manual Control and Adjust Joystick

Once the PC has booted up and the Main Menu of the Nano Software is displayed, enter the Manual Control option (see Sections 2.7.2.1 and Section 10.0). Once the manual control screen is displayed, observe the X-Y tables in the gantry. If the X-Y tables are moving, you may need to center the Joystick. Note the position counters displayed on the manual control screen on the monitor, as these are good indications of very small motion of the X-Y tables. If the tables are moving, use the "E" key on the keyboard to exit manual control. Refer to Section 10.2.3 of this manual for instructions on centering the joystick. If the tables are not moving, there is no need to adjust the joystick. Exit manual control, and proceed to the next step.

F.2.23 Run DAC Calibration

Run a DAC calibration. Refer to Section 11.2.2 of this manual for instructions on running a DAC calibration. If there is a problem or error with the DAC calibration, contact Nano Instruments immediately. If the DAC calibration completes it's operation, save the results and continue to the next step.

F.2.24 Test the Motion Of the Indenter

Enter the "Test Motion of Indenter" routine (see Section 11.2.3). Generate a Standard Test Motion Screen Plot, as described in Appendix E, Section E.6.5.1. Verify that the Test Motion Plot reveals proper indenter operation. If the indenter is not behaving in the proper manner, fax the plot you generated to Nano Instruments or your local representative and wait for further information before proceeding with the assembly process.

F.2.25 Attach Cabinet Front and Adjust

Attach the front of the cabinet. Close the cabinet door to verify that the door closes properly. If the door does not close properly, adjust the clips on the Left and Right panel until the door closes properly (see Figure F.8).

F.2.26 Load Samples Into Sample Tray

Load a standard sample into the Sample Tray. The recommended standard samples are the single crystal aluminum and fused silica samples provided with the Nano Indenter® XP. Refer to Section 3.3.3 of this manual for the proper method of loading samples.

F.2.27 Load Sample Tray Into Instrument

Refer to Section 3.3 of this manual for the proper method of loading the sample tray into the Nano Indenter® XP.

F.2.28 Verify the Height Of the Samples

Enter the manual control routine (see Section 10.0). Verify that the samples are not in contact with the microscope objective. Use the focus controls to verify that the samples are in focus range. Then move the X-Y positioning tables using the joystick, so that the sample tray is moving toward the indenter head in the X direction. Observe the height of the sample levelling arm as it approaches the indenter. If it appears that the sample levelling arm will strike the indenter's protective housing, then the sample height is incorrectly set. If the sample levelling arm will pass underneath the protective housing, then the sample height is properly set. Note that the indenter tip should be retracted inside the housing while you are in manual control. If the indenter tip is exposed, contact Nano Instruments immediately.

F.2.29 Perform a Microscope-To-Indenter Calibration

Refer to 11.2.1 of this manual for the proper method of calibrating the distance between the microscope and indenter.

F.2.30 Perform Standard Test

Perform a standard test on the fused silica sample. Refer to Section 7.0 of this manual for instructions. Examine the results. If you have any questions, contact Nano Instruments, or your local representative. Otherwise, the instrument is ready for use.

F.2.31 Attach The Instrument Nameplate

Once the instrument is fully operational, it is ready for the Nano Indenter® XP nameplate. Attach the nameplate to the isolation cabinet by removing the plastic on the double-sided tape. The nameplate is usually positioned on the left side of the isolation cabinet, directly below the access door's hinge.

F.2.32 Read This Manual

Now that your Nano Indenter® XP is ready for use, it is a very good idea to read Section 1.0 through Section 11.0, and Appendices A through C of this manual.

