Micro Materials

NanoTest On-line Help File Version 1.0 and NanoTest Manual Version 3.0

Ben Beake, Stephen Goodes, Steve Jones, Rob Parkinson, Nick Pickford and Jim Smith, Micro Materials Ltd, 2004.

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Introduction

The NanoTest is a nanomechanical property testing centre. The system has up to 3 separate modules (**indentation, scanning and impact**) depending on the version purchased. All three modules can be used in conjunction with the low load head (0.1-500 mN) or the high load "MicroTest" head (0.1-20 N).

There are a wide range of options which your system may have:-

Spherical indentation
Pin-on-Disc wear testing
Powder Adhesion Software

Continuous Contact Compliance Measurement
High temperature testing
Acoustic Emission system
2D levelling stage
High Resolution and Zoom microscopes
Humidity Control System

In most cases it is possible to upgrade your instrument to incorporate any of these options on-site without the need for return of your system to MML.

A brief introduction to depth-sensing indentation

The NanoTest measures the movement of a diamond probe in contact with a surface. In order to make a measurement an increasing load is applied and the probe indents the surface, as shown schematically below
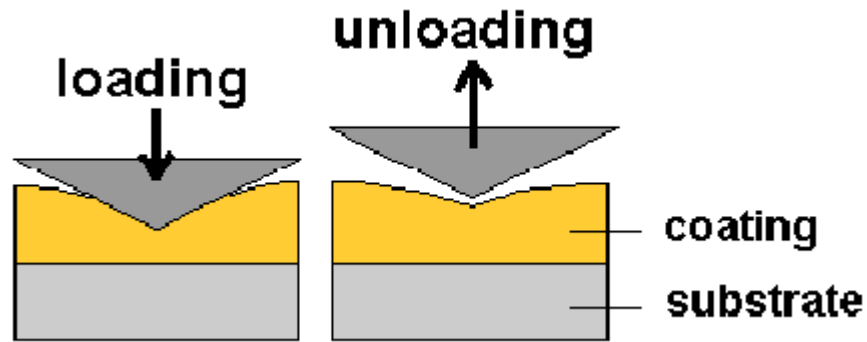

Force, displacement and time are recorded throughout the test. In an indentation, the elastic and plastic properties of the sample determine the shape of the unloading curve.

In addition to indentation testing, the versatility of the pendulum design allows for scanning and impact measurements as well :-

• Transverse sample movement during loading enables scanning measurements (scratch testing, fatigue wear testing)
• Sample oscillation at constant load for impact testing, contact fatigue testing and dynamic hardness

2 The NanoTest Concept and Hardware Description
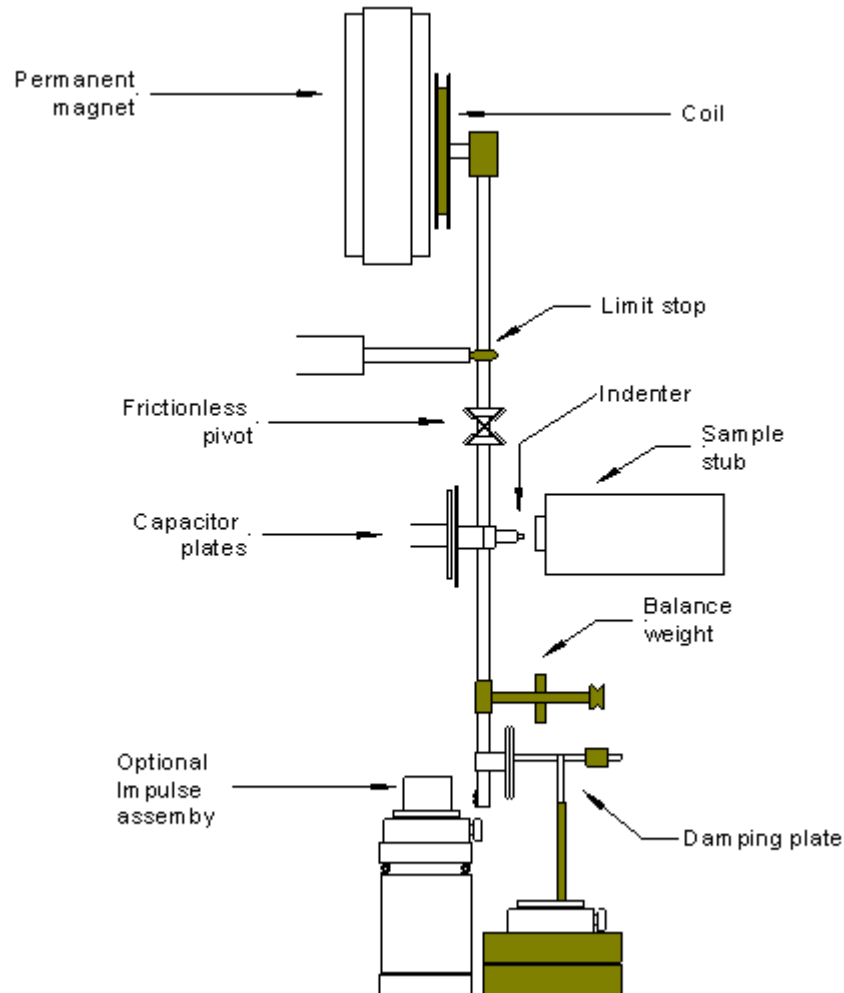
At the heart of the NanoTest system is a pendulum that can rotate on a friction-less pivot. A coil is mounted at the top of the pendulum; with a coil current present, the coil is attracted towards a permanent magnet, producing motion of the diamond towards the sample and into the sample surface. The displacement of the diamond is measured by means of a parallel plate capacitor, one plate of which is attached to the diamond holder; when the diamond moves, the capacitance changes, and this is measured by means of a capacitance bridge. The capacitance bridge unit is located close to the measuring capacitor in order to minimise stray capacitance effects.

Sample manipulation is by means of three DC motors driving micrometer stages in an XYZ configuration. The motor control electronics consists of a mother board containing three power modules, an IEEE interface module and a backlash control board. These plug into expansion slots in the computer. The motor power supply is derived from the computer, with motor positioning is by means of magnetic encoders. The motor control board communicates with the system computer via the IEEE bus.

The limit stop defines the maximum outward movement of the diamond, and also the operating orientation of the pendulum, when a load is applied. Its position is manually adjusted with a micrometer. The equilibrium position of the pendulum, with zero load current, is adjusted with balance weights which are movable along both the horizontal and vertical axes. A more detailed description of the equipment (electronics, stages, motors and pendulum) is given in the Technical Appendix (Section 12.2).

The importance of calibration The same NanoTest calibrations are common to all 3 operating modules. At first it may seem that there are a lot of calibrations, however, most are fully automated and easy to perform. It is *vital* that the instrument is *accurately* calibrated otherwise any data obtained will be inaccurate, or at worst, useless!

3 Finding your way around the configuration screen and platform software

Double-click on the NanoTest Platform Icon on the Windows desktop to enter the NanoTest system.

This brings up the **platform configuration screen** which allows the user to select the load range required (NT1, NT2 or MT), decide how the data is displayed during acquisition and analysis, and alerts the software to whether a microscope is to be used to position the probe over the area of interest.
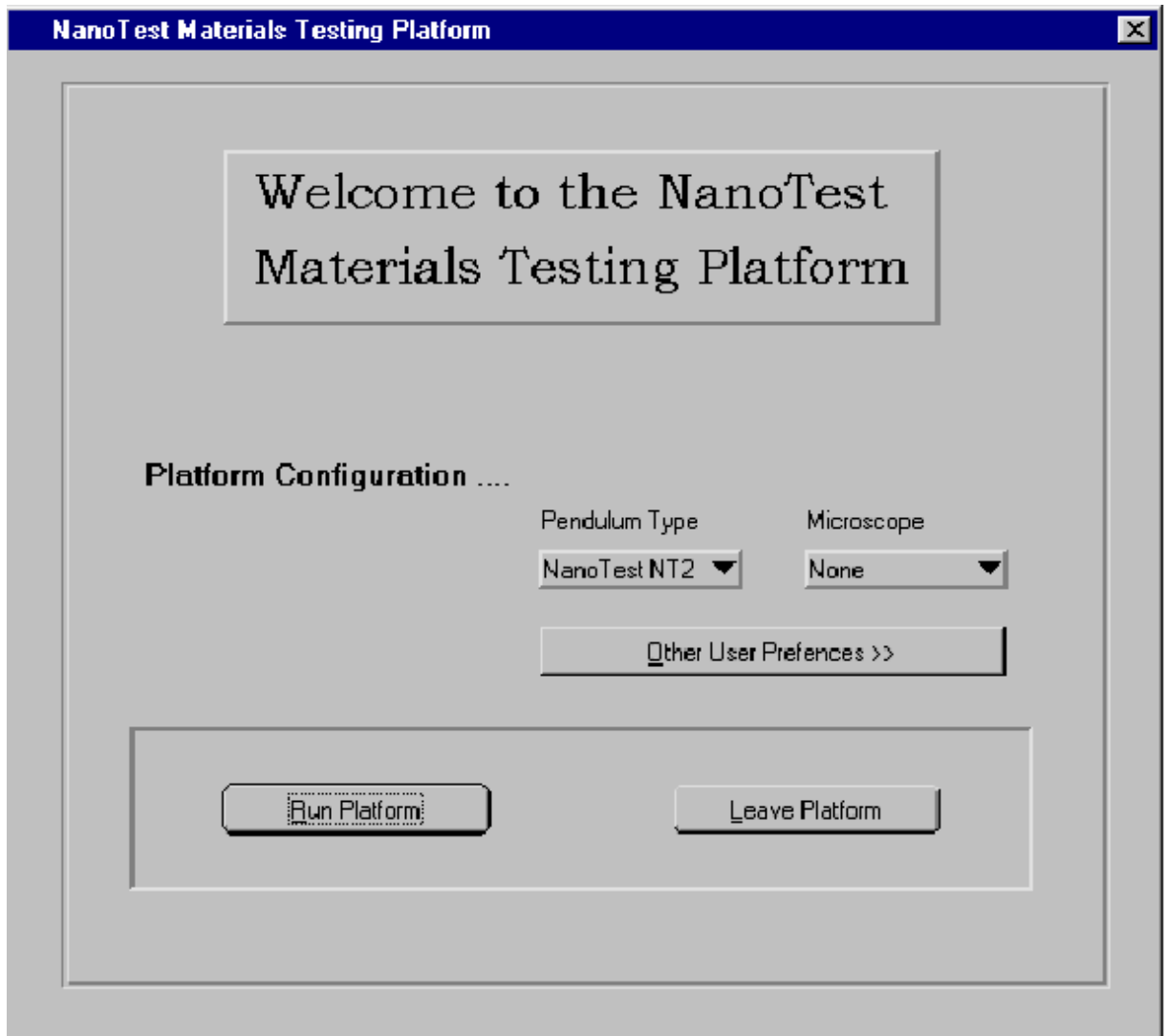
Choosing NT1 or NT2

The choice of load range depends on the type of test planned. The low load NanoTest pendulum has 2 load ranges, NT1 and NT2. NT1 has higher sensitivity but a lower maximum available load (typically 45 mN). Unless you will only be measuring very thin coatings (under ~200 nm), we suggest that the NT2 range is used in general, for greater flexibility. To switch between ranges manually switch to the other position on the front of the NanoTest control unit and click on the relevant option in the platform configuration screen.

MT refers to the high load or **M**icro**T**est pendulum.

NanoTest systems are supplied without microscopes or (more commonly) with either a *zoom* microscope or *high-resolution* microscope. Select the correct option for your configuration.
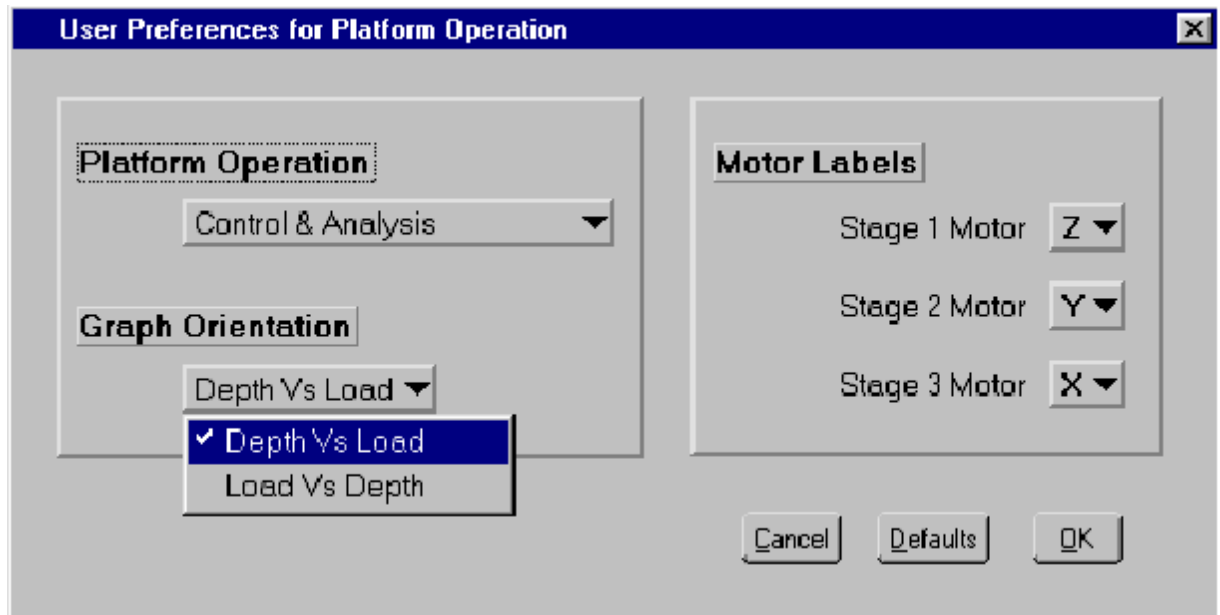
Once all these parameters have been set correctly, click-on *Run Platform* which brings up the NanoTest platform screen.

NanoTest Platform Screen
The screen is organised with 6 drop-down menus at the top. The picture below shows the top-left area of the screen which contains the menus. If you do not have a particular option/module on your system then it will be displayed in faint type and it will not be possible to click on it. The load range and microscope selected are indicated by red lights in the panel on the right of the screen.
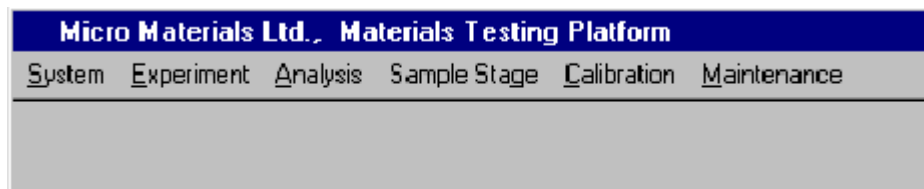
The 6 menus are *System*, *Experiment*, *Analysis*, *Sample Stage*, *Calibration* and *Maintenance*. These are described briefly below.

System
In day-to-day use it should not be necessary to alter anything in this menu. To leave the NanoTest system click-on the *Exit* key, and then enter *Yes* to *Do you want to leave the NanoTest platform?*

Experiment
The four types of experiment (*indentation, scanning, impact* and *special options*) correspond to the three operating modules and the separate special options (pin-on-disc, powder adhesion and 2D-levelling options). Schedule tests and run experiments from here. See sections 6-8 and 11 for full details.

Analysis
For analysis of indentation, scanning and impact data. Data can be reviewed with or without analysis. See section 12 for details.

Sample Stage
Sample movement for swapping samples, replacing indenters, moving between the microscope and indenter etc. See section 9 for details.

Calibration
Periodic calibrations (load, depth, friction transducer, machine compliance, diamond area function). These are described in section 4.

Maintenance
This includes the checks that should be performed on a daily basis to confirm the instrument is working at its optimum level (including bridge box adjustment, signal display, zero load calibration). The maintenance checks are described in section 4.

4 Essential Calibrations

The essential calibrations to ensure that the instrument is working correctly are found in the *Calibration* and *Maintenance* menus. The periodic calibrations are within the Calibration menu and the daily checks are within the Maintenance menu. This section shows how to carry out the calibration routines.

4.1 Calibrations

Load
Depth
      Stage Encoder Method
      Reference Sample Method
Friction Transducer
Machine Compliance
      Indirect machine compliance
      Direct machine compliance
Diamond Area Function
Microscope Calibration
      High Resolution Microscope
      Zoom Microscope

4.1.1 Load

You will need the 3 balance weights (typically just over 1, 2 and 3 g) supplied with your system and a pair of tweezers to place them onto/off the balance point. It is necessary to calibrate for each load range (NT1, NT2 and MT) being used (MT requires the set of three heavier balance weights).

Enter the *Calibration* menu and select *Load*. Press *Calibrate* and follow the on-screen instructions (3-5 determinations will suffice). Note that the calibration procedure continues after removal of the final weight, so allow the calibration to finish before using the system. This calibration takes about one hour and is very stable. It needs repeating once every 3 months.

> The Load Calibration establishes the forces that can be applied at the diamond tip during a measurement. Calibration consists of hanging a series of masses from a set point (the balance point) at the bottom of the pendulum while the coil applies a countering force equating to a known voltage for each mass. This calibration relies on a good pendulum test in *bridge box adjustment*.

> Typical results for the calibration are:-
> NT2   500mN maximum load (high load range) and 70mN maximum load (low load range)
> NT1   45mN maximum load (high load range) and 10mN maximum load (low load range)
> MT   20N maximum load (high load range) and 2N maximum load (low load range)

4.1.2 Depth

> The Depth Calibration is fairly sensitive to operating conditions and so should be performed frequently. We suggest that the calibration be done weekly.

Stage Encoder Method
Reference Sample Method

4.1.2.1 Stage Encoder Method

This method requires a large steel ball indenter (depth calibration probe) and a fused silica calibration sample.

It is recommended that this calibration is carried out without the microscope enabled in order to save time. Once the microscope is disables, select *calibration-depth-stage encoder* from the main menu and bring the probe to within 25 microns of the sample surface. Use the default settings (with 5 determinations) and select *calibrate*. The calibration will proceed and display the value (and deviation) when completed.

The Depth Calibration relates the change in capacitance to a known distance moved by a sample in contact with the pendulum. The sample movement is determined directly from the stage movement (encoder displacement). The sensitivity and hence the depth range can be altered in the Schedule by changing the value of Amplifier Gain, with maximum sensitivity at 100% (equating to maximum resolution) and minimum at 15% (~6.66 times less resolution which allows a greater available measurement depth).

The calibration routine involves moving the sample in until the signal coming from the measuring plates is 1/4 of the initial value. At this point, the motor movement reverses and between 1/3 and 2/3 of the maximum signal amplitude, the changing amplitude is monitored against the known distance moved by the fused silica sample. The sample stops at 10 points for collection of bridge output data versus sample position data as shown below:-
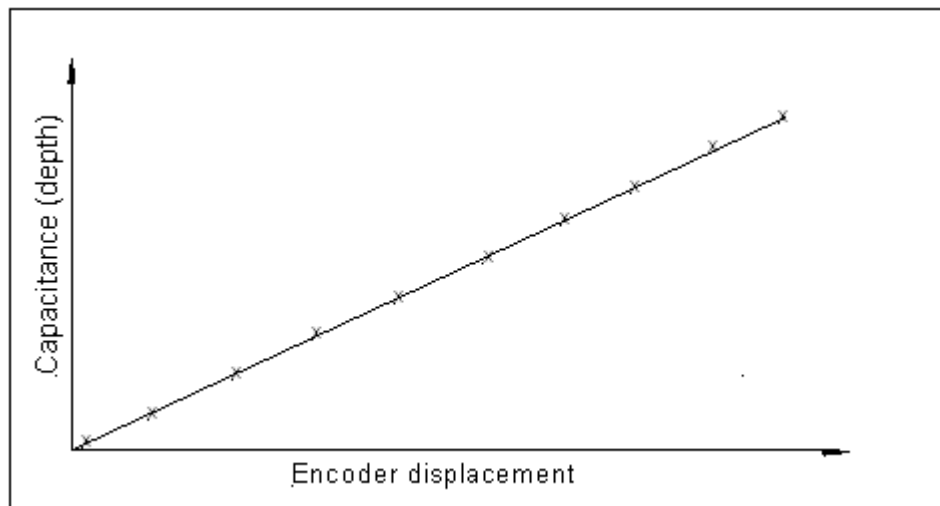
Typical results for the calibration are:-

NT   Depth Calibration Factor = 0.05-0.06 nm/bit (+/- 5 x 10^{-4})
**MT**   Depth Calibration Factor = 0.2-0.23 nm/bit (+/- 5 x 10^{-3})

Note that for ultra-low depth measurements the depth sensitivity can be improved (to less than 0.05 nm/bit) by reducing the capacitor plate spacing

4.1.2.2 Reference sample method

This software routine allows the NanoTest depth calibration factor to be re-calculated using indentation data into a calibration sample with reproducible, isotropic and well known mechanical properties (e.g. fused silica).

The depth calibration can be automatically modified and saved into the current calibration file for future use. All that is required is recent indentation data into fused silica at high loads (this also enables the system compliance to be re-calculated - see below). The *NT.Compliance.AIS* schedule which is pre-loaded onto your hard drive can be used to obtain the high load (150-200 mN) indents.
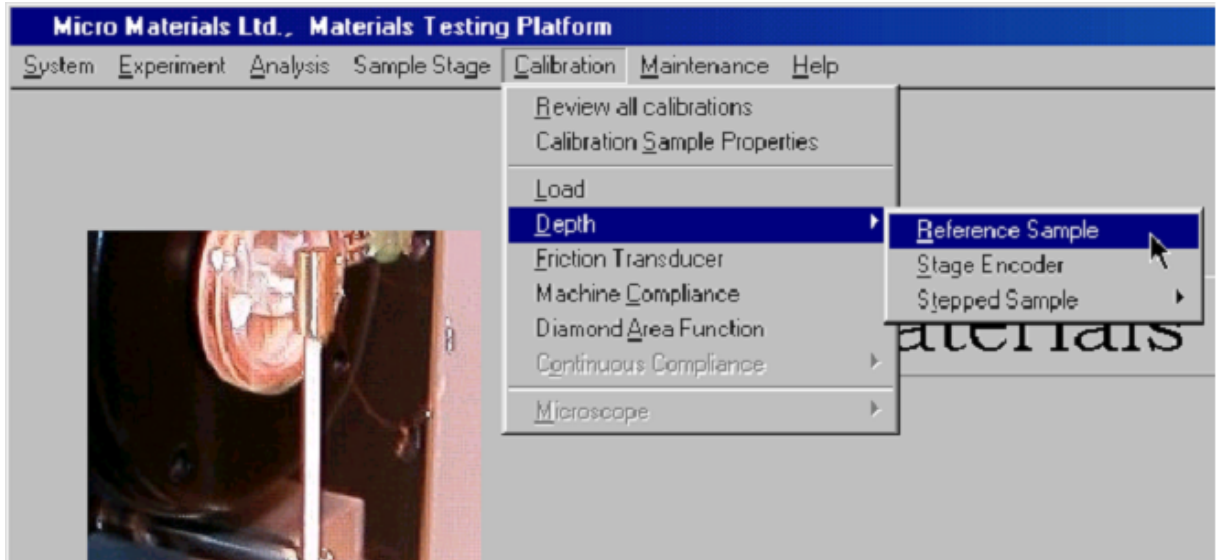
Follow the procedure shown below when the indentations have successfully completed.
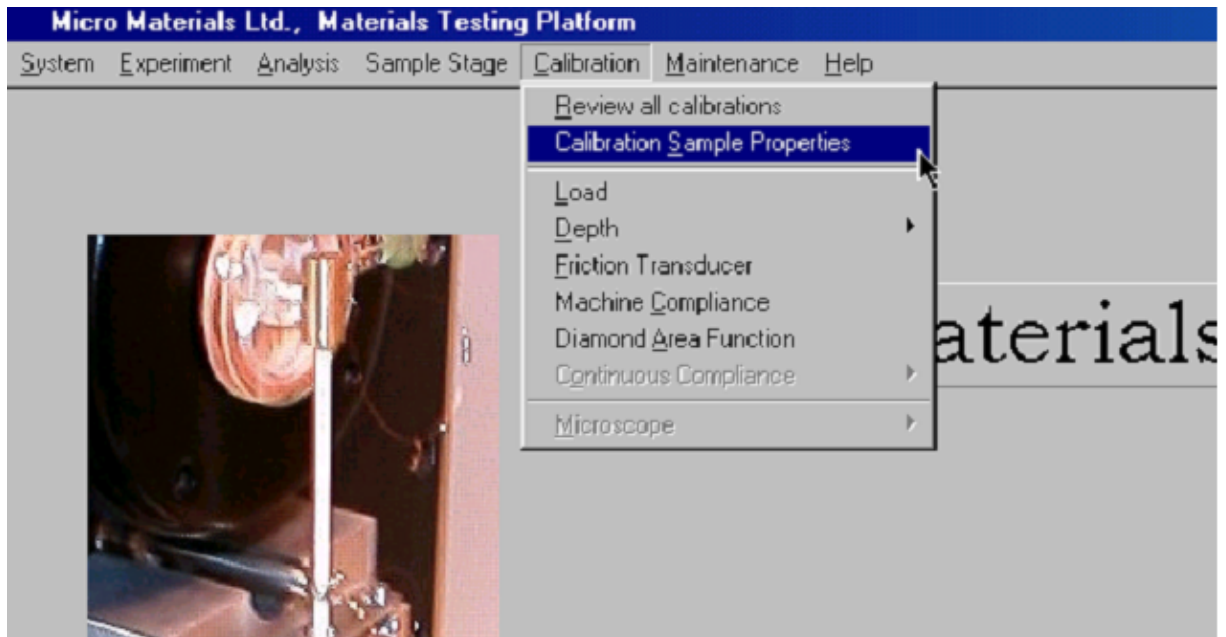
First check the sample properties are correct (the following are the default setting for fused silica)
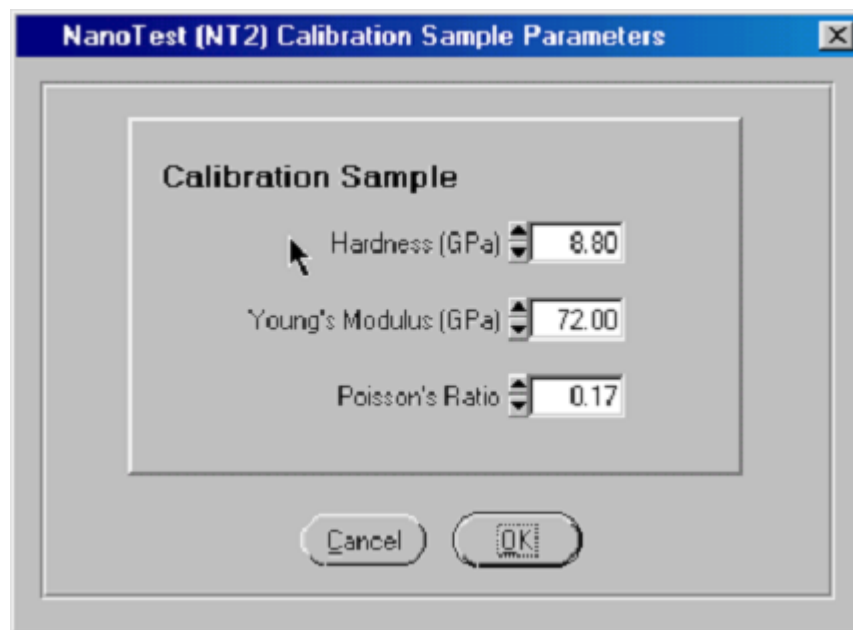
Go to the *depth* option within the calibration menu and select *reference sample*

You are now required to specify the range for the automatic software to perform the calculations (typically +/- 5% of the previous calibration value will suffice). The remaining values can be left as default (analysis definition and indenter area function – in this calibration it is assumed that the Berkovich indenter is essentially ideal at these depths).
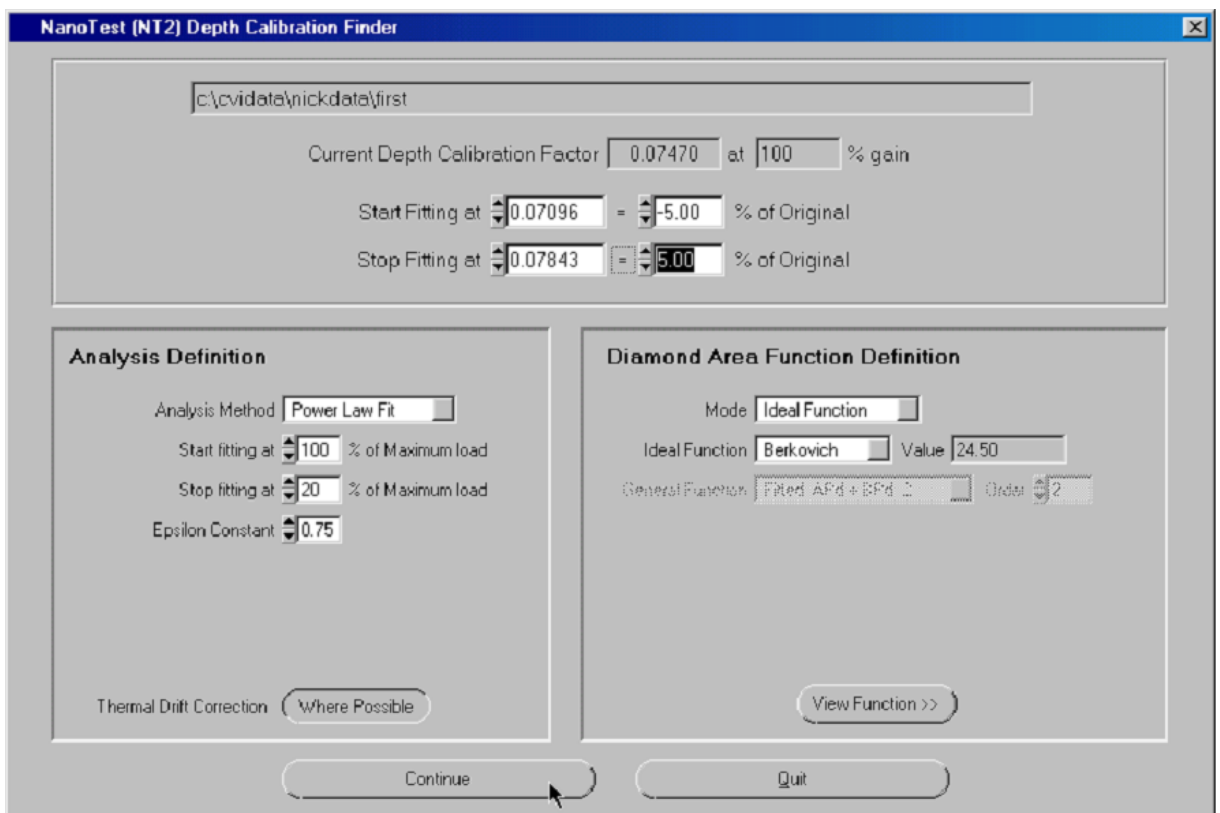
Select continue and the software will then proceed to calculate a more accurate depth calibration value, this will be displayed on screen when completed. The hardness, system compliance and new depth calibration are displayed and the depth calibration can be saved if acceptable.

If the mean hardness is 8.8 (e.g. shown as 8.80 or 8.799) then the calibration has found that the correct depth calibration is within the set limits (e.g. +/- 5%) of the current (previously saved) value. If not (e.g. H = 8.95), then the correct depth calibration is outside this range and the range must be increased (e.g. to +/- 10%) so that a hardness of 8.8 is returned.
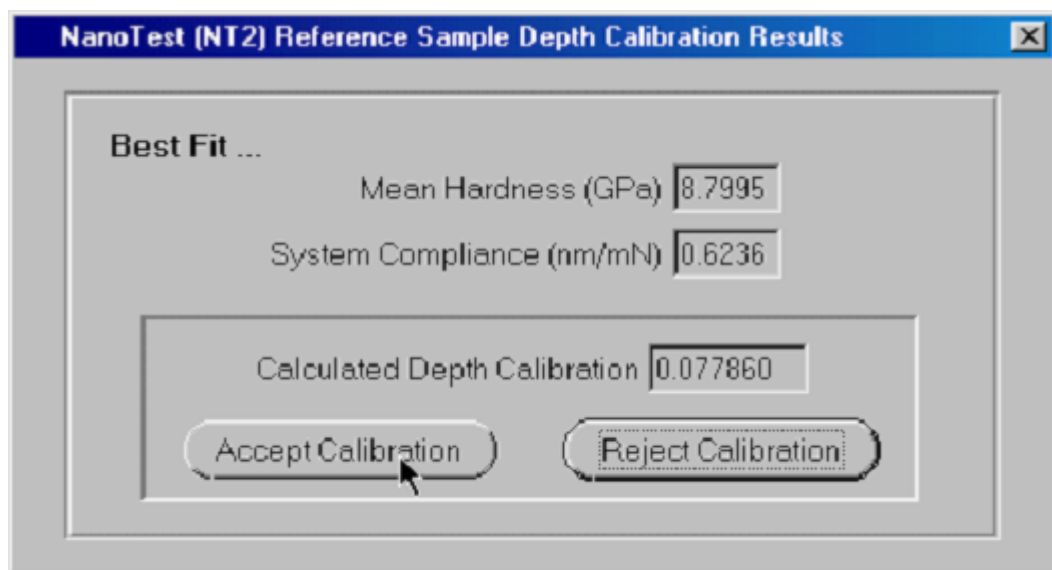
Once saved, the depth calibration is now set for future measurements. With the new depth calibration, a new compliance calculation will be needed (see also 4.1.4), and raw data with the correct depth calibration is needed for this.

To correct indentation data…
    (1) note down the depth calibration value (or highlight the value and press Ctrl+c to copy to the clip board),
    (2) review the data used for the reference sample calculation within the analysis menu.
    (3) Go to the edit drop down menu on the top of the screen and select depth calibration, highlight the value and press Ctrl+v or input the calculated value.
    (4) Exit and save the changes.

Perform the compliance calibration again as in 4.1.4 below with the updated indentation curves and save the new value of machine compliance.

Alternatively, the reference sample method allows for the system compliance to be updated directly, but it is important to note that the compliance is very sensitive to the quality of the raw input data and the exact depth calibration. Small errors in depth calibration can produce inaccurate frame compliance determinations by this approach. If the method returns frame compliance value far from the normal values for your system, then it is possible that the diamond needs cleaning and/or more accurate raw data obtained.

Note: for accurate diamond area functions (see 4.1.5) it is critical that data used in the DAF has the correct depth calibration and machine compliance, so if a DAF schedule has just been completed the data will need to be adjusted for the correct depth calibration.

4.1.3 Friction Transducer

It is only necessary to perform this calibration if you are planning to perform friction measurements!

WARNING – handling friction probes - the friction probes are designed to measure small forces. If a friction probe is dropped onto a hard surface (even from a height of less than 100 mm) the force will break the transducers. Placing a sheet of bubble wrap, bubble side up, onto the baseplate may provide some protection.

When installing the probe it is good practice to avoid applying any unnecessary force across the transducers (i.e. hold the end with the screw when inserting the calibration stub/indenter stub and tightening the screw, and the other end when placing it into the indenter mount on the pendulum). To save weight there is no strain relief between the wires and the solder. Care should be taken to avoid bending the wire at the joints.

Friction forces often increase dramatically when a coating fails, and so friction measurement during scanning/scratching enables accurate critical load values to be assigned.

For the frictional force calibration you will need a friction probe, masses and a calibration stub.

Before calibrating the probe, place it in the diamond holder with the friction calibration stub and allow the system to thermally stabilise. The friction signal can be observed in *maintenance-signal display*. When the signal drift is minimal (we recommend waiting ~15-30 minutes) go into *friction transducer* in the *calibration* menu and follow the 'on-screen' instructions.

We recommend that the calibration is done before each schedule of friction measurements.

4.1.4 Machine compliance

Why is accurate machine compliance important? From the measured data the contact compliance can be determined and then the sample compliance is obtained from *Total (measured) compliance (Ct) = contact compliance (C)+ Machine compliance (Cf)* as shown in the diagram below.

Note that contact compliance = sample compliance + indenter compliance; the indentation data analysis corrects for indenter compliance in the determination of sample hardness and modulus.

