Reset

Shift/reset

F2 — [MSI:,700,0,1] [Return]

F6 — [LOAD "MASTER"] — type this

F3 — [RUN]

Operating Instructions for the Mechanical Properties Microprobe

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PREFACE

About this Manual

This manual is not designed to be read straight through without reference to the Nano Indenter itself. Instead it is intended to be an adjunct of the extensive on-screen help available in the Nano Indenter program. The manual does contain a description of the instrument and technical explanations of how it works (Chapters 1 and 2). You don't absolutely have to read these chapters, but you will find it easier to appreciate the physical significance of the indenter operations if you do.

If you are an expert on H-P. computers, skip Chapter 3. On the other hand, if you are a novice, Chapter 3 ought to provide just enough instruction on running the computer to allow you to concentrate on running the indenter instead. Of course, the indenter comes with a full complement of H-P operating manuals that will provide all the instruction you want — and more!

Chapter 11 is also worth reading. It is a miscellaneous collection of hints and tips for making the indenter run smoothly. The Nano Indenter software is not without its idiosyncrasies, and Chapter 11 may contain an explanation or a fix for problems that otherwise seem without solution.

The "meat" of the manual is to be found in Chapters 4 through 10. The indenter program provides much on-screen help , and the material in these chapters enlarges on this help and provides explanations of the methods of operation and data manipulation employed in the software. These chapters are probably most helpful if you read them as you proceed to set up and run indent experiments and as you take advantage of the calculational abilities of the software.

Examples of the output available from the indenter programs are shown in the Appendix A. Sample data sets are presented both as plots and in tabular form. Also included in Appendix B is a discussion of the IEEE-488 interface from a programmer's perspective. If you know something about H-P Basic, this material will be of interest. Appendix C summarizes the data structure used in the Nano Indenter software.

The Nano Indenter software is menu driven. The Master Menu contains four entries, each of which is the subject of a chapter in this manual (Chapters 5 through 8). Selecting any one of these items brings up a submenu, and secondary submenus are brought up in response to choices in the primary submenus. The user is led through this "maze" by a series of prompts. This manual contains copies of all the menus and most of the prompts, and in an effort to minimize the confusion on the part of the reader, icons have been used to designate different levels of menus in Chapters 5 - 9. Thus in each chapter the components of the first submenu that appears when the subject of that chapter is chosen from the Master Menu are designated with a single "bullet" ("•"). Second level submenus are indicated by two bullets ("••"), third level by three ("•••") and so on.

Jumping In

The on-screen help in conjunction with this manual makes the operation of the Nano Indenter something that you can "learn by doing." The indenter is indeed a sensitive instrument — after all, it can measure distances smaller than a nanometer — yet it is a remarkably sturdy and simple instrument. As long as you avoid the few really serious no-nos listed below, it is unlikely that you can really damage the indenter. In short, if you will exercise just a little care,you can "jump in" and "learn by doing!"

- **Never allow the indenter to be dragged across a specimen surface.** Always be sure that the specimen surface is smooth and that the specimen is not tilted in the specimen holder.

- **Never allow the indenter to be struck from the side.** Always make sure that the indenter is high enough so that it is well above the plane of the sample when the sample is moved from the microscope to the indenter.

- **Avoid accidentally hitting the indenter when positioning the specimen holder on the Table.** It is good practice to insert the specimen holder into its mount from the right hand side of the instrument, i.e., from the side of the mount opposite the indenter.

- **Never tighten the sample mount with the indenter on or near the sample surface.** The best technique when inserting the sample holder into its mount on the Table is to do so when the mount is under the microscope, i.e., on the right hand side of the Table.

- **Do not attempt to lower the indenter onto a specimen surface manually.** Let the computer handle this delicate job unless you really know what you are doing. The displacement measuring system is made of glass. If the indenter head is run down toward the sample surface until the indenter itself is against its top stop, the system can be destroyed.

- **Never unplug any part of the system from another with power applied.** Several parts of the system will be damaged if connecters are removed with power applied to the mains. Always unplug the system from the wall socket prior to disconnecting cables.

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Chapter 1

GENERAL DESCRIPTION OF THE INSTRUMENT

The Mechanical Properties Microprobe or the Nano Indenter is a special microhardness tester capable of operating at loads in the microgram range. The instrument consists of three major components: 1) the indenter, 2) an optical microscope, and 3) a precision table that transports the specimen between the the microscope and the indenter. Unlike conventional hardness testers, it is not necessary to determine optically the area of an indent in order to calculate hardness. Instead, the position of the indenter relative to the surface of the specimen is constantly monitored, thus allowing the depth of an indent to be determined. The area of the indent is then calculated from a knowledge of the geometry of the tip of the diamond indenter. The fact that the position of the indenter can be determined to ± 0.2 nm means that the indenter can be used to sample very small volumes of material. Typical applications include the characterization of thin films, near surface properties, and the different phases in a multiphase material. The instrument is also useful in studies of composite materials and in the determination of interfacial strengths in such materials.

The Nano Indenter is essentially a load controlled system. However, a wide variety of indentation parameters may be prescribed, and through the use of feedback techniques, displacement controlled experiments may also be performed. Under both load control and displacement control conditions, load and displacement are measured continuously so that hardness can be determined as a function of indent depth on the basis of a single indent.

The Indenter

The indenter consists of a rod attached at its center to the middle plate of a special three-plate capacitive sensor. Movement of the capacitor plate is used to produce the displacement of the indenter. The total movement possible (i.e., the physical gap of the capacitor) is 100 µm. The depth resolution of the system is approximately ± 0.2 nm.

At the bottom of the indenter rod is a three-sided diamond pyramid ground so that the sides of the pyramid make an angle of 65.3° with the normal to the base. Thus the indents appear as equilateral triangles, and the length of a side of an indent is approximately 7.4 times its depth. The depth of an indent is related to the diameter of the circle that circumscribes the triangle the indent makes on a surface by the expression:

$$h = 0.113d,$$

where h is the depth of the indent and d is the diameter of the circumscribed circle.

A coil is attached to the top of the indenter rod and is held in a magnetic field. Thus, passage of a current through the coil can be used to apply a force to the indenter. There are two load ranges, 0 to 120 mN (0 to 12 gm) and 0 to 20 mN (0 to 2 gm). The load resolution for the more sensitive range is approximately ± 0.2 μN (± 20 μgm).

The gross vertical position of the entire indenter head is controlled by a motorized micrometer, referred to as the "Z motor." The Z motor may be operated either from the console or by a manual control box. Details of the operation of the manual control box are discussed in the Section on Electronics Cabinet below, and use of the computer console for this purpose is discussed in Chapter 8.

The Microscope*

The position of an indent on a specimen is selected using the optical microscope that is built into the system. A TV camera mounted atop the microscope permits the image of the specimen to be viewed remotely. The microscope features three turret-mounted objective lens that in combination with the eyepiece and the additional magnification produced by the TV camera provide magnifications of approximately 130, 270, and 2000 X on the TV screen. Both turret rotation and focussing are done remotely from the computer console, although it is possible to carry out both operations manually.

In systems without the remote option features, the specimen may be viewed directly through the eyepiece of the microscope. The available magnifications are then 100, 200, and 1500 ×. The microscope may be focussed with a focus knob mounted on the top of the enclosure, but the enclosure must be opened to change the objectives.

The Table

The specimens are held on an X-Y table whose position relative to the microscope or the indenter is controllable from the console. The spatial resolution of the position of the table is 0.1 μm, and its position is reported on the CRT as x and y coordinates in units of micrometers. The zero is arbitrary, and x and y values may be set to zero at any table position. This capability is useful in specifying the

* The Nano Indenter uses a microscope manufactured by Olympus. A number of additional attachments for the microscope can be obtained directly from Olympus. These options include other objective lenses (the nosepiece has mounting positions for 5 lenses) and interference contrast capabilities.

positions of a series of indents by specifying their x and y values relative to the user-selected zero point.

The specimen holder is a rectangular metal plate with four 1.25 in. diameter holes drilled in it. The hole size was chosen to match the dimensions of a standard metallographic mount. Samples may either be mounted metallographically or glued to special metal disks. Allen screws hold the specimens in place. The specimen holder fits under brackets attached to the Table and is held firmly in place by a screw that can be tightened against the bottom of the holder.

The Indenter Cabinet

The three components of the Nano Indenter described above are inclosed in a heavy wooden cabinet whose major purpose is to ensure the thermal stability of the samples. A hinged leaf on the front of the cabinet provides access to the indenter, and a small exit hole in the top of the cabinet permits the eyepiece of the microscope to be outside the cabinet. A light source for the microscope is also located outside the cabinet. The indenter itself, but not the cabinet, is suspended on a pneumatic vibration table to isolate it from building vibrations. As a further precaution against rapid temperature changes, it is recommended that the indenter cabinet itself be placed in a small "Isothermal Room" in which the temperature is controlled to ±0.5°C. Users should make sure that the door to such a room is kept closed and should take normal precautions to ensure that the temperature of the indenter environment remains constant

The Keyboard

A standard Hewlett Packard keyboard controls all the functions of the indenter including the focussing of the microscope (remote option), the movement of the specimen stage, and the selection of indent locations.

The Electronics Package

The electronic controls for the indenter are contained in a metal cabinet on the table to the right of the keyboard. A number of indicators including the displays of two digital voltmeters (DVMs) are mounted on the front face of the cabinet. These DVMs monitor the voltage supplied to the loading coil and to the displacement sensor. Many of the actions of the indenter are reflected in the readings of these instruments and indicators. However, with the exception of three buttons, a user can run the Nano Indenter without reference to these displays. The three exceptions are the amber "Scram" Switch" at the lower right front of the Cabinet, the green, square "Standby" button above the Displacement DVM, and the amber, square button at the front left of the cabinet above the Load DVM (see Fig. 1.1).