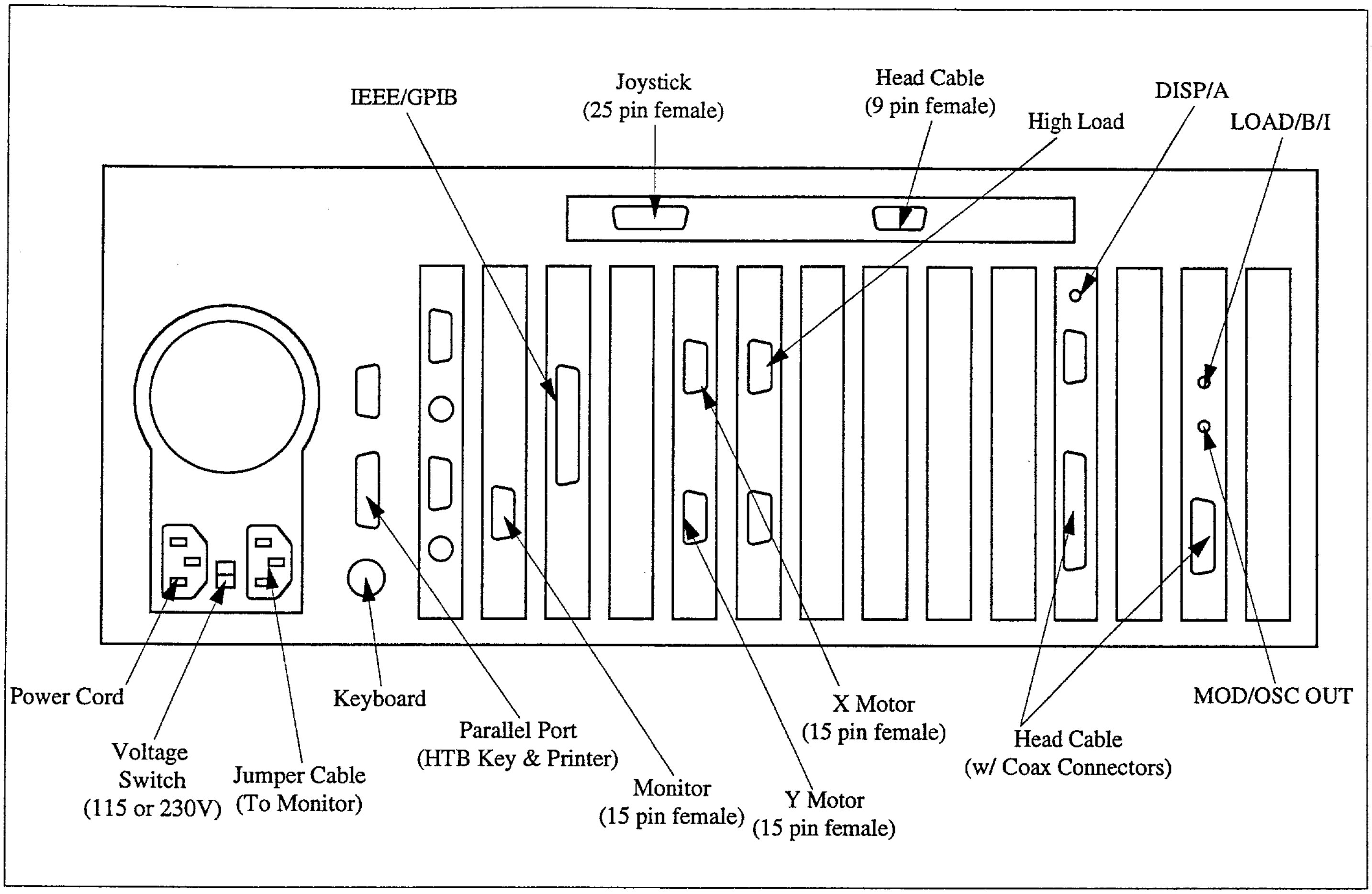


Figure F.1 Rear View Of The PC

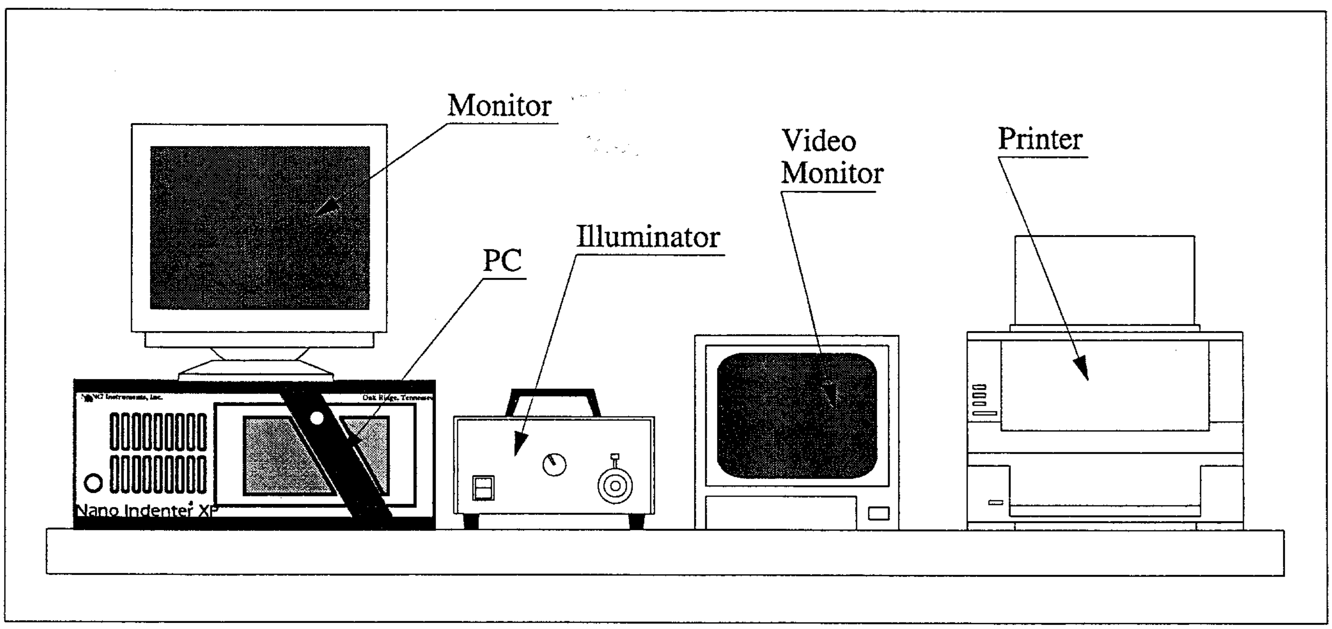


Figure F.2 The Desktop Components Setup On The Operator's Desk

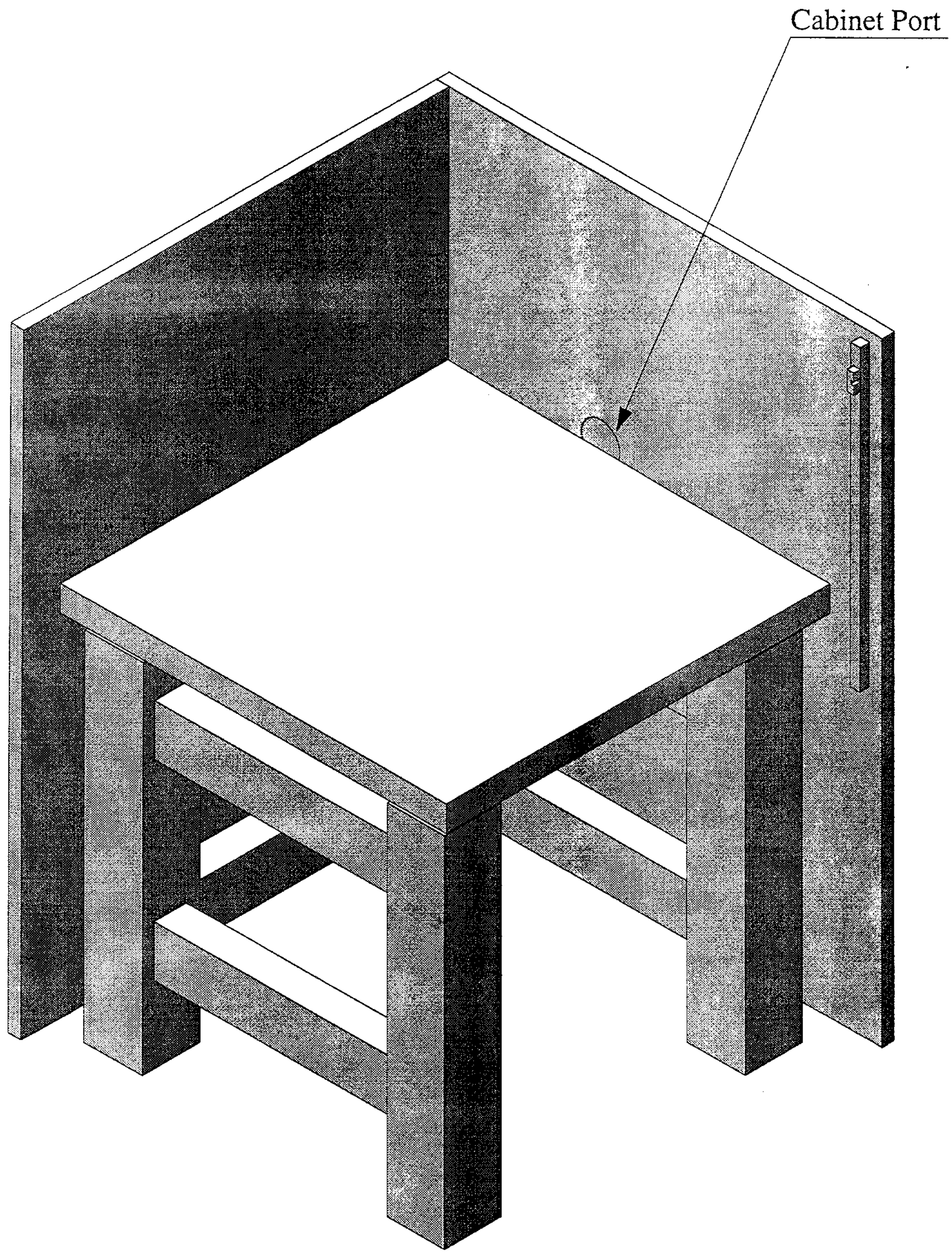


Figure F.3 Assembly Of Two Sides Of The Isolation Cabinet Around The Vibration Isolation Table