When the sample compliance is small (high modulus sample) the machine compliance makes up an appreciable fraction of the total measured deformation and so small errors in the machine compliance can affect the accuracy of the sample modulus determination.
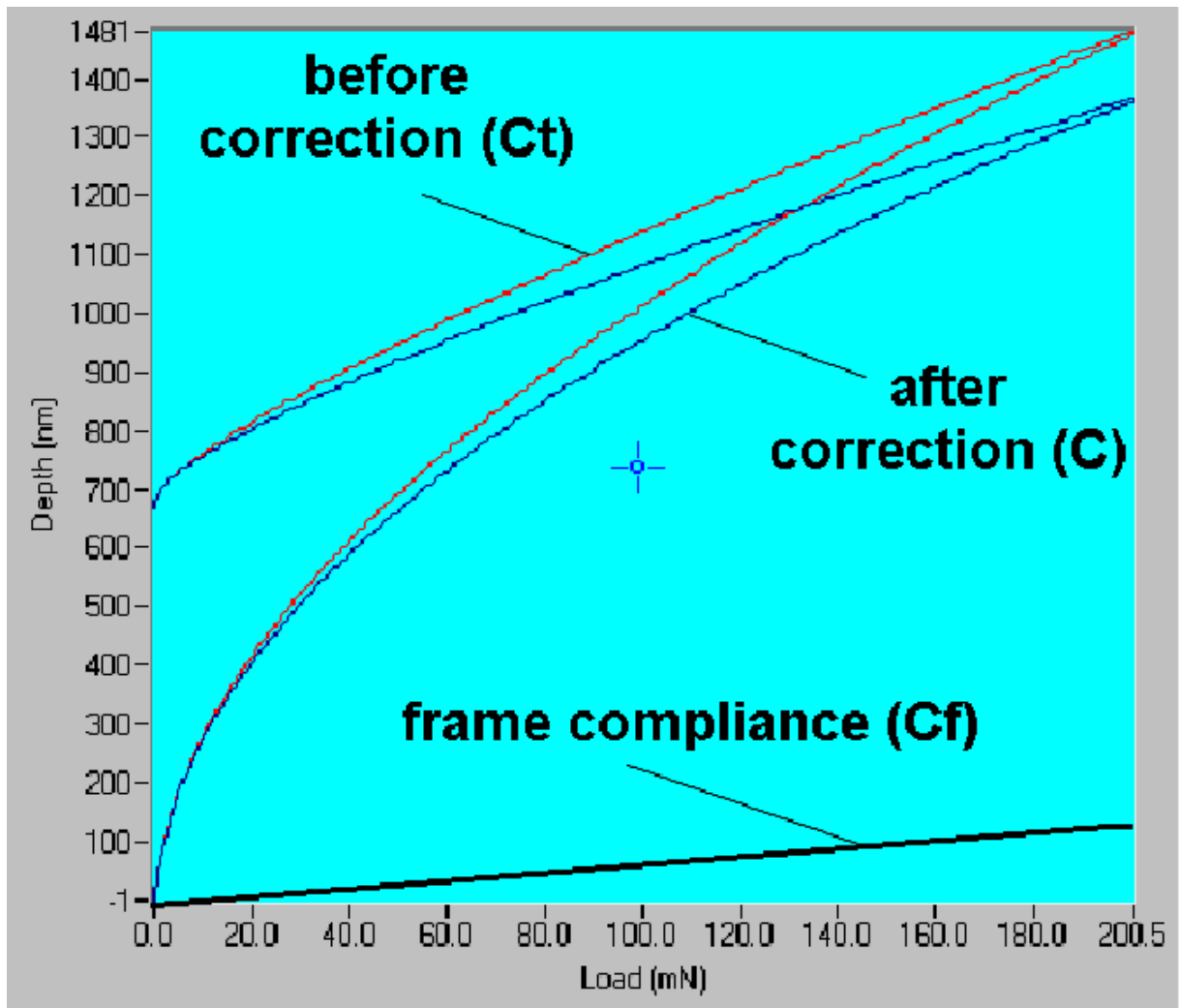
There are 2 different methods for accurately determining the machine compliance of your NanoTest system. These are (1) an indirect method using a reference sample of known modulus (2) a direct compliance method.

4.1.4.1 Indirect machine compliance calibration

This method is favoured in the current ISO standard for nanoindentation (ISO 14577) and works well provided that the diamond area function and depth calibration are known accurately.

For the indirect method you will need a Berkovitch indenter, fused silica calibration standard and test file (*NT Compliance.AIS*). Section 12.1.4 shows how to produce a test file if there is not one on your system. Alternatively, the tutorial in <u>section 6.1.1</u> produces a suitable test file.

Check that the instrument is working properly and the depth calibration is current. Mount a Berkovitch indenter and a fused silica test sample.

Go to *Experiment* then *Indentation* then click *Import Schedule* to bring up the *Indentation Experiment Definition Menu* of the test file. Contact the fused silica surface as described in <u>section 6.1.1</u> then retract 25 μm (or leave the sample in the focal plane with a valid 'sample stage calibration' if using the Hi-Res microscope). Move to a new area of the sample and exit. Run the test file. Review the data (editing out any poor curves) and analyse as described in <u>section 6.2</u>. Then go into *calibration- machine compliance* and add the file. Click on analyse now.
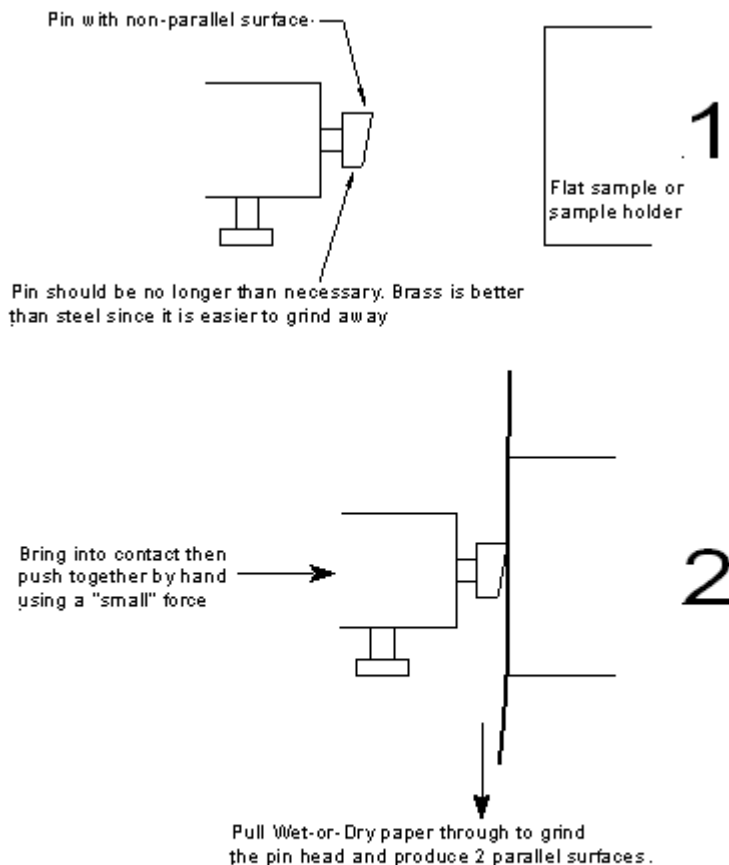
Now *save* the calculated value of machine compliance so that it will be recalled as the *default value* in the analysis of subsequent indentation data.

4.1.4.2 Direct Machine Compliance

For accurate measurement of the frame compliance by the direct method it is necessary that a hard contact exists between the stages and test probe. This is done by using a flat-ended ("pin") probe and gluing this to the Aluminium sample stub as described below. The load ramp test program is used to obtain load vs. depth data that are exported in ASCII and analysed in a spreadsheet to determine the frame compliance.

Procedure
The "pin" should be able to be screwed into the indenter holder just as a normal indenter. The "pin-head" should sit squarely against the indenter mount and then be tightly screwed in. It should have a much larger flat end (e.g. 2.5-5 mm in diameter). When this is brought into contact with the aluminium stub it is important that the opposing surfaces should be nearly parallel. If this is not the case, some deformation will occur and the measured compliance will be higher. To make sure that this is the case, the pin can be made to conform to the sample as shown below:-

Pin with non-parallel surface

.1

Flat sample or
sample holder

Pin should be no longer than necessary. Brass is better
than steel since it is easier to grind away

Bring into contact then
push together by hand
using a "small" force

2

Pull Wet-or-Dry paper through to grind
the pin head and produce 2 parallel surfaces.

Before glueing, rinse the
interface with alcohol and blow dry.

Once the pin has been made to conform to the sample place some superglue on the
end and use the X motor to approach. This can be done at 40 μm/s. Bring the
rectified output down to ~ 4 V.

> **NOTE: A higher initial load should be used so that most of the glue is forced out of the
> contact region and the contact is not compliant.**

Let the glue set for at least 90 minutes.

Then go *maintenance-load ramp test*. Select a filename, choose the maximum load,
ramp rate, number of cycles and hold period at maximum load. Typical values would
be maximum load = 200 mN, ramp rate = 4 mN/s, hold period = 20 s, number of
cycles = 3.

Once the experiment is complete exit the NanoTest program and open the .txt file
output in a spreadsheet such as MS Excel and linear fit to the loading or unloading
data. Typically the fit to linear behaviour is very good, and the slope gives the frame
compliance directly in nm/mN.

Once the compliance has been determined untighten the pin completely so that it can be removed by retracting the stage (move x motor positive).

4.1.5 Diamond area function

Accurate diamond area function determination requires that the load, depth and machine compliance calibrations have all been done previously. Inaccurate machine compliance will affect the accuracy of the diamond area function calibration.

You will need a Berkovich indenter, fused silica reference sample and a test file for this calibration.

The data acquisition procedure is similar to that of the indirect machine compliance procedure, although here, the indentations are to a much wider range of maximum load, which produces a dataset of different plastic depths.

Go to *Experiment* then *Indentation* then click *Import Schedule* to bring up the *Indentation Experiment Definition Menu* of the test file (Diamond Area Function.AIS). Contact the fused silica surface as described in section 6.1.1 then retract 25 μm. Move to a new area of the sample and exit. Run the test file.

If you prefer not to use the test file for DAF calibration, a suitable set of 5 indentation experiments (using **load vs. depth hysteresis**) which covers the range of plastic depths is:-

A) 8 indents, maximum load = 200 mN, minimum load = 150 mN, initial load = 0.05 mN, loading rate = 4 mN/s.
B) 8 indents, maximum load = 140 mN, minimum load = 40 mN, initial load = 0.05 mN, loading rate = 1.8 mN/s.
C) 8 indents, maximum load = 30 mN, minimum load = 5 mN, initial load = 0.03 mN, loading rate = 0.45 mN/s.
D) 8 indents, maximum load = 4 mN, minimum load = 2 mN, initial load = 0.03 mN, loading rate = 0.06 mN/s.
E) 8 indents, maximum load = 2 mN, minimum load = 0.5 mN, initial load = 0.03 mN, loading rate = 0.02 mN/s.

Dwell time at maximum load = 5 s for all experiments. Thermal drift correction can be set to 60 s if required.

Review the data and analyse as described in section 6.2. Then go into *calibration-diamond area function* and add the file. Click on analyse now.

Now review the fit of the fitted function to the raw data.
If there are any poor points that lie off the line and skew the fit, move the cursor to them and click on them to remove them from the fit.
When the fitted line overlays the raw data save with a new filename. The a and b parameters can be viewed when this function is used to analyse any indentation data:-
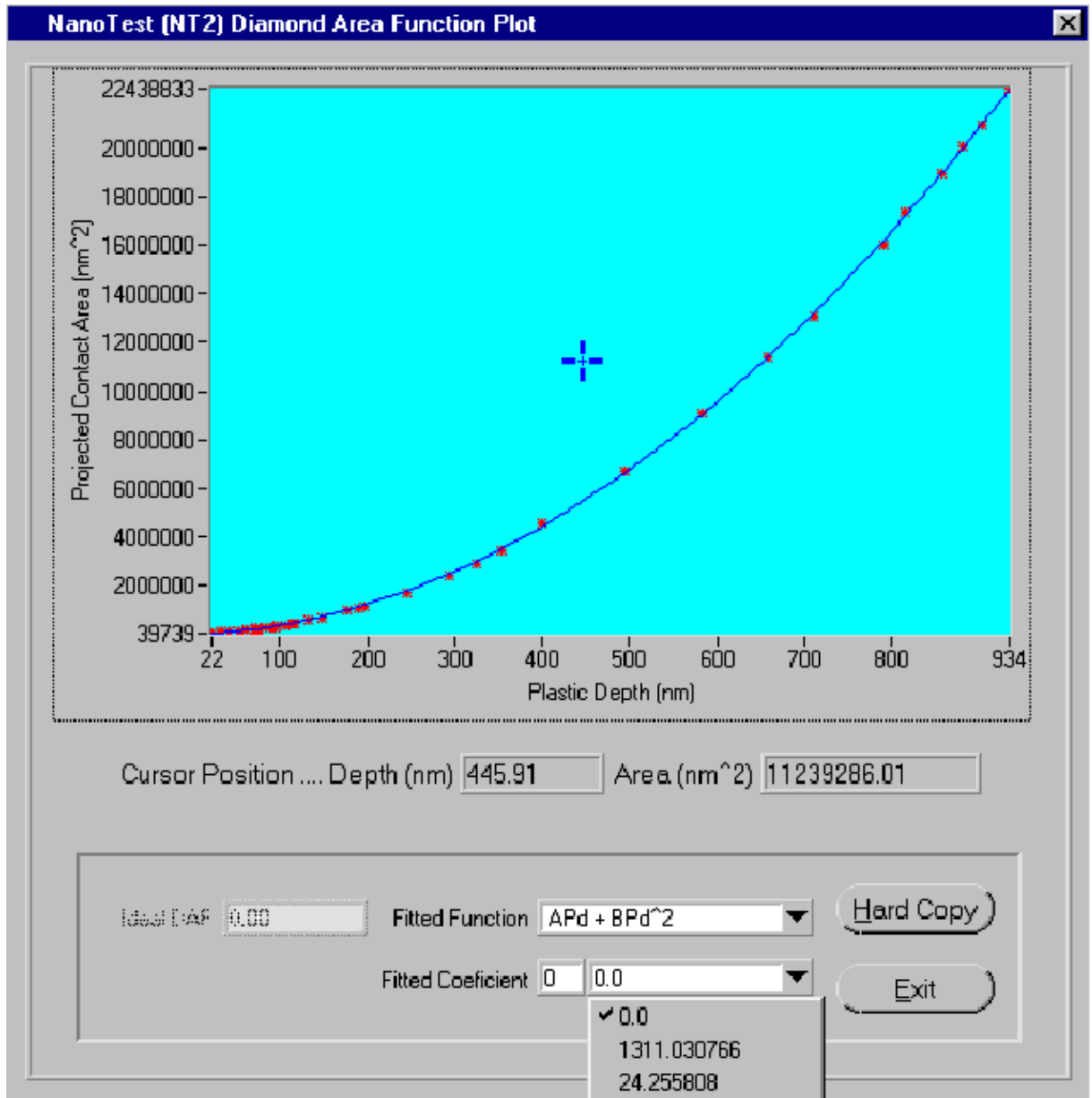
This graph shows a DAF for a sharper Berkovich indenter. Now reanalyse the data as described in 6.2 with the new DAF to assess the accuracy of the new function. For fused silica (quartz), a successful DAF will give constant hardness ~ 8.8 GPa, and constant reduced modulus ~ 69.6 GPa, across the load range 0.5-200 mN.

If the hardness and modulus values are too far out at low depths, then the automatic function can also be used as a "seed value" for a manual DAF calibration. In the manual DAF calibration the user can input the values of k_1 and k_2, and use the new function to fit the calibration data.

As an example, "typical" data for H and E_r from a DAF are shown below. Note that better quality raw indentation data would result in an improved DAF.

Typical data on fused silica
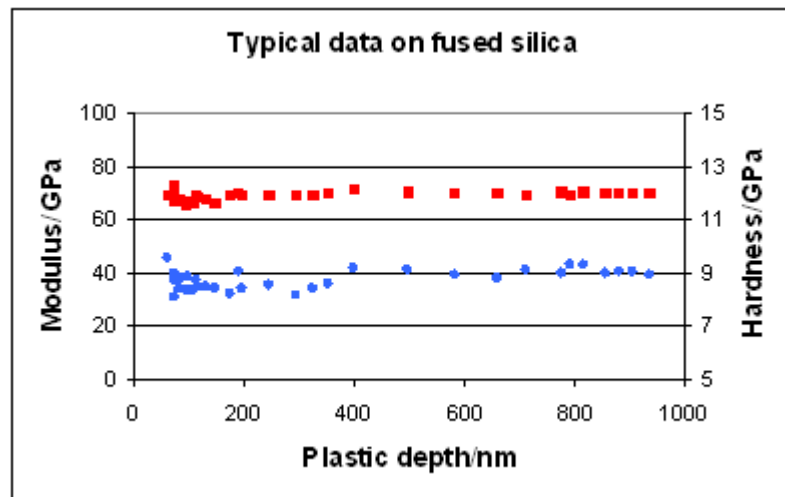
Mean values of Hardness and Modulus from these data:-

Hardness = 8.76 $\pm$ 0.06 GPa
Reduced modulus = 68.9 $\pm$ 0.3 GPa

The Diamond Area Calibration relates the actual, non-ideal diamond contact area to the depth of penetration. This involves performing a series of indentation experiments on a *fused silica* sample. As the largest indent made in this series of experiments should not be more than 2 μm, the measurements are best made with the Depth Amplifier at full gain (100% in the Indentation Schedule).

The target aim of the Diamond Area Calibration is to find the function which can be used across a wide indentation range (0.5-200 mN) to obtain accurate hardness and modulus values which allow for the changing indenter shape.

The calibration determines the constants k_1 and k_2 in the function $A = k_1 h_c^2 + k_2 h_c$, where A is the contact area and h_c is the contact depth. For an ideal Berkovitch indenter k_1 (=B) = 24.5 and k_2 (=A) = 0 nm. For a real Berkovitch indenter, values of k_1 = 21-24 and k_2 = 1500-2000 are typical, although since each diamond is slightly different, these values are only a guideline.

The DAF routine works best if the instrument is working well in a thermally equilibrated, vibration-free environment. Try running the calibration schedule overnight for optimum results. It is good practice to repeat the calibration once every 2-3months or if damage to the diamond is suspected.

Note that practically, since the transition from "ideal" to "rounded" indenter geometry can occur quite abruptly, often indentation data is best fit with the "combined" DAF. This uses the ideal function above the "crossover depth", which is typically about 400 nm.

4.1.6 Microscope

High resolution microscope calibration

Zoom microscope calibration

**4.1.6.1 High Resolution Microscope Calibration**

Important Note: When the high-resolution microscope has been successfully calibrated the sample is left in the **focal plane** before indentations. When the microscope is On the sample should be in the focal plane before performing any experiments. The NanoTest software makes the adjustment to bring it to the measurement plane automatically when experiments are performed. When the microscope has been calibrated NEVER start a schedule with the sample in the measurement plane. This will cause the pendulum to break as the software will think the sample is in the focal plane and apply the correction to bring it 30 mm closer to the pendulum.

The aim of the microscope calibrations are:-
(1) to accurately determine the distance between the focal plane and
    measurement plane (sample stage calibration)
(2) to accurately position indentations on features of interest (crosshair calibration)

To calibrate the high resolution microscope **follow the steps below in the order they are shown**:

1. calibrate the sample stage (section 4.1.6.1.1)
2. produce a series of indentations (see 4.1.6.1.2 crosshair calibration for details)
3. use these for the crosshair calibration (section 4.1.6.1.2)
4. save calibration and exit
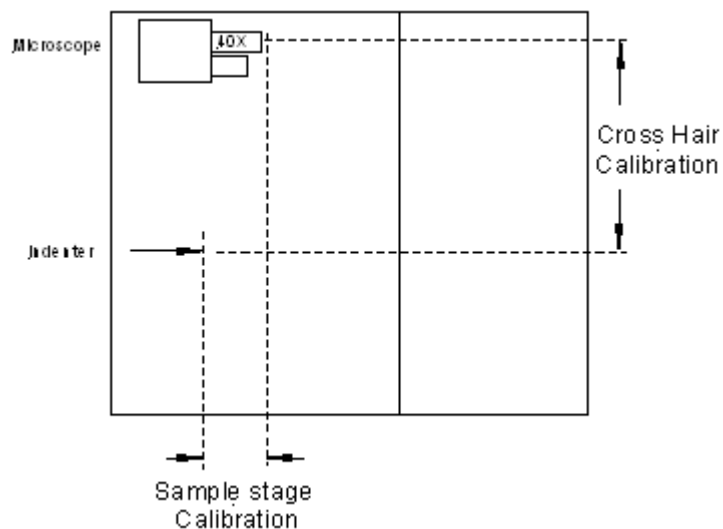
The calibration procedure is identical for both loading heads (NT and MT) and the displacement calibrations (as all other values) will be saved when alternating between the two options.

4.1.6.1.1 Sample Stage Calibration

Go into *Calibration-Microscope-Sample Stage*. You will be asked "are you sure you want to calibrate the sample stage again?" Press Enter.

Now move the x-motor positive so that the sample stage is clear of the microscope objective when translating to the microscope position (this will be about 30 mm). Move the transfer stage over to the high resolution microscope position by pressing the switch on the front of the Transverse stage control unit. Click OK when the motors have stopped moving.

Now focus the sample using the highest magnification objective lens. Focussing with this lens minimises error in determining the focal distance. The focal distance with the x40 objective is about 0.6 mm. The objectives are fully spring-loaded, and are not easily damaged.

Tip: practice moving the x motor positive and negative at very slow speed (e.g. 0.5 μm/s) from one side of the focal point to to the other. This will help you to gauge accurately when the sample is in focus. Press stop when in focus.

When you are happy that the sample is in focus click on the *sample in focus* button.

Now move the stage back to the indenter using the switch on the transfer stage as prompted.

Switch to x negative then press start to find the surface. Fast (250 μm/s) motor movement is fine when you are far from the surface. As you get to within 2-3 mm of the surface press **stop** then lower the speed to ~50 μm/s and re-start.

Make sure when contact is made the stage is travelling slowly by switching down again (to the safe contact speed of 3.8 μm/s) when a few hundred μm from the surface.

Using the x positive, retract 50 microns after making contact (a similar distance to the 25 microns used when the microscope is not being used, but a little larger to allow for any error in the focal plane determination) to define the measurement plane, and exit.

Press *Yes* to save. The system will now move from the measurement to the focal plane so that the sample surface is now left in the focal plane.

> **Remember the sample must always be in focus (+/- 5μm) on high magnification - any discrepancy in the in the focal plane calibration may damage the pendulum.**

4.1.6.1.2 Crosshair calibration

To calibrate the crosshairs it is necessary to use the probe to produce a feature (such

as a line or grid of deep indentations) easily seen with the microscope at low magnification.

In the indentation schedule create a series of high load indentations with a soft material that exhibits little elastic recovery (e.g. polished aluminium or copper). If possible use a highly polished smooth surface. A test file can be imported (Experiment-Indentation-Import schedule) and run if convenient.

DO NOT USE the fused silica reference sample for the crosshair calibration.

Note that having calibrated the sample stage the sample is left in the focal plane and therefore the indentation process involves automatic movement to the measurement plane (50 microns from the surface) first.

Select calibration/microscope/crosshair positioning and follow the on screen instructions.

Depending on the accuracy of (any) previous calibrations, it may be necessary to use the low magnification objectives to first find the indentations.

Once the indentations have been found use the y and z motors to position the crosshairs at the centre of the first indentation in the row.

Switch to higher power objectives making small adjustments until the centre of that indentation is at the crosshairs with the x 40 objective.

Press save to store the y and z coordinates.

Once the indenter has been changed, the microscope 'sample stage' calibration needs to be repeated (as different indenters will protrude out of the diamond holder different distances so the old calibration is no longer valid)   **If the sample is changed, ensure the new surface is in the focal plane (this will keep the sample stage calibration valid).**

Important Note: <u>When the high-resolution microscope has been calibrated the sample is left in the focal plane before indentations.</u> When the microscope is On the sample should be in the focal plane before performing any experiments. The NanoTest software makes the adjustment to bring it to the measurement plane automatically when experiments are performed. When the microscope has been calibrated NEVER start a schedule with the sample in the measurement plane. This will cause the pendulum to break as the software will think the sample is in the focal plane and apply the correction to bring it 30 mm closer to the pendulum.

4.1.6.2 Zoom Microscope Calibration

The zoom microscope calibration is similar to the crosshair calibration described above. Follow the 5-step procedure below.

1. In the indentation schedule create a series of high load indentations into a soft material that exhibits little elastic recovery (e.g. polished *aluminium* or copper). If possible use a *highly polished smooth surface*. Set the depth gain to 30% or less so that the loading curve stops when the maximum load rather than depth is reached.

2. Withdraw the sample about 20-30 mm in *sample positioning*. Then *Move the sample from indenter to microscope* in *Sample Positioning*.

3. Now enter *Calibration-Microscope-Crosshair calibration*. If simply re-calibrating (fine-tuning an existing calibration) go straight to step 4.

But if there is no current calibration (or the calibration has been reset to 0) then the indenter will have remained in front of the indenter at step 2. In this case you will need to move over to the microscope using the Y motor (a distance of about 30 mm as shown in the diagram below) before proceeding to step 4 below.

4. Use Y and Z motors to find the grid of indents.

> **Tip: to more easily find the indentations move the light sources slightly so that they do not shine directly on the sample surface, as this will show any gross surface scratches rather than the indentations. In reflected light it is often very easy to see the grid of indents which show up darker.**

5. When the indents have been found move Y and Z motors to position the *first* indent under the cross hair, then *save* the crosshair calibration.
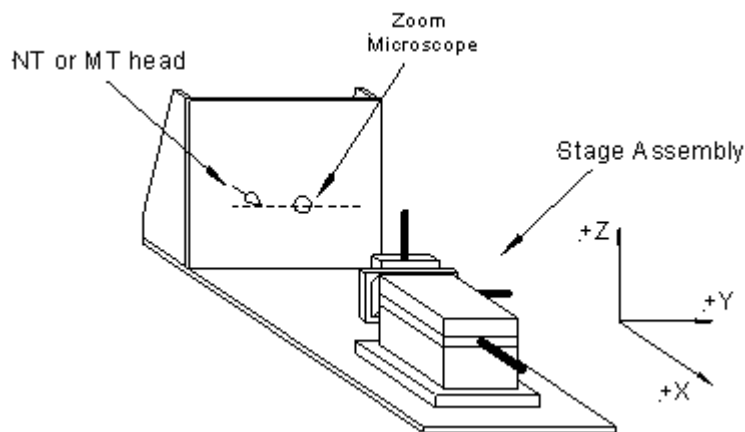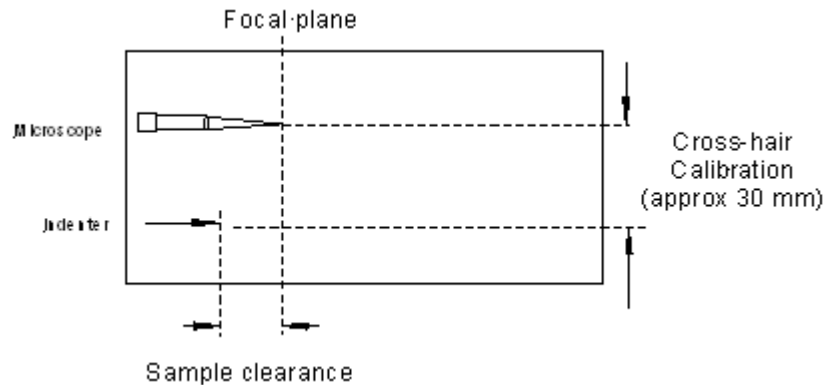
With the zoom microscope it is not possible to accurately calibrate the focal plane-measurement plane distance. The sample can appear "in focus" over a distance of several hundred microns since the objective has only low magnification. Using the same procedure as for the high resolution microscope would risk damaging the sample/indenter or even the pendulum, so the movement to the sample surface is done during the schedule set-up process.

The 'sample clearance' displacement to the microscope focal plane will vary depending on system configuration (with the scanning option, a distance of 25mm is set for friction transducer use and 5mm without). It is good practice to move x positive to ensure the sample will clear the indenter before clicking on *Move sample from microscope to indenter*.

> Important note:- with the zoom microscope <u>it is only possible to test 1 sample at a time</u>. Samples of different heights may appear to be in focus under the microscope due to the relatively large focal distance of the zoom lens. But damage to the pendulum (bending springs or even breaking the ceramic) will occur if samples are of different heights when moving between samples/approaching during a schedule.
>
> We do <u>not</u> recommend setting up schedules on more than 1 sample with the zoom microscope.

4.2 Maintenance Checks

The main daily checks are

1. bridge box adjustment
2. noise levels in signal display
3. zero load calibration test

4.2.1 Bridge Box Adjustment

The NanoTest uses a capacitive bridge circuit for displacement measurement. It is important to balance the bridge accurately to ensure maximum sensitivity to small displacements. The pendulum test is an indicator that the electronics are all plugged

in properly and functioning correctly, and secondly, it shows whether the pendulum is mobile or not. Operations such as sample contact and calibrations depend on the user being able to obtain an acceptable pendulum test.

Normal Balance procedure:

Use a combination of lower 2 knobs on the bridge box to
1)   adjust the rectified output so that a stable minimum value of <0.1 V can be reached
2)   set up a reference level (7.9V) with the lowest knob value only (usually counter-clockwise rotation)
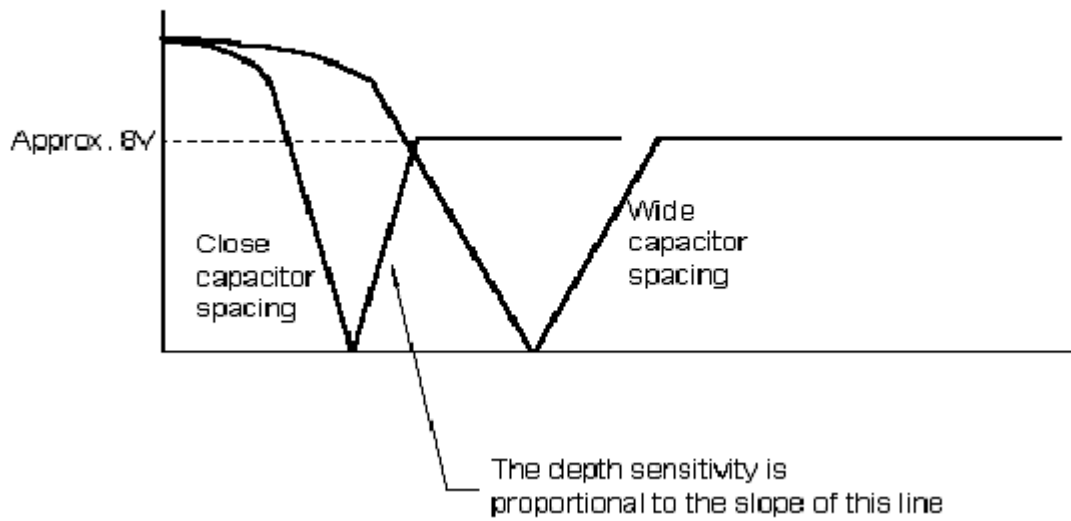
Now run the pendulum test (the signal should initially remain at 10V for 5-10 seconds before decreasing). The system lights on the left of the screen light up when a good reference and minimum level have been attained. The graph below shows a successful pendulum test. If both lights do not light up, then click on *Adjust Bridge Box* and re-tune the bridge box, then run another pendulum test. Once the test is being performed satisfactorily, click on *Quit* to exit bridge box adjustment.
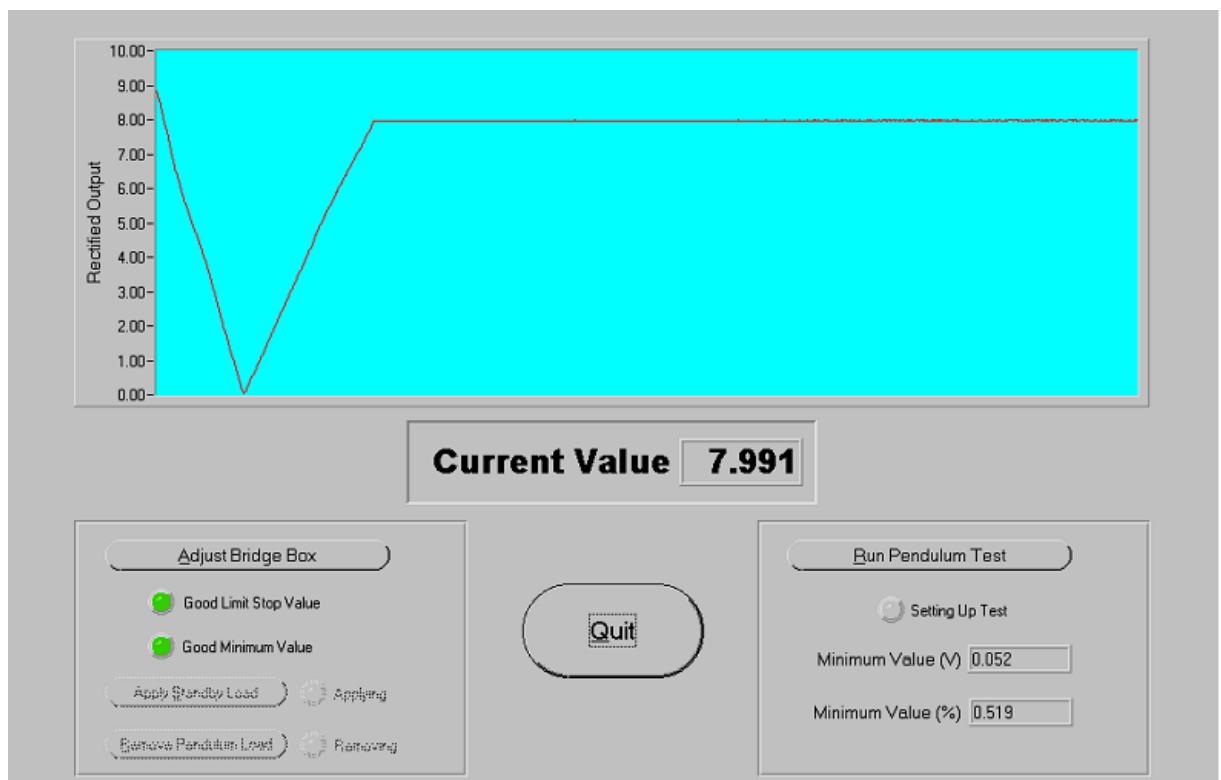
The following sketches show schematically what happens during a pendulum test.

Pendulum Test With 100% Gain

Approx. 8V

Close capacitor spacing

Wide capacitor spacing

The depth sensitivity is proportional to the slope of this line

Pendulum Test

Test location, with the pendulum just off the limit stop

Bridge is balanced at this point. The 3 bridge box components (knobs) plus the NanoTest capacitor when at this particular spacing give a balanced condition.