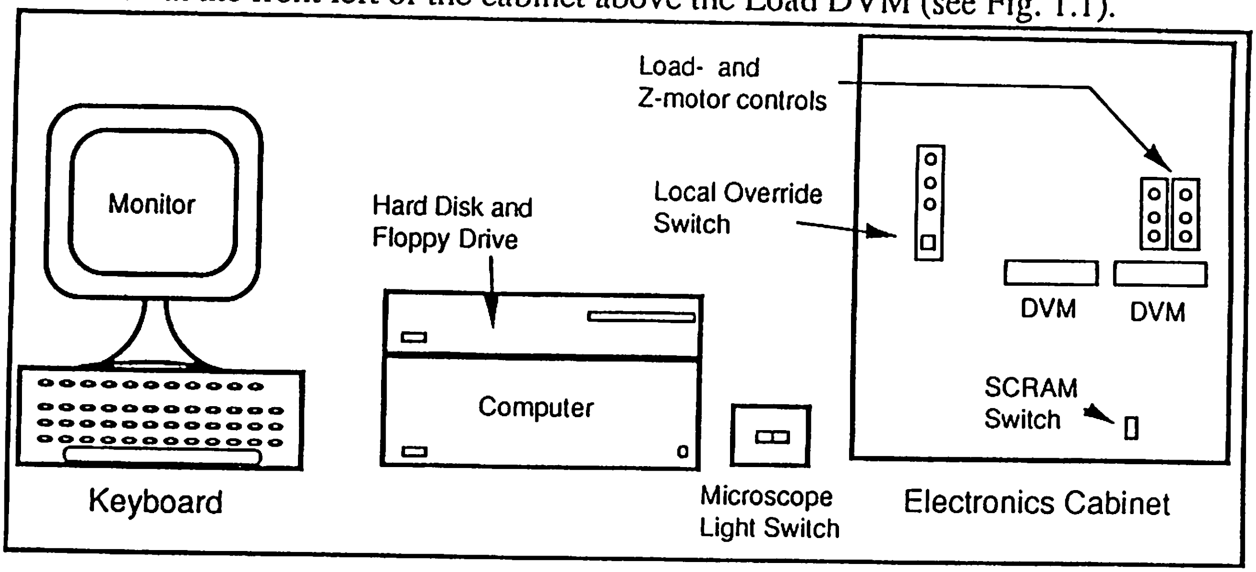


Fig. 1.1. Schematic drawing of the control area for the Nano Indenter.

The Scram switch is the master power control switch for all of the components of the indenter. This switch should be should be turned off only when the Nano Indenter is to be out of service for an extended period of time, e.g., over a weekend. However, in an emergency (most unlikely) it can serve as the "Scram Button" for the system. Turn it off, and all the power to the system is turned off immediately.

The Standby button controls power to the indenter coil and should be depressed when the system is left turned on but idle for a period of a day or so. If the red "Remote" light (just above the Local Override Button) is on at the time the Standby switch is depressed, the Standby switch will blink continuously.

The "Local Override Button," which is the square, amber button located on the left of the front face of the cabinet just above the Load DVM,is probably the most used of the three buttons . If the "Remote" light (the red light just above the Local Override Button) is on, depressing this button causes load and displacement control of the indenter to be transferred to the operator. (The Override Button begins to blink to indicate this condition.) A user should never have occasion to use this button except to raise the indenter head, i.e., control the "Z Motor." That operation is carried out by depressing the square, amber, "UP" or "DOWN" buttons on the manual control.box (see Fig. 2), which is the small black cylindrical device located on top of the TV monitor. Simultaneously depressing the "FAST" button and the

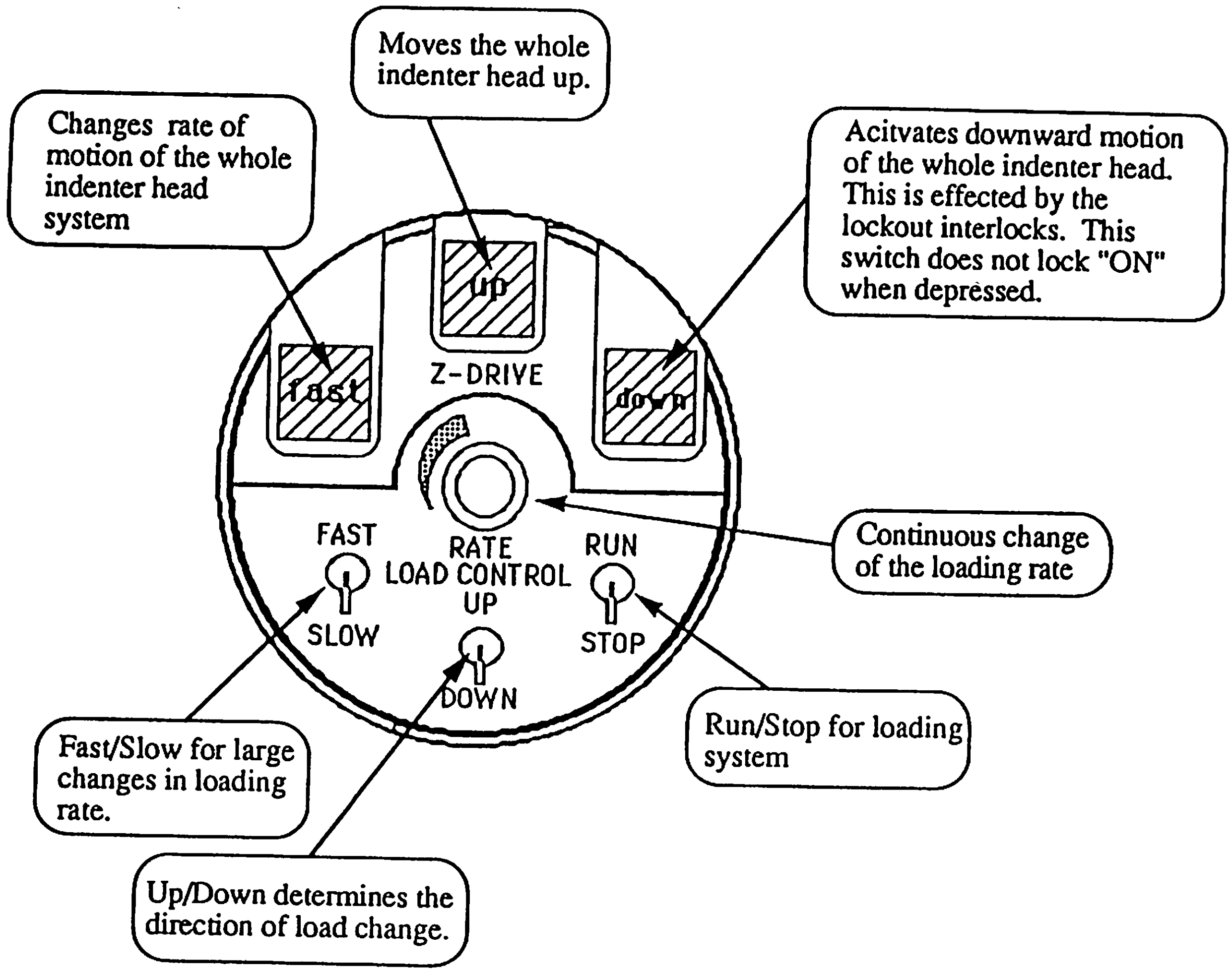


Fig. 1.2. Schematic diagram of the Manual Control Box. The controls in the upper half of the diagram move the entire indenter up and down with the Z motor. The lower controls affect the load applied through the coil. These controls move the indenter only (not the entire head) up and down as long as the indenter is not at its end stops.

"UP" or the "DOWN" button produces a more rapid response from the Z Motor. Note that the FAST and the UP buttons toggle on and off and thus must be depressed a second time to turn off the action of the Z Motor. The DOWN button turns off automatically when released. After the desired movement of the indenter head is completed, the Local Override Switch on the front of the Electronics Cabinet must be toggled to return to remote control. (Note:— If the remote light is not burning, strictly speaking, it is not necessary to depress the Local Override Button in order to activate the Z-Drive and Load Control Unit. However, it is recommended that the user do so each time before using the Z-Drive and Load Control Unit simply to make absolutely certain that the unit is energized when it is expected it to be, and that the indenter head really moves when it is expected to move. Otherwise, the indenter could be at a lower height than expected and be struck from the side by the specimen as the specimen is being moved into position for an indent.)

Do not change the positions of the switches on the Load Control side of the Z-Motor and Load Control Unit. These controls are used during the calibration of the indenter, and are not required during the normal operation of the instrument.

The Z Motor may also be controlled from the Keyboard when the Manual Control Mode is selected (see Chap. 8). However, there are times during the making of a series of indents when it is desirable to raise the indenter head without coming out of the indenter computer program.

As indicated above, a knowledge of the function of the other switches and indicator lights on the Electronics Cabinet is not essential to the operation of the indenter. However, the user will be able to follow the actions of the indenter by watching these lights, and additional information about them is provided below.