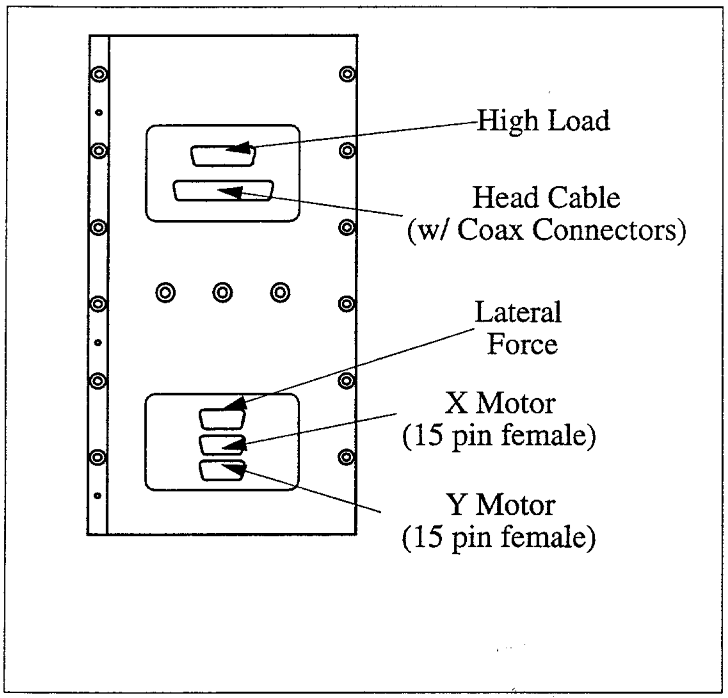


Figure F.4 The Gantry Left Side

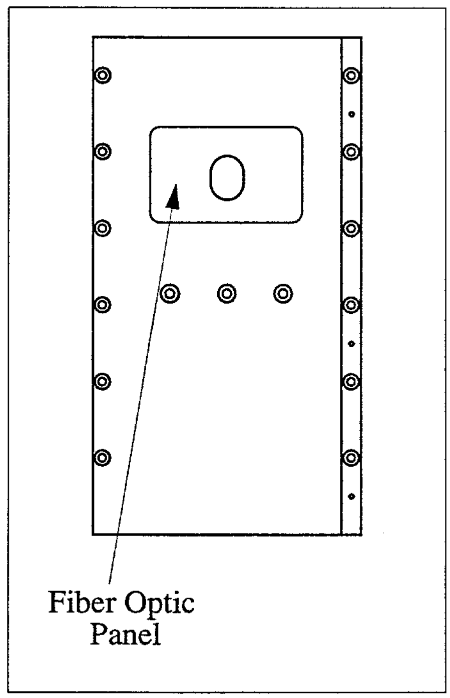


Figure F.5 The Gantry Right Side

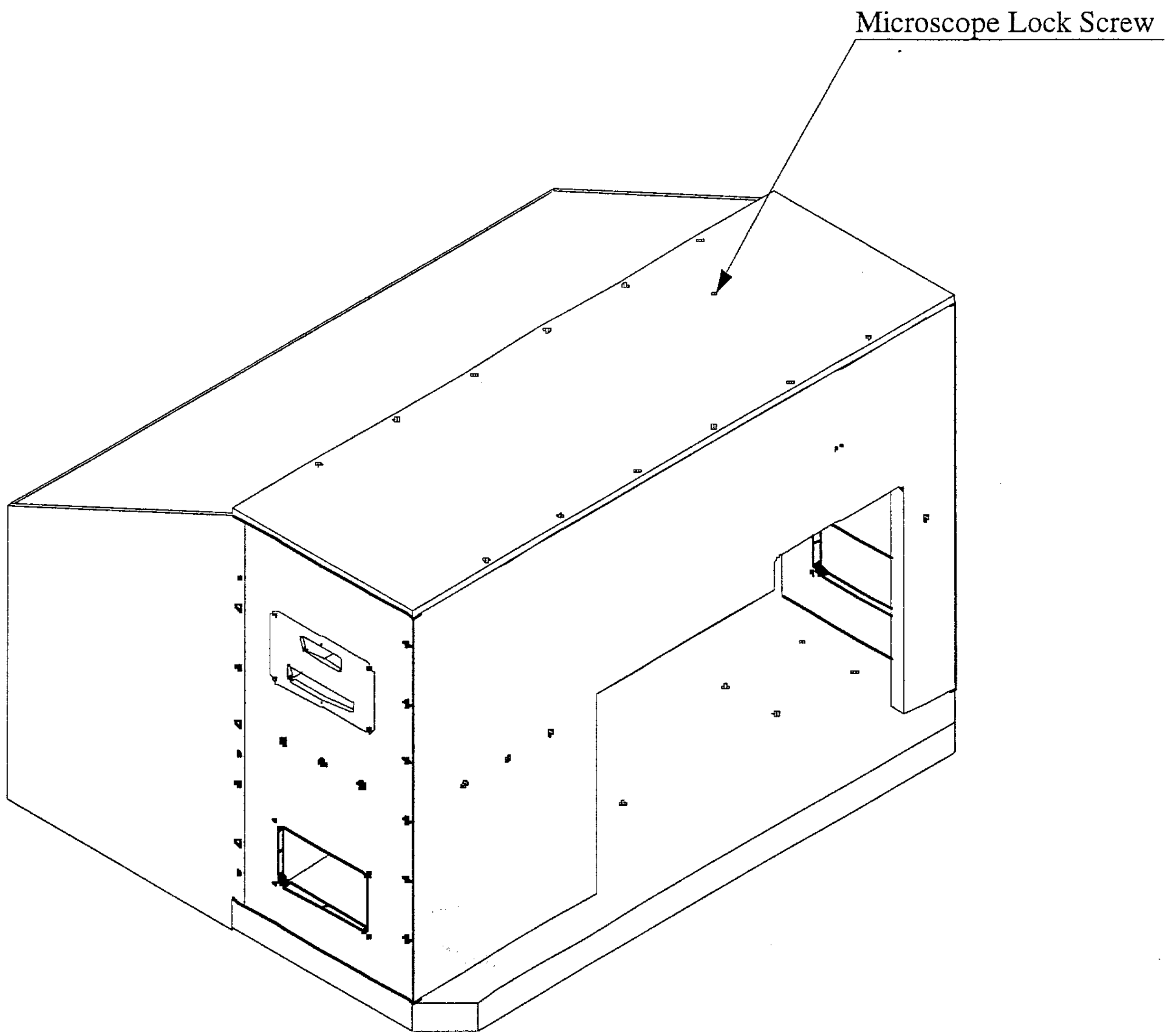


Figure F.6 Location Of The Microscope Lock Screw

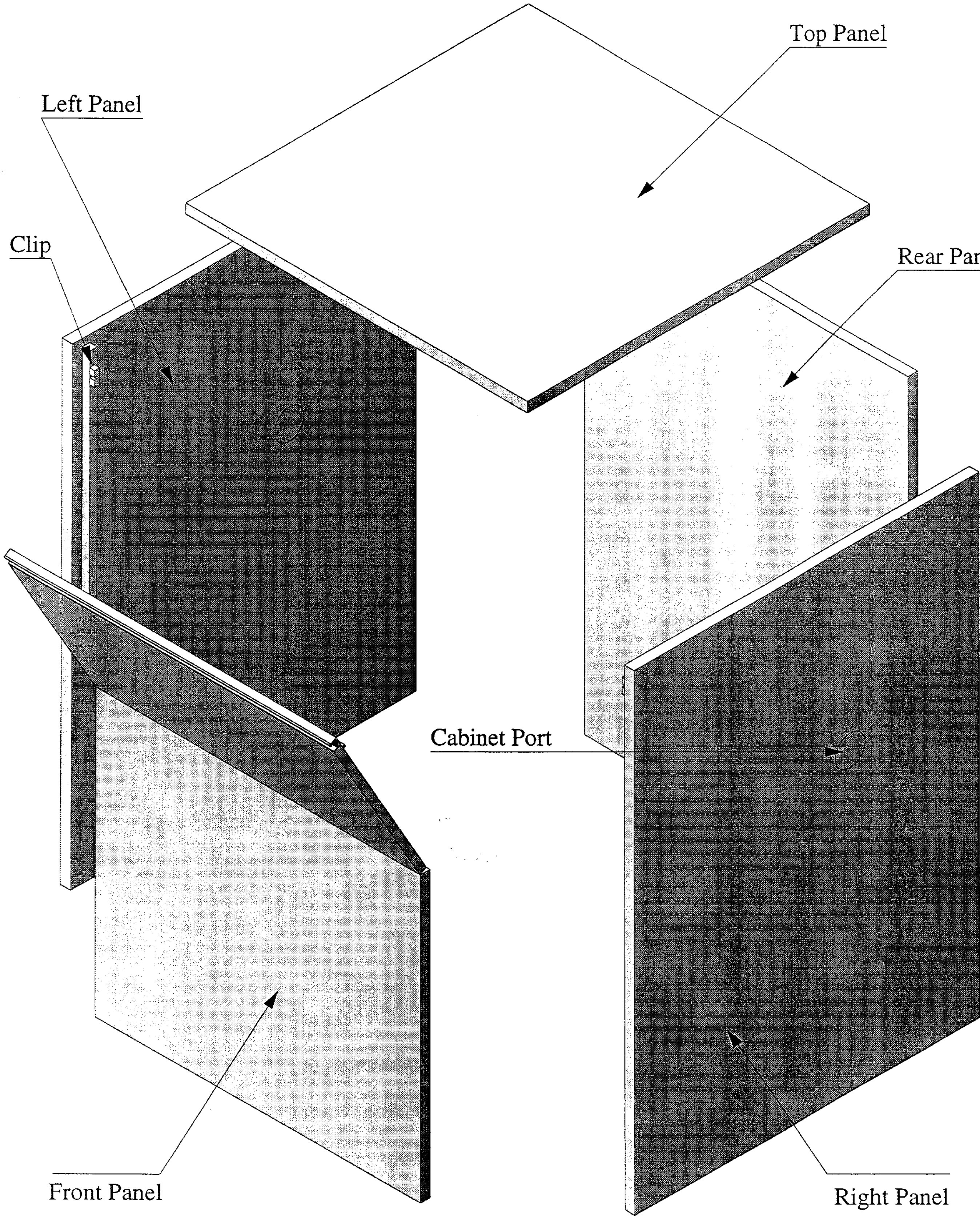


Figure F.7 Expanded View Of The Cabinet Assembly

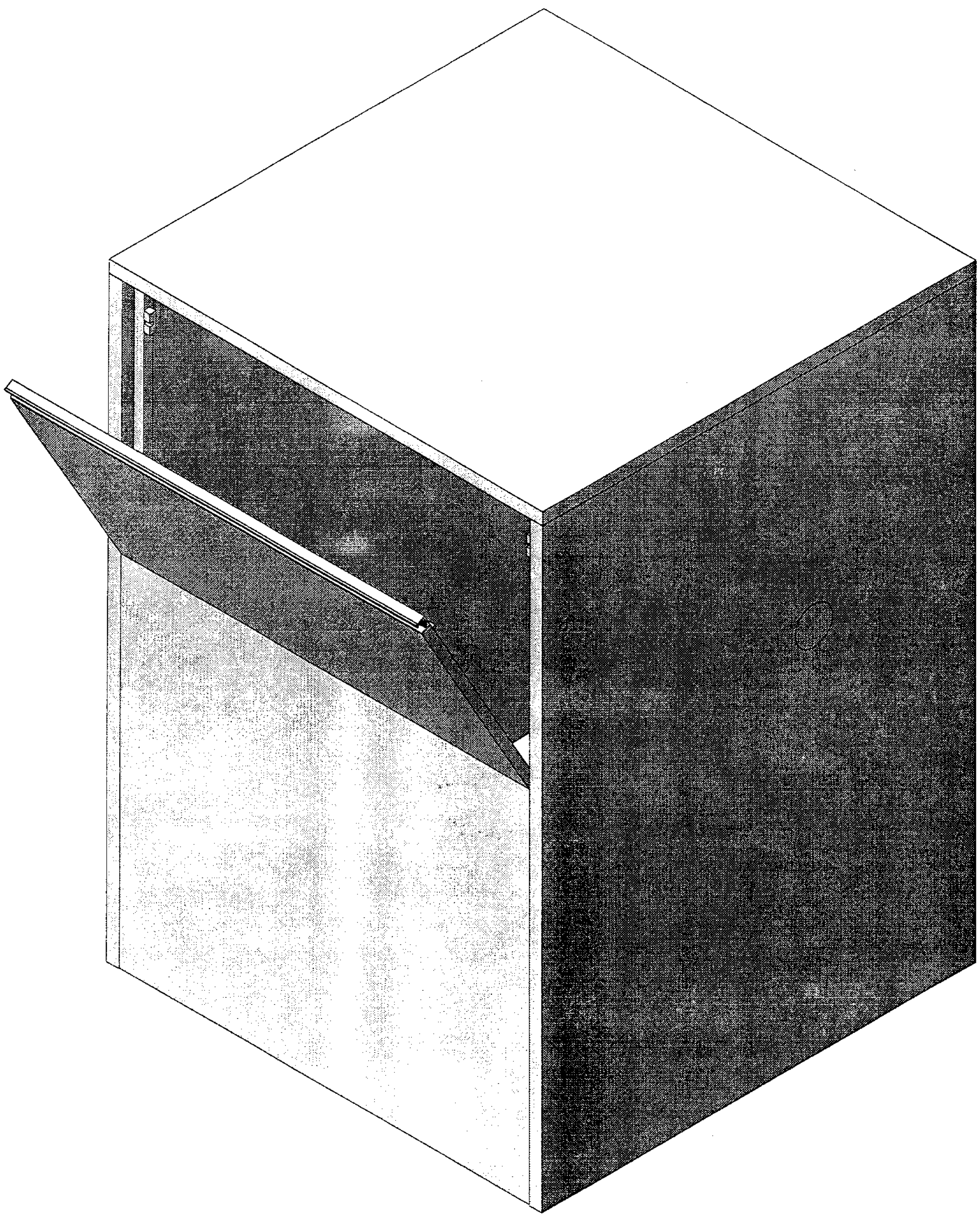


Figure F.8 The Assembled Cabinet

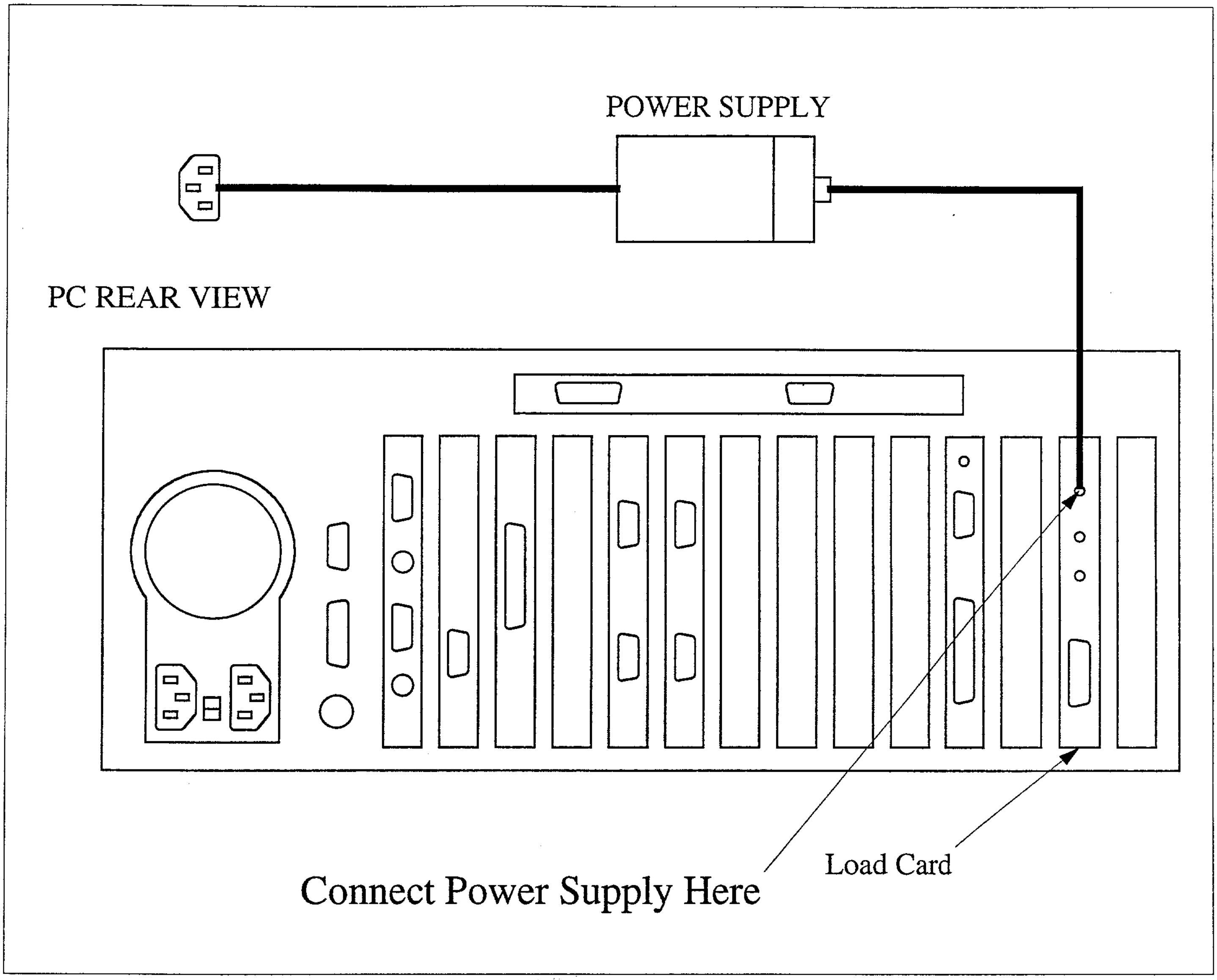


Figure F.9 Rear View Of The PC

Appendix G: Sample Data Reports

G.1 Overview

The following pages show some examples of output data files or sheets. Note that these reports do not relect actual test results. The values listed in these reports are shown only for the purpose of representing the format of the final test reports.

G.2 Sample Data Report

File: MH023002.BIN Date: 1 January 1996 Page:1
Specimen number: 001
Specimen name/Comments: Single Crystal Aluminum

Hardness Experiment No. =1 Target Load 10000 uN
NOTE: ** bracketing indent number indicates data thrown out of average

Indent No.	Depth (nm)	Load (mN)	L/S^2 (nm^2/mN)	Area (nm^2)	Modulus (GPa)	Hardness (GPa)
1	2.50E+03	1.00E+01	2.00E+03	1.40E+08	7.00E+01	2.22E-01
2	2.50E+03	1.00E+01	2.00E+03	1.40E+08	7.00E+01	2.22E-01
3	2.50E+03	1.00E+01	2.00E+03	1.40E+08	7.00E+01	2.22E-01
Average	2.50E+03	1.00E+01	2.00E+03	1.40E+08	7.00E+01	2.22E-01
Std.Dev.	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00

Hardness Experiment No. =2 Target Depth 2000 nm
NOTE: ** bracketing indent number indicates data thrown out of average