Increasing coil current
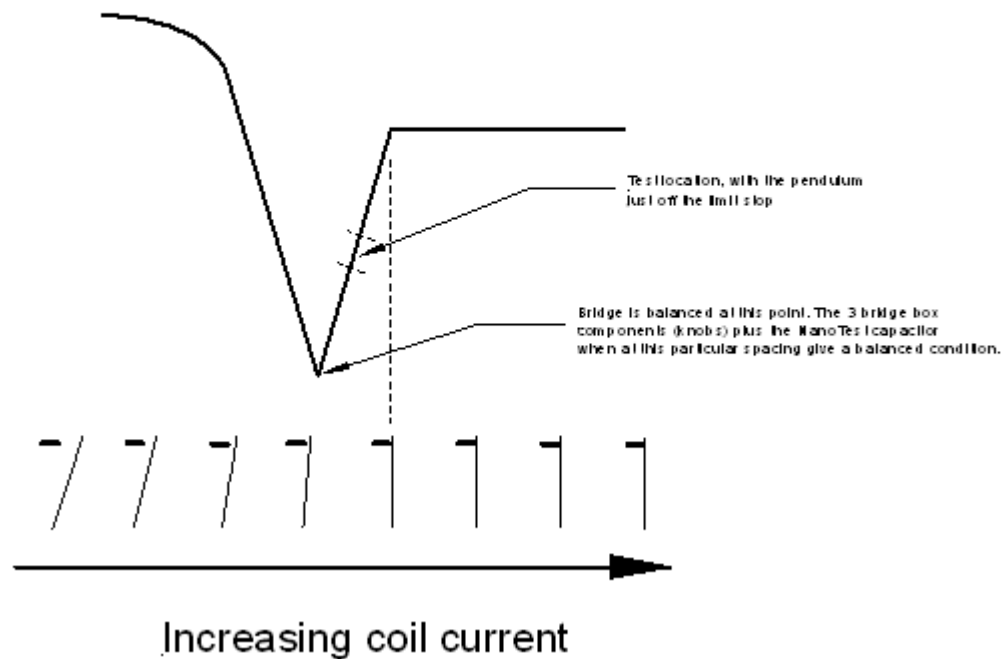
4.2.1.1 Bridge Box Adjustment - Troubleshooting (see also Appendix 14.4)

Problem	Likely Cause	Solution
signal does not go any further down than 8 V	you are on the wrong side of the balance position	turn the signal in the direction which causes it to minimise then go through the minimum point and back up to 8 V
signal remains at 10 V	balance weight is too far out.	move the counter-weight further in
signal remains at 8 V	pendulum stays on the limit-stop whether or not a coil current is applied - the counter-weight is having no effect	move the counter-weight much further out
rounding of the curve as contact is made	build-up of contamination on the surface of the limit-stop	clean the contacts using paper soaked in alcohol then immediately use an air spray to remove any liquid
signal oscillating on start of the pendulum test	counter-weight is too close to the mechanical balance of the pendulum causing it to swing excessively as the coil voltage is applied	counter-weight needs to be moved slightly further out. One scroll of the screen at 10V should suffice (5 to 10 seconds at maximum level)

Try not to alter the position of the top (coarse sensitivity) knob too much during the bridge box adjustment. If this knob is moved too far from the balance position it may prove difficult to quickly re-balance without the aid of an oscilloscope. If the bridge box is very far off 'tune', it can be difficult to adjust the signal down. In this case, it is easier to adjust while looking at the signal *NT Depth In* on an oscilloscope. At this stage, the signal is still very small and any adjustment made with the knobs will be seen on the oscilloscope. Adjust until the signal is minimised. Once this is done, reconnect the signal lead to the Control Box.

> **More on the bridge box**
> The bridge unit contains a 5 KHz sine wave generator. The 5 KHz signal is applied across a 4-element AC bridge. Three elements of the circuit are adjustable by means of the three bridge unit controls. The fourth element in the circuit is the NanoTest capacitor. By tuning the bridge components so that the bridge is close to its balance condition, it is possible to look at the very smallest change in signal due to a change in the capacitance of the NanoTest capacitor. This will correspond to very small changes in depth when the machine is being used. The signal that comes from the moving plates goes into the unit through a short, gold coaxial lead. This contributes a large part of the capacitance being 'tuned' by the other bridge components and it is very important that it is firmly connected for the best results.

When the pendulum has been removed, it may be necessary to use all 3 knobs on the bridge box to get the signal on-screen.

Note: There is more on troubleshooting the bridge box set up in Appendix 14.4.

4.2.2 Signal Display

Vibration levels can be checked here. Go to Maintenance then Signal Display and click on depth+offset. Switch the input range to +/- 100 mV and then Run. Now bring the signal on-screen with the 'auto-scale' and observe the electrical signal. It should appear fairly constant even at this high sensitivity. Press stop and look at the RMS variation - it should be less than about 0.3 mV for most NanoTest systems if the anti-vibration table is functioning effectively and the cables are not under tension.

In addition to looking at the level of out-of-contact noise, useful information regarding the effectiveness of the anti-vibration table can be gained by looking at the depth+offset signal whilst the indenter is in contact with a test sample. Use the sample stage position menu to bring the indenter in contact with the test sample at slow speed. Exit with the indenter in contact (rectified output at ~ 4 V). In signal display check the depth+offset signal. If the anti-vibration table is not functioning properly then values considerably greater than those out of contact will be seen.

Important - after exiting the signal display menu immediately go into sample stage and move the indenter out of contact (move x positive until rectified output reads 8 V and continue for 20 μm).

4.2.3 Zero Load Calibration Test

Important: The zero load calibration test should be done each time the indenter is changed (as the balance weight needs to be moved for different indenter masses). Watch the test as it runs and confirm that the zero load calibrations runs in the low and high load ranges and confirm it looks like a pendulum test (with no damping).

The zero load calibration test provides just enough constant coil current to bring the pendulum to a vertical, mechanical equilibrium position against the limit stop. The calibration is automatic and does not require a sample to be present. The calibration is done automatically (at least once, and we recommend 3 times) before each experiment in a schedule.

Poor zero load calibration is a often the result of the same factors which can lead to poor pendulum tests in bridge box adjustment and are a common cause of problems in subsequent indentations. If there are problems with the zero load calibration look at the table 4.2.1.1, and then re-try the calibration from the maintenance menu.

The Zero Load Calibration determines a minimum coil voltage necessary to hold the pendulum onto the limit-stop. The process is essentially the same as the pendulum test (Bridge Box Adjustment) but while the force is being applied to the coil, it is measured to extract the value required to just bring the pendulum into contact with the limit stop. This calibration benefits from the pendulum test characteristic being as close to ideal as possible.

As the calibration is very exacting (in the order a few μN), it needs to be done often. External factors such as temperature stability of the cabinet and of the electronics can cause the value to alter. In general carry out a minimum of 3 calibrations before measurements and other calibrations. The calibration does not make contact with the sample surface and so does not need a specific sample to be in the machine.

4.2.4 Other checks

In the *Maintenance* pull-down menu there are 3 other options:-

Load Ramp test
Test system components
Produce System Report

In general, these checks will only need to be done to determine if there is a problem with the system operation or optional components.

Load Ramp allows testing of the pendulum loading, useful for checking pivot springs for damage (typically by sample-diamond contact at too high speed).

Test system components allows various tests to be carried out on optional equipment supplied with the instrument (e.g. Pin-on-Disc motor or Impact signal generator)

System report creates a .txt file of the latest calibration and system parameter values, sometimes requested by Micro Materials to help diagnose problems.

5 Setting up

Before using the NanoTest system on test samples it is critical that the instrument is fully calibrated and that both diamond and sample are properly mounted in the instrument. This section describes the procedure for preparing to perform measurements. [Note this "pre-set up" is identical for indentation, scratch and impact tests] The actual scheduling and running of measurements is described in of this manual.

5.1 Sample and diamond mounting

Details of good sample mounting procedures are given in .

Which Diamond to use?
The choice of which type of diamond to use depends on the type of test planned and the nature of the test sample.

Indenter	Geometry	Typical Use	Typical Dimensions
Berkovich	Three Faced Pyramid (with same area to depth ratio as Vickers)	Nanoindentation	Tip radius 100 - 500 nm
Vickers	Four Faced Pyramid	Microindentation	Tip radius generally not defined
Rockwell 'C'	Conical with Spherical end form	Adhesion/Scratch Testing	Radius ~ 200 μm
Sharp Rockwell	Conical with Spherical end form	Scratch Testing Impact testing	Radius ~ 25 μm
Knoop	Four facets with the long axis seven times the short axis (rhombic)	Anisotropy	Tip radius generally not defined
Spherical	Round tip with specified radius	Stress/Strain Measurement Polymer testing	Selection between 5 μm and 100 μm
90° cubic	Three faces with cube corner (sharper than a Berkovich)	Fracture toughness	As Berkovich
Ceramic	Atomically sharp fractured alumina	Surface Roughness	Not defined

Note: Berkovich (pyramidal) indenters should **not** be used for scratch or impact testing unless the sample is very soft (e.g. polymeric) as practice has shown that they become very quickly damaged (blunted) during use. For these type of tests spherical/conical diamonds are preferred.

Once the calibrations have been completed satisfactorily and are "current", it is necessary to perform a few quick checks before attempting to run a schedule within the *Experiment* menu.

5.2 Daily checks

The main daily checks are

(1)   ensuring that the instrument can perform a good pendulum test
    (see Bridge Box Adjustment)
(2)   ensuring the instrument can perform a good zero load calibration
    (see section 4.2.3)
(3)   checking on noise levels (see signal display - section 4.2.2)

**As a final check before setting up any schedules:-**
4) Make sure the bridge box is set-up correctly - the system is on *standby* at 8V NT [or 4V for the MT]
5) If the microscope is 'enabled', the *sample stage calibration* must be valid and the sample placed in the focal plane (see 4.1.6 microscope calibration)
6) If the microscope is not being used, set up a nominal 20μm displacement between the indenter and the sample.

5.3 Sample positioning

This is primarily used for changing sample and/or indenter. Moving around samples/between samples is also possible from here, but note that this can also be done from within the *Experiment* menus during set up of schedules, and this is often more convenient.

5.4 Damping Plate configuration

The use of the damping plate may sometimes be required for low load experiments and the use of samples with a brittle surface. The plates minimise the pendulum oscillation prior to measurement, thus reducing the possibility of sample damage upon low load contact.
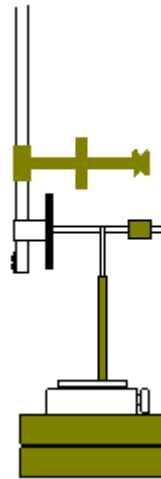
Set-up procedure (if not previously installed):

1. With the damping plate mounted to the machine base plate (2 x M4 screws), the glass slides should be 1-2mm apart and the mini stage at mid-travel (make sure the movable slide is directly opposing the pendulum slide and the stage is perpendicular to the pendulum, the two slides should be at the same height also).

2. Adjust the outer plate position using the manual stage so that it makes contact with the inner plate (blow off any dust first). The outer plate pivot should be quite loose, which allows plate alignment.
    Use the rectified output as a gauge for distance (when this drops below 8V, contact has been made between the two plates)

3. When the two plates are in contact (drive the stage counter-clockwise slowly until rectified output is approximately zero), drip a small amount of quick-setting epoxy over the bearing to lock it in position. (This can easily be broken

off later if necessary.)

4. When the adhesive has set (5-10 minutes should suffice), monitor the rectified output signal as the manual stage micrometer is moved clockwise. Just after the signal reaches 8V, stop. This separates the plates and provides a spacing in the order of μm's.

5. Carry out a pendulum test with the new set-up. The pendulum should only just hit the limit stop before the plates touch each other and plot should show more curvature than before as the limit stop is approached.

6. With the damping plate (and strong pendulum test curvature) you have to slow the zero load calibration down ('system'…'unprotected parameters' to 0.2s and 0.3s for the low and high load respectively). With no plate, the values should be returned to default…zero (low) and 0.1 (high).

> **The damping plate can easily be removed if not required, carefully lift the top section off the machine (be careful not to disturb the pendulum – this might alter the damping plate alignment and possibly damage the pendulum pivot) and store for future use.**

To install again, simply replace on the three grooves provided (this ensures exact replacement – therefore no need to set-up the displacement again)

The zero load calibration delay settings must be configured to the relevant hardware set-up (point 6).

5.5 Checklist

It is good practice to mentally check that the instrument is working at its optimum level before testing unknown samples, and also. Such a list includes…

1) correct type of diamond for the type of test
2) recent depth calibration
3) instrument noise level acceptable
4) bridge box adjustment is OK
5) sample properly mounted
6) microscope calibrations are valid (and instrument is in focal plane - high resolution microscope)
7) daily checks completed

If you are unsure as to whether a calibration or daily check needs re-doing, consult the calibration frequency table below:

Calibration/daily check	*How often?*	*Relevant section of this manual*

Bridge Box adjustment (& Pendulum Test)	*each morning*	*4.2.1*
Zero Load	*3 before each set of measurements*	*4.2.3*
Load	*every 3 months*	*4.1.1*
Depth	*weekly or whenever the indenter is changed*	*4.1.2*
Machine Compliance	*every 3 months or whenever the stage components are dismantled*	*4.1.4*
Diamond Area Function	*whenever the indenter is renewed or every 3 months*	*4.1.5*

You are now ready to perform indentation, scanning or impact measurements.

6 Running an indentation schedule and data analysis

Before performing any tests it is good practice to first confirm that the instrument passes the checklist described in section 5.2.

In the Windows NanoTest software all experiments are run as *schedules*.

A schedule is a series of experiments (which can be either on the same or different samples) which will be run in sequence at a time specified. Each experiment is saved separately with a different filename.

6.1 Setting up a new indentation schedule

Go to *Experiment* then *Indentation* then click on *Define new schedule*. In *select experiment type* choose the required experiment type to bring up the *Indentation Experiment Definition Menu*.

In *select experiment type* you can choose between the following different types of experiment:-
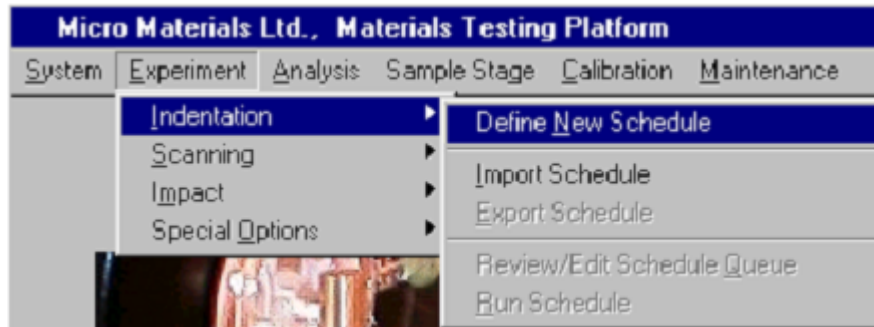
Load vs. depth hysteresis
Multiple Cycle with increasing Load
Multiple cycle to the same load
Creep Test
Spherical Indentation

These experiments are described as tutorial examples.

6.1.1 Tutorial:- setting up a load vs. depth hysteresis schedule

AIM: the following simple tutorial improves familiarity with the Indentation Schedule menu, and sets up a schedule which can be used to perform 200 mN indents into fused silica.

Go to *Experiment* then *Indentation* then click on *Define new schedule*.

In *select experiment type* choose *Load vs. Depth Hysteresis*.
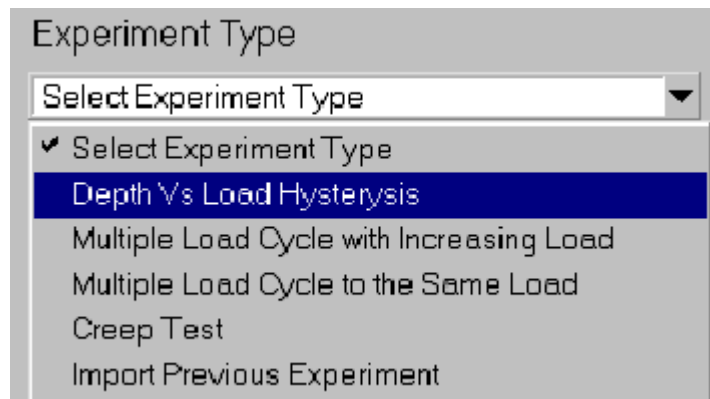
The *Indentation Experiment Definition Menu* contains 4 boxes: *Pre-experiment Operations; Load Ramp Termination; Load Ramp Control* and *Sample Stage Control*. Fields in all of these must be set up correctly before the experiment can be run.

In the *Pre-Experiment Operations* set the number of zero load calibrations to 3 and the experiment start delay to 10 s. Then in the *Load Ramp Termination Box* set the Method to *First condition*. This will stop the load ramp when whichever of the maximum load or the maximum depth are reached first. Now set the maximum and minimum load to 200 mN. Set the depth gain to 100 % then make sure the maximum and minimum depths are equal to this value. You should end up with a box looking something like:-

Load Ramp Termination

Method | First Condition ▼
Available Load (mN) | 470.00
Maximum Load (mN) ⬍ | 200.00
Minimum Load (mN) ⬍ | 200.00
Load Range | High ▼

Available Depth (nm) | 1742.00
Maximum Depth (nm) ⬍ | 1742.00
Minimum Depth (nm) ⬍ | 1742.00
Depth Gain % ⬍ | 100

Now find the *load ramp control box*. Set Method to *Standard* and then set the initial load to 0.1 mN and the limit stop load to 0.1 mN. Set the loading rate and unloading rate equal to a value around 3-4 mN/s, and set the dwell period at maximum load to 5 s. The test time (the total for the loading and unloading ramps and the dwell period at maximum load) will then be about 2 mins. Note: If the parameters are not relevant to the type of test then that field will remain in faint type and cannot be selected (for example, in the depth vs. Load hysteresis the parameters for multiple loading [unloading percentage etc] do not apply. The Load Ramp Control Box should look like this.

Load Ramp Control

Initial Load (mN) ⬍ | 0.10
Limit Stop Load (mN) ⬍ | 0.10

Loading Rate (mN/S) | 3.3 ▼
Unloading Rate (mN/S) | 3.3 ▼

Unloading Percentage (%) ⬍ | 30
Number of Load Cycles ⬍ | 2

Dwell Period at Maximum Load (S) ⬍ | 5
Dwell Period at % Unloading (S) ⬍ | 10

Now find the *sample stage control box*.

In this box you will need to:-
(1)   position the indenter ready for the first indentation
(2)   define the number of indents and their location relative to this first indent
(3)   choose the retraction distance between indentations
(4)   choose a file name for the saved indentation data.

(1) Click on *Define>>* to bring up the following screen….
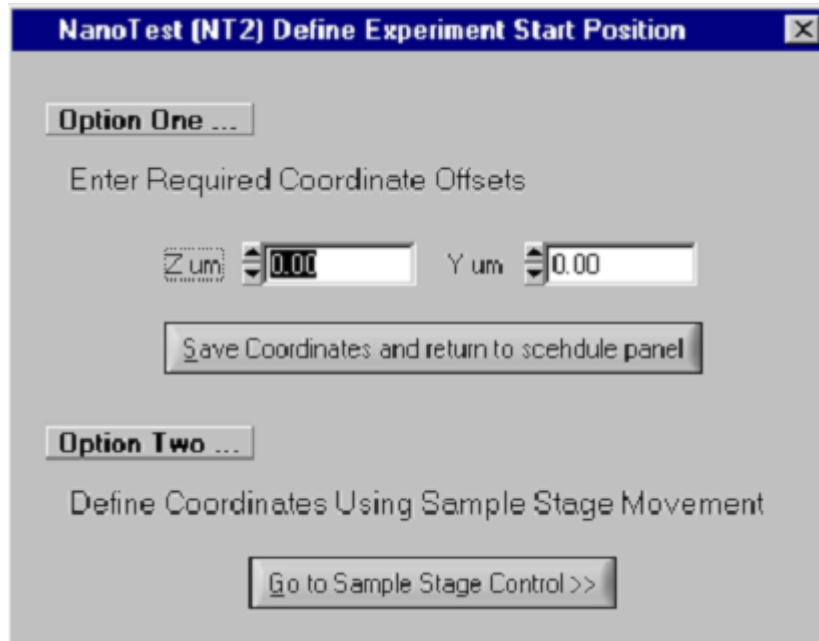
Choose **option two** to bring up the sample stage control screen.

Bring the diamond into contact (see box below) then retract about 25 microns and move in Y direction about 25 microns then exit.

Making contact with the sample
The motors can all move at up to 250 μm/s. If contact with the sample is made at this high speed damage to the instrument is very likely! Either the springs will be bent or worse, **the pendulum ceramic will break**. So it is important that when the indenter is quite close to the sample that it is travelling *slowly*. There is a safe contact speed button - at this speed (3.8 μm/s) damage will not occur. During the NanoTest training programme each user is shown how to bring the sample into contact safely. This is particularly important as there is a slight delay after pressing stop before the motors fully stop (at high speed the motor can travel several hundred microns in this time).

A **sensible method** for contacting safely is
1) approach at 250 μm/s until about 2 or 3 mm away from the sample. Now press stop.
2)   Lower the speed to about 50 μm/s and press start. When you are a few hundred microns from the surface switch to safe contact speed (3.8 μm/s) and allow the sample to contact the indenter. This is shown as a reduction in rectified output from 8 V to about 7 V, and the contact detected green light comes on.

Now define the number of indents. Set the number of columns to 1 and the number of

rows to 5 and the indentation offset to 25 micron in Z as shown below

Sample Stage Control

Start Position

Z (um) 25000.00   Y (um) 25000.00   X (um) 25000.00   Define >>

Indentation Grid Size   Column Offset   Indentation Offset

Number of Columns (Z) 1   Z (um) 0   Z (um) 25   Preview >>

Number of Rows (Y) 5   Y (um) 20   Y (um) 0

Number of Indentations 5   Retraction Distance (um) 20

Select Results Filename >>

This will produce a 1 x 5 array of indents at 25 micron intervals on the surface. Preview the indents with Preview>>.
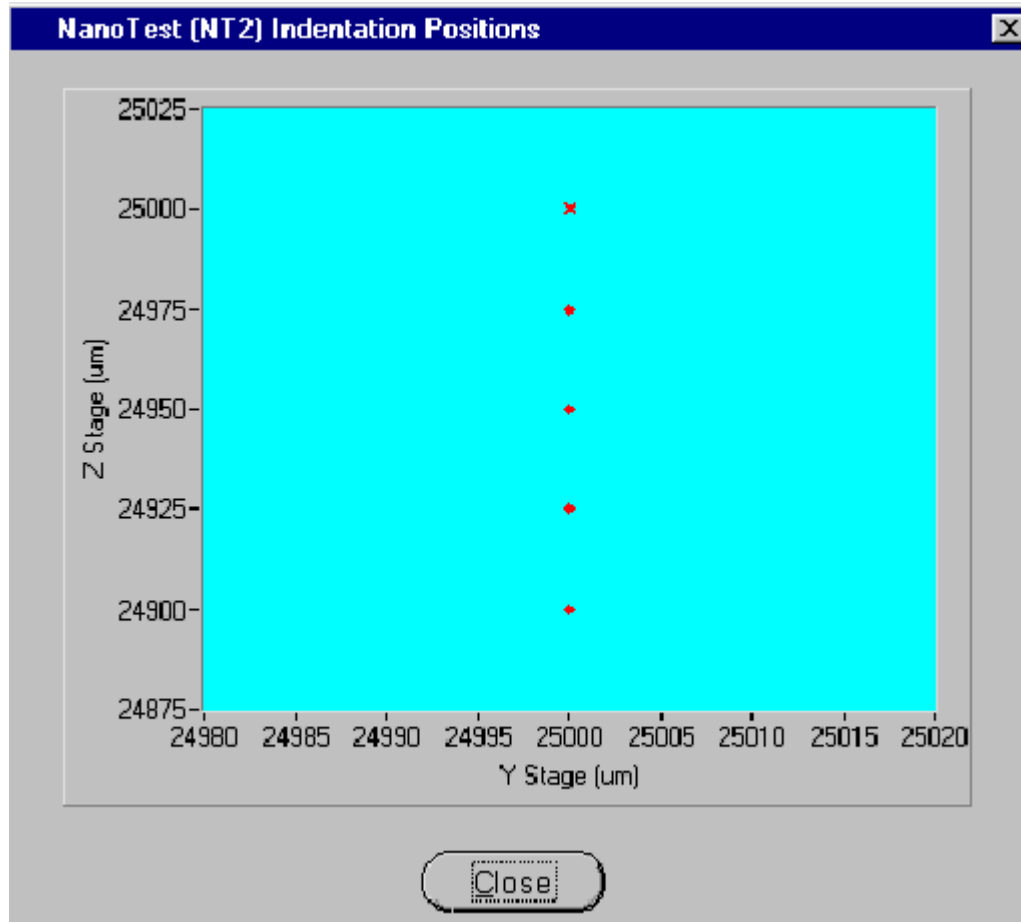
Set the retraction distance to at least 15 microns. Now choose the filename for this experiment (not the whole schedule). Once the file name has been chosen go to the bottom left of the screen and add the experiment to the sample queue and then select finished. The schedule is now ready to run (or to save and run later).

Enter *Experiment - Indentation Schedule - Run Schedule.*

It is possible to set the start time so that the schedule runs immediately or at some later point.

The possible advantage to delaying the start of a schedule is that ambient vibration levels are often lower overnight. The NanoTest scheduling allows for up to about 100 separate indentations to be performed over a single night.

What happens during an indentation procedure?
Finding surface - rectified output decreases from 8 V to ~ 6.5 V.
Moving sample into measurement position (rectified output ~ 0 V)
Pre-indentation delay (20 s)
Moving indenter to surface (rectified output to 4 V)
The loading ramp begins (red loading light comes on). The display 'autoscales' in real-time as the probe indents the sample. When the maximum load (or depth) is reached the ramp stops and the indenter is held at the maximum load for a specified dwell period before unloading. Once the indentation is complete the indenter is moved automatically to the next position to begin the next indentation.

Dwell periods
The hold time at maximum load is designed to allow for the sample to "creep out" before unloading. For a sample which is resistant to creep a short (e.g. 5 s) dwell time is sufficient, however, for materials which are susceptible to creep (e.g. metals and polymers) much longer hold periods may be needed.

Thermal drift correction
The NanoTest operates in a controlled-temperature cabinet to minimise thermal drift. However, in principle thermal drift can still affect results, particularly for slow-loading experiments. To allow for indentation data to be corrected for the effects of thermal drift it is possible to set a holding period at 80 % unloading for thermal drift evaluation. Typically the thermal drift time should be set to 60 seconds. The first 20 seconds of data sample creep dominates the change in depth. The thermal drift rate is calculated from the change in depth between 20 and 60 s (see also INDICOAT project report 2001).

Note that the method is not as effective for polymeric samples that exhibit significant creep recovery. Longer drift correction periods should be used.

Multiple experiments on a single sample: "Option 1"
When setting up a schedule of experiments on the same sample, once the indenter has been placed ~20 microns from the surface to define the initial position for the first experiment, all subsequent positioning can be entered as Y and Z co-ordinates without needing to move the motors during set up. Click on define then enter the required coordinates in the Option 1 dialogue box. For example, in the box below, the experiment will start 20 microns Y+ve from the previous experiment. This allows for schedules to be set up more quickly than by using option 2.
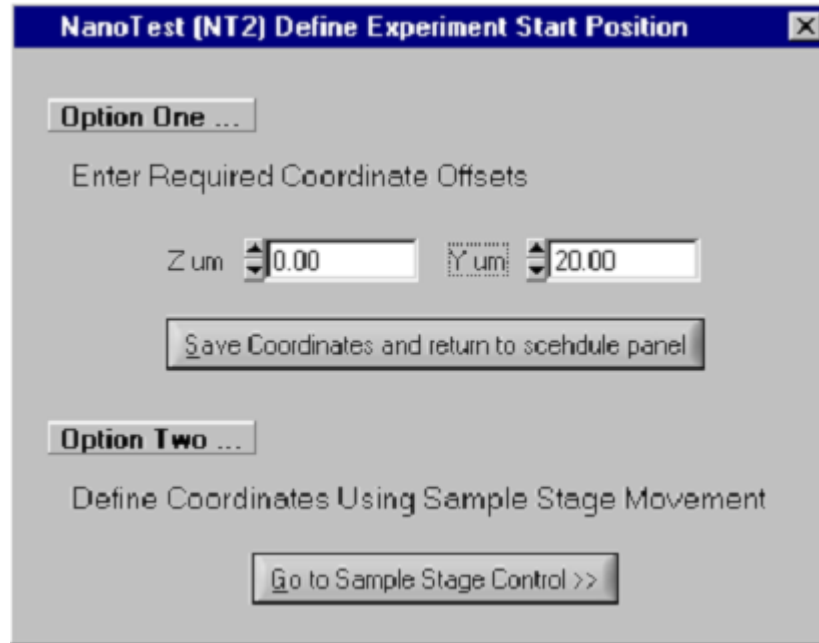
6.1.2 Tutorial:- setting up a load-partial-unload experiment

It is necessary to make the experiment depth-controlled or load controlled. This can be done by either:-
(1)   setting either the maximum and minimum depths to be different and the maximum and minimum load to be equal and high
(2)   setting either the maximum and minimum load to be different and the depths to be equal and high.

The following example shows how to make the load-partial-unload depth-controlled. In *experiment type* select *multiple loading cycle with increasing load*. Make the experiment *first condition* or *depth-controlled*. Set the maximum depth to 1000 nm and the minimum depth to 50 nm. Set the maximum and minimum loads to the maximum possible (e.g. 500 mN). Set the number of cycles to 20 and the unloading percentage to 20 % for that cycle.

This will produce a series of 20 load-unload cycles to gradually increasing depth at a single location on the sample surface. The minimum depth fixes the maximum depth for cycle 1 and the maximum depth fixes the maximum depth for the last cycle. The intermediate maximum depths are divided equally, so, in this example, the max. depths will be 50, 100, 150, 200…..800, 850, 900, 950 and 1000 nm. After the last loading the indenter unloads completely.

Curso Position .... Lcad (mN) [46.47]   Depth (nm) [504.97]
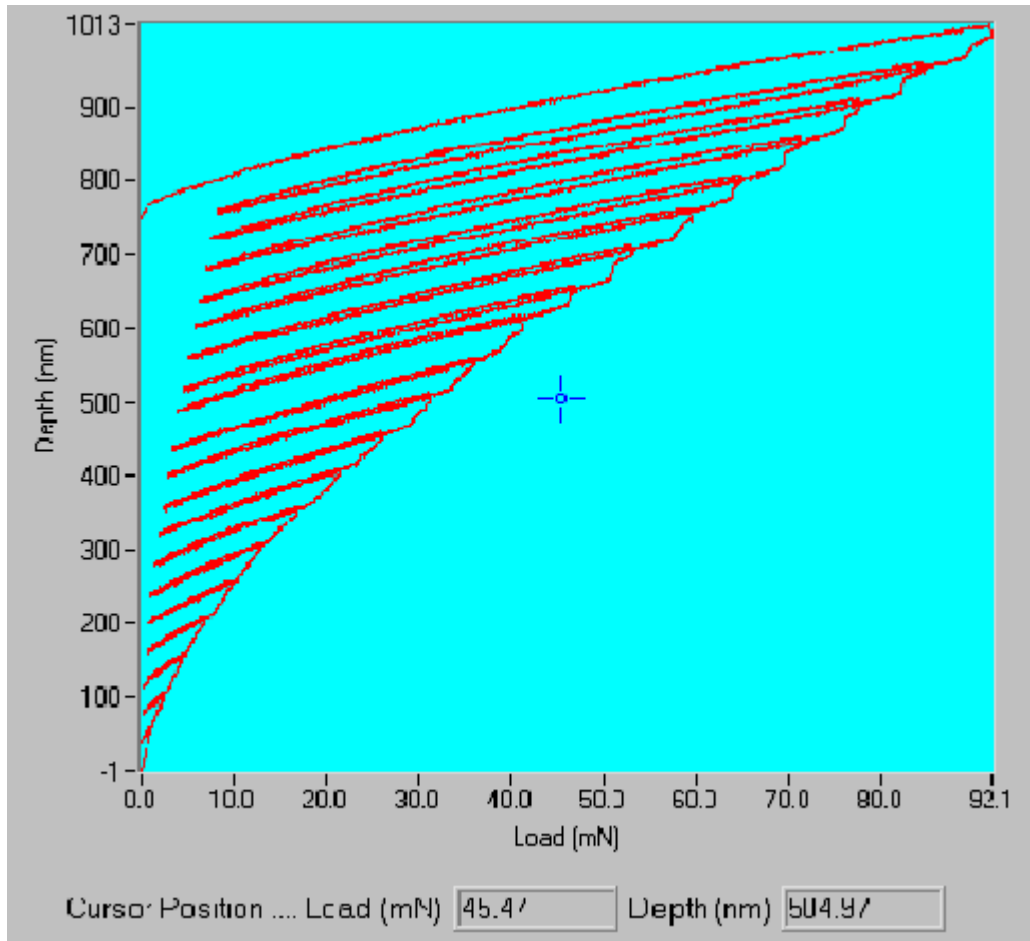
6.1.3 Multiple loading cycle to the same Load

The procedure is intended primarily for eliminating time-dependent effects, so that the final unloading data reflects only elastic behaviour.

In *Experiment-Indentation-Define Schedule-Select Experiment Type* choose *Multiple Cycle to the same Load*.

This enables a multiple loading cycle to be defined, with unloading occurring to 10% of the maximum load before the final complete unloading step, ensuring that the diamond remains precisely located throughout. 20 loading cycles are allowed, but data is displayed only for the initial loading stage and the final unloading stage.

6.1.4 Creep Test

In *Experiment-Indentation-Define Schedule-Select Experiment Type* choose *Creep Test*.

This test enables the variation of the depth of penetration vs. time under a static applied load to be investigated.

Note that it is also possible to perform creep tests in a depth vs. load hysteresis experiment. Set the loading and unloading rates will be dimmed out – they are set to abrupt automatically. Set the dwell time at maximum time to e.g. 300 s. The file can be reviewed in *Analysis* then select *Special Options* then *Review Creep data*. Data can be exported (ASCII) for further analysis.

Setting up more complex schedules and importing schedules
The Windows software allows for considerable versatility in designing complex schedules of indentation experiments. Each experiment within an indentation schedule does not have to be of the same type (e.g. experiment 1 could be load vs. depth hysteresis curves and experiment 2 could be a load-partial-unload experiment). Any schedule can be saved so that it can be imported at a later date for subsequent tests on different samples. This ensures consistency in experimental parameter choice, and improves throughout. At any stage experiments can be run either by (1) setting up a new schedule or (2) importing an existing schedule.

A warning!
When the high resolution microscope has been calibrated the system will automatically move the indenter from the focal to measurement plane at the beginning of each schedule. If, therefore, you bring the sample into contact then retract 20 microns then run a schedule the system will assume that the indenter is in the focal plane and will attempt to make the focal plane-measurement plane correction and WILL DAMAGE THE PENDULUM.
If you have a high resolution microscope but are planning not to use it and are not sure whether the system currently has this calibration then it is important to check in calibration review rather than risk damaging the delicate NanoTest pendulum.

6.1.5 Ultra-low Load Indentation

It is possible to use a different contact procedure described as the Ultra-Low Load method, which can produce more accurate results on smoother samples when indentations to small depths are required (e.g. for very thin coatings). The ultra-low load method works by performing a modified zero load calibration at the beginning and end of a scheduled line of indents. The surface positions defined in this way are then stored. The local surface position and slope in the region of the planned indentations are now known with precision, and for each indentation, contact can be now made without applying a contact load.
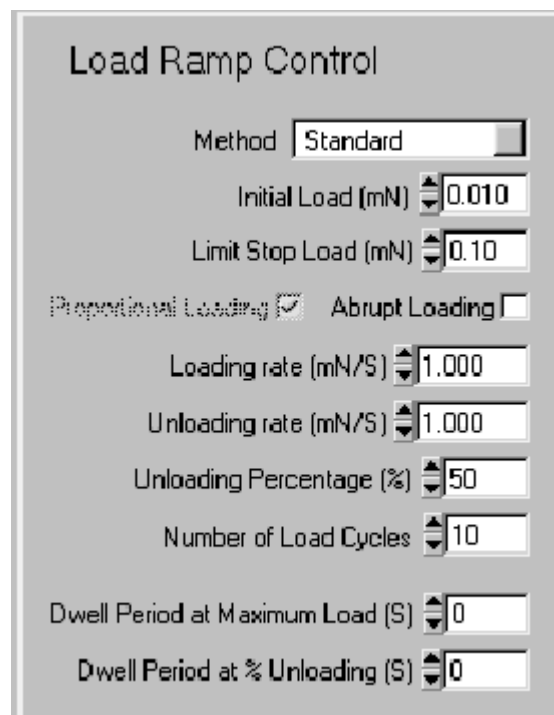
In the Load Ramp Control box set method to Ultra Low Load. The initial load can then be set to a lower value (e.g. 0 or 10 μN). The schedules are then set up in an identical way to above. The only restriction is that a grid of indents is not possible within a single experiment. The method works best when the sample has very low roughness.

6.1.6 Spherical Indentation

This should only be done with a highly polished spherical diamond. Use of the 25 micron radius scratch probes is not recommended.

In *Experiment-Indentation-Define Schedule-Select Experiment Type* choose *Spherical Indentation*. Specify the radius of your spherical indenter. If required a calibration check in the elastic regime on a material of known modulus (fused silica) can be carried out to verify/optimise the value used.