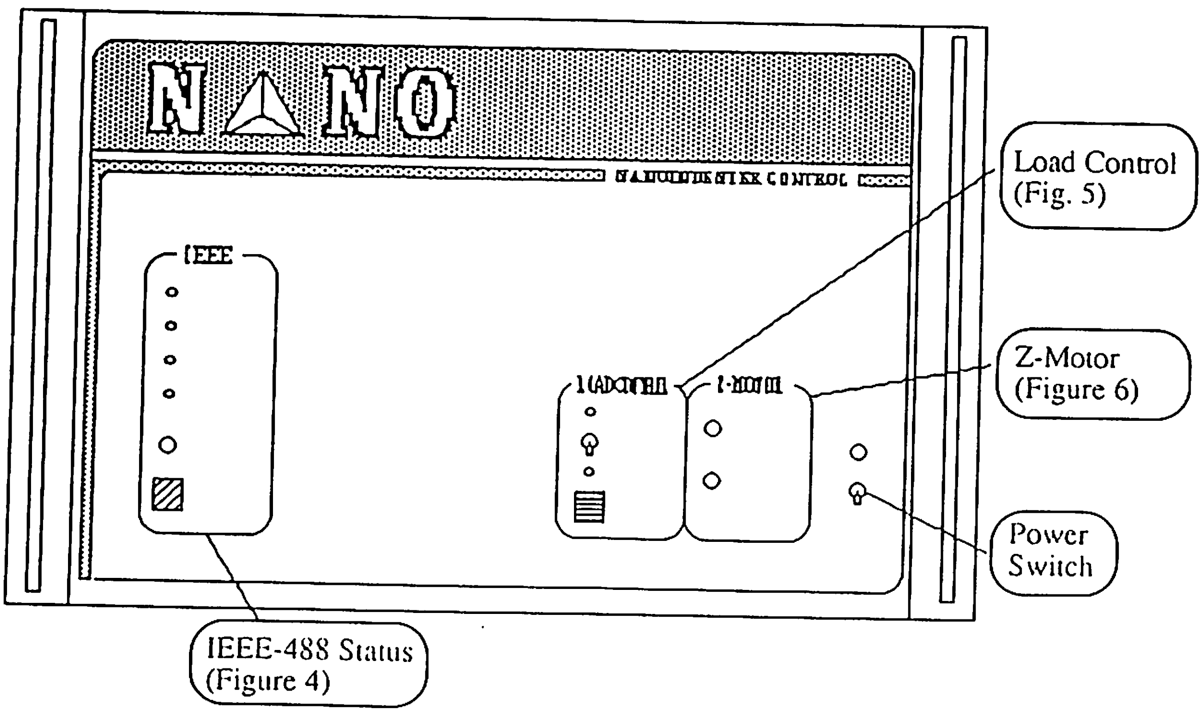


Fig. 1.3. Schematic of the control panels for the Nano Indenter electronics.

gure 1.3 shows the control panel for the electronics of the Nano Indenter system. This panel occupies the upper half of the Electronics Cabinet (see Fig. 1.1) and contains three smaller panels: the IEEE-488 status board on the left and the Load Control and Z-Motor status and control boards on the right.

Figure 1.4 is a diagram of the IEEE-488 status panel. In the Nano Indenter system, the computer interfaces with the system hardware through an IEEE-488 interface. When the two are communicating properly, the top light ("System Active") is lit. The "Reset Button lights if the IEEE-488 interface is RESET

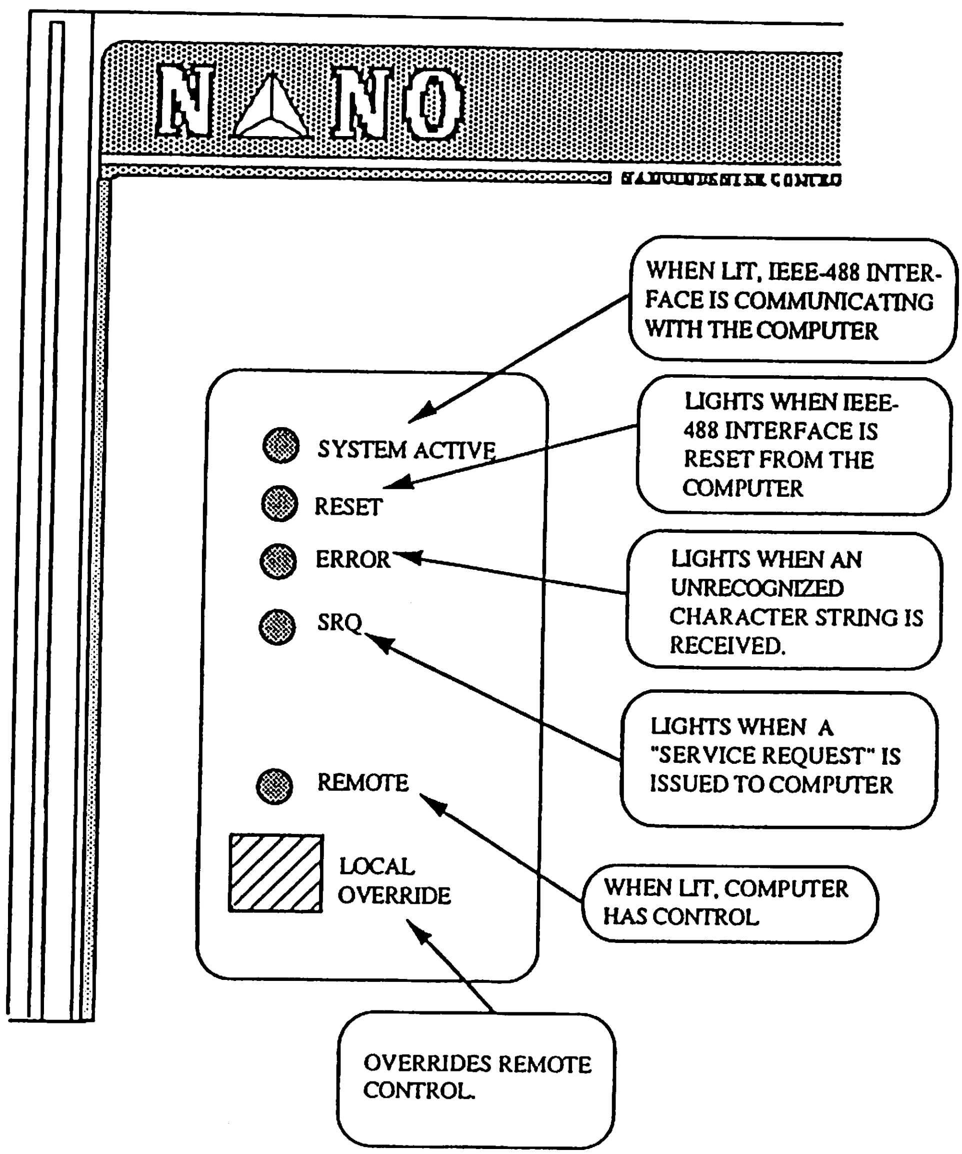


Fig. 1.4. Diagram of the IEEE-488 status panel.

from the computer. The Error Indicator lights when the interface receives a character string (i.e. a computer command) that it does not recognize. The SRQ light goes on when a service request is issued by some component of the system to the computer. When the remote light is on, the computer has control of the system, but this control can be overridden by depressing the Local Override button.

Figure 1.5 is a diagram of the Load Control panel It contains the standby switch, which, when depressed, removes power from the loading coil of the indenter with the result that the indenter comes to rest on its bottom stop. The switch can be used to change from the high load range to the low range. In high range the load on the indenter may be varied from 0 to 120 mN (0 to 12 gm) and from 0 to 20 mN (0 to 2 gm) in the low range. The load resolution for the more sensitive range is approximately ± 0.2 μN (± 20 μgm). The indicator lights show which range has been selected.

This panel allows the user to control load ranges manually. The load range can also be set from the keyboard. Keyboard range selection, which is one of the

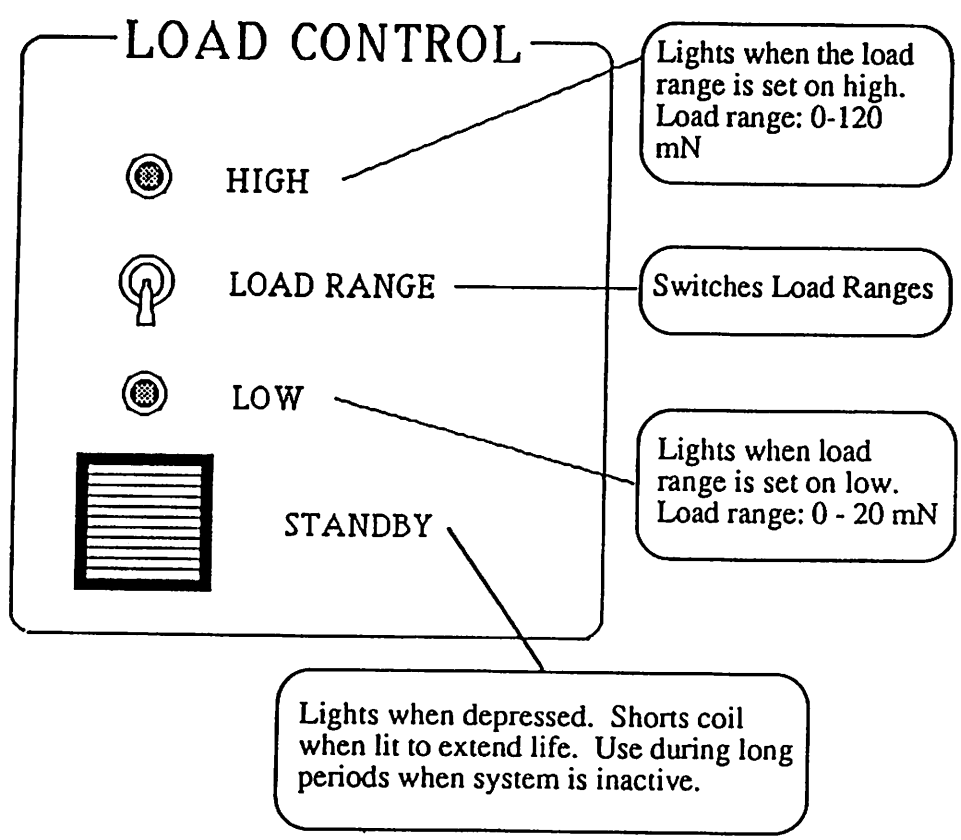


Fig. 1.5. Load control panel on the electronics control panel of the Nano indenter.