Indent No.	Depth (nm)	Load (mN)	L/S^2 (nm^2/mN)	Area (nm^2)	Modulus (GPa)	Hardness (GPa)
4	2.00E+03	7.00E+00	2.50E+02	1.00E+08	7.00E+01	2.22E-01
5	2.00E+03	7.00E+00	2.50E+02	1.00E+08	7.00E+01	2.22E-01
6	2.00E+03	7.00E+00	2.50E+02	1.00E+08	7.00E+01	2.22E-01
Average	2.00E+03	7.00E+00	2.50E+02	1.00E+08	7.00E+01	2.22E-01
Std.Dev.	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00

G.3 Sample Reprinted Data Sheet

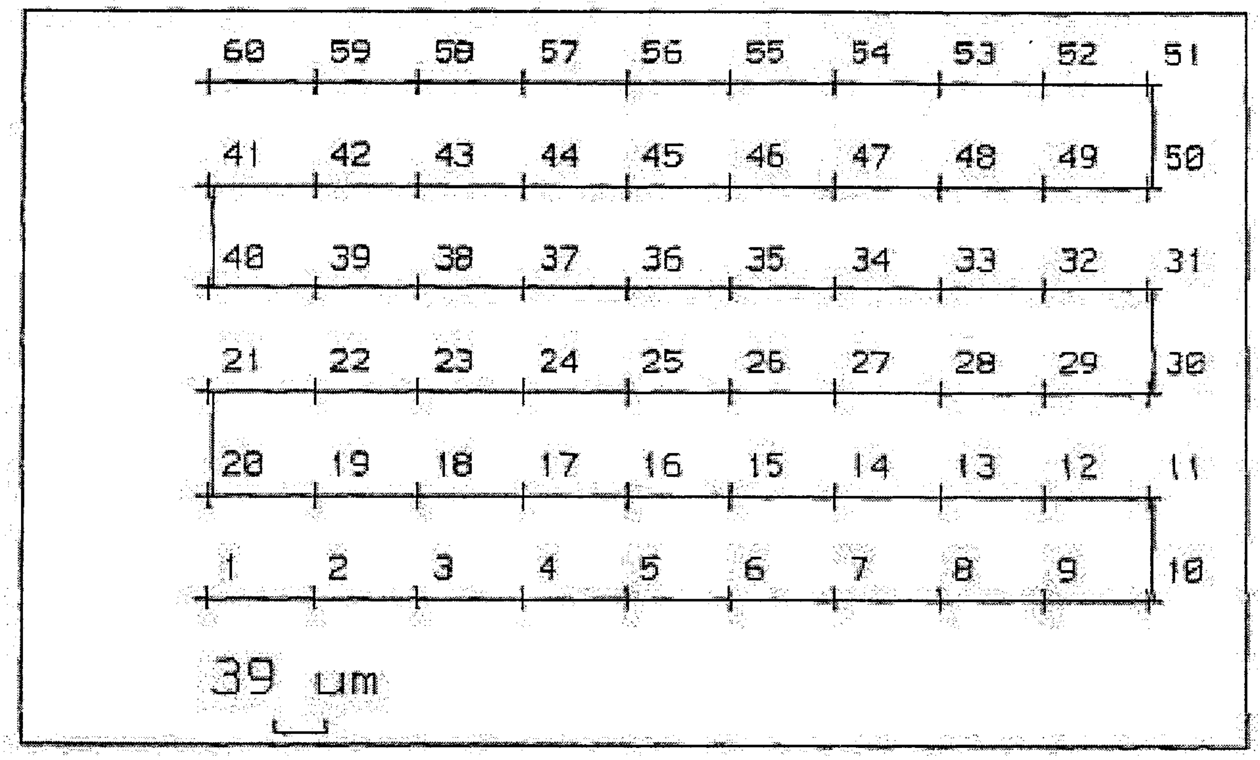
```
******REPRINTED*****DATA SHEET************************************
7 Mar 1997,10:47:15
******************************************************************

Surface search distance (nm)= 200
Radius to initial impact (um)= 50
Angle to initial impact (Deg)= 270
Maximum drift rate prior to tests (nm/sec)= .1
Number of diamond used for these tests = 1

******************Data filing information************************
File base name R_OOC '_' replaced with experiment type code.
Files numbered: 601 to 060 will result from this run.

**********************Specimen information***********************
Specimen number- 1     Specimen name- fused silica
```

```
*******************************************************************
This is the geometric configuration of indents
*******************************************************************
```



These are the exact positions of this geometric configuration

If the experiment type is prefixed 'S' then a surface finding procedure is done prior to begining the experiment.

	Measurements in Microns			
Position Number	x Position	y Position	Exper. Type	Specimen Number
1	0.00	0.00 6	1	1
2	50.00	0.00 6	1	1
3	100.00	0.00 6	1	1
4	150.00	0.00 6	1	1
5	200.00	0.00 6	1	1
6	250.00	0.00 6	1	1
7	300.00	0.00 6	1	1
8	350.00	0.00 6	1	1
9	400.00	0.00 6	1	1
10	450.00	0.00 6	1	1
11	450.00	50.006	1	1
12	400.00	50.006	1	1
13	350.00	50.006	1	1
14	300.00	50.006	1	1
15	250.00	50.006	1	1
16	200.00	50.006	1	1
17	150.00	50.006	1	1
18	100.00	50.006	1	1
19	50.00	50.006	1	1
20	0.00	50.006	1	1
21	0.00	100.00	6	1
22	50.00	100.00	6	1
23	100.00	100.00	6	1
24	150.00	100.00	6	1
25	200.00	100.00	6	1
26	250.00	100.00	6	1
27	300.00	100.00	6	1
28	350.00	100.00	6	1
29	400.00	100.00	6	1
30	450.00	100.00	6	1
31	450.00	150.00	6	1
32	400.00	150.00	6	1
33	350.00	150.00	6	1
34	300.00	150.00	6	1
35	250.00	150.00	6	1
36	200.00	150.00	6	1
37	150.00	150.00	6	1
38	100.00	150.00	6	1
39	50.00	150.00	6	1
40	0.00	150.00	6	1
41	0.00	200.00	6	1
42	50.00	200.00	6	1
43	100.00	200.00	6	1
44	150.00	200.00	6	1
45	200.00	200.00	6	1
46	250.00	200.00	6	1