The experiment is very similar to the load partial unload experiment for pyramidal indenters, and produces a series of load- or depth-controlled load-partial unload cycles to gradually increasing depth. Set up the load ramp control panel with the required unload % (often 50%). For example, 20% unloading means unload to 20% of maximum load/depth. A typical screen would look as follows…

Load Ramp Control	
Method	Standard
Initial Load (mN)	0.010
Limit Stop Load (mN)	0.10
Proportional Loading ☑ Abrupt Loading ☐	
Loading rate (mN/S)	1.000
Unloading rate (mN/S)	1.000
Unloading Percentage (%)	50
Number of Load Cycles	10
Dwell Period at Maximum Load (S)	0
Dwell Period at % Unloading (S)	0

6.2 Indentation data analysis

There are 3 simple steps to data analysis:-
1)   preview the data without machine compliance correction (and remove any curves if deemed necessary)
2)   analyse data
3)   review and export the indentation curves and the derived mechanical property data

Both pyramidal and spherical analysis are possible and are described in turn below.

6.2.1 Pyramidal analysis

First review the indentation data by in *Analysis* selecting *Indentation* then *Indentation*

review and open up the filename. The data will be shown as acquired (i.e. before machine compliance correction). It is possible to edit out any unrepresentative curves if necessary.

Then go into *pyramidal analysis*. The Pyramidal Analysis window has 3 main areas - *files for analysis* at the top, *analysis definition* at lower left and *diamond area function definition* at the lower right.

Select the data file in *files for analysis* using *add file to list*.

Tip: If a complex multi-experiment schedule has been set up, analyse all the experiments together in one go (using the same parameters) then review the results separately.

In *analysis definition* set the analysis method to *Power Law fit* and then set the start fitting at 100 % of maximum load and stop fitting at 20 % of maximum load. Use an Epsilon constant of 0.75.

The energy calculations box can be left un-ticked.

In *Use Original Compliance* unclick the box and then click on *set default* to bring up the previously calibrated value of the machine compliance.

[If the use original compliance box is left clicked the data will be analysed with the machine compliance set at zero (i.e. as acquired) giving inaccurate values for the sample's mechanical properties.]

Analysis Definition

Analysis Method	Power Law Fit ▼
Start fitting at	100 % of Maximum load
Stop fitting at	20 % of Maximum load
Epsilon Constant	0.75
Energy Calculations	☐
Use Original Compliance	☐
Frame Compliance (nm/mN)	0.377325 (Set Default)

In diamond area function definition set *Mode* to *Ideal Function* and select *Berkovich* in the box below. The function can be viewed and a hard copy printed. The function relates the plastic depth (in nm) to the projected contact area in (nm^2).
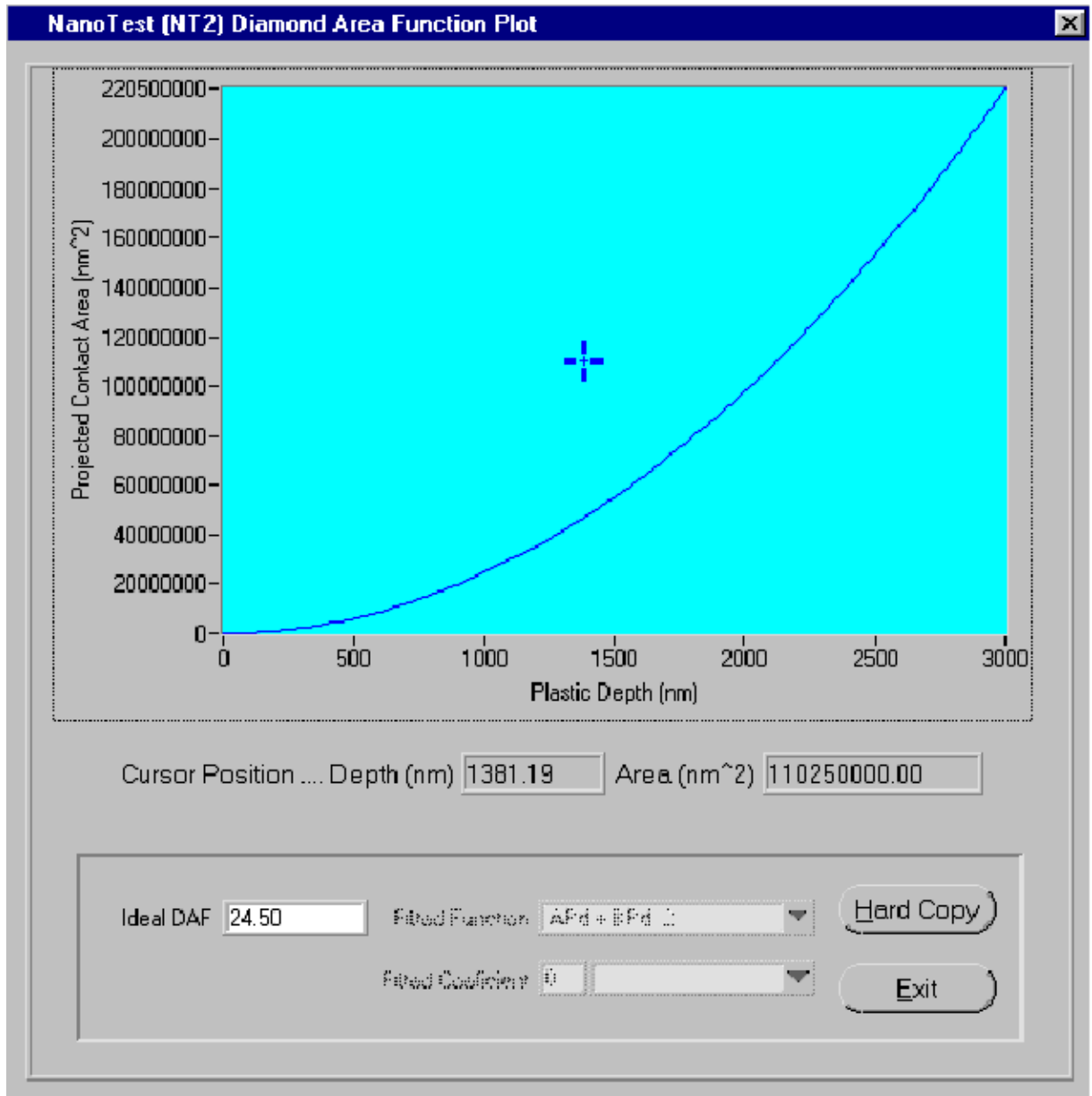
Now click *analyse*. The curves will now be analysed one by one. The results can then be reviewed in *review data file* after *quit* from this menu.


A warning - using CDs (and neighbourhood networks) to transfer large amounts of data.
Data is stored on CD in read-only format. When data from CD is transferred to another PC for off-line analysis it needs to be made not read-only before analysing otherwise the NanoTest software will not be able to overwrite the files (e.g. to allow for compliance correction etc) and data will be saved incorrectly. Simply select all the files on the target directory and right click then go to Properties. Now click on the *read-only* and *archive* boxes to **remove** any faint (or full) ticks then click *apply*. The data can now be analysed as normal.

Use a similar procedure when copying files across an internal network.

Using reference curves
In *review data file* click on *make reference* to keep the currently displayed indentation as a reference (shown in blue). It is now possible to look at other curves in the experiment and compare directly, as illustrated below.
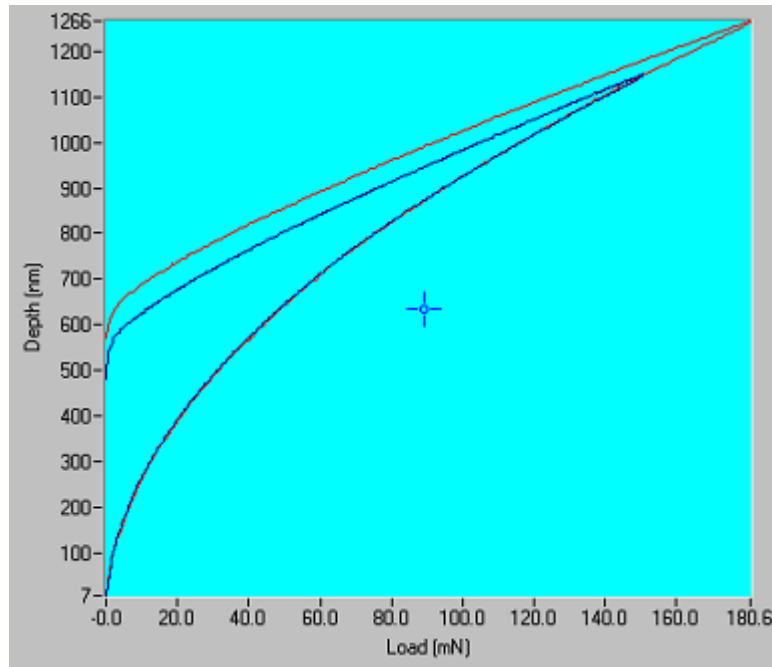
Also, by selecting *file-new* and loading a new experiment, the reference indentation can be compared with any indentation in a different file.

6.2.2 Spherical data analysis

If required, the data can be previewed as for pyramidal indentation by *analysis-indentation-review data file*. To analyse spherical data, go into *analysis-spherical analysis*. The Spherical Analysis window has 3 main areas - *files for analysis* at the top, *analysis definition* at lower left and *diamond area function definition* at the lower right.

Select the data file in *files for analysis* using *add file to list*. In *Use Original Compliance* unclick the box and then click on *set default* to bring up the previously calibrated value of the machine compliance. In *diamond area function definition* the tip radius defined when the experiment was set up is used in the analysis (this can be altered by un-clicking the box and typing a new effective radius in). The contact radius can be corrected for piling up/sinking in if necessary by clicking on the box (see below for more details on this). After analysis data can be reviewed as for pyramidal indentation. Stress-strain curves are found in *analysis-indentation-review data file.* Select the analysed file then *view-stress vs strain curve*.

6.2.2.1 Spherical Indentation Theory

The elastic behaviour of contacting spheres was first described by Heinrich Hertz in

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1882. **Hertzian analysis** predicts a contact circle of radius **a** given by the equation.

$$a = (3PR/4E^*)^{1/3}$$

where **P** is the applied load, **R** is the radius of the sphere, and **E*** is the reduced modulus given by

$$1/E^* = (1-v_1^2)/E_1 + (1-v_2^2)/E_2$$

and the subscripts 1 and 2 refer to the two contacting materials.

In static Hertzian contact, the maximum shear stress occurs at a distance of about half the contact radius immediately below the point of contact. Yielding therefore first takes place below the surface, and generally this occurs when

$$P_m = 1.1Y$$

where P_m is the mean pressure and **Y** is the yield stress. If the mean pressure is less than this, the contact remains elastic.

An indentation process may produce: (i) a reversible, elastic deformation; (ii) a permanent, plastic deformation; or (iii) both plastic and elastic deformation. The detailed behaviour depends on the ratio of the actual strain to the yield strain of the material. Low ratios (< 2) produce elastic behaviour; high ratios (> 50) produce plastic behaviour. The actual strain is given by $\tan\beta$, where β is the angle between the indenter and sample surfaces, so it is clear that a spherical indenter will behave in a fundamentally different way from a pyramidal indenter. In the case of a Vickers pyramid, for example, β is constant and the strain is therefore constant (about 8%), regardless of depth.

> For a spherical indenter, β, and therefore the strain, increases as the indentation depth increases. A series of spherical indentations with progressively higher maximum load can therefore produce results ranging from purely elastic to elastic + plastic, and, furthermore, can yield curves of Stress vs. Strain.

In the NanoTest, multiple load - unload cycles with increasing maximum loads are performed at a single point using the spherical indentation package. From this data and the known diamond radius, the contact area is determined and hardness, Young's modulus, stress and strain are calculated. The contact areas can be corrected for possible "piling-up" or "sinking-in" around the contact perimeter.

6.2.2.2 Spherical Indentation Analysis Procedure

For a given experiment comprising **n** indentation cycles there will be an associated total penetration depth h_t at a load P_t and a partially recovered depth h_s at a reduced load P_s (P_s would normally be 50% of P_t). The depth h_r of the residual impression relative to the original surface that would be made if the indenter were fully unloaded

is calculated from

$$Hr = \frac{r.Hs - Ht}{t - 1}$$

where

$$r = \left(\frac{Pt}{Ps}\right)^{\frac{2}{3}}$$

The elastic component **h$_e$** and plastic component **h$_p$** of the indentation at each indentation cycle can be calculated from

$$He = Ht - Hr$$
$$Hp = \frac{Ht + Hr}{2}$$

The radius of the circle of contact **a** in the plane of the original surface at each indentation cycle is calculated from

$$a = \sqrt{2.R.Hp - Hp^2}$$

where **R** is the radius of the spherical indenter.

Hardness **H**, reduced modulus **E$_r$**, representative flow stress **Y$_r$** and corresponding representative strain ε**$_r$** at each load step can be calculated as follows

$$H_i = \frac{Pt_i}{A_i} = \frac{Pt_i}{\pi a_i^2}$$
$$Er_i = \frac{3}{4} \frac{Pt_i}{a_i.He_i}$$
$$Yr_i = \frac{H_i}{3}$$
$$\varepsilon r_i = 0.2 \frac{a_i}{R}$$

where **i = 1 to n**

Because of possible piling-up or sinking-in around the perimeter of the spherical indenter contact, the true radius of the circle of contact **a$_r$** is related to **a** as determined above by the function **a$_r$ = ca**. The **c** value can be determined from plotting **log(Pt$_i$)** verses **log(a$_i$)**, the slope giving Meyer's index **(2+1/n)**, thus allowing **c** to be calculated as

$$c^2 = \frac{5}{2} \frac{2n - 1}{4n + 1}$$

Thus **H** and **E$_r$** calculated above have to be corrected by the factors 1/**c^2** and 1/**c**, respectively.

7 Running a scanning schedule and data analysis

Berkovich (pyramidal) indenters should **not** be used for scratch testing unless the sample is very soft (e.g. polymeric) as practice has shown that they become very quickly damaged (blunted) during use. Use spherical or conical (e.g. Rockwell geometry) diamonds instead which are blunter.

If friction forces are to be measured, a friction probe must be used. See section 4.1.3 for details of how to handle and calibrate the friction transducer. The *friction* box in *additional data collection* should be clicked when the experiment is being set up.

Before setting up a scanning schedule first check that the instrument is working at its optimum level - see checklist in section 5.2.

7.1 Setting up a new scanning schedule

Go to *Experiment* then *Scanning* then click on *Define new schedule*. In *select experiment type* choose the required experiment type (e.g. *Single Pass Scratch*) to bring up the Scanning Experiment Definition Menu. This screen contains 3 boxes: *Pre-experiment Operations*; *Scan Definition* and *Sample Stage Control*.

The types of experiment possible with scanning package are illustrated with the help of the four tutorials shown below.

Scratch Test
Topography Test
Topography-Scratch-Topography
Multi-pass wear test

7.1.1 Tutorial:- setting up a simple scratch test

AIM: the following simple tutorial improves familiarity with the Scanning Schedule menu, and sets up a schedule which can be used to perform 100 μm ramped load scratches.

In *Select Experiment Type* select *Single Pass Scratch*. In the *Pre-Experiment Operations* set the number of zero load calibrations to 3 and the experiment start delay to 30 s.

In Scan Definition set the scan velocity to 1 μm/s and the scan direction to **-Z**. Now set the scan length to 100 μm and the depth gain to 15 %.

Setting a lower depth gain gives a greater possible maximum scratch depth if needed.

Set the scratch load to 200 mN and applied after to 20 μm. Now set the loading rate to about 1.5 mN/s. Set the topography and limit stop load both equal at 0.1 mN. Since

the maximum scratch load is 200 mN the load range will be shown as high. Leave the Additional Hardware boxes (acoustic emission and friction) un-ticked.

The Scan Definition part of the screen should look something like this:

This will produce a scan where between 0-20 μm the applied load on the probe is the topography load and after 20 μm the load is ramped progressively upwards at 1.5 mN/s.

With the sample stage control box make contact with the surface then retract about 25 microns and move away in the Y direction (e.g. move 50 microns in Y positive).

Set the number of repeats to 1 and the retraction distance to at least 15 microns. Now choose the filename for this experiment. Once the filename has been chosen go to the bottom left of the screen and add the experiment to the sample queue and then select finished. The schedule is now ready to run (or to save and run later).

Now run the scratch test by *Experiment - Scanning Schedule - Run Schedule*. Click on start now.

The contact procedure is similar to that in indentation. Either the depth or load can be displayed during the scratch test.

7.1.2 Topography Test

The NanoTest can be used as a nano-/micro-scale profilometer. In *Select Experiment Type* select *Single Pass Topography*.

7.1.3 Topography-Scratch-Topography (3 scan multi-pass wear test)

This procedure is similar to a scratch test but also provides additional information regarding the amount of elastic recovery which occurs. Critical load values are obtained from discontinuities in the final topographic scan.

In *Select Experiment Type* select *Multipass Wear Test*. Set the number of passes to 3 and the number of scratches/topography to 1. Set the topography load to a very low value (e.g. 0.2 mN).

Using the scratch ramp preview

The scratch preview button is found in the bottom right hand corner of the scan definition box. Clicking on this allows the user to check whether the maximum scratch load is reached in the test. The load, scan velocity, loading rate can all be varied whilst this plot is open so their effect on the scratch ramp can be seen.

The example conditions above would be suitable for producing scratches into a rough sample where a long levelling distance is necessary.

7.1.4 Multi-pass wear for fatigue studies

Unlike a scratch test or topography-scratch-topography experiment where the scans

are investigated for critical load effects (e.g. due to adhesion failure), the multi-pass test uses a constant load for the scratches so that slower fatigue processes (e.g. crack growth) can be investigated.

In *Select Experiment Type* select *Multipass Wear Test*. Set the number of passes to 11 and the number of scratches/topography to 1.

This will produce a multipass wear experiment consisting of alternate topography scans (scans 1, 3, 5, 7, 9 and 11) and scratches (scans 2, 4, 6, 8 and 10).

7.2 Scanning Data Analysis

Results of scratch, topography and multipass wear tests can be reviewed and analysed in *Analysis-Scanning-Review Data File*.

Scratch Tests and Multipass Data Analysis
Topography Statistics

7.2.1 Scratch tests and multipass wear experiments

Select datafile to enter the *scanning review screen*. At this stage the first topography and first scratch data are shown unlevelled. To level the data go to select display and switch to topography. Now switch data levelling to over defined surface which brings up to bars. Move the 2 bars to the region before the scratch ramp commenced.

For example, for an experiment where the levelling distance (distance before scratch ramp starts) is 20 microns, pull one bar to about 2 microns scan distance and one to about 18 microns then click on *level now*.

To look at the on and off-load deformation go to select display and select both then put topography number to All.

The on-screen window can be exported to the clipboard (Export-Window to Clipboard) and/or printed directly (Export-Window to Printer). To Export the levelled data in ASCII format it is necessary to Export each scan individually. Display a single scan on the screen then go Export-Displayed Scan Data and choose the format of the exported data and click on Export.

7.2.2 Topography Statistics

In the scanning review the topography statistics (measures of sample roughness) can be viewed. Switch *select display* to *topography*. The scan will look something like this:
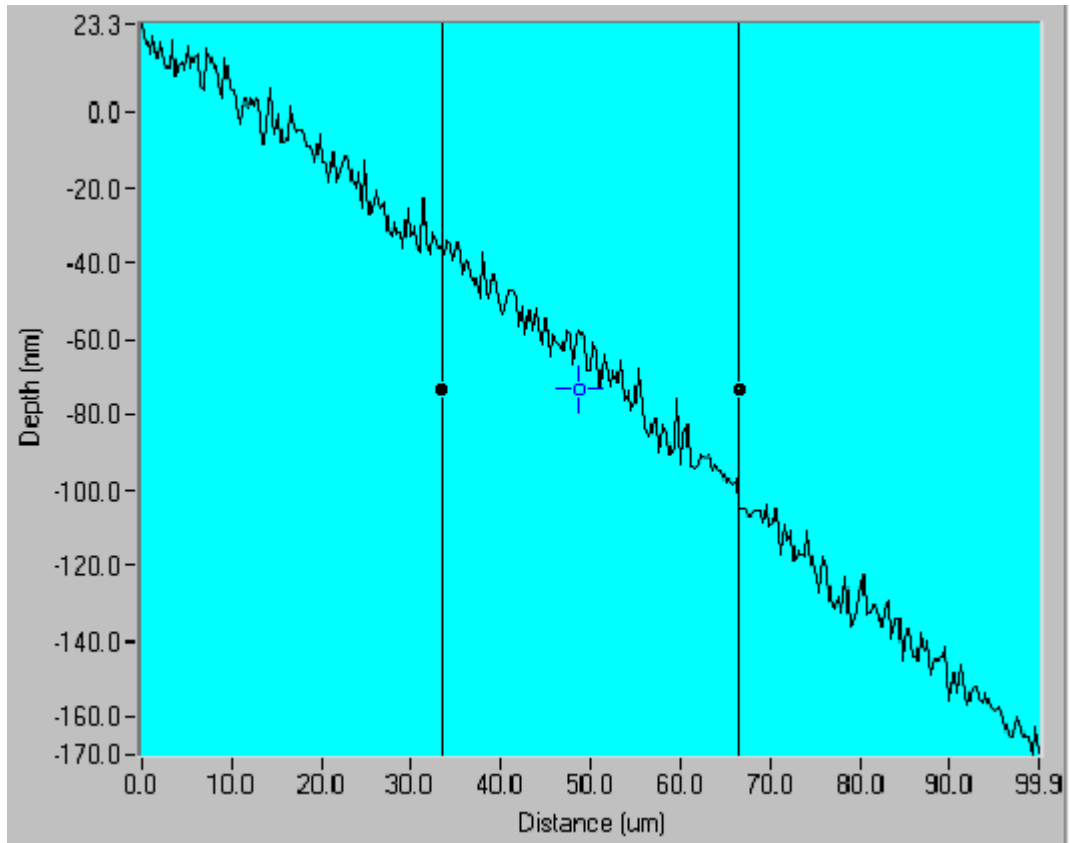
Now level on the whole of the scan (by selecting *over defined period* and moving the bars to the edges of the scan area and pressing *level now*).
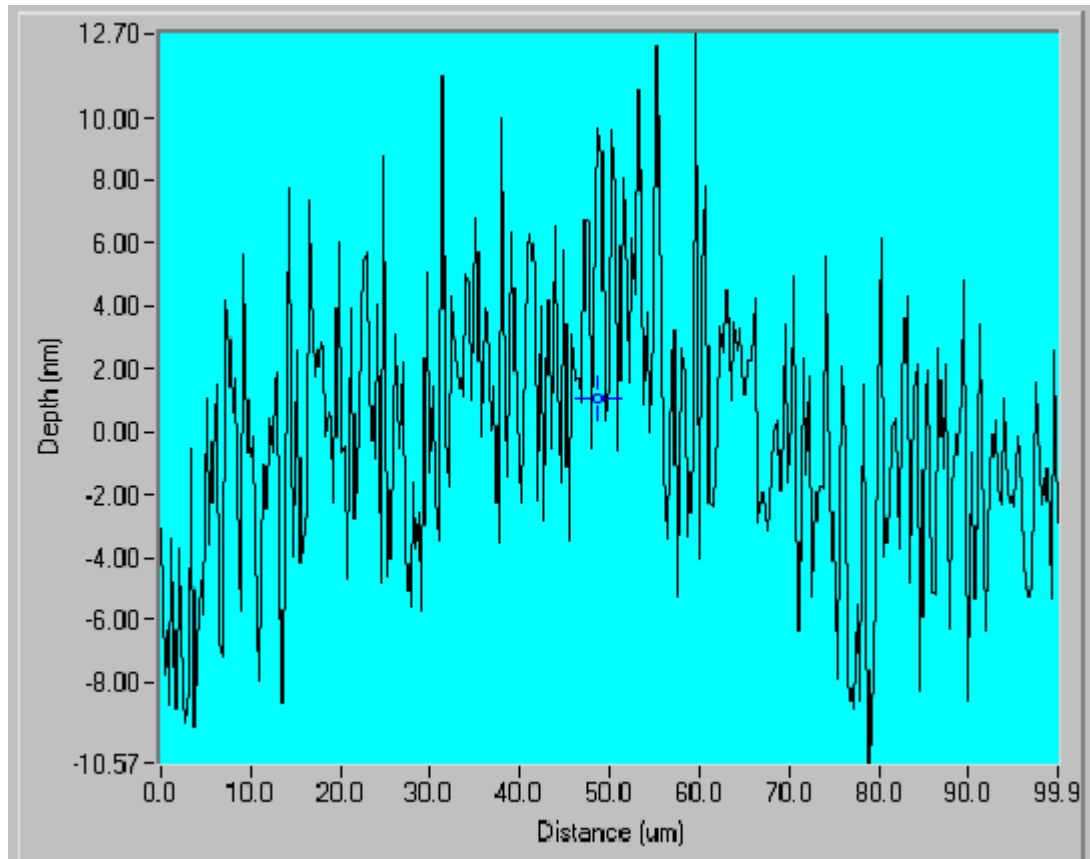
Now select *topography statistics* and then click on *between the cursors*. Move the cursors to define the area of interest. As the bars are moved the topography statistics update automatically.

8 Running an impact schedule and data analysis

Berkovitch (pyramidal) indenters should **not** be used for impact testing unless the sample is very soft (e.g. polymeric) as they become very quickly damaged during use. In general, spherical/conical diamonds are used for impact testing.
Also, the pendulum damping plate has to be removed from the machine for Impact/Impulse experiments.

8.1 Tutorial - Multiple Impulse

Before setting up a *multiple impulse impact schedule* it is necessary to first set the *impulse displacement*.

Go *experiment-impact-Adjust Impulse Displacement* and enter Yes to continue to set up impulse displacement.

Now move the impact solenoid away (counter-clockwise rotation of thumb screw) from the pendulum so that the instrument can perform a zero load calibration.

After the zero load calibration move the sample into contact slowly and continue at slow speed until the rectified output drops to 4 V then quit.

Select 'Genny gate on' with the software (**this should trigger a green light**).

Wind the micrometer screw in – you will see the rectified output go "off-scale" (i.e. 10 V) before the solenoid touches the bottom of the pendulum (typically about 1 mm apart when goes off scale). Continue to wind up whilst watching the rectified output signal. It will begin to come on-scale and decrease as the solenoid approaches the bottom of the pendulum.

With small movements move the micrometer so that the rectified output drops to 0V then becomes just slightly positive again (~0.5 V). This ensures that the depth signal is measurable, and fixes the depth from which the pendulum approaches the surface during the impulse test (typically ~ 12 μm).

Make sure that the solenoid stage is not moved too far with pendulum contact (i.e. impulse displacement < 5μm) – as damage to the pendulum pivot may occur.

Switching the on-screen toggle switch on-off should cause the pendulum to move on and off the surface. How easy it is to do this depends on the applied load. If the applied load is too small the pendulum will remain stuck to the solenoid so increase this until it breaks free (typically 1-1.5 mN). If the load is too high the pendulum remains on the surface so decrease it until it breaks free (typically 8 mN). By altering the value of the load you will be able to determine the maximum and minimum possible applied load for impulse testing with your pendulum and solenoid.

Exit and now follow the software instruction to move the sample stage back until it is 20 μm away from the sample surface (20 μm after it reaches 8 V). *or to the focal plane of the microscope.* The impulse displacement is now set.

Note that you need to reset the impulse displacement if the indenter is changed.

To set up a multiple impulse experiment select *Define New Schedule* and then select *Multiple Impulse*.

Note that since the solenoid is now close to the pendulum it is not possible to do a zero load calibration at this stage so the instrument uses the value determined during the set up procedure.

Definitions:-
- Impulse load – is the load applied to the indenter during the test
- Total sampling time – is the total time allowed for the whole experiment

Set the experiment start delay to 10 s. Set the limit stop load to 0.1 mN and the impulse load to 5 mN and the total sampling time to 10 min. (Note that the pendulum is held away from the sample for 4.5 s so that the minimum test time is 5 s.) Move the stage in the Y positive direction so that the probe will impact at a fresh region of the sample surface.

The impulse control window allows designation of the 'on' and 'off' times of the solenoid (i.e. the energised and de-energised state).

Save the filename for the experiment, and exit.

Run the impulse schedule.

The data can be analysed with *Analysis-Analyse Impact data*. Graphs can be re-scaled (e.g. set depth minimum to zero) to examine areas of interest. The cursor position shows the depth and time and the data can be exported to file similarly to the indentation and scratch testing experiments.

8.1.1 Diamond choice for nano-impact testing

The choice of indenter geometry for impact testing is sample dependent, but in general, 25 μm spherical probes are more suitable for inducing failure of hard and brittle coatings of ~ 1-2 μm thickness with relatively poor adhesion on brittle substrates such as silicon or glass.

To fracture thinner coatings, or to fracture/debond coatings with better adhesion, or coatings on more ductile substrates such as tool steel a sharper geometry is usually required. *For 95% of nano-impact testing at MML a cube corner indenter has been used with excellent results (see list of publications).* This is sharper (lower end radius) and also has greater contact strain due to the different angle from the probe to the surface around the contact (higher contact strain is known to promote brittle fracture

in scratch testing, even on soft materials).

An illustrative example on a 2 μm DLC coating on tool steel is shown below. When a 25 μm spherical probe was used even a 2 hour test at high load did not result in fracture, only a blistering of the surface (first 600s only shown). In contrast, when a cube corner indenter is used the sample fails dramatically in ~ 100s. At 5mN force (not shown) failure is within a few seconds.
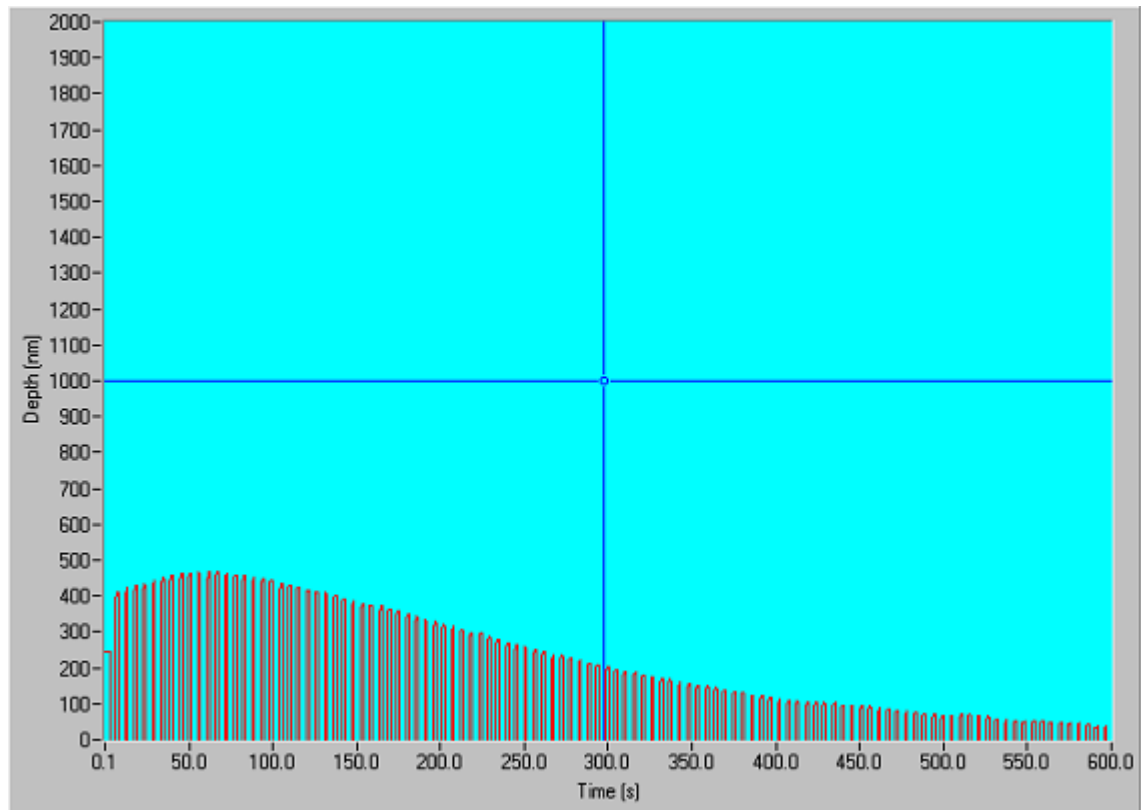

No failure with 25μm spherical probe at 20 mN force
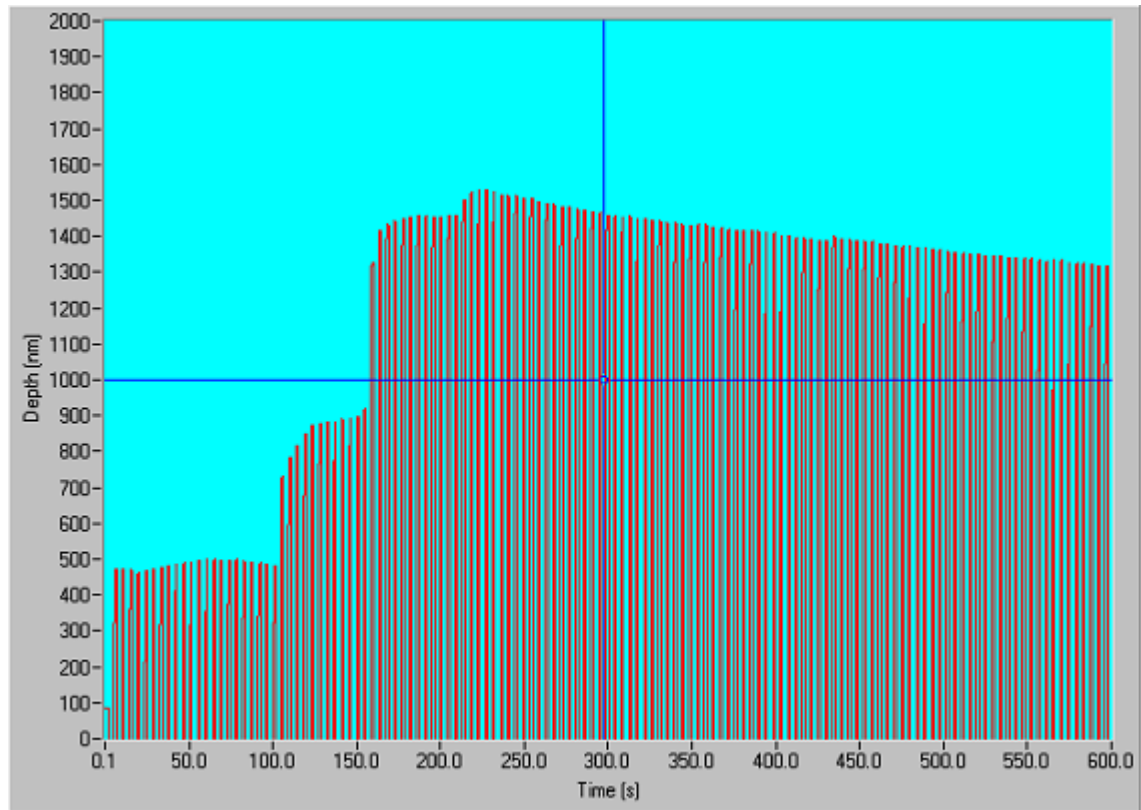
With cube corner, 1 mN force causes rapid failure

8.2 Tutorial - Impact (Sample Oscillation)

With the Impact (Sample Oscillation) Test it is not necessary to first set the impulse distance. So go directly *experiment-impact-Define schedule- impact(sample oscillation) experiment* to schedule the impact test(s). Set the experiment start delay to 10 s.