Execution Parameters (see Menu of Execution Parameters, Chapter 6), is almost always used in setting up indent experiments.

Figure 1.6 shows the Z-motor control panel. The two indicator lights are labeled "Total Lockout" and "Fast Lockout." The former comes on when the displacement sensing system is near the middle of its travel. When this light is lit, the Z-motor, which moves the indenter head up and down, cannot be used to move the head down toward the specimen. The "Fast Lockout" light is lighted at those times when the Z-motor can be run in the down direction at slow speed only. The light comes on when the displacement sensing system comes off it bottom stop.

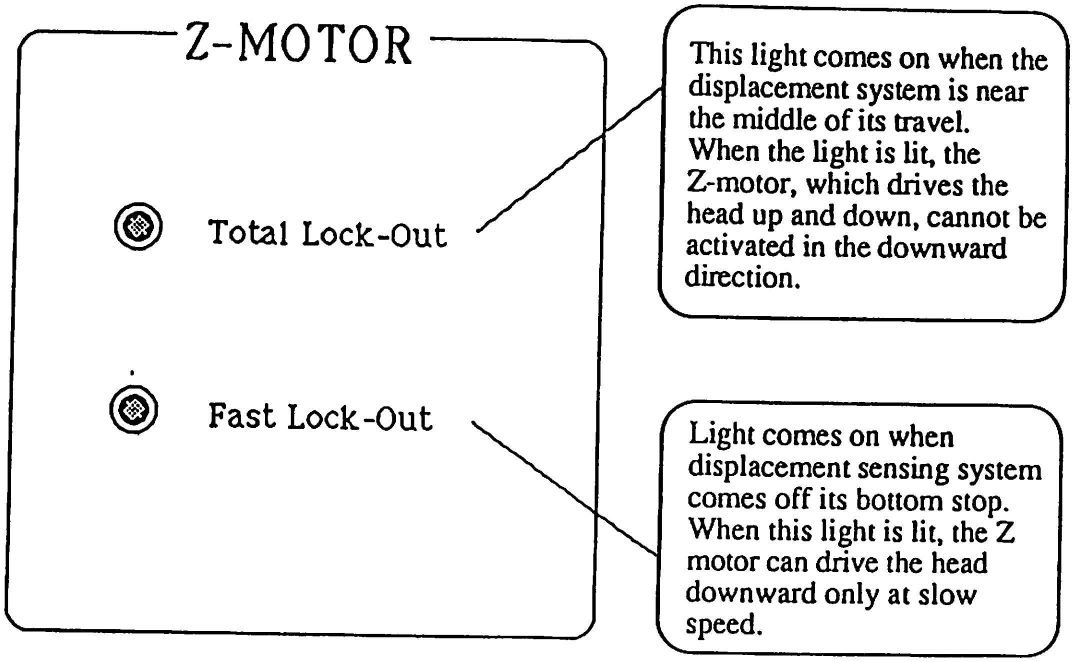


Fig. 1.6. The Z-motor section of the Nano Indenter electronics control panel.

Chapter 2

PRINCIPLES OF OPERATION

This chapter presents a discussion of the principles of operation of the main components of the Nano Indenter System: 1) the Loading System, 2) the Displacement Sensing System, 3) the Indenter Head, and 4) the X-Y Tables. In the last section the details of the making of an indent are described.

The Loading System

The system used to apply the load to the indenter consists of a magnet and coil in the indentation head and a high precision current source. These two items are shown schematically in figure 2.1.

The current source is applied across a coil in a magnetic field, thus generating a force. The current from the source, after passing through the coil, passes through a precision resistor across which the voltage is measured. It is this voltage that is displayed on the displacement voltmeter. The current source is programmable with a resulting range of displayed voltage of approximately -2.0 to +2.0 volts in the high load range and from approximately +0.8 to +1.7 in the low range. The voltage is controlled through a 16 bit DAC; therefore, the maximum resolution is 10 μV. During experiments, the computer controls the voltage source through the IEEE-488 interface. It can also be manually controlled using the manual control box (see Fig. 1.2)

Displacement Sensing System

The displacement sensing system is at the heart of the Nano Indenter system. It is shown highly schematically in Fig. 2.2 and consists of a special three-plate capacitive sensor. All three plates are circular disks approximately 1.5 mm thick. The two outer plates have a diameter of 50 mm, and the inner, moving plate is half that size. All three plates have a hole in their centers large enough to accommodate the indenter column, which is actually attached to the moving plate. This plate-and-indenter assembly is supported by two leaf springs cut in such a fashion as to have very low stiffness.

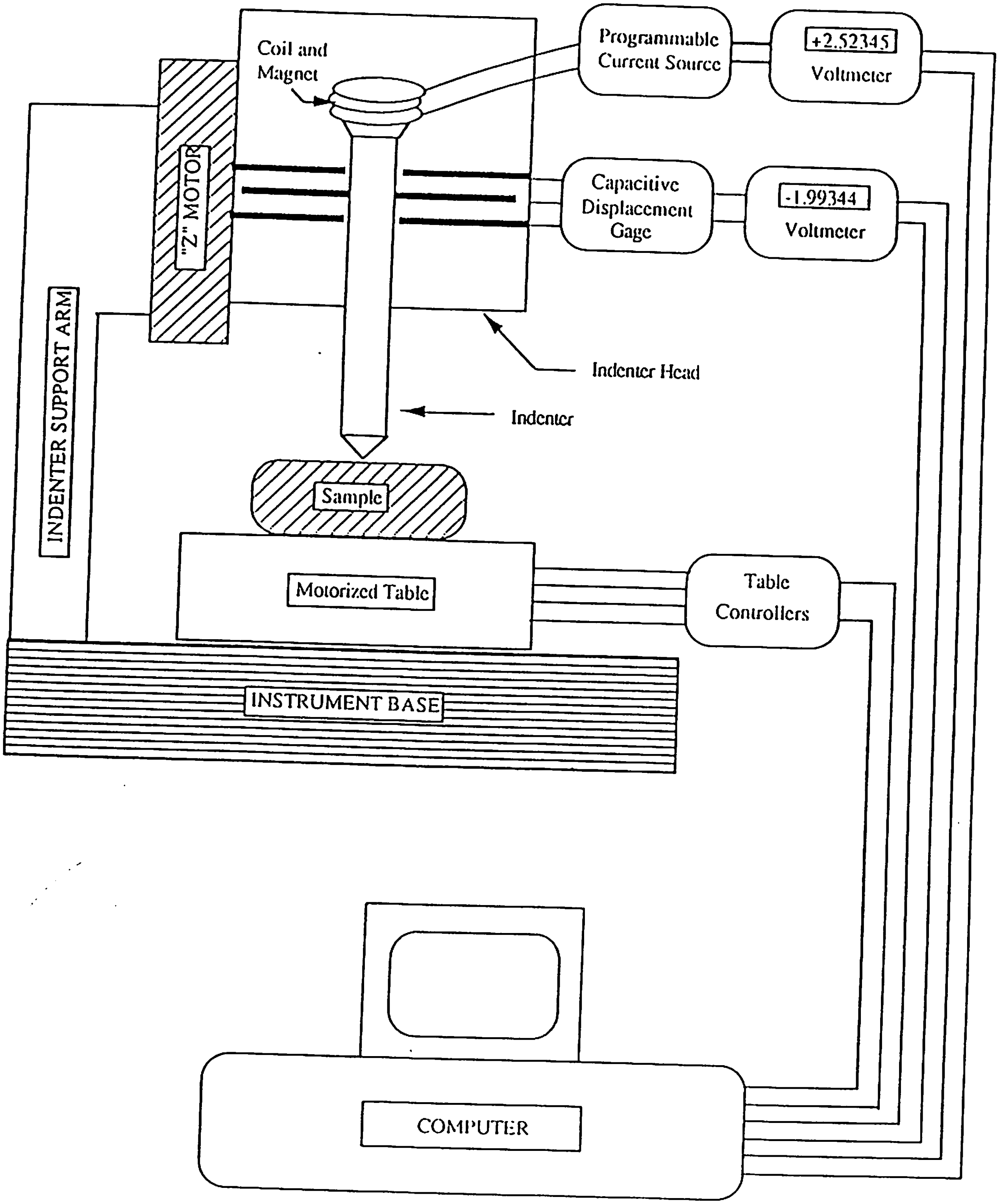
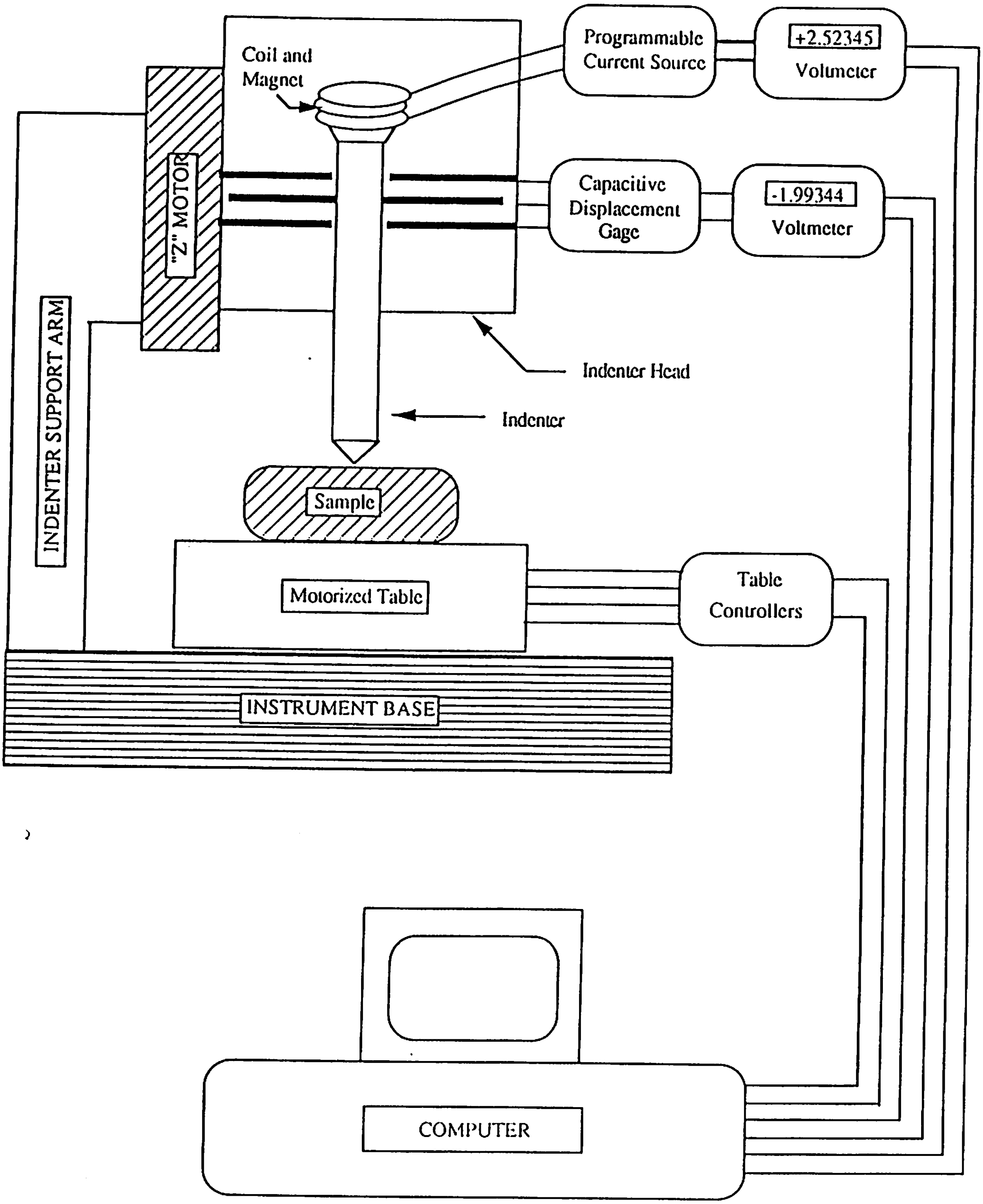