47	300.00	200.00	6	1
48	350.00	200.00	6	1
49	400.00	200.00	6	1
50	450.00	200.00	6	1
51	450.00	250.00	6	1
52	400.00	250.00	6	1
53	350.00	250.00	6	1
54	300.00	250.00	6	1
55	250.00	250.00	6	1
56	200.00	250.00	6	1
57	150.00	250.00	6	1
58	100.00	250.00	6	1
59	50.00	250.00	6	1
60	0.00	250.00	6	1

```
*****************************************************************
     This is the No. 1 type of experiment that will be performed.
     It was defined under the name: HL 50000 uN
*****************************************************************

Segment:
1 -A
2-LL,5%Rl,50000For
3-H,ORat,15Tim
4-UL,90%Ra,90%un
5-H,ORat,2Log,5OPoi
6-UL,90%Ra

*****************************************************************
     This is the No. 2 type of experiment that will be performed.
     It was defined under the name: HL 500000 uN
*****************************************************************

Segment:
I -A
2-LL,5%Rl,500000For
3-H,ORat,15Tim
4-UL,90%Ra,90%un
5-H,ORat,2Log,5OPoi
6-UL,90%Ra

*****************************************************************
     This is the No. 3 type of experiment that will be performed.
     It was defined under the name: HD 500 nm
*****************************************************************

Segment:
1 -A
2-LD,5%Rl,500Dep
3-H,ORat,15Tim
4-UL,50%.Ra,90%un
5-H,ORat,2Log,5OPoi
6-UL,90%Ra

*****************************************************************
     This is the No. 4 type of experiment that will be performed.
     It was defined under the name: HD 1000 nm
*****************************************************************

Segment:
1 -A
2-LD,5%Rl,1000Dep
3-H,ORat,15Tim
4-UL,50%Ra,90%un
5-H,ORat,2Log,5OPoi
```

```
6-UL,90%Ra


*****************************************************************
     This is the No. 5 type of experiment that will be performed.
     It was defined under the name: SD 500 nm
*****************************************************************

Segment:
1-A,2Con
2-LS,.05Rat,50ODep
3-H,ORat,15Tim
4-UL,70%Ra,80%un
5-H,ORat,2Log,5OPoi
6-UL,IOO%Ra


*****************************************************************
     This is the No. 6 type of experiment that will be performed.
     It was defined under the name: SD 2500 nm
*****************************************************************

Segment:
1-A,2Con
2-LS,.05Rat,250ODep
3-H,ORat,15Tim
4-UL,70%Ra,80%un
5-H,ORat,2Log,5OPoi
6-UL,100%Ra
```

Appendix H: File Structure

H.1 Overview

Data files that have been created or manipulated by the Nano Software are always stored in the appropriate subdirectories. These subdirectories are transparent to the user, unless there is a need to identify or move a specific file. Following is the file structure for data file storage on the hard drive.

H.2 File Structure

C:\XPCODE
- INDENLIB — *Experiment Library*
- EXPLIB — *Test Definition Library*
- XPCALS — *Calibration Storage*
- SHAPELIB — *Stored Shape Library*
- XPDATA — *Generated Data Files*
    - RAW — *Raw Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - DLT — *Load/Displacement/Time Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - STF — *Stiffness Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - MOD — *Hardness/Modulus Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - AVG — *Averaged Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - BRK — *Break Point Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - RZR — *Razor Edge Test Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - ELA — *Hardness/Elastic Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*
    - PLA — *Hardness/Plastic Data Files*
        - BIN — *Binary*
        - TXT — *ASCII*

Appendix I: Updates

I.1 Overview

Updates to these instructions should be inserted following this header page.

Appendix J: Technical References

The following list of technical references comprises a very small number of the total available literature associated with the Nano Indenter®. If you are unable to find one of the references listed on this page, or if you are interested in technical topics not addressed in this brief list, contact Nano Instruments or your local representative.

J.1 An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments

W.C. Oliver, G.M. Pharr, J. Mater. Res., Vol. 7, No. 6, June 1992

J.2 Spatially Resolved Mechanical Properties of a "TPO" Using a Frequency Specific Depth-Sensing Indentation Technique

B.N. Lucas, W.C. Oliver, A.C. Ramamurthy, SPE ANTEC '97, Toronto, Ontario, Canada

J.3 Time Dependent Deformation During Indentation Testing

B.N. Lucas, W.C. Oliver, G.M. Pharr, J-L. Loubet, MRS Symposium Proceedings, Vol. 436: Thin Films: Stresses and Mechanical Properties VI, April 1996

J.4 Some Measurements of Viscoelastic Properties with the Help of Nanoindentation

J-L. Loubet, B.N. Lucas, and W.C. Oliver, Extended Abstracts, Proceedings of the Workshop on Indentation Techniques, ICMCTF-95, San Diego, CA, 1995.

J.5 Measurement of Fracture Toughness in Thin Films and Small Volumes Using Nanoindentation Methods

G.M. Pharr, D.S. Harding, W.C. Oliver, Materials Research Society Symposium Proceedings, Materials Research Society Fall Meeting, 1994.

J.6 Understanding and Quantification of Elastic and Plastic Deformation during a Scratch Test

V. Jardret, H. Zahouani, J.L. Loubet, T.G. Mathia

J.7 Mechanical Characterization of Ultra-Thin, Hard-Disk Overcoats Using a Reciprocating Wear Test and Depth-Sensing Indentation

J.L. Hay, R.L. White, B.N. Lucas, and W.C. Oliver, MRS Fall Meeting, Boston, MA, Dec., 1997

J.8 Method For Continuous Determination of the Elastic Stiffness of Contact Between Two Bodies

Warren C. Oliver, John B. Pethica, United States Patent Number 4,848,141

Appendix K: Variable Definitions

K.1 Variables By Mode Of Operation

Depending on the type of test being performed, and the configuration of the system (what type of options are present, if any), the variables acquired during a test will change. The following sections list the variables associated with different types of tests. Note that these variables are cumulative. That is, if a test is performed on an instrument with the CSM option and the High Force option, the Basic variables, as well as the variables for these options will be acquired.

K.1.1 Basic Variables

Following are the variables associated with the Basic instrument (the variables acquired even if no options are present on the instrument).

Basic Variables			
Variable Code	**Variable Name**	**Raw Units**	**Units**
Load	Load	V	μN
Displ.	Displacement	V	nm
Time	Time	s	s

Table K.1 Basic Variable Definition

K.1.1.1 Load

The Load variable refers to the applied force used during an experiment.

K.1.1.2 Displacement

The Displacement variable refers to the displacement achieved during an experiment.

K.1.1.3 Time

The Time variable is a time reference for each load/displacement data point collected during an experiment. The variable is recorded in terms of time into the test.

K.1.2 Continuous Stiffness Measurement Option Variables

Following are the variables associated with the Continuous Stiffness Measurement option.

CSM Option Variables			
Variable Code	**Variable Name**	**Raw Units**	**Units**
Amplit	Amplitude	V	-
Phase Deg.	Phase Angle	Degrees	-
Excite	Excitation	V	-

Table K.2 CSM Option Variable Definition

K.1.2.1 Amplitude

The amplitude variable refers to the amplitude of displacement oscillation recorded during a continuous stiffness experiment.

K.1.2.2 Phase Angle

The phase angle variable refers to the phase difference between the input (force) and output (displacement) oscillations as recorded during a continuous stiffness experiment.

K.1.2.3 Excitation

The excitation variable refers to the input or applied (force) excitation used during a continuous stiffness experiment.