Preview the *Signal Generator Control Display* to set the Sample Oscillation On and Off periods. For example, a 180 s test with the sample oscillation on period set to 15 s and the off period set to 10 s would look like this:-

Genny Gate Signal ✕
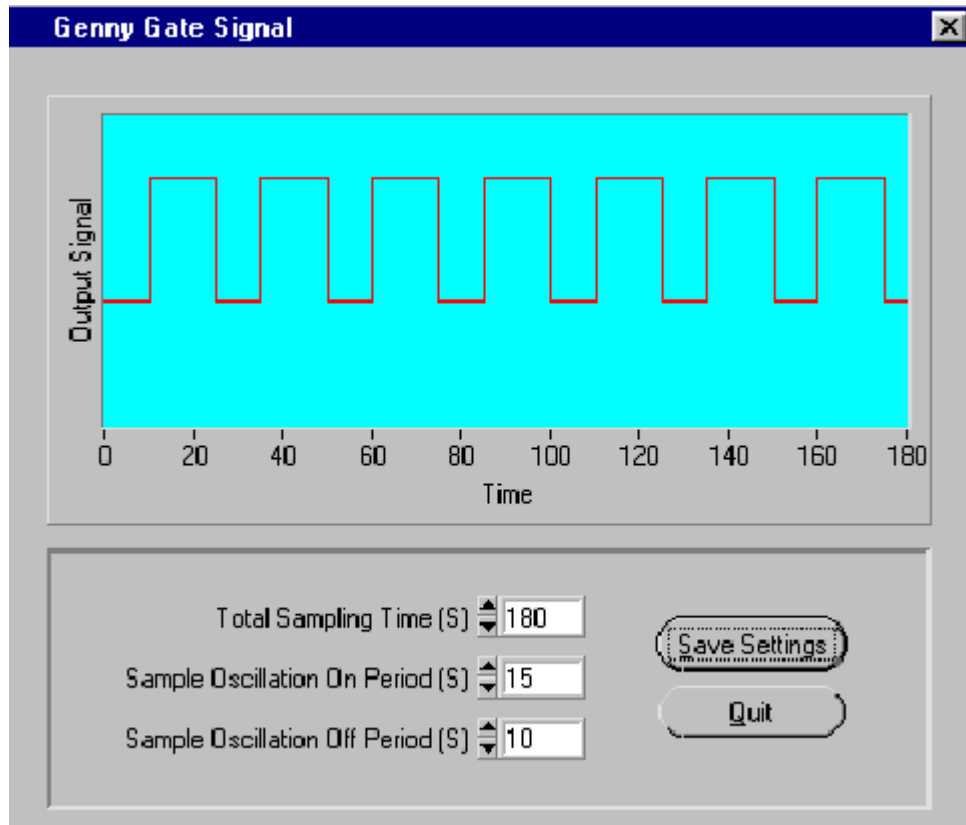
Note that the sample oscillation should always be *off* at the beginning and end of the impact test. When you have set the desired settings click on save settings and these values are entered automatically in the Sample Oscillation On Period (s) and Sample Oscillation Off Period (s) boxes. The *Optional Notes* allow for the signal generator frequency and amplitude used in the test to be entered so that they are saved with the experiment parameters although they do not influence the actual software for running the impact tests.

Set the limit stop load to 0.1 mN and the impulse load to 5 mN and the total sampling time to 5 min. Move Y positive so that the probe will impact at a fresh region of the sample surface.

Save the filename for the experiment, and exit.

Run the impact schedule and analyse by *Analysis-Impact-Review* and selecting the file. The impact results can be exported to file or sent to your printer and/or Windows clipboard after zooming in on features of interest.

8.2.1 Scanning Impact Tests

It is also possible to move the sample in the +Y, -Y or –Z directions whilst impacting to obtain a scanning impact test. Use a similar scanning speed as in a normal scratch or topography test (see section 7.1 for more details).

Switch *Sample Scanning* to *On*, then select the scan direction (e.g. –Z) and scan velocity (e.g. to 100 μm/s). The scan distance is defined by the total test time.

Results are analysed by *Analysis-Impact-Review* and selecting the file. Results are plotted as depth vs. time, and after ASCII export can be converted to depth vs. displacement in a spreadsheet such as MS Excel.

8.2.2 Post Impact Indentation

If a post-impact indentation is required then switch *Post Impact Indentation* to *On* in the multiple impulse set up. Set the indentation load, ramp rate as in a normal indentation test. Set the depth gain to 15 % i.e. the maximum possible depth, so that the indentation will be "on-scale". When the impact test is run, the instrument will perform the indentation immediately afterwards.

9 Sample Mounting/Diamond mounting

Diamond indenters are fragile and it is important to take great care when handling them. Always use a pair of precision tweezers and grip the diamond by the holder shaft.

Theoretically the diamond will not wear, but in practice small levels of wear do occur over time, particular if repeatedly indenting very hard materials such as silicon carbide. The main factor affecting the performance of the diamond will be debris sticking to it, particular after indenting into soft materials and plastics. Normally the diamond is checked and cleaned when a change in result for fused silica is observed that cannot be attributable to another part of the instrument's operation. Using an optical microscope to check the diamond's three facets may show debris but will not show tip blunting if it has occurred on a small scale.

9.1 Diamond cleaning

To clean the diamond, several approaches are possible:-

1)   use a fine camel paint-brush and alcohol. Immerse the diamond into a bath of alcohol and gently brush towards the tip. On removing the diamond, quickly blow off the fluid using an air spray. If the debris clings on, press the tip into a piece of hard polystyrene foam and then repeat the cleaning procedure.
2)   carry out some high load indents (200mN) on a hard material such as fused silica to push back the debris.
3)   deep indents into a sticky polymer such as PET.
4)   high load indents in Si wafer – the Si indentation-induced cracking sweeps debris from the diamond

After cleaning perform a diamond area function calibration as described in section 4. If the data still suggest that the diamond is blunted, it may have to be re-polished

(send back to MML) before it can be re-used for hardness measurement.

NOTE: When the diamond is inserted back into the pendulum's diamond-holder, The diamond stub should be pushed in until it is flush and the screw delicately tightened onto its pin (excess sideways force while tightening onto the diamond stub may bend the pivot.) For consistency, it is best to always keep the diamond orientation constant. Experimentally, it has sometimes been found that data reproducibility can be improved by keeping the diamond in the orientation shown in the figure below (with the edge vertical and pointing directly downwards).

preferred diamond orientation
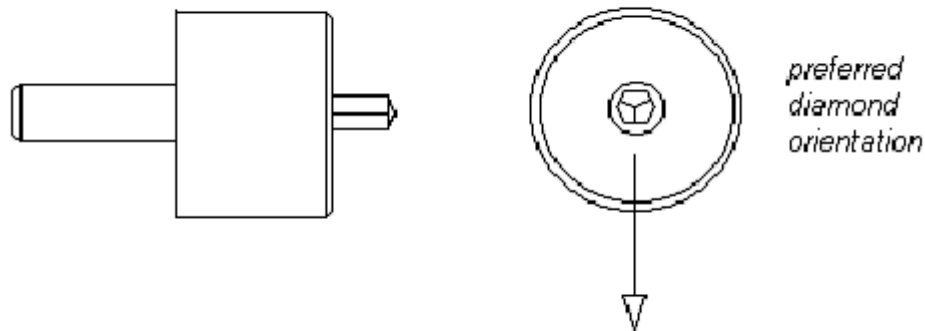
9.2 Sample Preparation

As a general rule, the better finished a sample's surface the more consistent the results are likely to be, so it is worthwhile spending time on specimen preparation. There is no point in measuring 100 nm indents when the sample roughness is 10 μm! Obviously care should be taken not to contaminate the sample with fingerprints and suchlike before it is used for testing.

NOTE: It is very important when a microscope is being used that either one sample is attached to the sample stub, or, if a number of samples are attached, the surfaces are on the same plane. The high resolution microscope's highest magnification objective has a working distance of only ~ 0.6 mm.

9.3 Sample Mounting

Samples are normally attached directly to an aluminium stub by means of a suitable adhesive. Cyanoacrylate "Superglue" is the most common choice. Adhesives such as Indian Wax can also be used to remove samples more easily once the testing is complete. A relatively large area should be covered with adhesive. If a polished stub is used, it is usually straightforward to remove samples by causing delamination at the glue-aluminium interface with a razor blade.

The main criterion when attaching samples is that the adhesive layer is thin and stiff once set and doesn't contribute significantly to system compliance.

The sample holder should be retracted (click on Sample stage then on Position

sample stage then move x positive) and the sample carrier carefully inserted such that the raised, hemi-cylindrical strip on the fixed plate locates in the hemi-cylindrical channel in the carrier plate. The carrier is locked in position by simply screwing the small knob through the carrier plate and into the backplate attached to the vertical micrometer stage. The knob should not be over-tightened.

> When inserting the sample stub, make sure no contact with the balance weight arm or other pendulum part occurs (potentially damaging the pivot).

10 Environment

The instrument is housed in an environmental cabinet which

- Reduces air turbulence upsetting the NanoTest pendulum.
- Provides a thermally controlled environment.
- Provides sound-proofing to reduce acoustic disturbance.

10.1 Temperature Control

The temperature within the cabinet is controlled. This minimises electronic drift of the capacitance bridge unit and thermal drift due to expansion.

> The standard set-up for the Temperature Controller is to set it to a value about 2 or 3 °C above the maximum room temperature, with expected stability at ± 0.1°C. It is important to remember to allow time for the instrument to thermally stabilise after the cabinet has been opened. The Instrument base is a large mass of aluminium with large thermal inertia and so is thermally quite stable. However, as there are also small components with low heat capacity the instrument should be left to settle for at least half an hour after the cabinet has been opened before any serious measurements are made. The internal/external thermometer provided should be regularly inspected.

Two small heaters are placed at the front of the cabinet away from the machine's base to provide a thermal barrier, while the temperature controller's probe (and temperature sensor) is placed towards the centre of the machine at the height of the measurement in order to give an average value for the cabinet temperature.

> To adjust the cabinet temperature, press and hold either of the two arrow buttons on the digital display unit (the upwards/downwards arrows increase/decrease the target temperature respectively).

10.2 Vibration Isolation

All instruments need vibration isolation to produce reproducible, noise-free results. If vibration stability is thought to be a problem, the vibration levels can be checked in *Signal Display* within the *Maintenance* menu. Choose *Depth + offset* to investigate how the signal varies with time, and movement within the laboratory.

Switch the input range to +/- 100 mV. If the deviation is less than 0.3 mV this is acceptable.

If there is a problem, check that the anti-vibration table is operating correctly.

11 NanoTest Options

High Temperature Testing

Pin on Disc Testing

2D Levelling Stage

Acoustic Emission

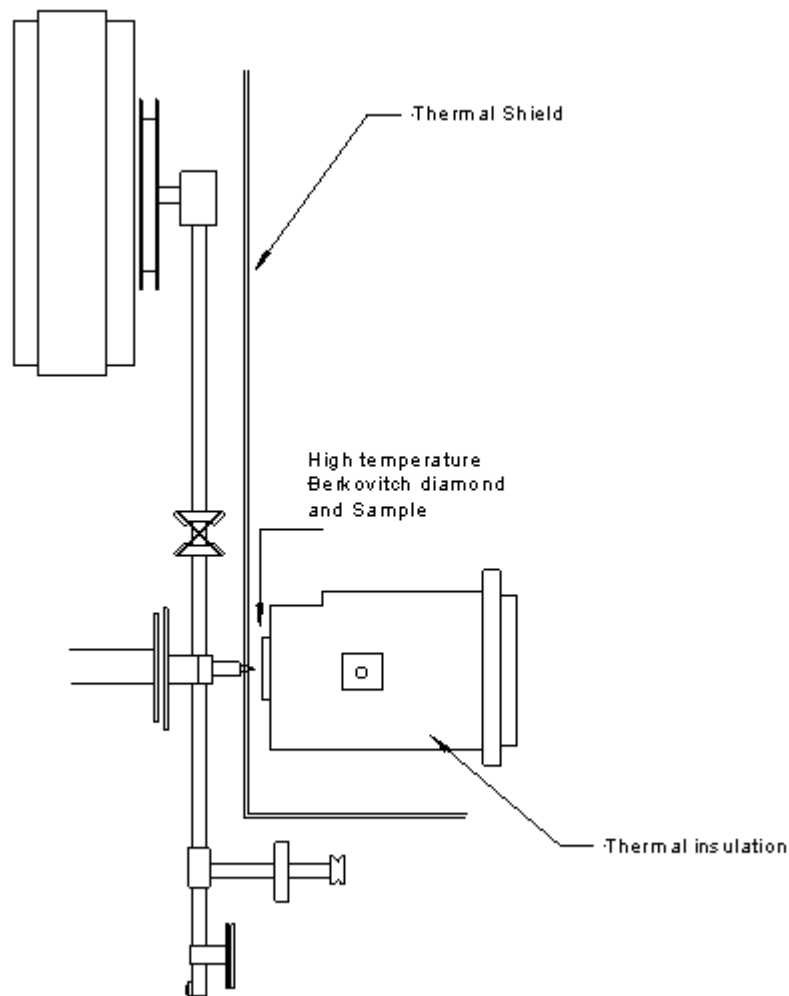Continuous Compliance Measurement

11.1 High Temperature Testing

The hot-stage should ONLY be used when the customer is satisfied with the operation of the instrument at room temperature (e.g. by testing the hardness and modulus of a fused silica sample).

Using the NanoTest with the hot stage is a lot slower than room temperature testing. Below are some approximate times.
A   Adhesive curing time – 24hrs (6 hours if warmed GENTLY)
B   Set up of the heat shield and indenter – up to 4 hours depending on dexterity and experience
C   Heating sample to target temperature – up to 5 hours depending on temperature
D   Tuning – ½ hour
E   Stabilisation time up to 18 hrs depending on temperature change and maximum thermal drift
F   Cooling time - ½ to 5 hours
G   Removing sample – ½ hour

Some time can be saved, for instance, waiting for sample adhesive to cure can be done while setting up the shield and indenter, and the computer can do C,D&E while the operator is elsewhere.

Using the microscope with the hot stage may damage the machine. Contact Micro Materials for advice.

NanoTest High Temperature Testing Configuration

Installation
Heating Procedure
Performing High Temperature Measurements

11.1.1 Installation

Sample mounting
1. Mix the adhesive.
2. Apply to the stage.
3. If the compliance of the stage is unknown use a piece of fused silica to calibrate it in the usual way.
4. Press sample into the adhesive by the corners ensuring that the sample is level and that if two different thickness samples are to be tested, they are packed with extra adhesive to the same level.
5. Leave to dry for a minimum of 24 – 48 hrs at room temp, or increase the

temperature slowly to 80 °C for 6 hrs.

Diamond heater & shield

The aluminium shield is to prevent any thermal interference with the ambient electronics and depth sensing equipment. The wires to the diamond heater need to pass through the slot in the heat shield while **not** making contact with it.

1. Make sure that the hole in the heat shield is at the correct height. If the hole is too low, spacers can be placed under the top and bottom of the shield. However, there is no reason why there should be a significant error here.
2. The slot at the top of the heat shield allows some front-to-back adjustment of position.
3. Take a piece of card about 40 x 60 x 1 mm and cut a 'V' slot several mm long from the centre of the 40 mm edge.
4. Using PVC tape, attach the card to the front of the heat shield such that the V is horizontal across the diameter of the through hole. The card should be placed on the side opposite the vertical slot in the aluminium, i.e. it should not cover the slot.
5. Push the 1 mm dia. pin of the diamond stub through the card such that it is central in the through hole. The four leads from the heater/thermocouple should fall across the vertical slot in the heat shield.
6. Apply a small piece of PVC tape across the edge of the heater ceramic and over the edges of the card to prevent any further movement.
7. Carefully pass the four leads through the slot and bend into position using tweezers such that they do not make contact with each other or with the edge of the slot. On the hot side, the leads should be well behind the diamond. On the cool side, the leads should be 5 - 10 mm from the heat shield surface.
8. Trim the copper leads such that when they are placed in the top and bottom locations of the chocolate block connector there is no tension. The centre of each lead should sag down by about 1 cm.
9. Pass both thermocouple leads through the central location of the chocolate block (this section of the thermocouple will still be insulated.). Gently increase the pressure on the clamping screw to preclude any movement of the leads. Again, there should be no tension in the leads and a 1 cm sag should be allowed.
10. Put metal cover over wires.
11. Carefully move the heat shield into position on the instrument and insert the diamond stub into the diamond holder. As mentioned above, some front-to-back adjustment of the shield position is available to achieve this.
12. Tighten the top clamping screw. In view of the width of the slot, a large washer will be necessary.
13. Carefully remove the PVC tape attached to the diamond ceramic.
14. Carefully remove the PVC tape attached to the end of the card and slide the card sideways until the pendulum is released.
15. With the pendulum on the limit stop there should now be a gap between the heater ceramic and the heat shield equivalent to the card thickness.
16. Enter *Maintenance-Bridge Box Adjust* and click on *remove pendulum load.* Rotate the balance weight in until the signal drops below 9V then rotate it back

until the signal reaches 9V. Rotate it a further 180° - 360°.

17. Perform a *Pendulum Test*. Because of the extra stiffness of the wires, the pendulum test should take longer (it should *look* like the plates are slightly wider). If there are unusual changes in slope, the wires are probably contacting the shield. Adjust the shield or the wires until no interference is seen
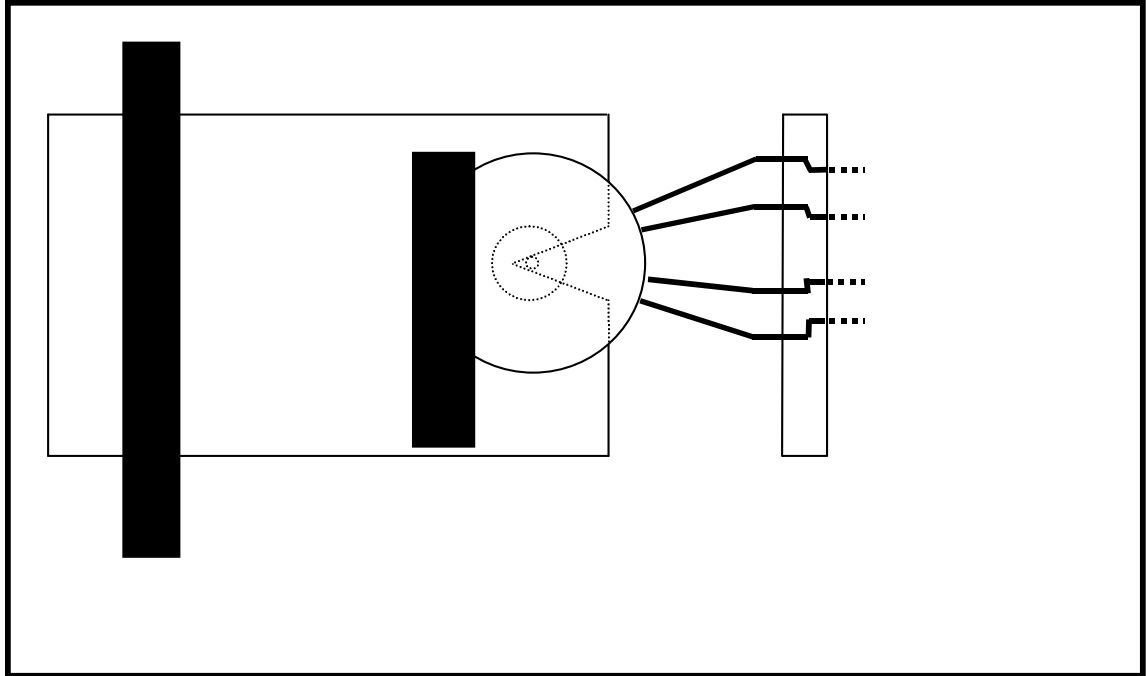
Stage and insulation installation

1. Retract the X motor almost fully (~45 mm position), also adjust the sample stage assembly if necessary (loosen the 4 x M8 screws on the 'transverse stage' and slide the stage assembly back away from the pendulum until there is sufficient clearance).
2. Clamp the stage in the usual way
3. Calibrate the compliance in the normal way.
4. Enter s*ample stage control* and make contact with the areas of interest noting their coordinates.
5. Move X +ve 25um, exit and re enter *sample stage control*. (this resets the counters)
6. Move the stage away by 5-10mm.
7. Place 4 strips of glass fibre insulation between the stage and shield (these are to stop any air flow around the sample)
8. Make contact (slowly over the last few um). If contact is detected before the counter returns to   -25um, adjust the insulation.
9. Perform a *pendulum test.* If the curve has changed, adjust the insulation.
10. **Retract the stage by 1um for every 3 degrees of temperature increase**.

11.1.2 Heating Procedure

There are 3 important considerations when using the hot stage controller:-

·   **Rapid changes in temperature can crack the glue under samples and even the hot stage itself, so set the rate limit when changing temperature.**
·   **Best results are achieved when the controller has been tuned for the temperature it is being used at.**
·   **To tune the hot stage for a particular temperature the controller must force the temperature oscillate through two cycles, when it is doing this the rate limit must be switched off. To prevent damage the max and min power must be temporarily limited. This is explained step-by-step in Auto Tuning below.**

Warning
The controller remembers all the parameter (including current temperature) when it was last on. To prevent the controller compensating for a perceived sudden drop in temperature it is advised to switch the controller on and connect the thermocouple, then set the set point (see below) to below the current temperature. The power will gradually fall and the out put indicator (OP1) will turn off. While this is happening other parameters may be checked/changed. The white power lead can then safely be connected. Alternatively the power can be reduced manually – see below.

Getting started
There are four ways to change the parameters in the controllers -

1. *Experiment/Special Options/Manual Hot Stage Control/Simple.*
2. In a schedule.
3. Manually pressing the 4 buttons on the front.
4. Manually adjusting the parameters through the computer using *Experiment/Special Options/Manual Hot Stage Control/Advanced.*

Simple

In the 'simple' the user types the desired temperature and clicks Continue. Accessing this through '**Manual** Hot Stage Control' is a misnomer as the computer then takes control adjusting the parameters, gently increases the temperature then tuning the parameters for good control
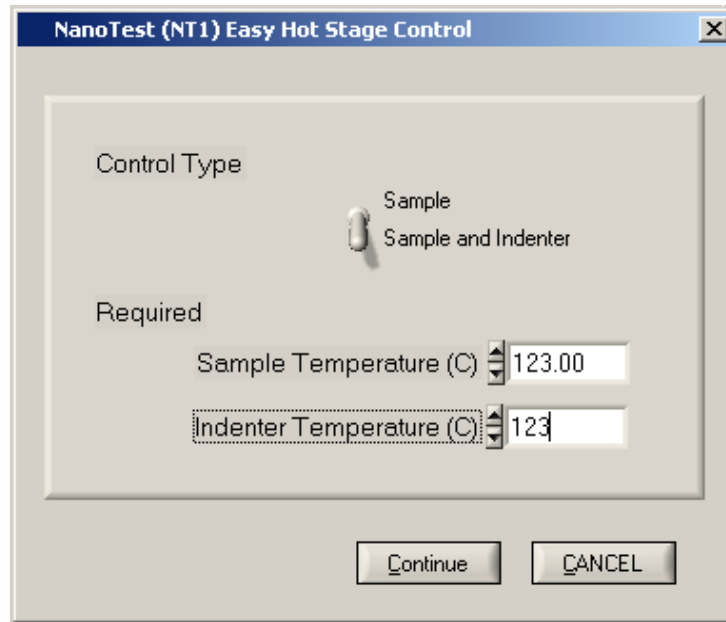
The progress can be seen on screen.
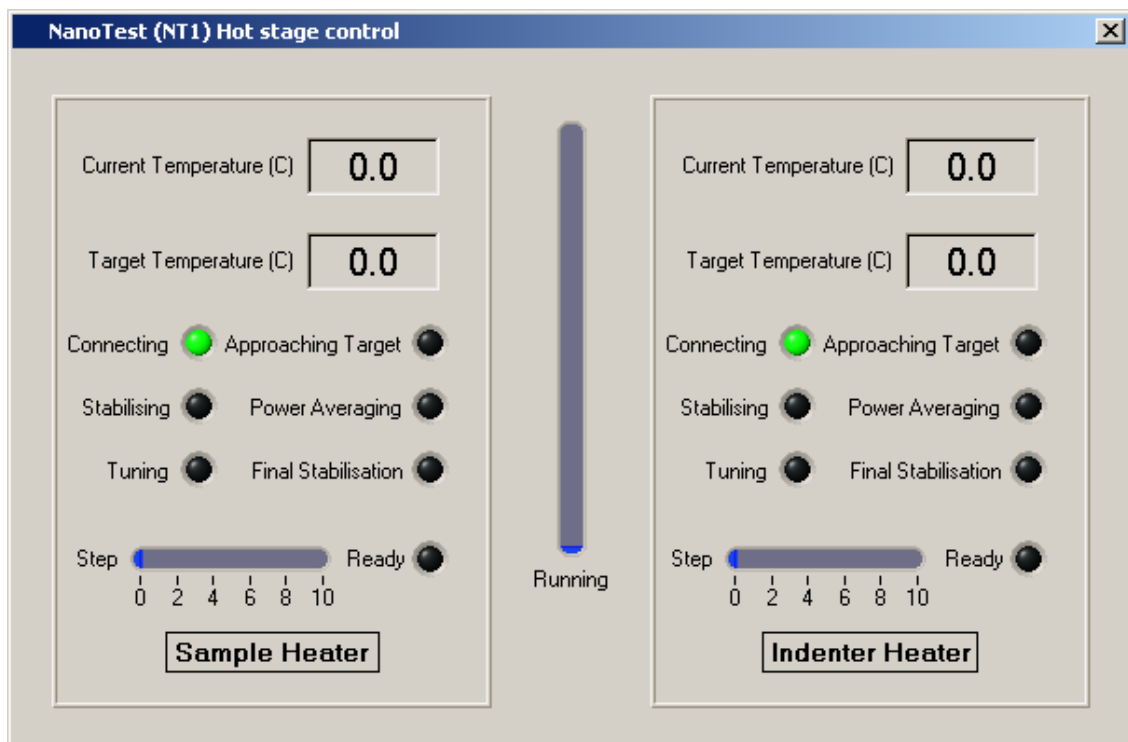
Schedule

The software moves the sample to the start of the experiment, uses the 'simple' routine then starts the experiment.
Remember that the stage expands, so withdraw the sample 1 micron for every

3 degrees as mentioned in the stage and insulation installation section in section 11.1.1 (point 10).
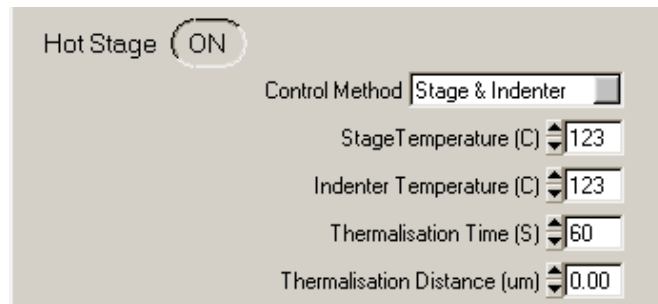
Pressing the buttons

The Eurotherm parameters are grouped into lists.
To cycle through the lists press the page button (far left).
To cycle through a list press the scroll button (next to the page button).
To change a value, press the up and down buttons.

1. Setting the temperature
(Set the rate before the temperature, but read about the temperature first)
The temperature that the Eurotherm is aiming for is called the 'Set Point' or 'SP'. This can be changed by pressing and holding or repeatedly pressing the up and down buttons in the home display (this is the display seen after the initial self-checking routine when you first switch on. The controller will return to the home display if no button is pressed for 45 seconds, or if both the 'page' and 'scroll' buttons are pressed together).

2. Setting the rate
The rate at which the working set point is ramped is the 'Set Point Ramp Rate' or 'SPrr'. The rate is in degrees centigrade per minute. It is common to use a rate of 1.6 (i.e. 100 degrees per hour).

1. Press 'page' until 'SP', the set point list is displayed.
2. Press 'scroll' until 'SPrr' is displayed.
3. Use the up and down arrows to change the value.

3. Auto Tuning
To calculate the parameters needed for control of the temperature, the controller must oscillate the temperature.

Because samples can debond if the temperature change is too rapid, the max and minimum power must be limited.

1. When at the desired temperature, and in the 'home display', press the 'scroll' button until OP is displayed. The lower display is the out put %. Note this number.

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2. Press the 'page' button until 'OP', the out put list is displayed.
3. Press the 'scroll' button until 'OP.Lo' is displayed.
4. Change this value to 2% below the out put level.
5. Press the 'scroll' button until 'OP.Hi is displayed.
6. Change this value to 2% above the out put level. The output is now limited.
7. Use **setting the rate** procedure above to reduce 'SPrr' until it is 'OFF'.
8. Press 'page' until 'Atun', the auto tune list is displayed.
9. Press 'scroll' until 'tunE' is displayed
10. Change this from 'OFF' to 'ON'.
11. Press 'page' and 'scroll' together. The controller will return to the 'home' display. 'tunE' will flash while it is tuning.
12. Once it has finished, press 'page' until the 'Pid' list is displayed.
13. Press 'scroll' until 'Pb' is displayed.
14. Double this value, i.e. if it is 30.0 increase it to 60.0.
15. Do not forget to set OPLo and OPHi to 0.0 and 100 respectively (or the temperature will not change much) and set the ramp rate (otherwise the temperature may change too fast.)

4. Manual power control.
1. Press 'page' until 'ACCS', is displayed.
2. Press 'scroll' until 'CodE' is displayed.
3. Use the up and down arrows to change the value to 1.
4. Press 'scroll' until 'Goto' is displayed.
5. Use the up and down arrows to change the value to 'Full'.
6. Press 'page' until the temperature is displayed.
7. Press 'scroll' until 'm-A' is displayed.
8. Press 'up' to change to 'mAn'
9. Press 'page' to return to the home screen. The lower number is the percentage power.
10. Use the up and down arrows to change the value.
11. Press 'scroll' until 'm-A' is displayed.
12. Press 'up' to change to 'Auto'

5. Switching off
Before switching off, set the set point to below room temp, and the ramp rate to off, or the controller will try to use the settings when next switched on which could damage the hot stage.

Two point operation
The controller has the ability to store two set points (e.g. room temperature and the experiment temperature) and switch between them.

A set point can be altered in the home display if it is in use, or in the 'SP' list. To switch between 'SP 1' and 'SP 2' change 'SSEL' in the 'SP' list.

Advanced

Advanced is almost identical to using the buttons. It is the fastest way of changing or checking several parameters as entire lists and their limits are displayed on screen. If a typed value is inappropriate it will be rejected e.g. if you set temperature to 501 when it is limited to 500.

11.1.3 Performing high temperature measurements

Hot Stage Calibration

Setting up Indentation or Scanning Experiments

11.1.3.1 Hot-Stage Calibration

Once the high temperature components are installed, calibration (at room temperature) is necessary to ensure correct assembly of the hardware. General instrument calibration steps include:-

• Bridge Box adjustment (Pendulum test) - to check that the instrument is operating correctly.

• Signal display - check the Depth + offset signal (at +/- 100 mV level) for noise (Standard Deviation < 0.25 mV).

• Depth Calibration (also done at target temperature)

• Sample positioning - Locate the sample surface with the diamond (safe contact speed) and retract 20 μm.

• Zero load calibration - Carry out three zero load calibrations to ensure probe/sample contact is accurate.

• Machine Compliance Calibration - Perform 5 200mN indents into fused silica to determine the machine compliance when using the Hot-Stage (in Machine Compliance calibration), checking the hysteresis curves for correct operation of the instrument. It is recommended to cement a small piece of fused silica to the sample plate in addition to the sample under test to avoid the need for removing the stage to swap samples. The **additional** compliance from the hot stage is typically ~0.3-0.5 nm/mN (typically the total compliance is approximately 0.9 nm/mN).

11.1.3.2 Setting up indentation or scanning experiments

1. After successful tuning (the Eurotherm stops flashing TUNE, temperature is within $\pm$ 0.1°C of the target) retract the stage and position it for experiments.
2. Carry out 5 depth calibrations (the depth calibration factor should be near to that at room temperature with a typical good standard deviation < 5 x 10^{-4} nm/bit)
3. Perform indentations or scanning tests as shown below:-

In *Experiment-Indentation* click on the *Hot Stage* button to turn this to *On*, and set the values of hover distance and hover time. To ensure thermal stabilisation between the indenter and the sample, set the *thermalisation distance* and *thermalisation time*.

- *Thermalisation Distance* (in microns) - the indenter is held a fixed distance above the surface before indentation. Typically this parameter is set to zero.
- *Thermalisation Time* - the time the indenter is held at the thermalisation distance before indentation. Typically this parameter is set to 300 s.

Once these values are set the schedule can then be run and analysed as normal. (i.e. high temperature data is analysed in the same way as room temperature data.)

Follow a similar procedure for high temperature scanning tests.

For further details of published high temperature test results see *High temperature Nanoscale Mechanical Property Measurements*, JF Smith and S Zheng, Surface Engineering 16 (2000) 143-146; and *High temperature nanoindentation testing of fused silica and other materials*, BD Beake and JF Smith, Phil. Mag. A, 82 (2002) 2179-2186.

11.2 Pin-On-Disc Wear testing

The Pin-On-Disc (POD) option is designed for tribological studies and is usually used in conjunction with friction measurement transducers. There are high speed (120-3600 rpm) and low speed (1-120 rpm) motor options, and depending on the experiment required the relevant motor has to be installed on to the spindle assembly.

Insert the POD assembly with sample and the friction transducer with a spherical probe (25μm). Note that the stage assembly may need adjustment (retract the X motor until sufficient installation space). Then set up the friction probe as described in <u>section 4.1.3</u>.

Make sure the bridge box is set-up correctly (the system is on 'standby' at 8V NT or 4V MT). If the microscope is 'enabled', the *sample stage calibration* must be valid and the sample placed in the focal plane (see *microscope calibration*). If the microscope is not being used, set up a nominal 20μm displacement between the indenter and the sample as usual.

To set up and run Pin-On-Disc or Ball-on-block experiments, enter *experiment - pin-on-disc - set up and run*, and choose the experiment planned from the four choices (low speed pin-on-disc, high speed pin-on-disc, low speed ball-on-block, high speed ball-on-block). Note that these experiments are not scheduled in the same way as the indentation, scanning or impact.

Depending upon the type of experiment, the parameters to be set will vary…

1. *Pre-experiment;*

(zero load calibration and contact velocity)

2. Post contact;
(friction bridge stability delay time)

3. Definition;
(Applied force, rpm and experiment time)

4. Start Position;
(Locate the sample surface and measurement position)

Set contact velocity to 0.5 microns/s.

Disc Translation
Disc translation allows for a spiral track to be produced, this can be profiled later using the scanning module (using a low contact load in *topography scan*) to accurately determine the extent of wear, which occurred during the pin-on-disc experiment. It is necessary to define the *start position, end position* and *rotation speed*. The software will determine the necessary track pitch.

Once these parameters have been run the experiment can be run (to start/set the schedule select continue, now choose the start time and date) and the friction signal will be recorded throughout the test.

Data is analysed in *Analysis-Pin-on-disc* where *frictional force, displacement & friction coefficient* can be plotted and can be exported to ASCII for subsequent processing.

11.3 2D levelling stage

The 2D stage is used to manipulate the sample surface until it is perpendicular to the probe surface (typically $< 0.01^{0}$ of residual tilt).

Once the unit is installed on the machine with the sample attached and the probe mounted (usually a 25μm conical diamond), select 'special options' and then 'sample levelling'.

Now you have the choice to level the Y or Z axis, choose one axis and follow the on-screen procedures until completed. With the levelling complete, select the other axis and repeat the steps.

To test the 'levelling' and residual tilt, perform a topography scan (typically 500μm in the –Z direction).

11.4 Acoustic Emission

Acoustic Emission is commonly recorded, together with friction, during a ramped load scratch test, since bursts of AE often accompany coating failures, and their position can be used to accurately pin-point the onset of a failure event, and hence the critical load at which it occurs.

Acoustic emission detection utilises a special sample stub that contains piezoelectric elements. These generate high voltage signals when they are subjected to acoustic vibrations. The signals derived from the piezoelectric elements are pre-amplified and then filtered to remove any effects from low frequency ambient vibrations. Due to the fact that AE signals can be extremely fast, whereas any A-D electronics will have a finite response time, the AE signal is used to produce a 0-10 V analogue voltage proportional to the energy content of the signal that is readily detected and displayed.

11.4.1 AE Operation

1.   Attach the sample to the AE sample stub in the usual way. Use the minimum thickness of adhesive and place the sample at the centre of the stub.
2.   Attach the sample stub to the sample holder removable plate and connect the shielded cable provided. Connect the other end to the AE control unit. (The cable has snap-on connectors at each end.)
3.   Connect the AE control unit to the main system control unit by means of the low noise BNC cable provided.
4.   Switch the control unit on and allow at least 30 min for warming up.
5.   Enter *Signal Display-Acoustic Emission*.
6.   Adjust the voltage trigger level control on the AE control unit until square pulses do not appear on the signal display.