Fig. 2.1 Schematic representation of the Nano Indenter system viewed from its left side. The relationship of the of the indenter proper to the indenter head and the Z motor is illustrated. The Z motor moves the head and indenter simultaneous. The coil and magnet move the indenter only. For the sake of clarity, the microscope is not shown. It is directly behind the indenter in this drawing.



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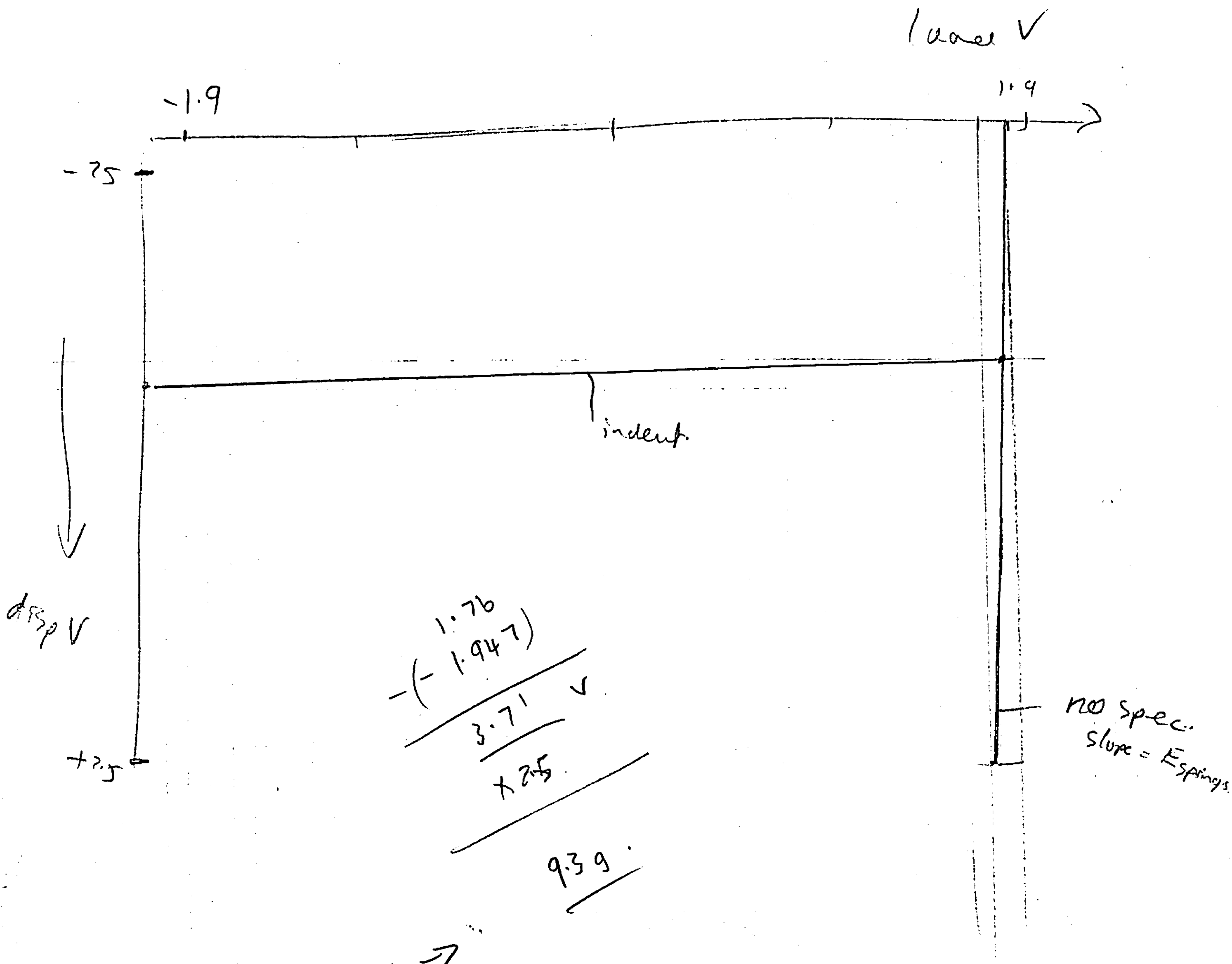
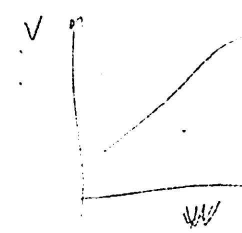
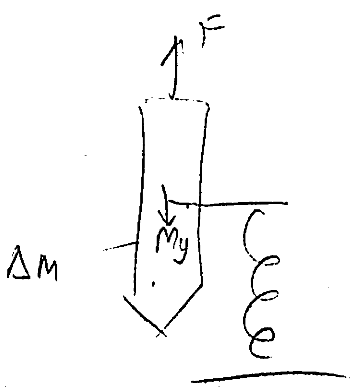