K.1.3 Tangential Force Measurement Option Variables

Following are the variables associated with the Tangnetial Force Measurement option.

TFM Option Variables			
Variable Code	**Variable Name**	**Raw Units**	**Units**
Xforce	X Lateral Force	V	µN
Yforce	Y Lateral Force	V	µN
Xpos Tb	X Table Position	µm	µm
YPos Tb	Y Table Position	µm	µm

Table K.3 TFM Option Variable Definition

K.1.3.1 X & Y Force

The X and Y Force variable contain different data before and after data reduction. When raw data is acquired, the X & Y Force variables refer to the lateral deflection of the indenter shaft in terms of the voltage output of the lateral proximity sensors.

Once data is manipulated, however, the X & Y Force variables refer to the actual lateral force on the indenter shaft, as calculated through the lateral displacement and the known lateral stiffness of the indenter shaft.

K.1.3.2 X & Y Position

The X & Y Position variables refer to the table position as recorded through the motion system of the Nano Indenter® XP. These variables can be referred to as the position in the X & Y directions along the scratch.

K.1.4 High Force Option Variables

There are no unique variables associated with converted High Force Option data.

K.2 Variables By Manipulation Routine

As data is processed through the various manipulation routines, additional variables may be created. The following sections describe variables created by the different routines.

K.2.1. Load Displacement Time

There are no new basic data variables created in the Load/Displacement/Time routine (see Section 9.4.7.1). Table K.4 displays the input and output variables for this routine.

Load/Displacement/Time Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	V	Load	Load	µN
Displacement	V	Displacement	Disp.	nm
Time	s	Time	Time	s
Amplitude&Excitation	V	Stiffness	S.	N/m
Phase Angle	Deg.	Stiffness	Dw	N/m
Phase Angle	Deg.	Tangent(Phase Angle)	Tan Phi	
X Force	V	X Lateral Force	Xforce	µN
Y Force	V	Y Lateral Force	Yforce	µN
X Tip Position	µm	X Tip Position	Xtip	µm
Y Tip Position	µm	Y Tip Position	Ytip	µm

Table K.4 The Load Displacement Time variable list.

Note that Table K.4 shows the variables associated with Basic Data, Continuous Stiffness Measurement data, Tangential Force Measurement data, and High Force data.

Note that continuous stiffness data is converted when the "D" file is created. The Amplitude of oscillation, Excitation voltage, and Phase Angle are used to generate Stiffness and Phase Difference data (see Section 17.0).

In addition, if tangential force measurement data is present in the "R" file, the X and Y Force variables will be converted from a voltage reading of indenter shaft deflection into X & Y lateral force variables (see Section 18.0).

K.2.2 Stiffness From Unloading

The Stiffness From Unloading routine uses load, displacement, and time data to generate the stiffness of contact from the unloading segment of an experiment. Refer to Section 9.4.7.2 for more information about this routine.

Stiffness From Unloading Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	µN	Load	Load	mN
Displacement	nm	Displacement	Disp.	nm
Time	s			
		Stiffness	Stif.	N/m

Figure K.5 The Stiffness From Unloading variable list.

K.2.3 Hardness/Modulus/Depth

The Hardness/Modulus/Depth routine calculates the hardness and modulus from load, displacement, and stiffness data. In addition to generating the hardness and modulus, the contact depth, area of contact, compliance, and several other variables are created See Section 9.4.7.3 for more information about this routine.

Hardness/Modulus/Depth Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	mN	Load	Load	mN
Displacement	nm	Displacement	Disp.	nm
Stiffness	N/m	Stiffness	S	mN/nm
		Load/Stiffness^2	L/S^2	mN/nm^2
		Contact Depth/Displacement	C.D./DISP	
		Contact Depth	Cont.D.	nm
		Area	A	nm^2
		Modulus of Elasticity	E(F)	GPa
		Hardness	H(F)	GPa
		Inverse Square Root of Area	1/SQRT(A)	nm
		Compliance	C	nm/mN
		Area for Constant Modulus	A(E)	nm^2

Figure K.6 The Hardness/Modulus/Depth variable list.

K.2.4 Hardness/Displacement/Elastic

The Hardness/Displacement/Elastic routine generates the area of contact, contact depth and Hardness from load, displacement and time data. Refer to Section 14.4.7.5 for more information on this routine.

Hardness/Displacment/Elastic Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	µN	Load	Load	mN
Displacement	nm	Contact Depth	Cont.D	nm
Time	s	Time	Time	s
		Area	Area	µm^2
		Hardness	Hard.	GPa

Figure K.7 The Hardness/Displacement/Elastic variable list.

K.2.5 Hardness/Displacement/Plastic

The Hardness/Displacement/Plastic routine generates the area of contact and Hardness from load, displacement and time data. Refer to Section 14.4.7.4 for more information on this routine.

Hardness/Displacment/Plastic Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	µN	Load	Load	mN
Displacement	nm	Displacement	Disp.	nm
Time	s	Time	Time	s
		Area	Area	μm^2
		Hardness	Hard.	GPa

Figure K.8 The Hardness/Displacement/Plastic variable list.

K.2.6 Hardness/Modulus From Lock-In

Although the input variables for the Hardness/Modulus from Lock-In are different than those typically used in the Hardness/Modulus/Depth (in that the continuous stiffness data is used in the Hardness/Modulus From Lock-In routine), the output data is the same. See Section 17.2.1 for more information on this routine.

K.2.7 Average Data

Average data output depends upon the input data. The number of variables from the input file that are included in the output file depends on the user's selection of output variables. See Section 9.4.8.3 for more information about this routine.

K.2.8 Break Points

Break Point data output depends upon the input data and the selected variables for break point searching. The number of variables from the input file that are included in the output file depends on the user's selection of variables. See Section 14.4.10.2 for more information about this routine.

K.2.9 Standard Scratch Friction & Profile

The Standard Scratch Friction & Profile routine generates the scratch profile, vectoral and perpendicular forces, vector position and coefficient of friction data from load, displacement, X&Y Force, and X&Y position data. Refer to Section 18.8.1.2 for more information on this routine.

Standard Scratch Friction & Profile Variables				
Input Variable(s)	**Units**	**Output Variable**	**Code**	**Units**
Load	V	Load	Load	µN
Displacement	V	Profile	Prof.	nm
Time	s	Time	Time	s
X&Y Forces	µN	Perpendicular Force	perp.F.	µN
X&Y Forces	µN	Vectoral Force	vect.F.	µN
X Tip Position	µm	X Tip	Xtip	µm
Y Tip Position	µm	Y Tip	Ytip	µm
X&Y Tip Positions	µm	Vector Position	Vpos	µm
Load, X&Y Forces	µN	Coefficient Of Friction	Coef.Fric.	

Figure K.10 The Standard Scratch Friction & Profile Variable List

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