An alternative set-up method is to perform a very low load scanning experiment (topography test) with force and acoustic emission as the plotted variables and to adjust the trigger level until square pulses do not appear on the display.
To record the AE during a scratch test, simply click on *Acoustic Emission* in *Additional Data Collection* in the *Scanning experiment Set up*.

Frequency response of acoustic emission detector
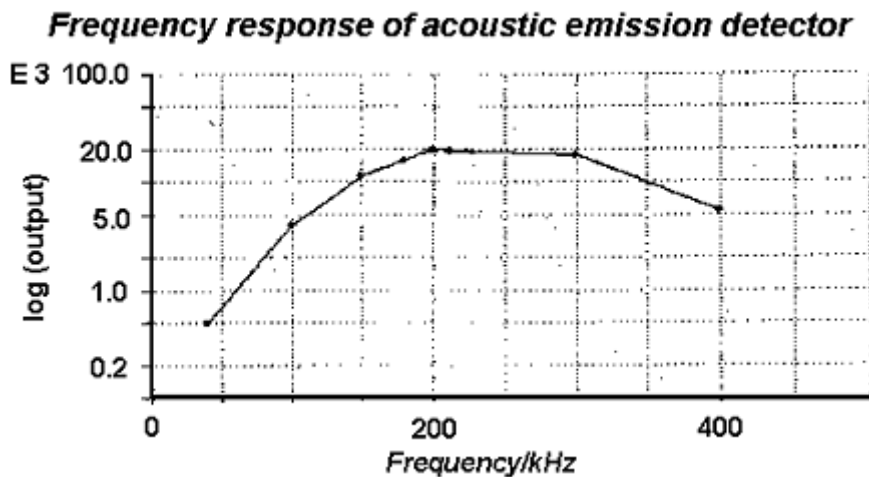
11.5 Continuous Compliance Measurement

> This technique utilises high frequency sample oscillation to determine the contact compliance (and hence derive hardness and modulus) from the measured change in the phase signal without the need to produce the loading unloading part of an indentation curve. Section 11.5.2 describes how the technique can be used to determine the complex modulus of polymers at different frequencies.

The technique uses controlled sample oscillation and measurement of the resulting pendulum response. The phase difference between the sample oscillation and the pendulum motion is related to the elastic properties of the contact. To determine the hardness and modulus of materials which store and release energy without loss during oscillation (such as metals and ceramics), requires the contact compliance and the total depth of penetration. The contact compliance is found from the phase difference as shown below and the total depth is a basic NanoTest measurement.

The *Sine Out* signal from the Lock-in Amplifier is fed into the piezo stack. The phase signal output (from the depth + offset of the NanoTest control box) is fed back to the *A input* of the lock-in amplifier.

The procedure has three main steps:-

1. Determine the resonant frequency of the system
2. Determine a calibration curve by performing CCCC measurements on fused quartz over the entire load range
3. Perform CCCC measurements on the test sample

11.5.1 Determination of the Resonant Frequency

Set the amplitude to 5V. Bring the Berkovich diamond into contact with the quartz surface - set a 10 mN initial load (the high load is to keep the probe in contact), and

continue motor movement (continue X negative) until the rectified output reaches half the initial amplitude (i.e. ~4 V). Once the surface is reached the phase signal becomes stable. Now turn the frequency knob until the amplitude signal reaches a maximum (the resonant frequency) and note down this frequency (it is used in 11.5.2).

> **Typically, the frequency is about 75-85 Hz giving a maximum amplitude of ~ 0.5 mV.**

11.5.2 Determination of the Calibration Curve

A fused quartz sample is used as a calibration standard. The phase angle data are collected during the second half of the dwell period at maximum load (i.e. allowing an initial period for stabilisation). It is necessary to set up schedules to find how the phase angle varies with load over the entire range. Enter **Schedule** then go to **Define Continuous Compliance Determination Schedule** and, after positioning the sample, choose **Collect Calibration Data**.

Set up three schedules using the **Multiple Menu Grid** option, one covering 10 indentations in the range 200-40 mN, one covering 10 indentations in the range 40-10 mN (where the largest phase changes occur) and lastly 10 indentations in the range 1-10 mN.

Typical system data are:-
Number of zero calibrations 3.
Experiment Start Delay 300 s.
Dwell time 30 s.
Drift data acquisition 0 s.
Oscillation amplitude 5V.
Oscillation frequency 80 Hz. (set to the resonant frequency determined in 11.5.1)
Sampling time constant 1 s. (the time constant is the response time of the amplifier; longer time constants mean that short fluctuations will have no effect on the phase reading)

The phase data are fitted to produce the calibration curve, as illustrated below. The fit is excellent across the whole compliance range. Extrapolation to zero compliance gives the instrument contribution to the phase angle.

```
Oscillation frequency .. 83 Hz
      35 Calibration points
```
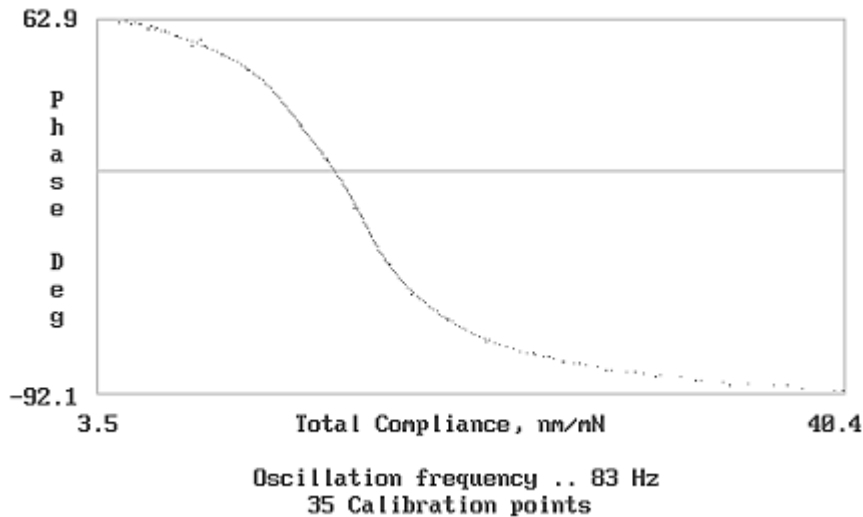
Complex Modulus Determination

The continuous compliance technique can also be used to determine the complex modulus of polymers at different frequencies. For polymers some energy is lost due to 'internal friction' as the material flows under pressure. Clearly, for viscoelastic materials the deformation response under an applied stress will be due to the combined effect of elastic strain and flow. This results in the stress and strain curves being out of phase as shown. The apparent modulus is resolved into a component in phase with the stress (the storage modulus) and a component out of phase (the loss modulus). The so-called loss tangent, which is the energy dissipated per cycle, is given by:

$$\tan\delta = \textbf{Loss modulus/Storage modulus}$$
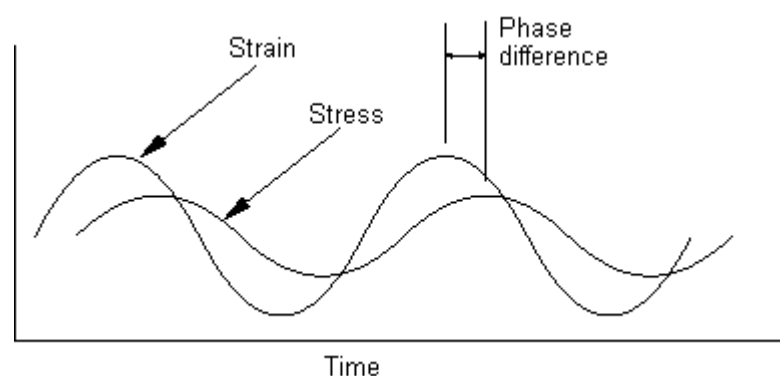
Applied stress and resulting strain of a viscoelastic material

11.6 High resolution Imaging Stage - set up and background

Starting Platform with the Imaging option active

When the platform system is started with the High Resolution Imaging Stage option set up in the Platform Configuration Panel, two different system initialisation paths will be taken. If the imaging stage control unit is not switched on then Platform will initialise as normal i.e. set up the standby machine etc, before displaying the main system menu. In this case all of the high resolution image acquisition options will be dimmed out.   If the imaging stage control unit is switched on before Platform is started then the following message will be displayed during the system initialisation.
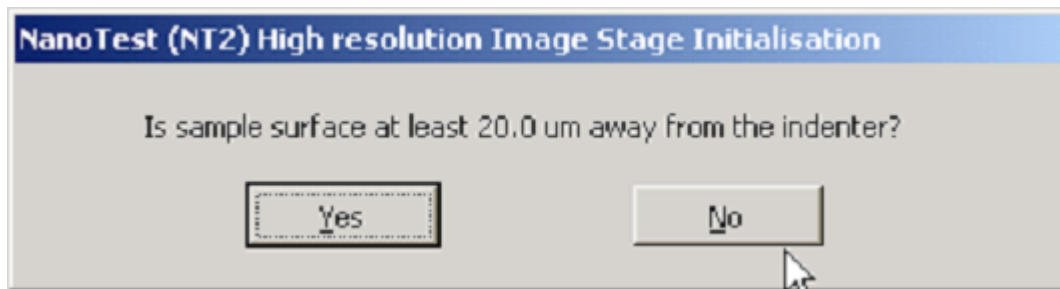
This question has to be asked because during the initialisation of the PI stage an 'auto zero' is performed. The 'auto zero' involves running each axis to the extremes of its travel. In the case of the 'X' axis (that in the line with the X stage on the NanoTest) the extreme of travel is 20um and so if the indenter is left any closer than this to the sample surface then 'auto zeroing' the 'X' axis will push the sample into the indenter! If you reply "yes" to the question then the image stage will continue as shown below otherwise the following appears on the screen

Pressing "OK" causes the motor control panel to be displayed to allow you to move the sample away from the indenter.
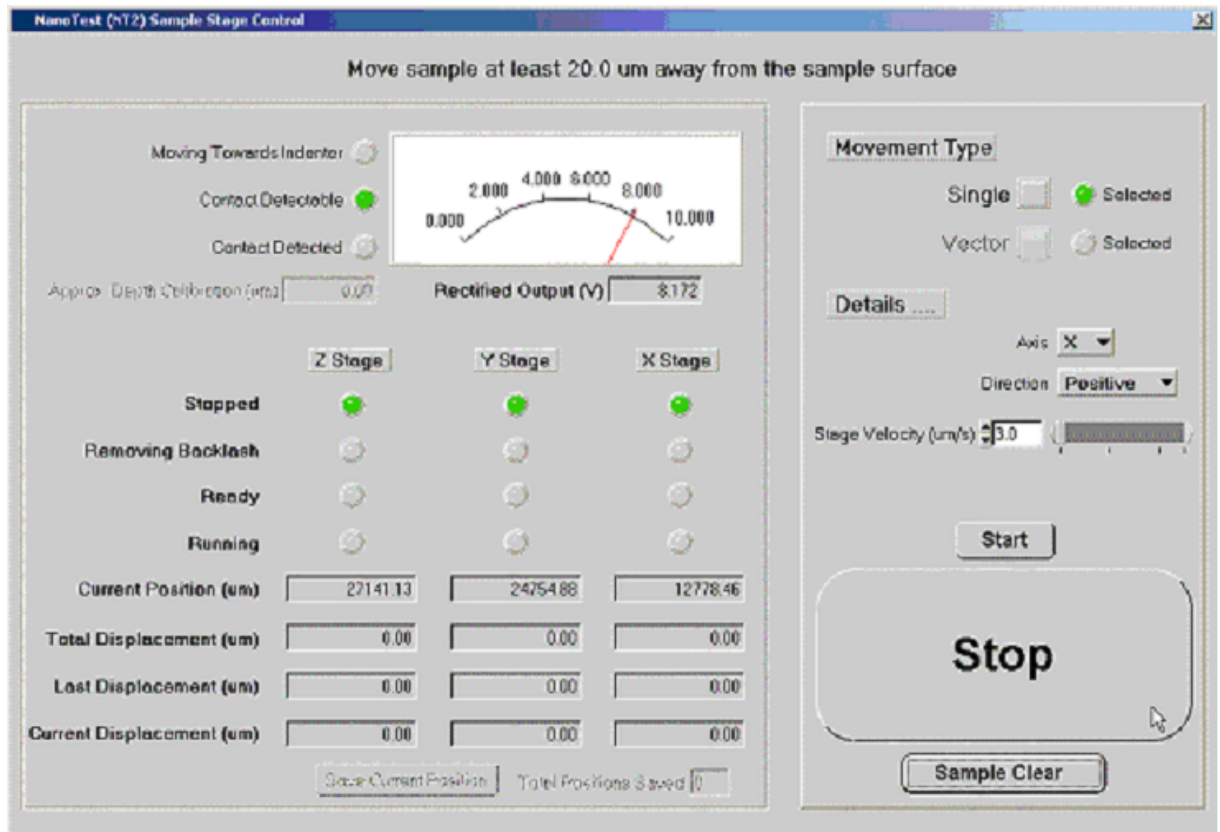
Note that the message at the top of the panel reminds the operator how far to move and that the "Axis" and "Direction" controls are set up in the correct sense automatically.

Once you're happy that the sample surface is the required distance away from the indenter click on "Sample Clear" and the initialisation procedure will continue whilst the following progress panel displayed on the screen
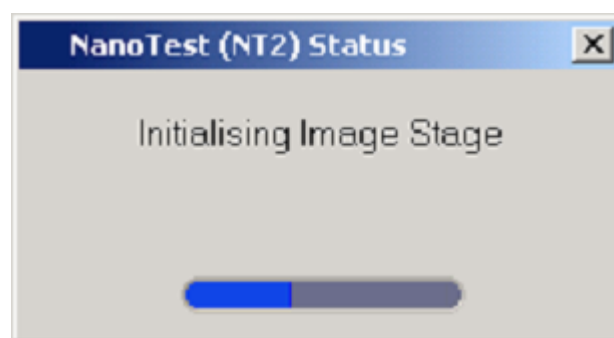
Important Note…The imaging stage control unit MUST NOT be switched on after the Platform system has been started!

Stage Coordinate System

The full range of the imaging stage is 0 to 200 um in the 'Z' and 'Y' directions. On power up and at all times when the stage is not in use Platform maintains the image stage at position (100um,100um,10um) (i.e. Z = 100um, Y=100um, X=10um) thus I have defined the 'image coordinates to run from –100 to 100um in 'Z' and 'Y' directions and the standby image coordinate as (0,0).   In essence this means that if you start Platform with the imaging stage installed and its control units switched on, the first indentation of your experiment will be at image coordinate (0,0). See example "How to produce and image an indentation"

Image Acquisition Panel

High resolution image data acquisition is set up by selecting the following option
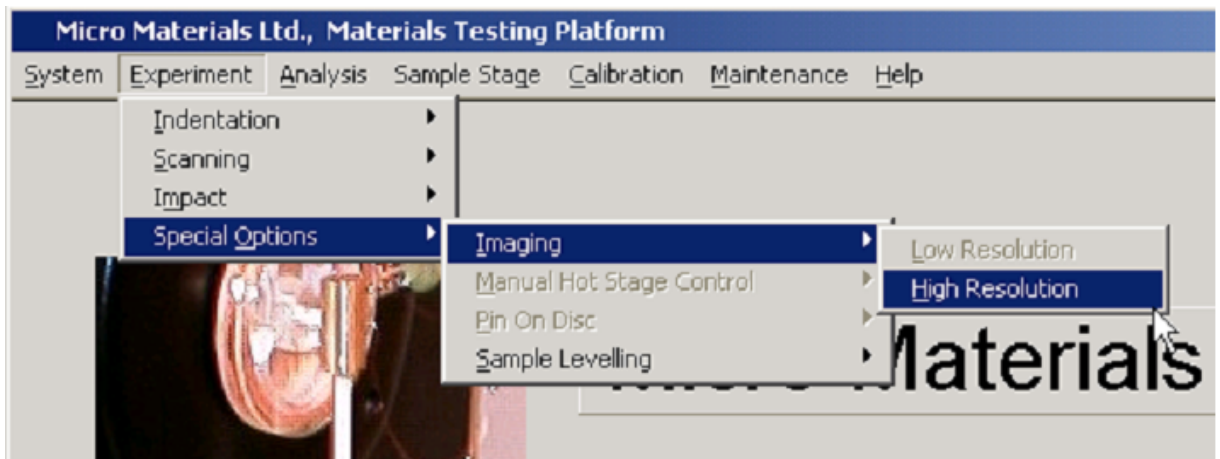
The system will check that he image stage control unit is connected and switched on and then display the following set up menu
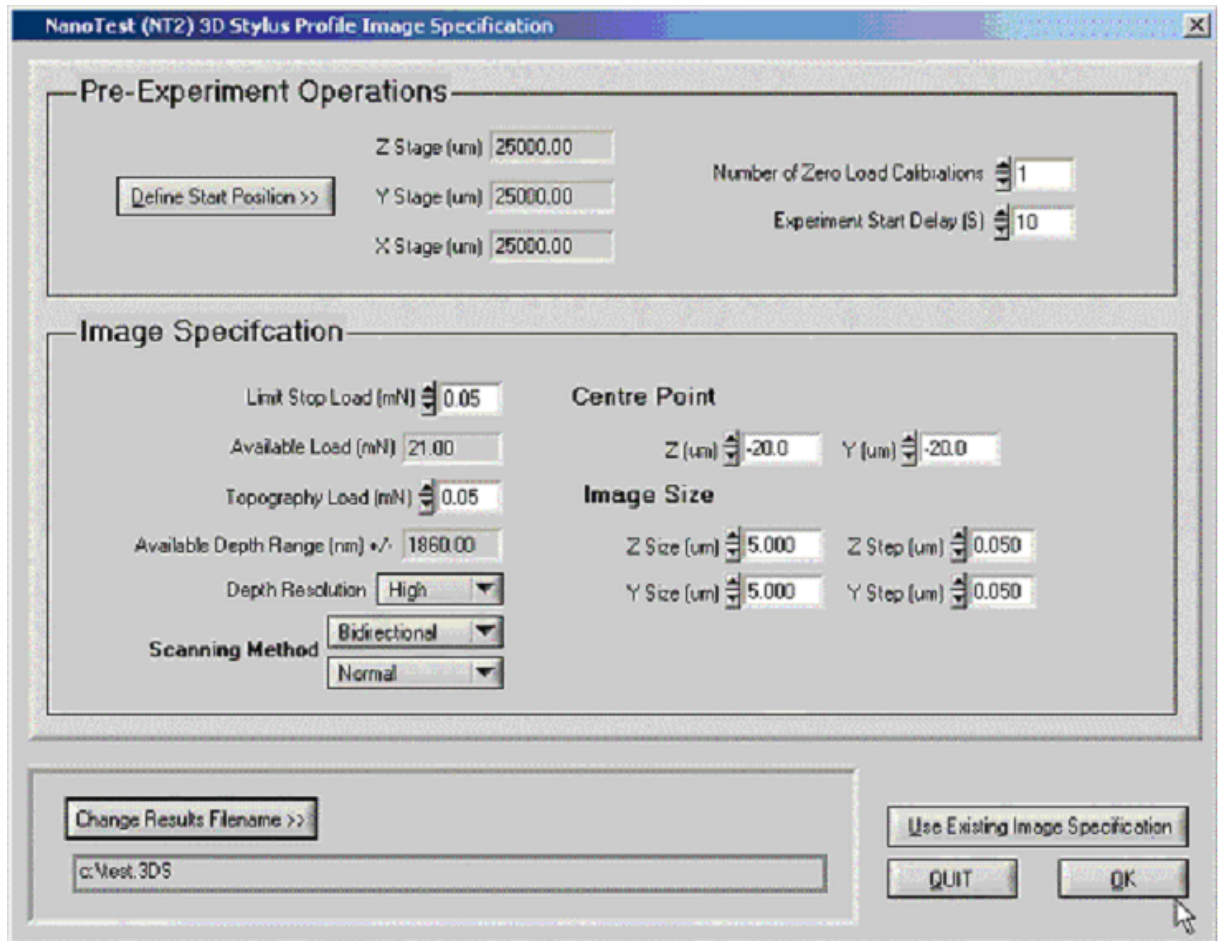
Pre-Experiment Operations

"Define Start Position" This control is used to call the normal stage control panel to move the NanoTest sample stage.

"Number of Zero Load Calibrations" and "Experiment Start Delay" these have the same meanings as within the other schedules.

Image Specification

"Limit Stop Load" is the load applied during the contact procedure.

"Topography Load" is the load applied during image data acquisition.

"Depth Resolution" this has two states 'high' and 'low'. With 'High' selected the depth is read with the same resolution used during an indentation at 100% depth gain (i.e. around +/- 2um for a typical set up). If 'Low' is selected then the depth is read on the 10 volt range rather than the standard 1 volt which will increase the available depth range but reduce the resolution by a factor of 10. In any setting the actual available depth range is shown in the "Available Depth Range" control

Scanning Method

With these controls you can select between 'bi-directional' and

'raster' scanning modes. In bi-directional, the fastest method, the image is created by effectively 'zig-zaging' across the surface and data being collected in both directions. In raster mode the image in formed by scanning rather like a TV screen with data always colleted in the same direction.

Centre Point

These controls are used to specify the required coordinates of the centre of the image.

Image Size

These controls are used to define the size and resolution of the image. For example the image specified in the menu above will contain 10000 points and the Z coordinates with run from –22.5 to –17.5 while the Y coordinates with run from –22.5 to –17.5. The coordinates are defined such that they correlate with those entered into the indentation schedule set up menu. See example "How to produce and image an indentation"

Image Display Panel

Image data can be reviewed using the image data review option
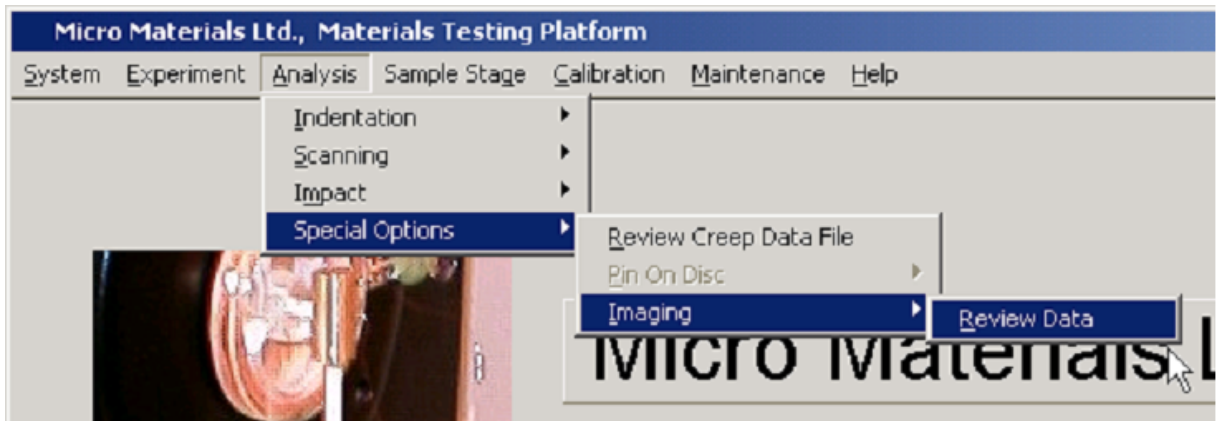
After which you will be prompted to select a filename for review.
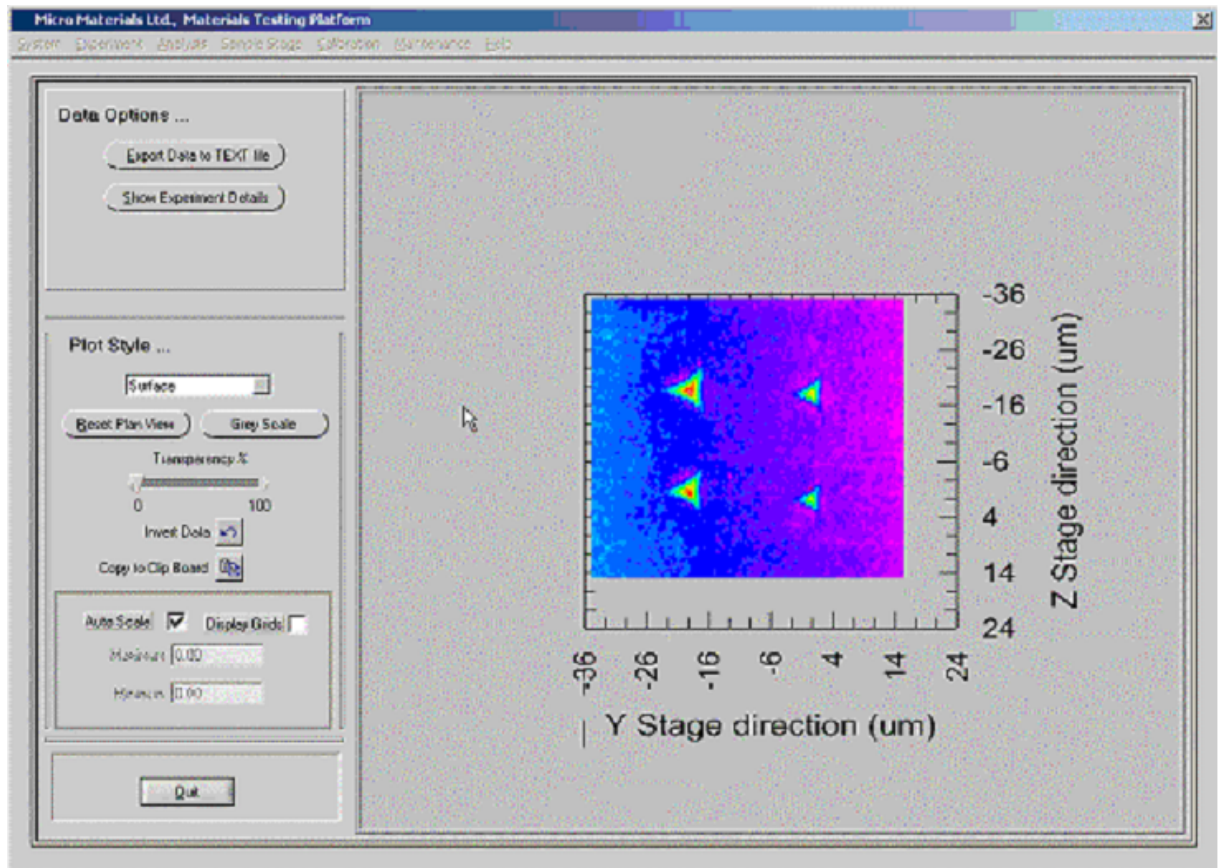
Most of the controls are self explanatory.

"Export Data to TEXT file" enables the operator to produce a text data file that can be used within other graphics packages.

The layout is one line per point Z value <TAB> Y value <TAB> depth value.

"Show Experiment Details" reproduces the menu used to specify the image capture.

The image itself can be manipulated using the mouse by dragging the mouse over it with the left button held down. The scroll wheel can be used to zoom in and out of the picture.

How to produce and image an indentation (see also 11.6.1)

As described above when prompted to do so make sure that the surface is at least 20 um away from the indenter.

Set up an indentation schedule in the usual way for example
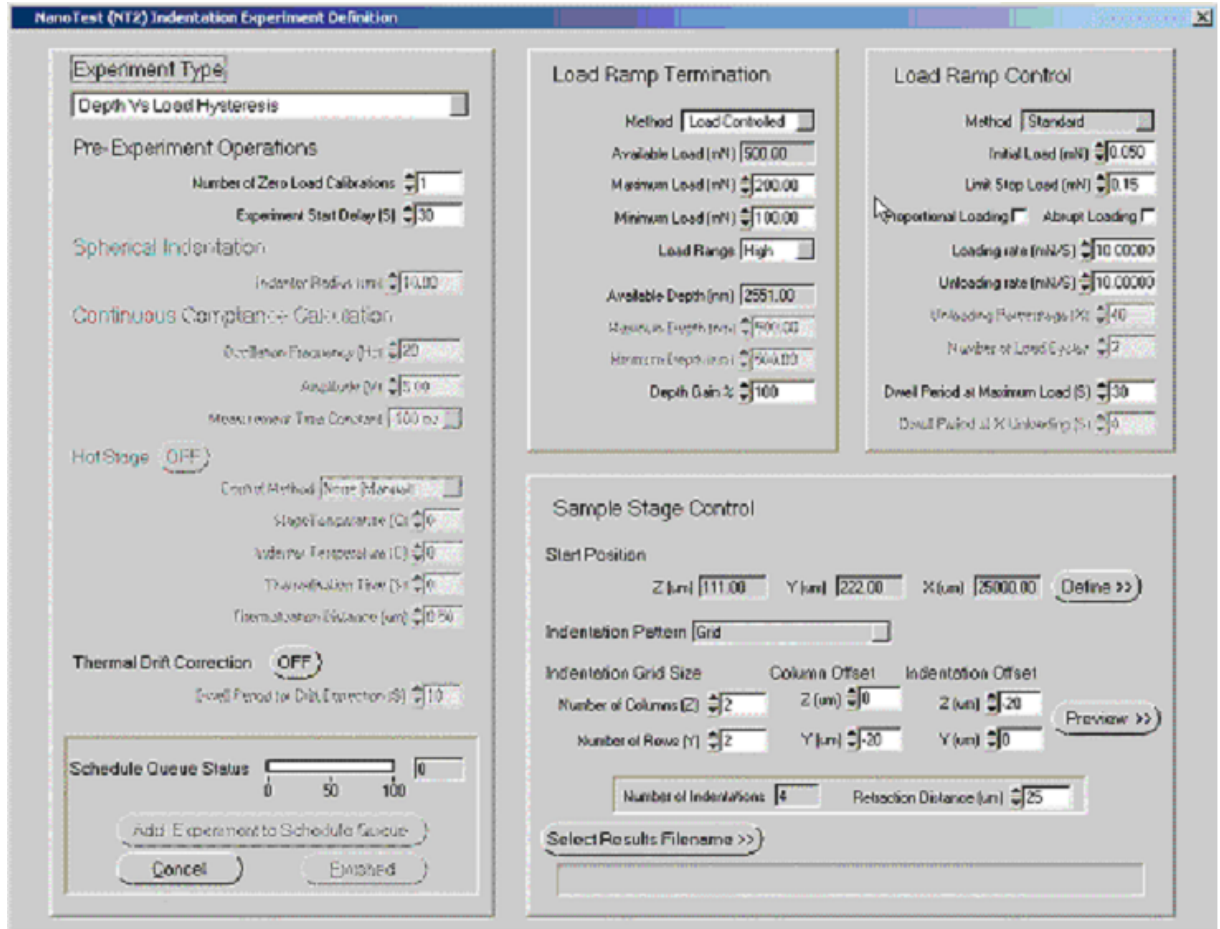
This should produce a small grid of indentations whose depth increases
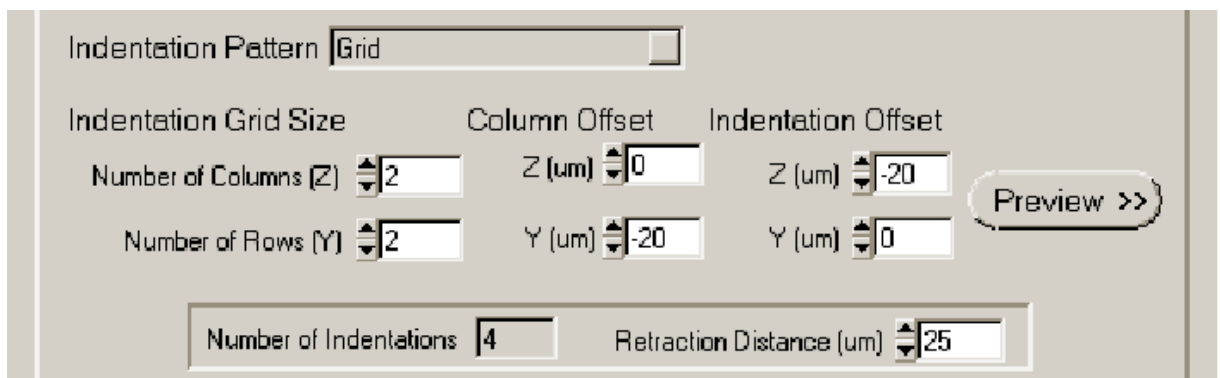
The locations of the indents (Z,Y), in order, will be (0,0) (-20,0) (0,-20) and (-20,-20)