1.76
$-(-1.947)$
3.71 V
× 2.5
9.39

2.59 N





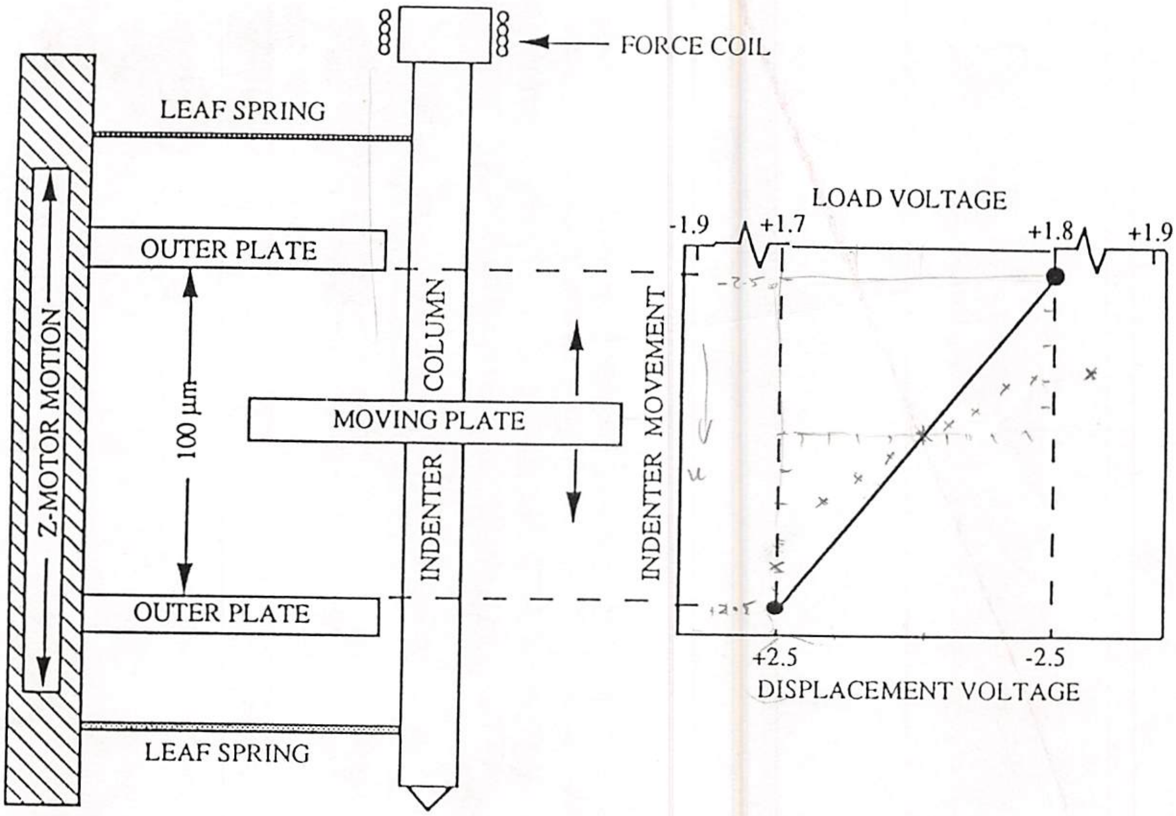


Fig. 2.2. Schematic drawing of the three-plate capacitor and indenter column that form the displacement sensing system of the Nano Indenter. This drawing is highly schematic. For the sake of clarity, only the left halves of the 50 mm diameter, circular outer plates of the capacitor are shown. For the same reason, the vertical scale in the drawing is also grossly

At the top of the indenter column is a load coil that is used to raise and lower the plate and indenter assembly through its 100 μm travel between the outer plates of the capacitor. This same coil applies the force required to make an indent. As may be seen in the plot at the right side of Fig. 2.2, a load voltage of 1.7 V will just lift the indenter off its bottom stop, and 1.8 volts suffice to bring it to the top of its travel. It should be emphasize that while the Z-motor can be used to raise or lower the entire indenter head, only the motion of the indenter column as controlled by the load coil is used in the actual making of an indent. the entire indenter head should be handled with care, as this capacitive device is made of glass.

The voltage output range of the displacement sensing system is approximately -2.5 to +2.5 v. The system is most linear from +1.5 to -1.5 v. The voltage is measured using a 5 1/2 digit DVM. The resulting displacement resolution is approximately ±0.2 nm.

Positioning the Specimen in the X-Y Plane

The X-Y tables have a real resolution of 0.1 micrometer. The motor controllers can be controlled by the computer via the IEEE-488 bus or from the front panel. The screws have a thread pitch of 40 threads per inch. They have a real accuracy of 20 millionths of an inch per inch and are mounted on bearing with a maximum of 15 millionths of an inch horizontal play. Although the backlash in the stage is virtually nonexistent due to special zero backlash nuts on the screws, it is recommended that each position be approached from the same X and Y directions (removal of backlash is taken care of by the computer during experiments). The stepping motors have 50,000 steps per revolution. The motors must be de-energized between moves to prevent excessive heating during the experiment, which leads to thermal drift in the displacement measurement.

Making an Indent

This section discusses the making an indent. It describes the process and indicates the programmable features of the system. The whole procedure is automated and, therefore, not essential information for the user. It is included here to help the user better understand the workings of the Nano Indenter.

Once the tables have been positioned so that the indenter is above the area of the sample to be indented and the table motors have been powered down, the indenter is lowered close to the specimen surface, and a wait period of a minute is taken to allow the system to come to equilibrium.

Clearly, some estimate of the relative heights of the indenter and the sample surface must be available to accomplish this movement. This estimate is obtained for the first indent by setting the center plate of the capacitor (to which the indenter is attached) on its bottom stop and moving the entire head down (using the Z-motor) until the indenter contacts the surface. When contact occurs, the indenter is pushed upward, tripping the Z-motor interrupts and stopping the Z-motor. The head is then moved upward at slow speed for 2 seconds,the tables are moved slightly, and the process is repeated with the slow Z-motor speed. This second step gives the best estimate of the surface position that can be obtained when one is moving the entire indentation head.

After this second indent is made, the indenter is left in contact with the surface under a very small load. The Z-motor is then used to move the head up or down so that the displacement sensing capacitor is near the center of its travel.

At this point the system is allowed to monitor the thermal drift rate in the displacement measurement, and when the drift rate becomes smaller than the maximum prescribed by the user, the displacement is read, establishing an initial height for the surface. The indenter is then raised using the coil/magnet assembly (the head position remains fixed for the rest of the experiment), and the Table is moved so that the chosen location for the first indent of the specified shape is under the indenter. For subsequent indents, the estimate of surface position used is that found in the previous indentation experiment. As stated above, this procedure is performed before the first indent in a series. The user can also designate other indentations in a series before which this procedure will be performed (see "**Head Repositioning During a Shape**" in Chapter 6).

Continuing from the wait period described above, the indenter is lowered slowly toward the surface at a constant rate while the computer checks to see if surface contact has occurred. The indenter should be lowered at less than 15 nm per second to avoid significant impact effects when it arrives at the surface. The computer determines contact by constantly checking the last ten displacement readings to determine if the average stiffness sensed by the system has increased above a certain critical value (usually four times the stiffness of the indenter support springs).

Once contact is established, the rate of change of the load is increased in such a way as to perform the prescribed indentation experiment. The load is then removed from the indenter. When the voltage on the indenter passes the displacement voltage at which the surface was detected on the loading portion of the cycle, the current through the coil is fixed while the data is reduced, recorded and plotted on the screen. The indenter is then raised well away from the surface in preparation for moving the sample to the position of the next indent.

The data are reduced by removing all data points before 17 points prior to detection of the surface . The raw data are recorded on a disk and all calculations involving calibration constants are done down stream in other programs.

All of the displacement rates and other critical values are programmable. Data files are given a base name of choice and are amended with sequential number representing their position in the series of indents.

Chapter 3

OPERATING A HEWLETT-PACKARD COMPUTER

Configuration of the Computer System

The computer system for the Nano Indenter consists of a Model 300, Series 9000 Hewlett-Packard computer with monitor, a printer, a hard disk drive, and a floppy disk drive. The two drives are contained in a single cabinet, which rests on top of the computer itself just to the right of the monitor (see Fig. 1). The printer is an H-P "Think Jet" printer.

Volume Specifiers and MSI

Various components of the Nano Indenter system, including such "mass storage" devices as the floppy and hard disk drives, are tied together through their connection to the HP-IB buss of the computer. In order for software commands to be routed to these components, each must have an "address" or in the case of mass storage devices, a "volume specifier." In its most complete form a "Mass Storage Volume Specifier" (MSVS) contains designations for the device type, the device selector, and the unit number. For our purposes, however, a simplified version of the MSVS will suffice, and the user is referred to the Hewlett-Packard manual, *Installing, Using, and Maintaining the Basic 5.0 System* , for a more complete discussion of the subject. All that is required is a designation of the device selector (which consists of interface select code and a device address) and, where necessary a unit number.

In the Nano Indenter system, the "interface select code is always "7" and the device address is always a two digit number appended to the "7." For example, the device selector for the printer is "701". The cabinet containing the disk drives is designated "700." Because it contains two "units," the hard disk drive and the floppy disk drive, an additional number (the "Unit Number") is required to define uniquely the address of the two drives. The hard disk is "700,0" while the floppy is "700,1." The hard disk is divided into four "volumes" denoted as "0", "1", "2", and "3," and the unit number of the hard disk is further elaborated by appending a volume number to it separated by a comma. Thus the MSVS of Volume 3 of the hard disk is "700,0,3". The floppy is normally not divided into volumes and, therefore, does not need a third specifier digit. Its MSVS is "700,1." The addresses for the various components of the Nano Indenter system are shown in Table 1 below.

Table 1. HPIB Buss Addresses of Nano Indenter Components

Device	Address
Hard Disk	700,0
Floppy Disk	700,1
Printer	701
Plotter (optional)	705
Nano Indenter	707
Displacement DVM	708
Load DVM	709

The MSVS designations are used to send commands to the various components of the computer system. For example, if one wishes to run a program stored in volume 1 of the hard disk, the initial command takes the form

MSI ":,700,0,1"

followed by a carriage return. MSI is an acronym for "Mass Storage Is," and the command declares that the Mass Storage Unit "700,0,1" is to be accessed. (Note the colon and the apostrophe following the first quote (") marks. It is essential to include the colon and apostrophe in all references to MSVS's.)

MSI ":,700,1"

transfers control to the floppy disk drive, and

MSI ":,700,0,3"

allows access to files in volume 3 of the hard disk, for example.

Printed Output Devices

Either the printer or the monitor may be designated as the output device. The command

PRINTER IS 701

will cause subsequent print commands to be carried out on the printer. On the other hand,

PRINTER IS CRT ⇒ printer is 1

will result in printouts on the monitor screen.

Soft Keys

The eight gray keys across the top of the keyboard numbered *f1* to *f8* are user-definable "soft keys." The function served by each soft key is printed in the green boxes at the bottom of the monitor. The box at the extreme left refers to key *f1* , the next box to key *f2* and so forth. Four levels of commands may be accessed through the use of the "MENU" and "USER/SYSTEM" keys located between soft keys *f4* and *f5*. Pressing the MENU key toggles the green boxes on and off. Pressing the USER/SYSTEM key returns a selection of green box options relating to the system, e.g, commands such as RECALL, CONTINUE, RUN, etc. Pressing SHIFT and USER/SYSTEM simultaneously calls up the "User 1" set of command options. "User 2" and "User 3" command lists may then be recalled by pressing SHIFT-MENU.

The soft keys can eliminate much of the tedium in working with the computer, and users should familiarize themselves with the various functions carried out by these keys. For example, in the User 1 Level, pressing key *f8* issues the command 'MSI ":,700,0,1"', which immediately puts the computer in volume one of the hard disk where all of the Nano Indenter programs files are stored. The RECALL command (key *f8* in the System Level) can be used to recall any one of the last several commands, thus avoiding the retyping of the commands. A number of other functions are also available such as LOAD, PURGE, RUN, etc.

Copying or Purging Files

Two methods are available for copying or purging files from the hard and floppy disks. Individual files may be dealt with using either the COPY or PURGE commands. The formats used are illustrated below:

COPY "Myfile" to "Myfile:,700,1"

This command copies the file "Myfile" located in the current multiple storage area (assumed in this example to be one of the volumes of the hard disk) to the floppy disk (MSVS "700,1") where it is also given the name "Myfile" (In typing these commands, note that H-P Basic is sensitive to case.)

Another example assumes that the computer is in multiple storage area "700,0,1", i.e., volume 1 of the hard disk. Then the command

```
COPY "Myfile:,700,0,3" to "Myfile:,700,1"
```

copies the file "Myfile" from volume 3 of the hard disk to the floppy. The use of the quotes, the colon, and the comma as shown is essential.

There is a corresponding PURGE command. Thus

```
PURGE "Myfile"
```

will delete the file "Myfile" from the current multiple storage area.

```
PURGE "Myfile:,700,0,3"
```

will delete "Myfile" from volume 3 of the hard disk. Implicit in this command structure is the assumption that the current multiple storage area is something other than "700,0,3."

For copying or purging large numbers of files, it is most convenient to use the special program "HBACKUP." The HBACKUP program is located in volume 1 of the hard disk. It may be accessed by the following series of commands:

```
MSI ":,700,0,1"   (RETURN)
LOAD "HBACKUP"  (RETURN)
RUN
```

The user is then queried as to whether copying (C) or purging (P) of files is desired. The multiple storage source from which files are to be copied/purged must be specified as must the destination storage area. In both instances the proper format is ":, 700,#,@", e.g, ":,700,0,3" or ":,700,1". In all cases the quotes, the colon, and the comma must be used. The user is then prompted for the names of the first and last files of the sequence to be copied/purged. The program begins with that file name and copies/purges all subsequent files listed below it in the CATALOG for the source multiple storage area until the last file specified is reached. The default file names are FIRST and LAST. If the default values are accepted, all files in the CATALOG will be copied/purged. However, in all cases the user is prompted for a decision as to whether copying/purging shall proceed automatically to the end of the CATALOG or whether the user is to be asked before each file is copied/purged.

Chapter 4

PREPARING THE NANO INDENTER FOR OPERATION

Loading Samples

Indent specimens are normally in the form of polished metallographic samples mounted in standard 1.25 in. diameter plastic mounts. However, samples may also be glued to 1.25 in. dia. metal disks. The specimen holder is a metal plate

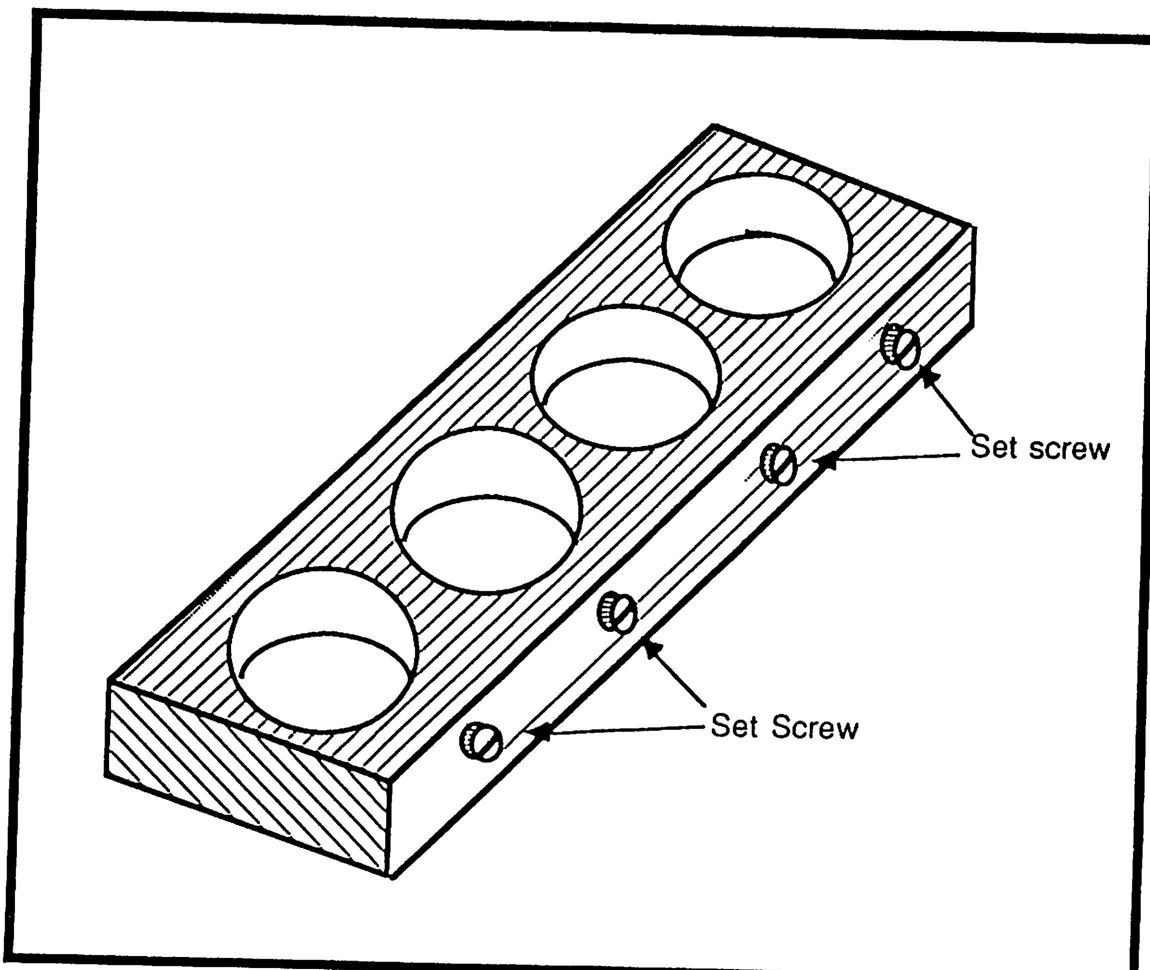


Fig. 4.1. Schematic diagram of the Nano Indenter specimen holder.

2 x 6 x 0.75 in. into which four 1.25 in. diameter holes have been drilled. Set screws hold the samples in place.

It is particularly important that the top surfaces of the samples all be at the same height so as to eliminate the danger of the indenter hitting the side of a sample as the Table changes the position of the specimens under the indenter. A relatively easy method for achieving this alignment is as follows. Place two strips (~1/16 in. thick) of cardboard on a flat surface, spacing them so that when the specimen holder is placed on top of the strips, the strips are aligned just under the long edges of the holder. Mounted samples may now be slipped face down into the holes in the holder and secured by the set screws. Careful visual inspection of the samples after mounting is advised to make sure that the samples did not tilt slightly when the set screws were tightened. It is also good practice to blow off any dust or lint on the surfaces of the specimens before they are inserted into the Nano Indenter.

As noted above, the specimen holder fits under brackets attached to the Table and is held firmly in place by a screw that can be tightened against the bottom of the holder by means of a knurled knob. The fit of the holder is fairly tight, and care and patience should be exercised when reinserting the holder into the brackets. If the ends of the holder are straight with respect to the brackets, the holder will slip into place with minimum difficulty.

Note that the set screws are off-set slightly from the center line of the side of the holder. This design was adopted to allow for variations in the thickness of specimens and specimen mounts. That is, the holder may be loaded in an orientation with the screws below the center line or above it depending on which arrangement is best given the sample height.

Specimens must be thermally equilibrated with the Nano Indenter prior to the initiation of indentation. It is a good practice to store specimens inside the Indenter cabinet before loading them into the specimen holder. A loaded holder should be left in place for several hours before beginning indentation, and when convenient should be left overnight before beginning an experiment.