Run the schedule and when it has completed select the imaging option
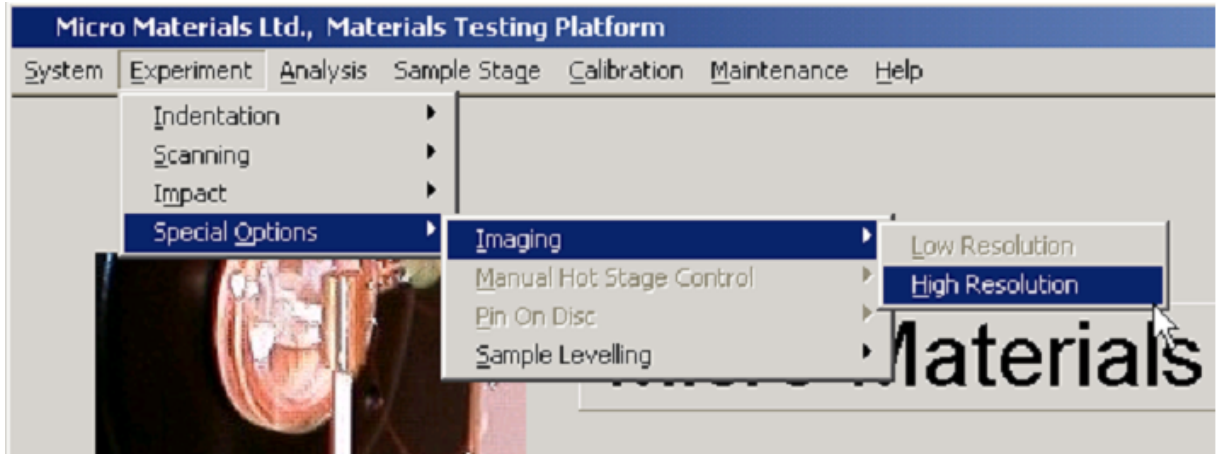
The following menu entries should produce an image centred in the centre of the grid showing the indentations at the correct coordinates.
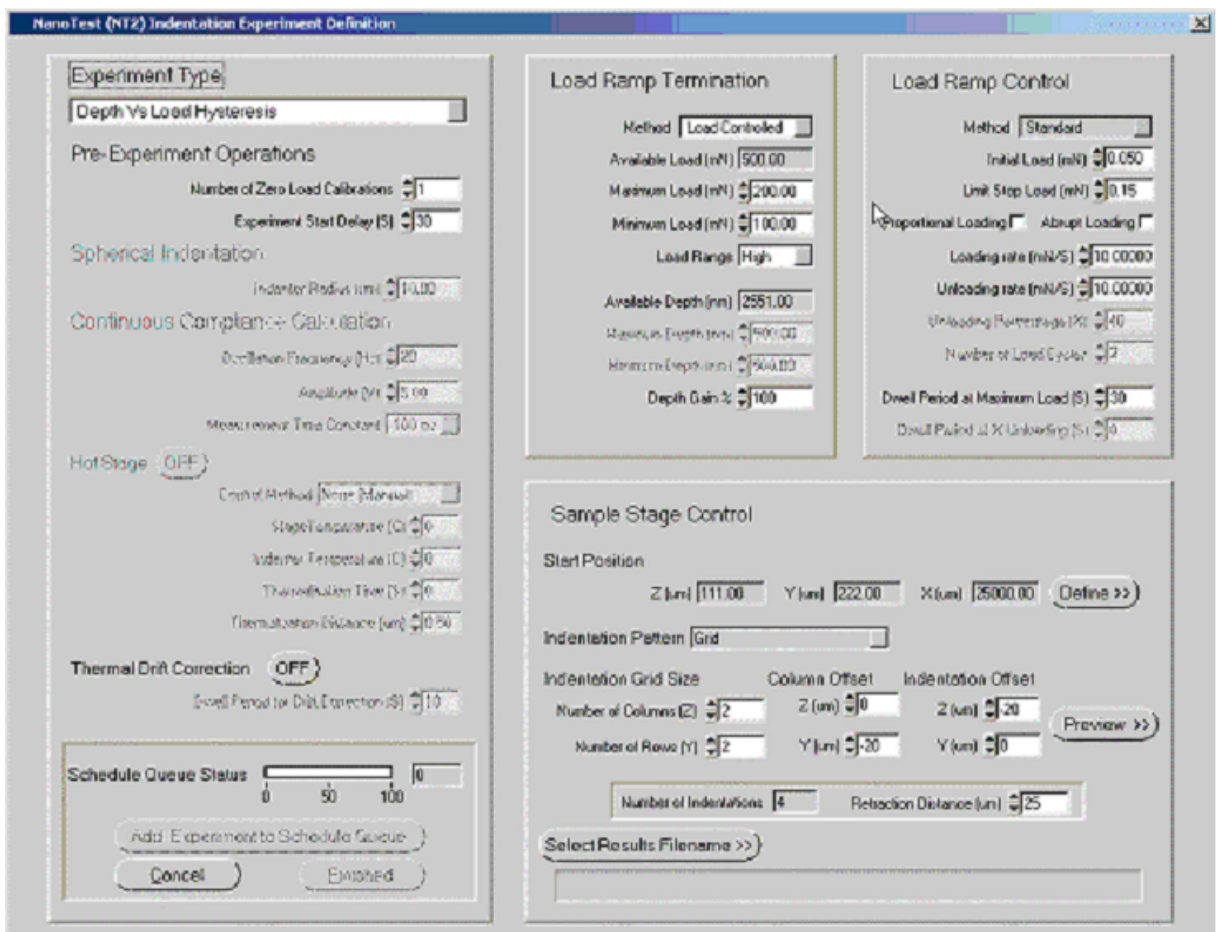
After entering a filename (click on "Select Image Filename") click on "OK". While the data acquisition is running a real time display is produced. However the display uses only 12 colours to cover the whole depth range and so it may not always be possible to see anything on this display
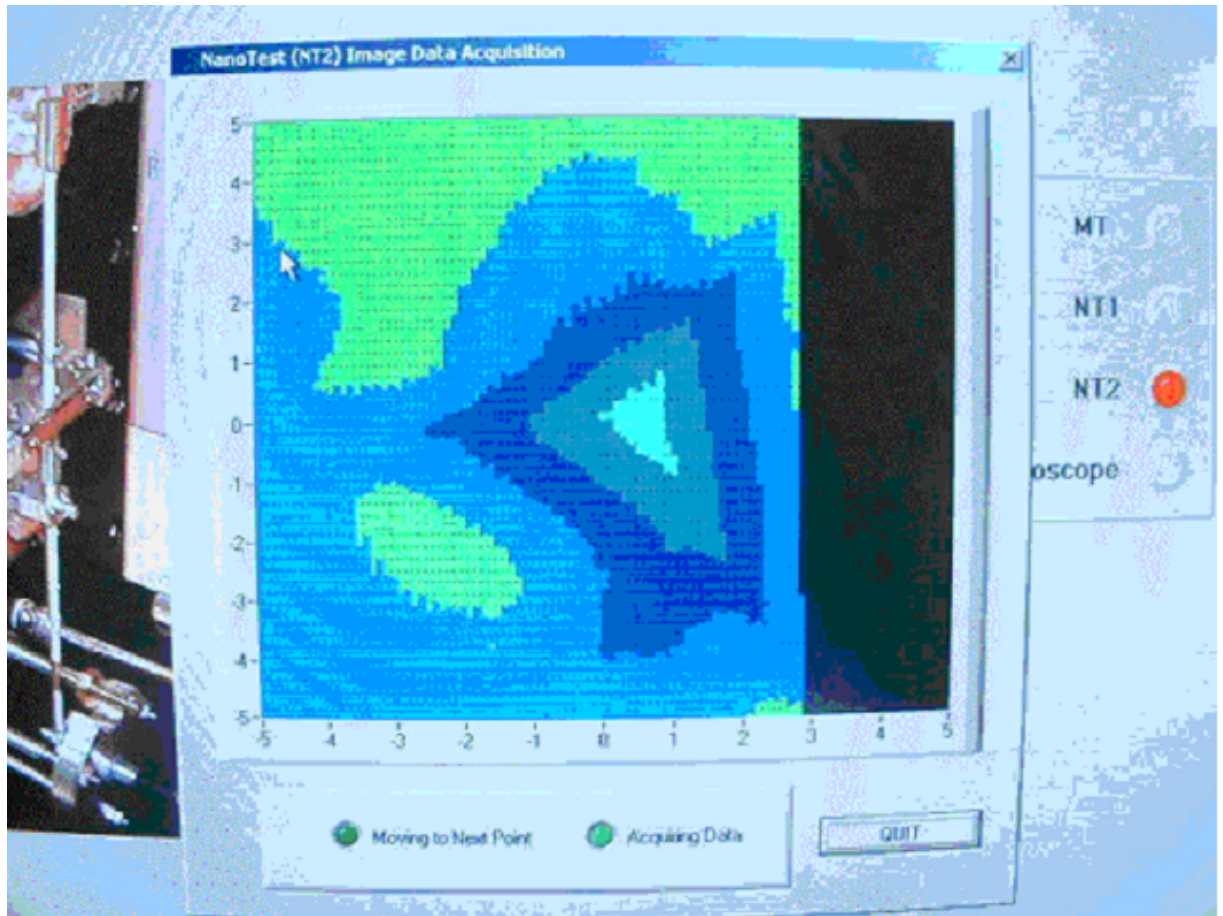
When the acquisition is completed the following will be displayed showing the result with the best possible colour resolution
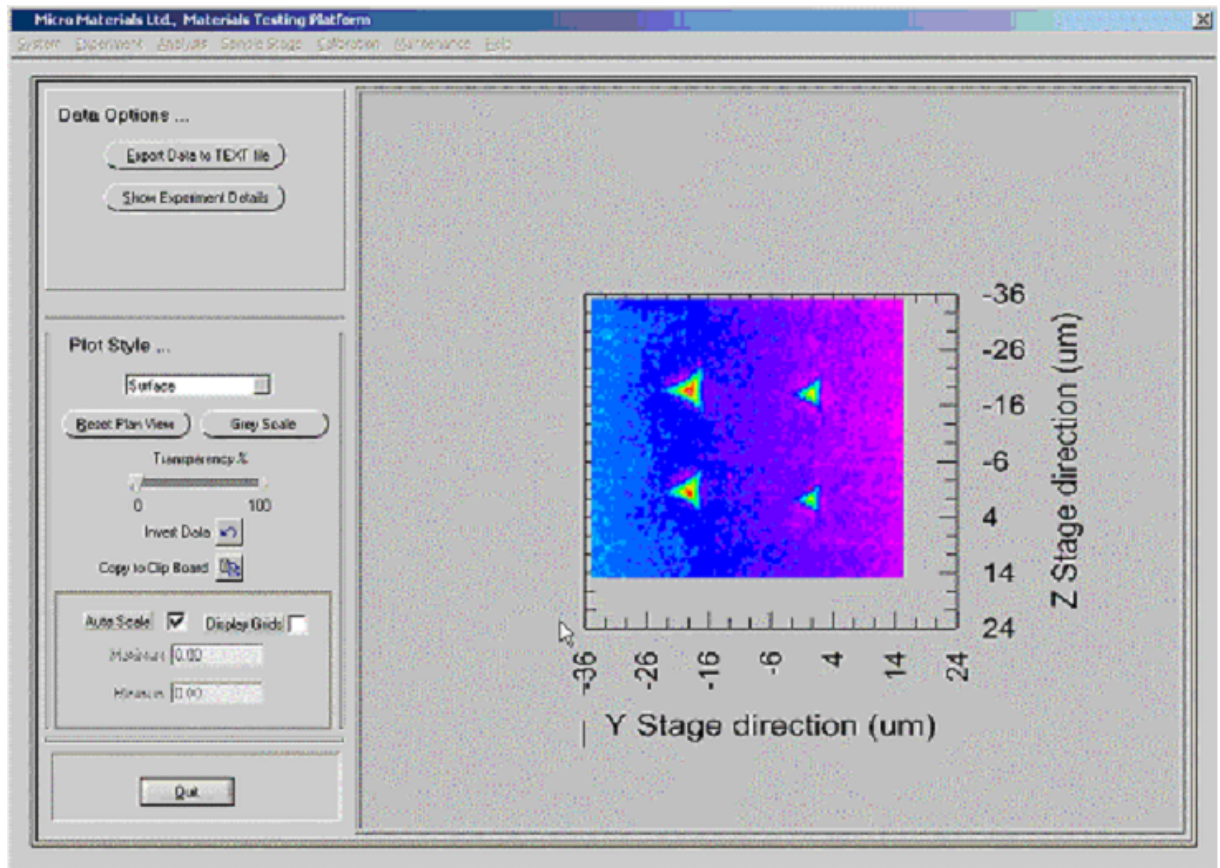
You can see that the image is centred on (-10,-10) at the first (smallest) indentation is located at (0,0) with the last (largest) at (-20,-20). The default display is depth verses position this can be inverted by clicking on the "invert data" control. The Depth axis can be scaled manually by un-selecting "Auto Scale" and entering the required maximum and minimum values. I plan to extend the manual scaling option to include the Z and Y-axes.

Further images can be set up and acquired because the coordinates of the indentation will remain fixed until the NanoTest sample stage itself is moved.

11.6.1 More on the Piezo stage (nanopositioner)

The 'point and click' option allows the operator to-

- · Produce a high resolution 3D topography image of the surface of a sample
- · Use the image to define the location on the surface at which scheduled indentation experiments will be performed.
- · Allow the operator to rescan the surface to 'image' the resulting indention features.
- · Provide the option to repeat steps 2 to 3 all the time using the latest image obtained from the sample.

Method

1. Select the high resolution imaging option from the Experiment menu bar
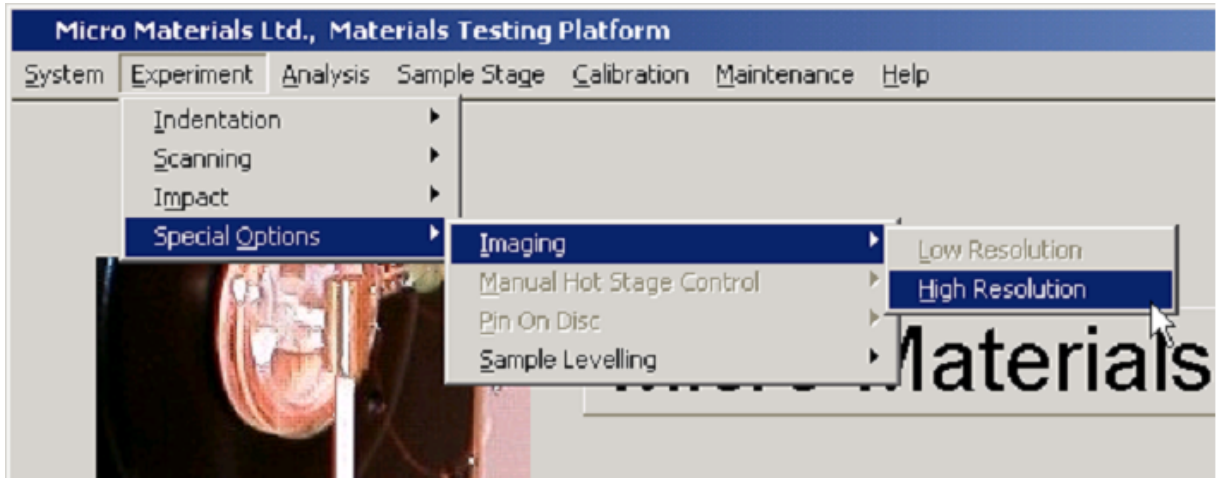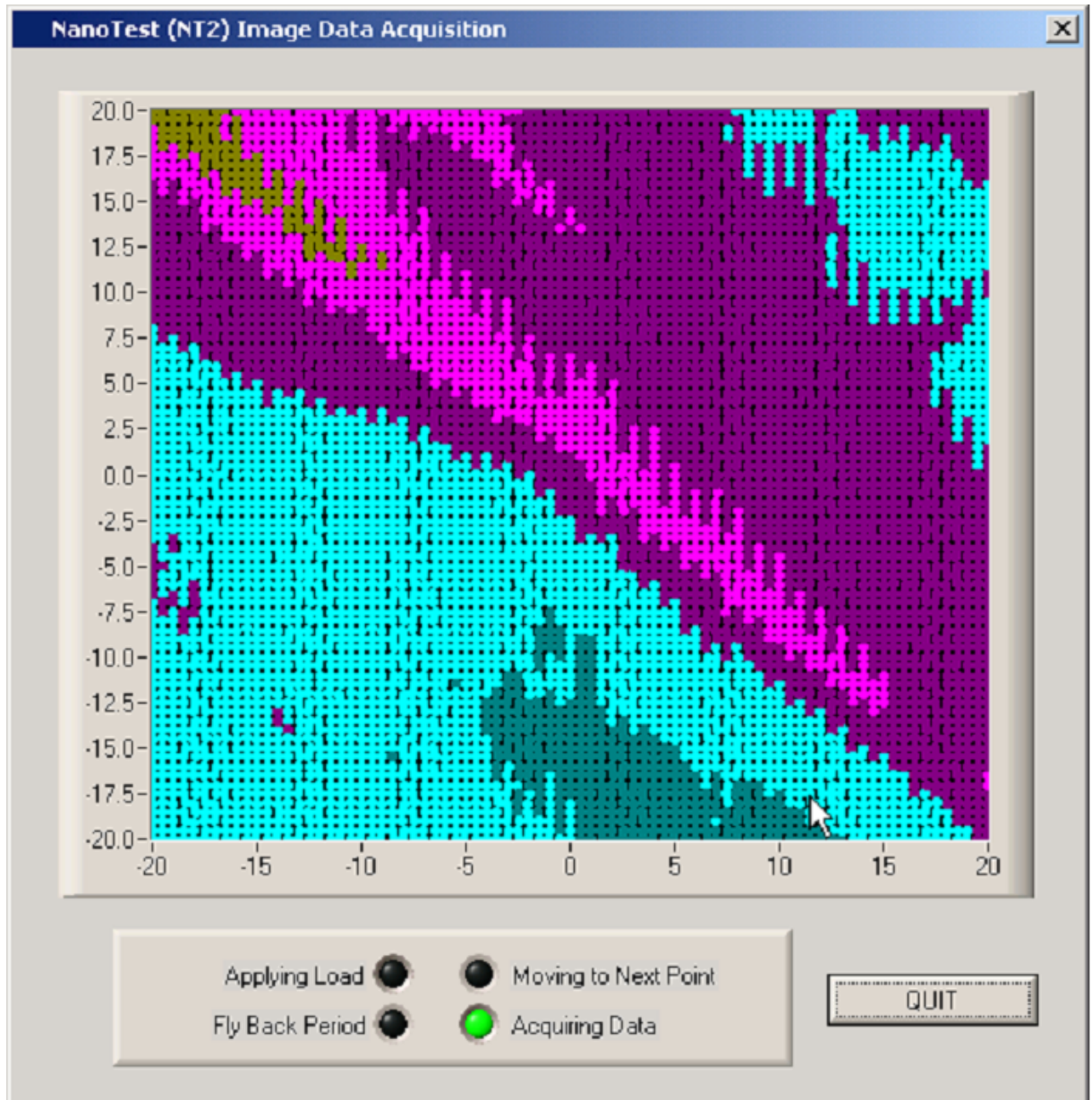

A high resolution scanning menu will be displayed
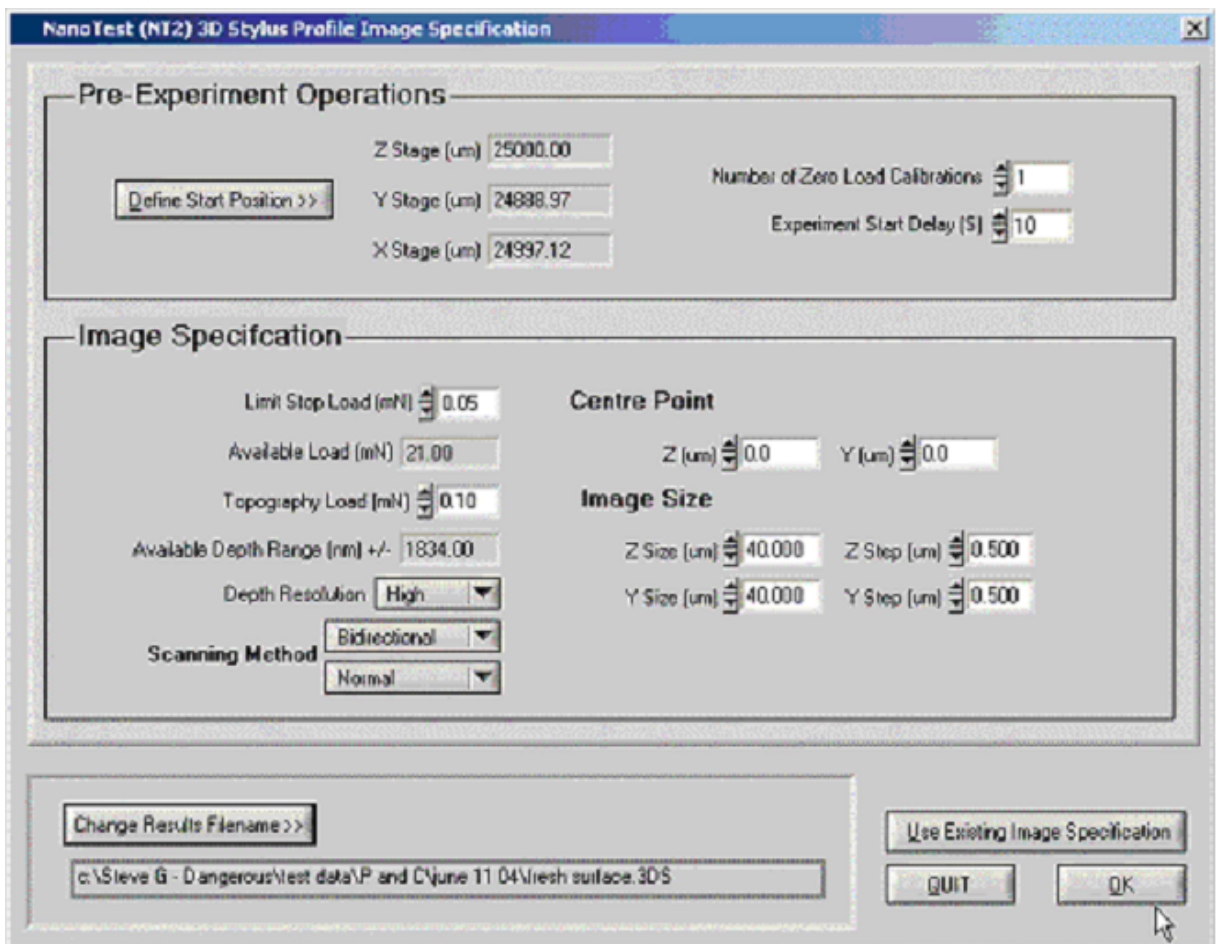
Use the controls on this menu to define the initial scan of the sample surface. Note.
As normal, "The Define Start Position Control" should be used at this stage to place
the surface of the sample around 25um from the sample surface.
Click "OK" to continue with the experiment and as normal enter the start time when
the schedule start window is displayed.

2. When the experiment is running a low resolution image will be displayed

3. When the scan has finished a high resolution image will be displayed
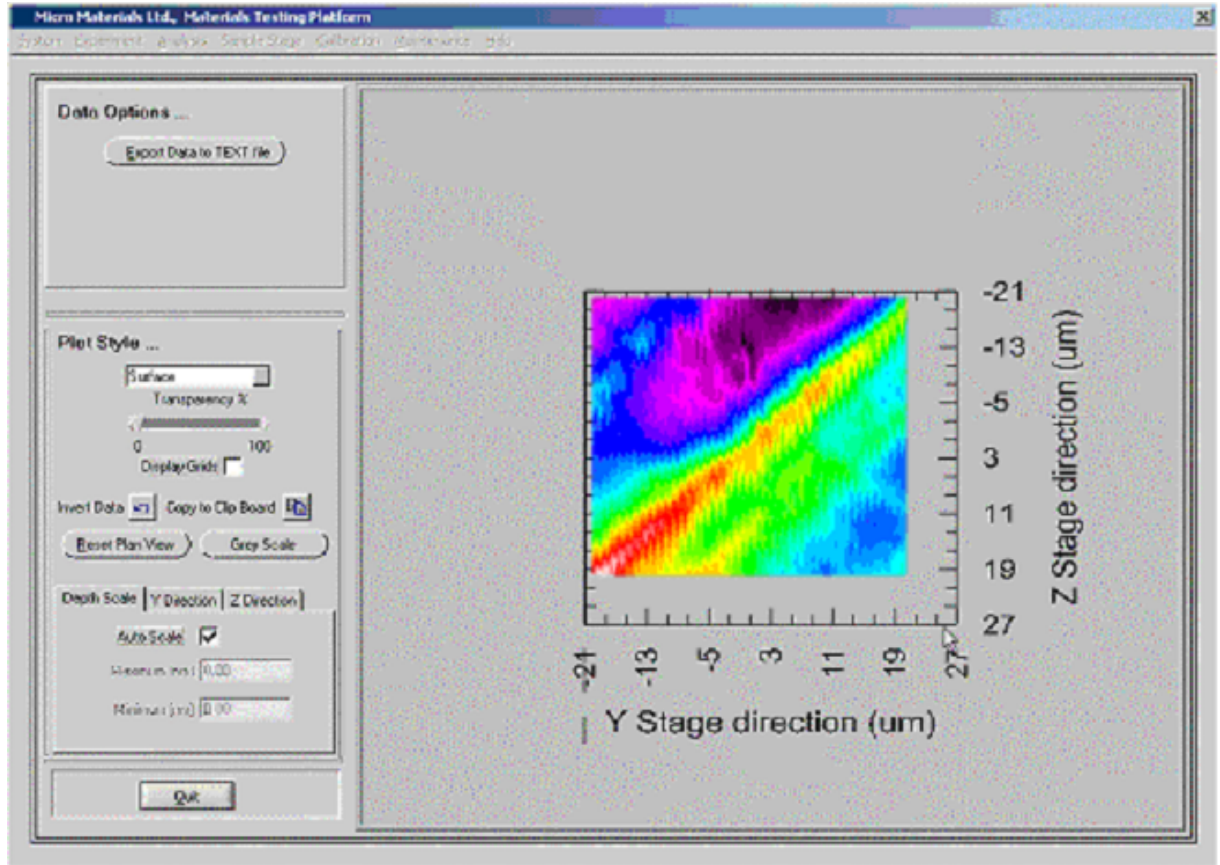
The image can be manipulated in the usual ways – scaled, display type etc.

4. When "Quit" is clicked the following question will be displayed
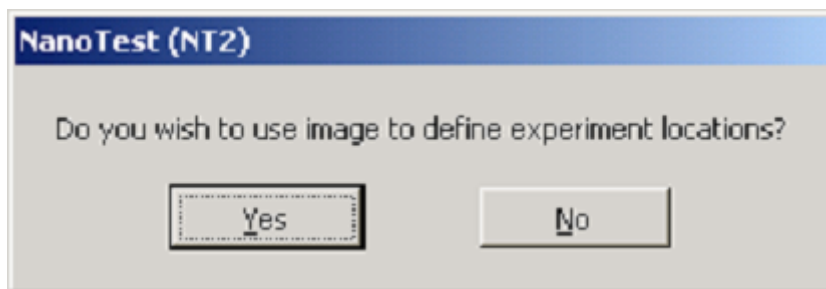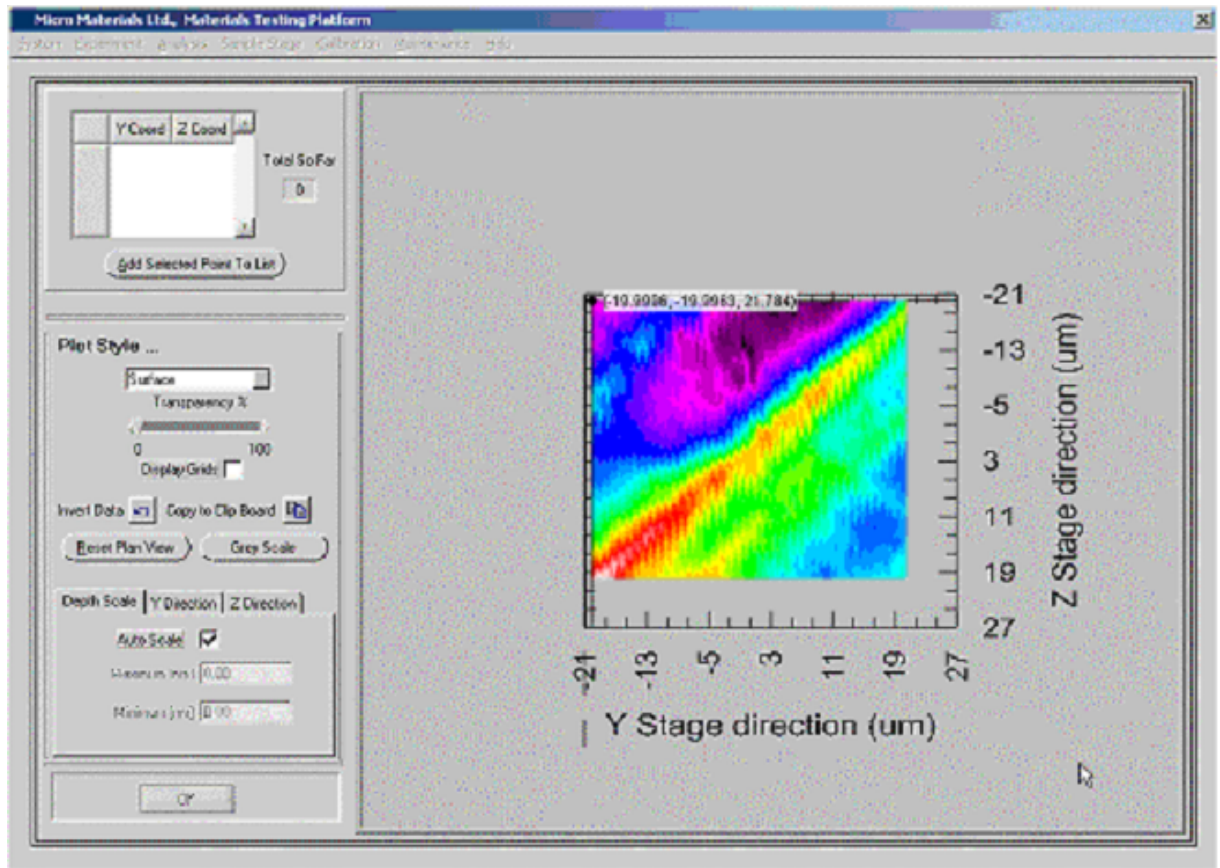

Click "No" if you're only interested in the image or not happy with its position on the surface.
Click "Yes" if you want to continue to define indentation locations on the image.

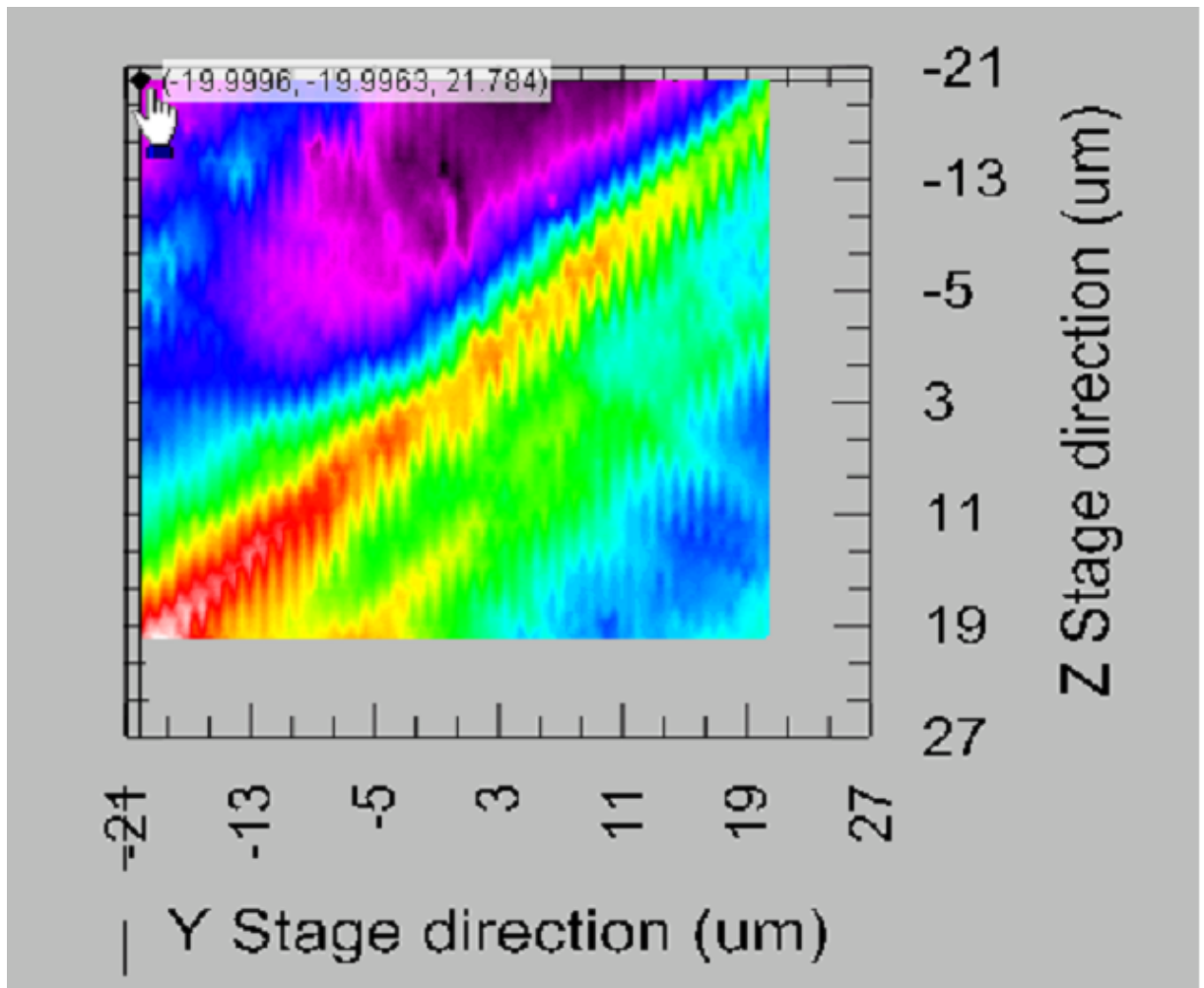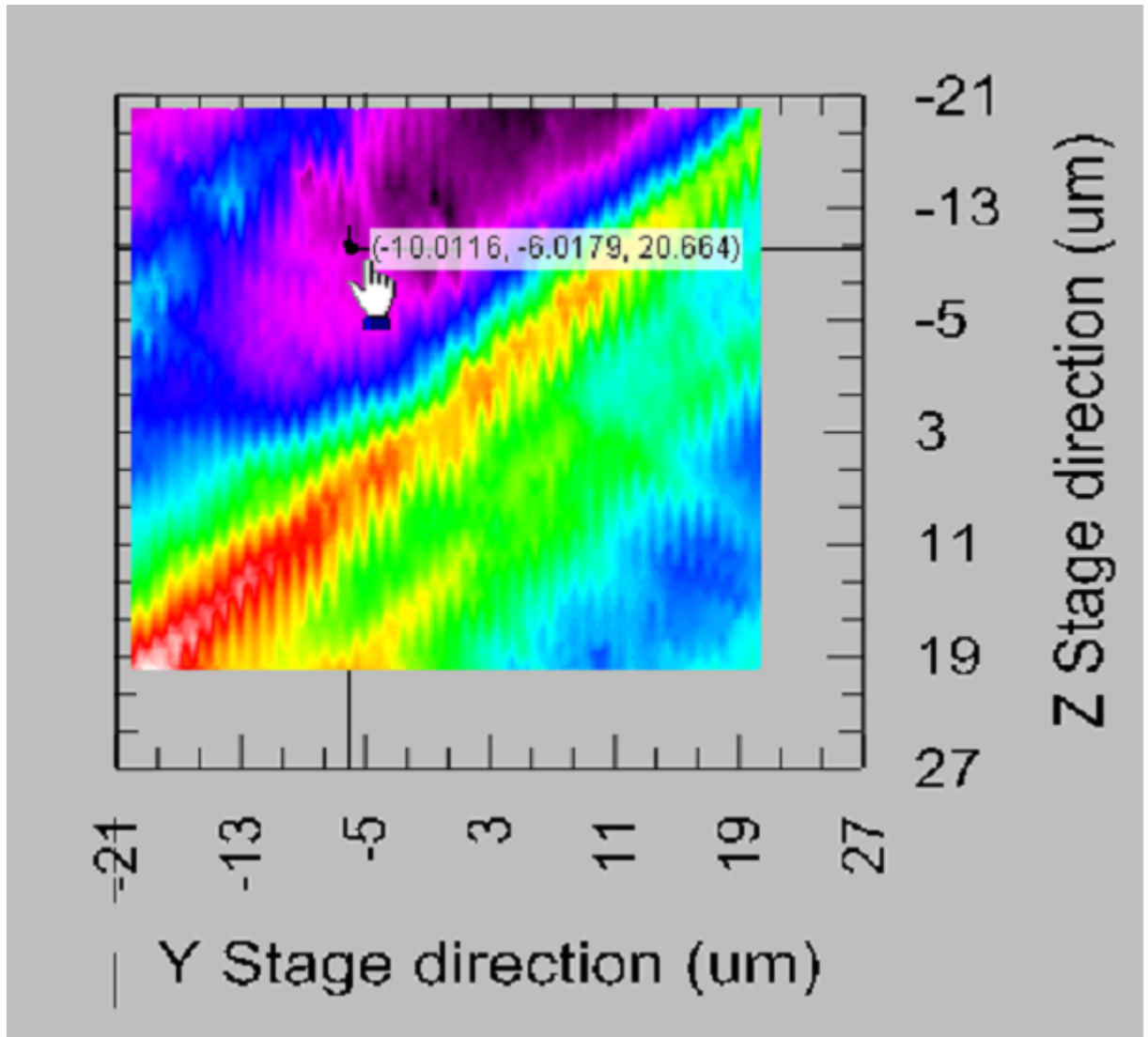5. If you click "Yes" an experiment definition menu will be displayed.
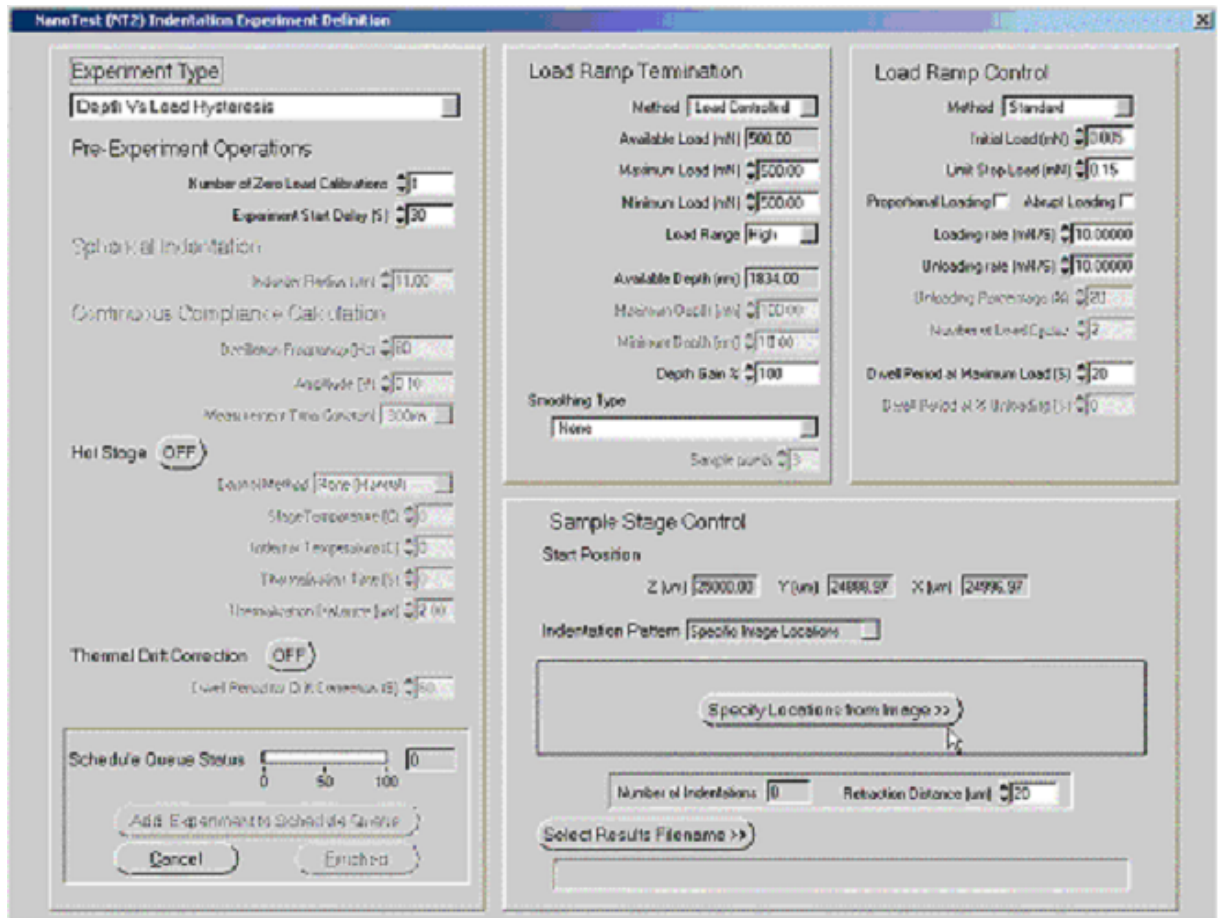
After selecting the indentation experiment type the click "Specify Locations from Image" button to re-display the image
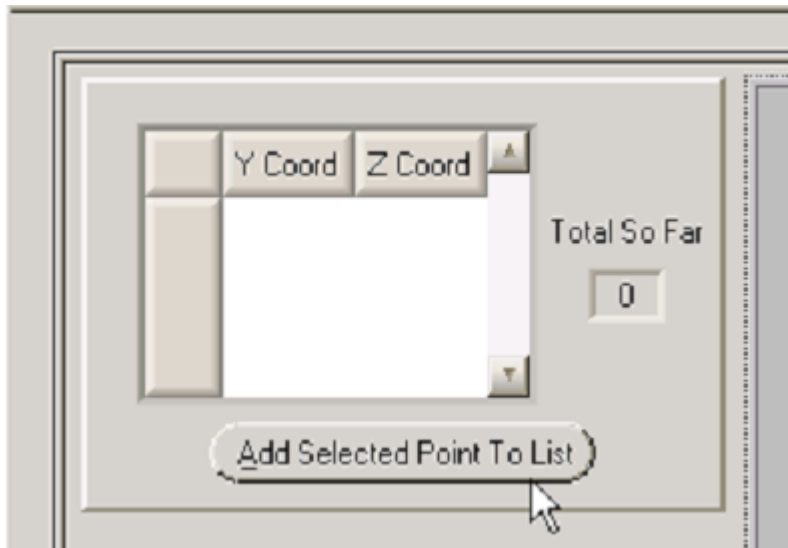
The image will be displayed with a cursor located at its top left hand corner and the display panel modified to contain a 'selected point list' in the top left hand corner.