Turning on the Indenter

The main power switch (the "Scram Switch") for the Nano Indenter system is located in the lower right hand corner of the front face of the electronics cabinet (see Fig. 1.1) This switch is normally left on all the time unless the indenter is to be out of use for several days. There are also a number of auxiliary power switches on the front of the electronics cabinet that are always left in the "on" position. These include the power switch for the topmost electronics package in the cabinet. The switch is located on the upper right hand side of the cabinet front, just to the right of the "Z-motor" indicator lights. The "up" position of the switch is "on," and when the power is on, the red light above the switch should be burning. The power switches for the DVM's are located at the extreme left side of each DVM control panel; power is "on" when the switches are depressed. The small switches marked "TERM" on the right side of each DVM control panel should also always be in the depressed position. Under normal conditions all of these auxiliary switches should be left in the "on" position all the time.

On-off switches for the computer, the hard disk, and the monitor are all located on the front of each instrument in the lower left corner. Press the switches in to turn the units on. The power switch for the TV screen is the leftmost small black knob located directly under the screen. The on-off switch for the microscope light is on the transformer that powers the light, and there is an auxiliary remote switch on a power cord between the computer and the electronics cabinet (see Fig. 1.1). Both the TV screen and the microscope light should be turned off except when actually in use.

Special Note: At startup, be sure that all components of the system, including the printer, are on and operational. Also be certain that the printer has paper in it.

Turning Off The Indenter

IN AN EMERGENCY TURN OFF THE INDENTER BY TURNING OFF THE SCRAM SWITCH. This action cuts all power to the indenter system and associated computer and peripherals.

Normal shutdown procedures vary depending on the length of time the instrument will be out of service. For periods of a few hours, simply turn off the monitor and the remote TV and leave the rest of the power on. For an overnight shutdown, depress the "Standby" switch on the front of the electronics cabinet, and leave the rest of the power on. If the shutdown is expected to last several days, turn off the main power switch (the SCRAM switch) as in the case of an emergency shutdown.

Chapter 5

RUNNING THE NANO INDENTER

The Nano Indenter software is fully menu-driven and, with a little practice, easy to use. Although you may not think so the first time through, the program is very user-friendly with lots of on-screen help. At almost every step along the way, you are prompted as to the appropriate action to take. For example, when you first boot the computer, you are queried,

"Do you want to use the Nano Indenter?"

If you then type Y (for "yes") and hit RETURN, the computer will automatically load and run the main Indenter program, which is named "MASTER," and you are presented with the Master Menu (see below). If you type N (for "no"), the computer is free to perform whatever other functions you have in mind.

Getting to the Master Menu

As just indicated, when you first boot the computer, you are led directly into the master menu. Later on , however, after a RESET or other action that terminates "MASTER", it will be necessary to load and run MASTER yourself in order to continue operating the Nano Indenter. To do so, you must first enter Volume 1 of the hard disk where MASTER is stored. Type

MSI ":,700,0,1"   (RETURN)

or enter this command by pressing the *f8* key in the User 1 Level. Next type

LOAD "MASTER"   (RETURN)
RUN   (RETURN)

(Note that the LOAD and RUN commands may also be entered with the *f6* and *f3* soft keys, respectively.) The Master Menu will then appear on the monitor. MASTER may also be loaded and caused to run automatically if the *f1* soft key is pressed followed by (RETURN) while the softkeys are in the User 3 mode.

Master Menu

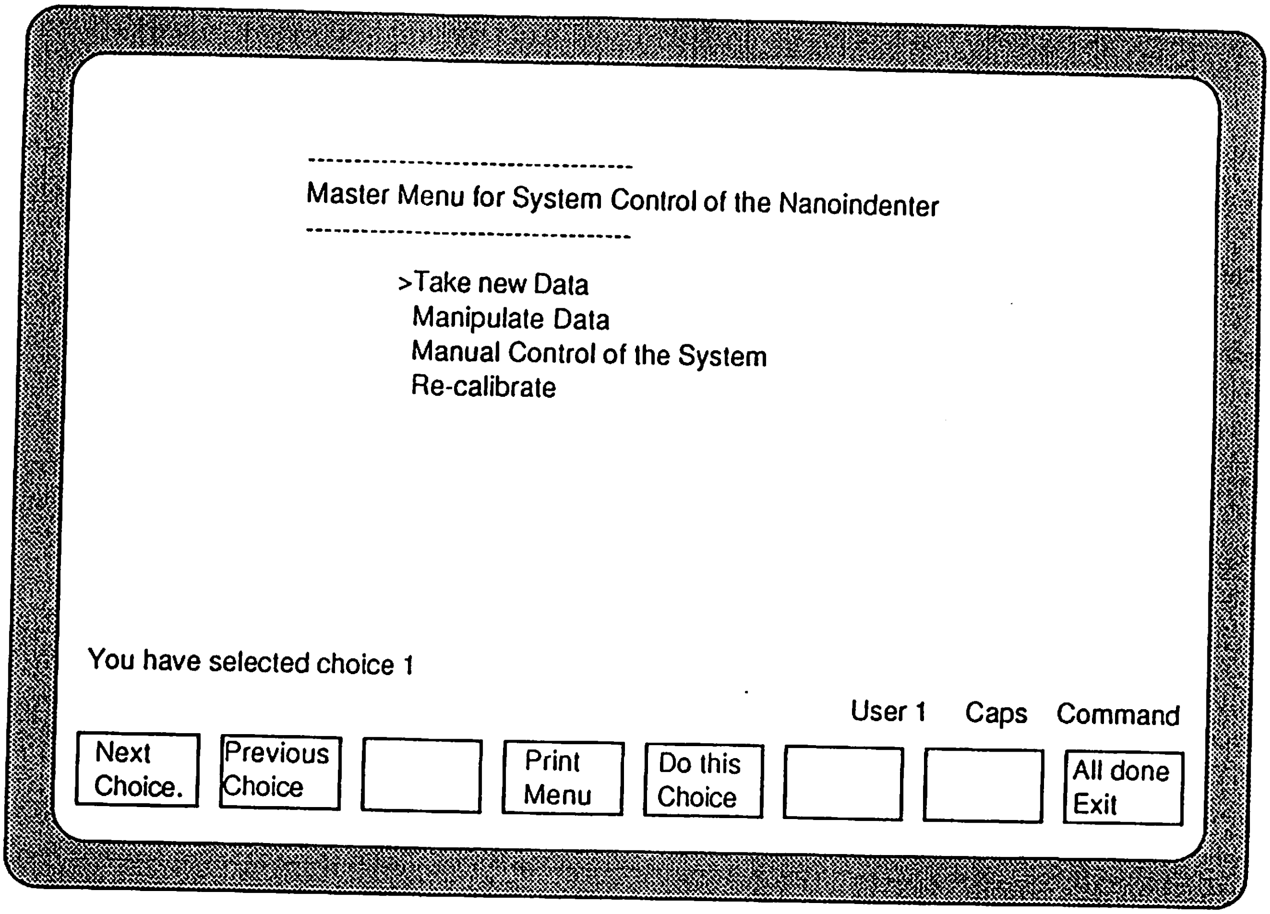


Fig. 3. The Master Menu as it will appear on the monitor. Note the soft key boxes at the bottom of the screen. There are several unoutlined message boxes just above the soft keys boxes. The message at the lower left indicates the number of the opposite the cursor. At the lower right the first notation shows the User Mode for the soft keys. "Caps" indicates that the Caps Lock is on. If the caps lock is off, the space is left blank. The last message box indicates the status of the computer operation; "Command" indicates that a command has been given the computer. A "Running" entry indicates that a program is running, etc.

Notice the soft key functions. The cursor (>) may be moved downward by pressing the *f1* key ("Next Choice") or upward by pressing *f2* ("Previous Choice"). You select the action you wish to take by moving the cursor to a position opposite that action and pressing soft key *f5* (:Do This Choice"). A new menu appropriate to your choice will appear.

The Master Menu contains four entries. Each is discussed in turn below. Each entry on the Master Menu has one or more submenus associated with it, and these submenus are discussed in detail in the Chapters that follow.

"Take New Data" — This branch of the program permits you to define an indent or series of indents and to specify the location of the indents on the specimen. A variety of information about the specimen is also entered and becomes a part of the hard copy output of the experiment. The actual making of indents and the accumulation of raw load-displacement data are carried out in this segment of the program. (See Chapter 6)

"Manipulate Data" — This choice is selected when raw data is to be converted into Load vs. Displacement and Hardness vs. Displacement plots. Stiffness and Modulus calculations may also be made, and options are available for selecting and listing hardnesses at preselected displacements. (See Chapter 7)

"Manual Control of the System" — This choice allows the user to control various operations of the indenter system from the external keyboard. For example, different parts of a specimen can be viewed on the TV screen by moving the Table appropriately. Selection of indent sites is carried out in this manner. The microscope may also be focused, and the specimen can be moved back and forth between indenter and microscope. The indenter head may also be raised or lowered by pressing appropriate keys. (See Chapter 8.)

"Re-calibrate" — This branch of the program contains subprograms used in the calibration of various functions of the indenter, e.g., load and displacement systems. Only two choices, "Recalibrate Distance from Microscope to Indenter" and "Alter DAC Positions" should be of concern to users. Both these routines should be run at the beginning of an extensive series of indents. Accurate calibration of the distance between the microscope and the indenter is essential if the positions of the indents are to be determined precisely. The recalibration is carried out more or less automatically, and the on-screen instructions are clear and easy to follow. For the ultimate in accuracy in the positioning of indents, this calibration procedures should be carried out with the same microscope objective lens as will be used in the selection of indent locations on the specimen.

The alteration of the DAC (Digital-to-Analog-Converter) refers to the recalibration of some of the position sensors on the indenter. The recalibration is also done automatically and results in a speed up of the operation of the indenter. (See Chapter 9.)