    6. To define an indentation position first 'drag' the cursor to the required position on the image
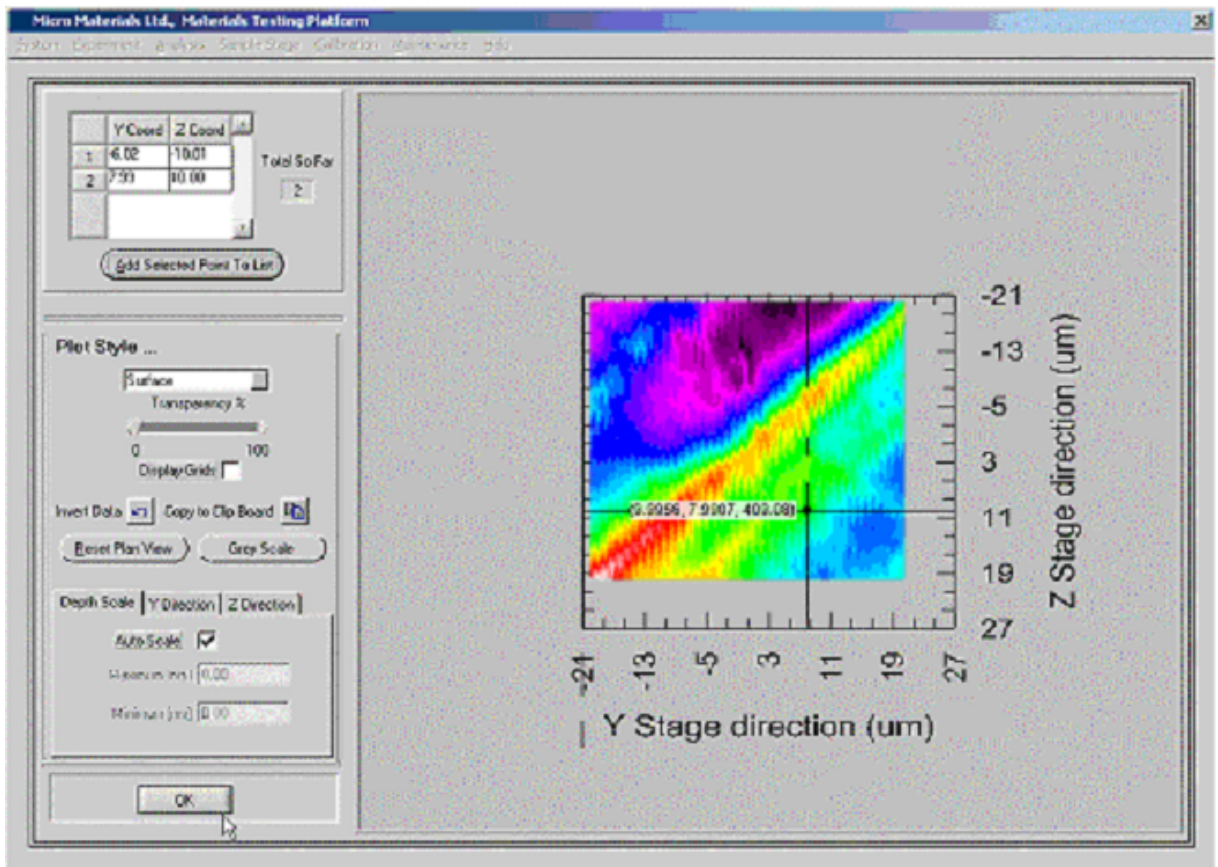
and then click on "Add Selected point to List"

Repeat for each location, here two points have been selected

Click "OK" to return to the experiment definition menu panel
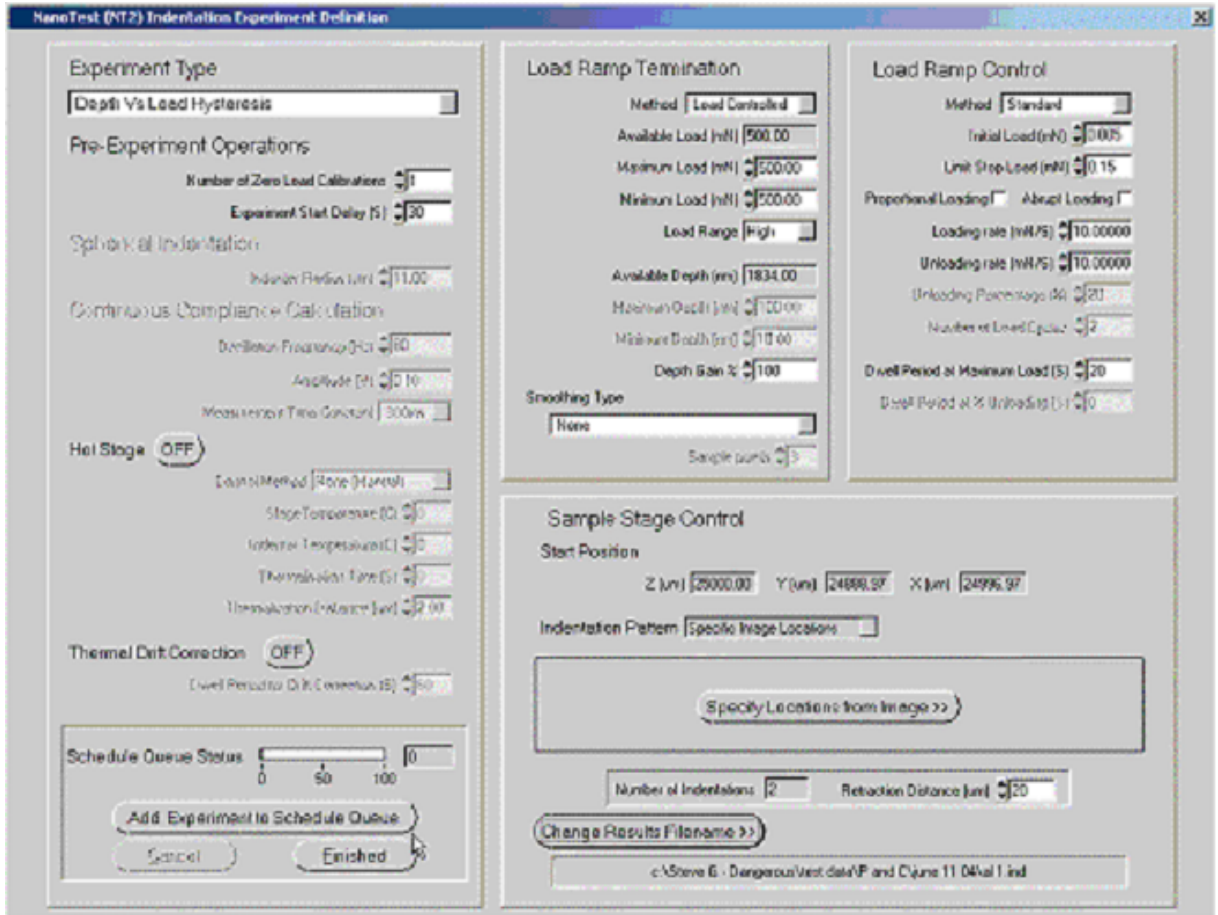
The experiment is added to the schedule queue in the normal way by clicking on "Add Experiment to Schedule Queue". Other experiments can be added to the schedule in the normal way.

7. When you've finished creating the schedule click on "Finished" in the normal way.
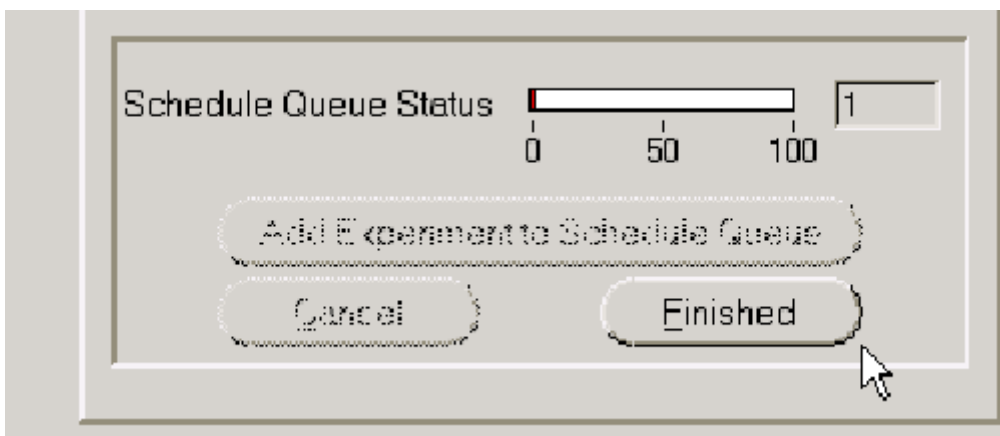
8. When the schedule has finished
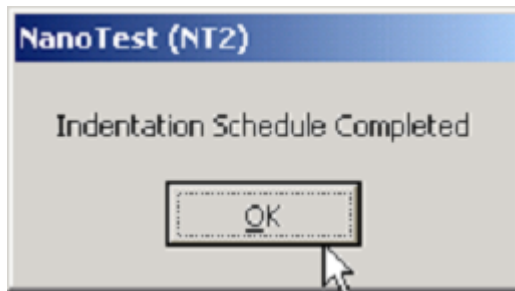
To create an image of the surface with the new indentations select the high resolution imaging option from the main menu BEFORE any movements of the standard sample stage are made.
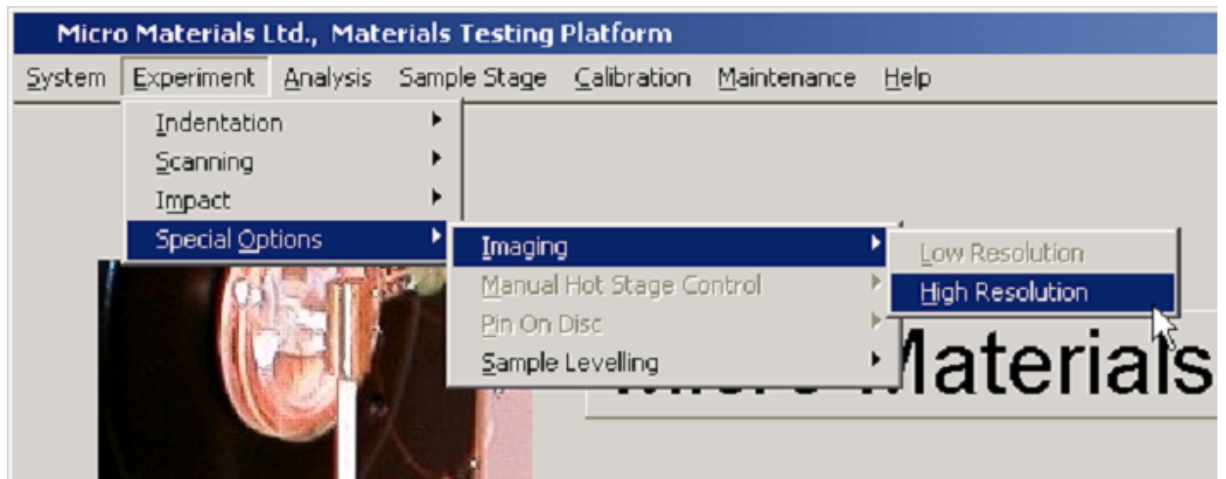
Then define another image scan using the displayed panel.

Note DO NOT use the "Define Start Position" button since moving the standard sample stage at this point would lose the registration between the initial and the defined image.

When the procedure has completed the displayed image should contain the indentations at the locations defined at stage 7.
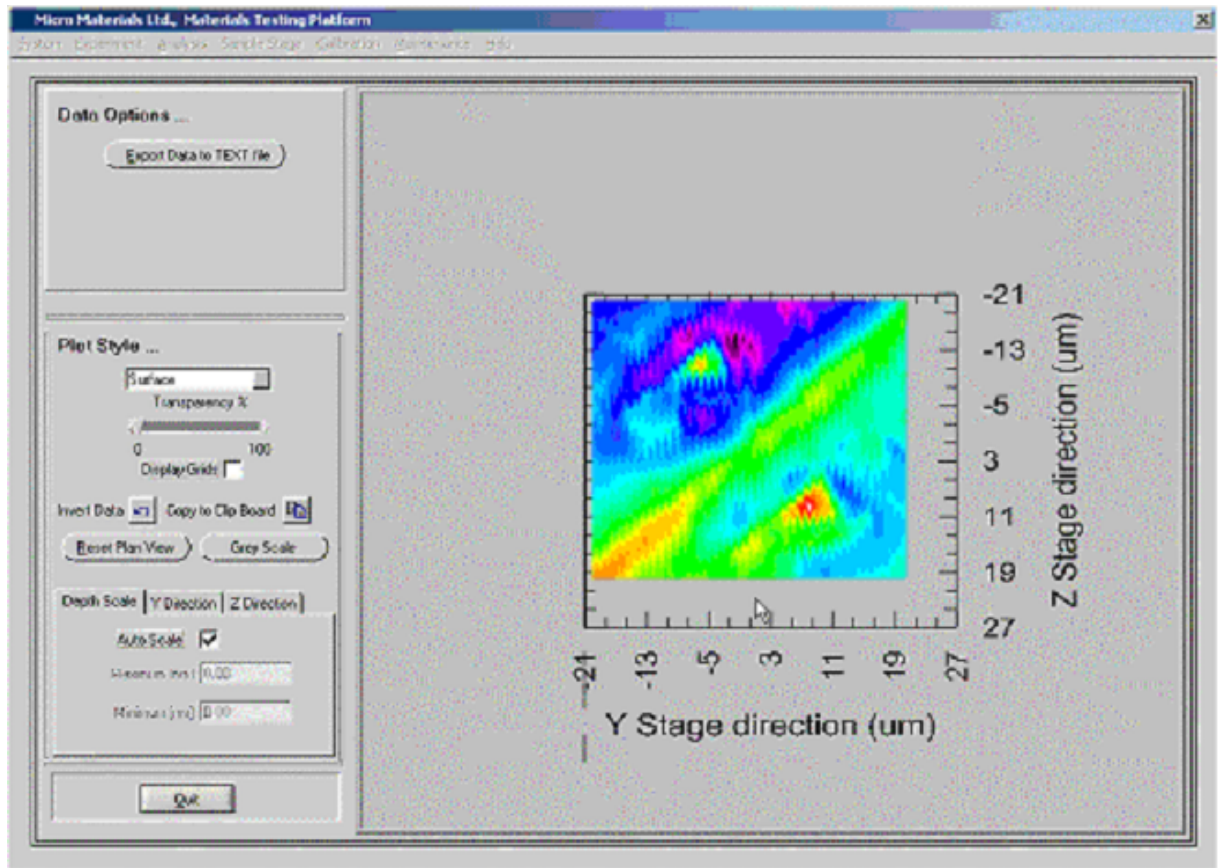
When the "Quit" control is clicked, as at step 5, you will be given the option to use this image to define further indentation locations if required. All images and indentation data can be reviewed and analysed using the appropriate review and analysis options from the main menu bar.

12 Technical Appendix

Data Analysis

Hardware Description

12.1.1 Data Analysis

Indentation Data Analysis - Pyramidal Analysis

From *Analysis* select *Indentation* then *Pyramidal Analysis*. The user specifies how the data is to be analysed (see options below) and the analysis is performed automatically in the software.

In a pyramidal indentation, the NanoTest measures the penetration depth of the

calibrated diamond indenter as a function of the applied load during a load-unload cycle. On unloading the elastic component of the displacement starts to recover producing a sloped rather than horizontal unloading curve. **It is from this slope that the elastic and plastic properties can be derived.**

For statistical purposes, it is good practice to quote mean values of hardness and reduced modulus from 5-10 indentation curves acquired under the same parameters.
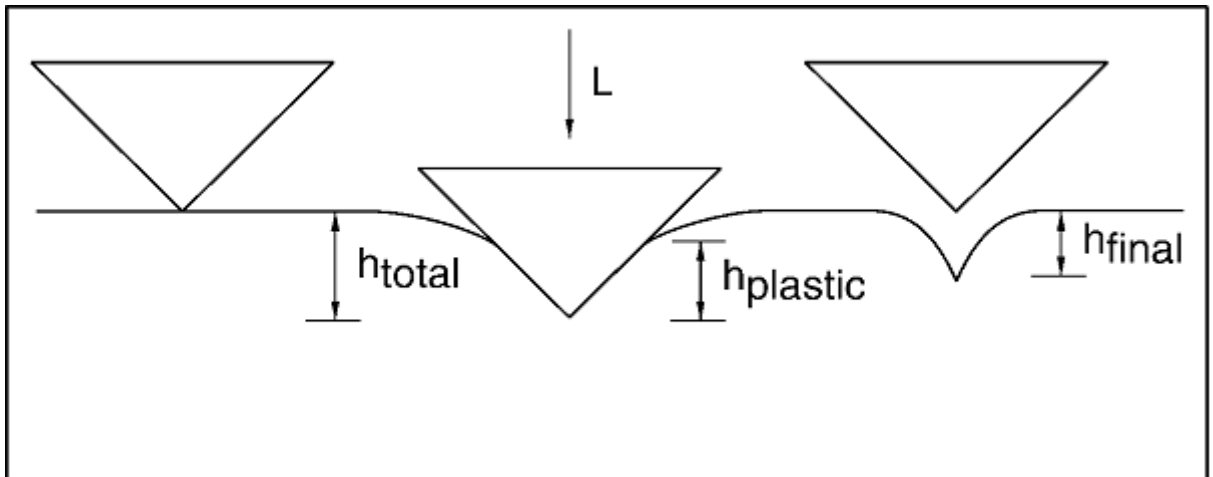
The NanoTest data analysis software produces values of h_{total} (h_{max}) and $h_{plastic}$ (h_c, contact depth) together with the derived hardness and reduced modulus.
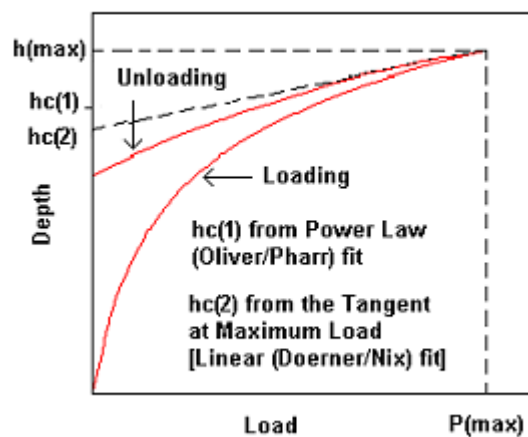
Plastic depth determination

In *Pyramidal Analysis*, the depth vs. load unloading data can be fitted to either

(1)   A **Linear fit** - a tangent to the unloading curve at the maximum load (Doerner and Nix fit).

(2)   A **power law** fit - (Oliver and Pharr fit) - this method is preferred and is described in detail below.

to determine the mechanical properties of the test sample.

Power-law fitting: Oliver and Pharr method
The power law function has the form

$$P = a(h - h_f)^m$$

where **a**, **h$_c$** and **m** are constants. The plastic depth is determined from the expression:

$$h_c = h_{max} - \varepsilon(C\ P_{max})$$

where **C** is the contact compliance equal to the tangent at maximum load. The value of ε depends on the indenter geometry. For a Berkovich indenter ε is 0.75. The diamond area function **A(h$_c$)** has been previously determined, according to the procedure described in section 7.5.

The hardness (**H**) is determined from the peak load (**P$_{max}$**) and the projected area of contact, **A**:

$$H = P_{max}/A$$

To obtain the elastic modulus, the unloading portion of the depth-load curve is analysed according to a relation which depends on the contact area:

$$C = \pi^{0.5}/(2E_r\ A^{0.5})$$

where **C** is the contact compliance and **E$_r$** is the reduced modulus defined by

$$1/\ E_r = (1 - \nu_s^2)/\ E_s + (1 - \nu_i^2)/\ E_i$$

where ν_s = Poisson's ratio for the sample, ν_i = Poisson's ratio for the indenter (0.07), **E$_s$** = Young's modulus for the sample and **E$_i$** = Young's modulus for the indenter (1141 GPa).

For further details consult Oliver and Pharr's original paper (*J. Mat. Res. Vol. 7, (1992) pp1564-1583*).

12.2.1 Hardware description
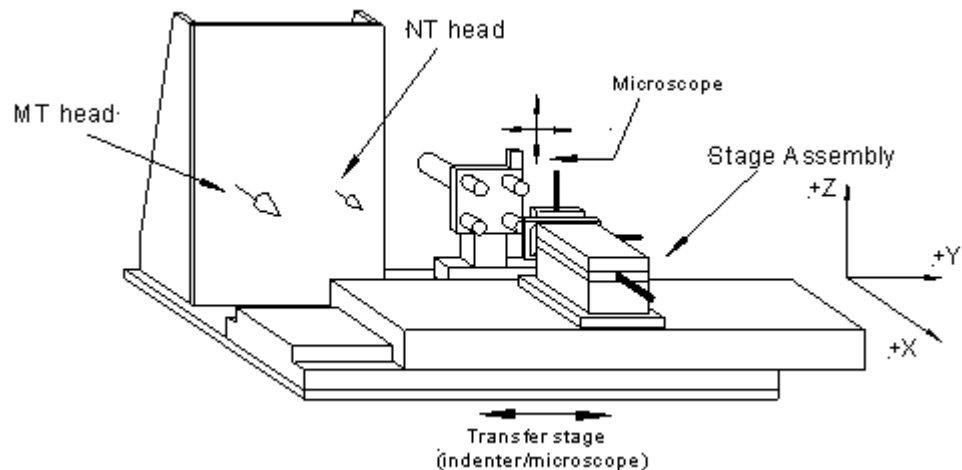
A sketch showing the position of the NT head (Nano) and MT head (Micro) on the instrument (shown here fitted with the larger base-plate for the high resolution microscope).

Stages and Motors

The motors used for manipulation of the sample are precision DC type using a magnetic encoder and high gearing to achieve a resolution per encoder count of just 17.3 nm. These are coupled with precision stages. These are arranged to control the sample in the X, Y and Z planes, with X moving the sample towards and away from the diamond or microscope objective, Y moving the sample across and Z moving the sample vertically.

The Pendulum

The pendulum is designed to be light-weight but stiff enough for the maximum (500mN) load that can be applied. The shaft is a solid length of cylindrical ceramic. Loads are applied during measurement when ramping the current in the coil causes its attraction towards a permanent magnet. The pivot is a friction-free design comprising four stainless steel leaf springs clamped in an X pattern. This is very compliant in rotation, but is easily damaged if care is not taken when handling the pendulum, i.e. when diamonds are being changed and when the occasional collision occurs between sample and diamond.

The depth measurement transducer is located just below the pivot and comprises two parallel circular discs, which are about 0.3 to 0.5mm apart when the system is at full sensitivity. The maximum measurement depth of the system when it is set at full sensitivity is ~1.5 μm. Directly in front of the plates is the diamond holder, situated so that the plate moves by the same amount as the diamond. The diamond holder and the moving capacitor plate are earthed.

Below the plates there is a counter-balance weight, necessary to counter the mass of the coil and any probe that is inserted into the diamond holder. Under proper balance conditions the pendulum, with no voltage across the coil, has the tendency to gently fall clockwise. Initial load calibration provides just enough constant coil current to bring the pendulum to a vertical, mechanical equilibrium position against the limit stop.

The Electronics

- Control unit, a multi-function *Analogue-Digital (A/D) board* acting as the interface between the instrument and the computer. The computer has an GPIB IEEE board, converting the analogue information coming from the machine.

- Capacitance Bridge Unit, the bridge unit contains a 5 KHz sine wave generator. The 5 KHz signal is applied across a 4-element AC bridge. Three elements of the circuit are adjustable by means of the three bridge unit controls. The fourth element in the circuit is the NanoTest capacitor. By tuning the bridge components so that the bridge is close to its balance condition, it is possible to look at the very smallest change in signal due to a change in the capacitance of the NanoTest capacitor. This will correspond to very small changes in depth when the machine is being used.

  The signal that comes from the moving plates goes into the unit through a short, gold coaxial lead. This contributes a large part of the capacitance being 'tuned' by the other bridge components and it is very important that it is firmly connected for the best results.

- Motor-box: there is also an additional unit, which houses the motor control boards and the anti-backlash system.

13 Micro Test high load head

The operation of the MT loading head is very similar in principle to the low load head (NT) and the pendulum has to be calibrated for load and depth like the NanoTest. Similarly, a compliance calibration is necessary - although not with fused silica, as the large forces required will 'crack' the surface. For this calibration a steel sample is provided.

Switching between NT and MT

The sample stage assembly is transferred between the two loading heads and the microscope by means of an independent linear stage. Selecting the relevant position (NT, MT or Microscope) is controlled by the *transverse stage control unit*.

IMPORTANT: Before any translation can occur the sample must be retracted beyond all three planes to avoid any lateral contact, as failure to do so may result in damage to the instrument (see diagram).

Retract the sample until is reaches a point where sufficient visual clearance is present (usually the X motor will read ~ 40-45mm)

The unit has two switches *indenter/microscope* and *low/high head*. To select a

loading head, choose either *low* or *high* and initiate the linear stage by selecting *microscope* until the micro-switch has been triggered (this is audible) and then *indenter* position can be selected.
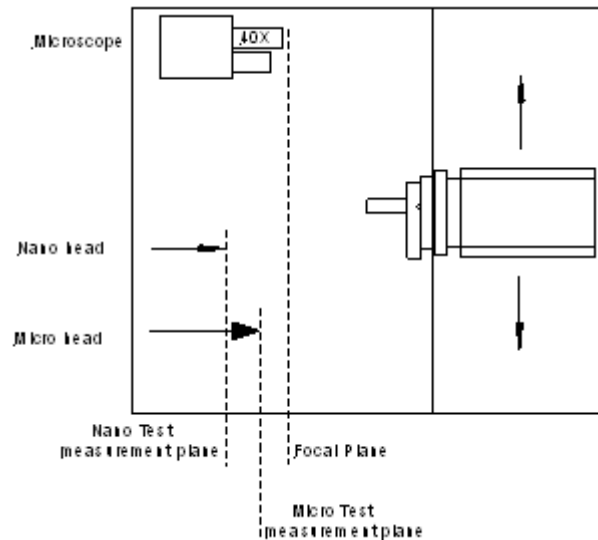
Using the MT head:

1. Select **New** from the *System* menu and then select the pendulum type (*MicroTest*). The software will now only allow for MT use (NT calibrations saved). To revert to the NT head simply repeat the procedure selecting *NanoTest* for the pendulum type. Follow the on-screen instruction by selecting the MT configuration on the *Electronic control system*.
2. Make sure the bridge box is set-up correctly (the system is on 'standby' at 4V, *unlike 8V for NT*)
3. If the microscope is *enabled*, the *sample stage calibration* must be valid and the sample placed in the focal plane (see microscope calibration for further detail). If the microscope is not being used, set up a nominal 20μm displacement between the indenter and the sample.

The operating procedure is now identical to the NT head:-

Calibration

Load: as NT head (although with a different mass range, 10-100g)

Depth: as NT head

Compliance: Using provided steel sample (and schedule), indentation carried out over 1-20N load range.

Note: we recommend performing compliance calculations at the loads which you will be using in the testing since in view of the large depths involved, results can be VERY sensitive to the exact value of the frame compliance which typically varies very slightly (1-2%) over the range 1-20 N.

The DAF is not required, as at the indentation depth produced at these higher forces the tip correction is insignificant and for all practical measurements the ideal function will be satisfactory.

Hardness Scale Conversion

MicroTest data can be compared to microhardness data from conventional microhardness testers. However, note that conversion between the different hardness scales is not exact since the definition of hardness varies with the type of test used. It is a system property not an intrinsic material property like modulus. Also note that for many applications involving thick coatings, the modulus is a more important measure of coating quality and performance than the hardness.

The Vickers hardness number HV (or H_V) is the test force divided by the sloping (i.e. surface) area of the indenter up to a depth where it has the same cross-sectional area as the residual indent:-

$$H_V = \text{constant x test force/surface area of indentation.}$$

In contrast, indentation hardness, H_{IT}, is defined as the test force divided by the projected area of contact:-

$$H_{IT} = F/A(h_c)$$

The projected contact area A(hc) is calculated from knowledge of the geometry of the indenter and stiffness of the contact

From ISO/DIS 14577-1.2 (draft indentation standard):-

The ratio of surface area (A_s) to projected area (A_p) is a constant for a perfect Vickers or modified Berkovich indenter:-

$$A_s/A_p = 26.43/24.5 = 1.08$$

The diagonal length measured in conventional Vickers tests is related to Ap by:-

$$d^2 = 2 A_p$$

which leads to:-

$$H_V = 0.0945 \text{ x } H_{IT} \text{ (in MPa)}$$

BUT, it is also necessary to check units. The above equation converts a Vickers hardness number **to a H_{IT} value in MPa** (as kg/mm^2 = 10^6 N/m).

(note: it is common practice for HV to be quoted in MPa and HIT in GPa – so the above equation becomes:-

$$\mathbf{H_V = 94.5 \text{ x } H_{IT} \text{ (in GPa)}}$$

Example:- HV 700 is approximately equivalent to H_{IT} of 7.5 GPa)

Note that this correlation does not allow for **tip blunting at small depths** and the two scales *diverge* as the indentation contact becomes more elastic.

14 Appendices

14.1 Spring Change Procedure - Low Load Pendulum

Troubleshooting: Bent Springs

Bent springs are usually caused by lack of sufficient care in operating the instrument and will lead to poor performance and unreliable data.

If you suspect that the springs may be damaged, there are a couple of checks that can be made:

1) The Bridge Box adjustment (see 4.2.1) is likely to be far off-scale.
2) The Pendulum may have moved to a different position, leading to a large change from the previous Depth calibration.

If the springs are damaged, replacements can be obtained from Micro Materials. The spring change procedure is illustrated below:-

1.Move sample out of way

2.Turn off all electronics (NOT THE CABINET HEATER)

5.Note which way round the wires are then de-solder from vero board

3.Back limit stop away

6.Remove hinge bolts

4.Remove Indenter

7.Remove pendulum and CLOSE THE CABINET DOORS
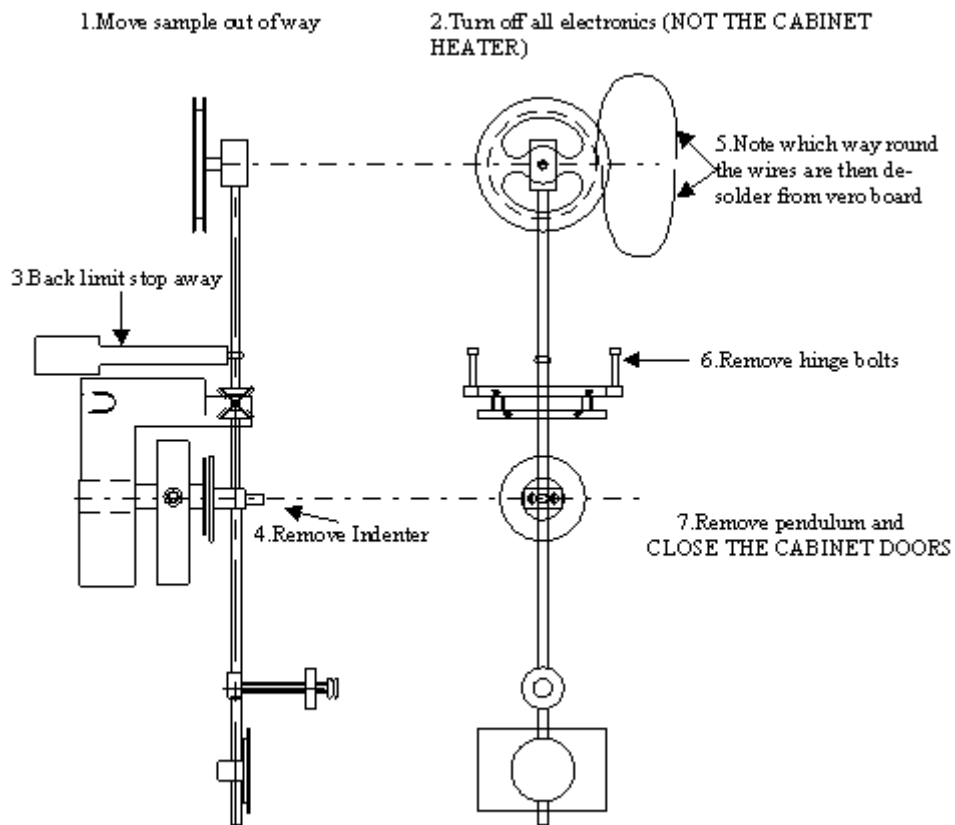
8.Loosen the 4 x 12BA brass screws on the front of the hinge, remove the screws and plates from the back of the hinge.

9.Remove all the springs. Keep any good ones

11.With the top and bottom hinge plates on a flat surface to keep them orthogonal, tighten the bolts

10.Insert the spacers and the bolts
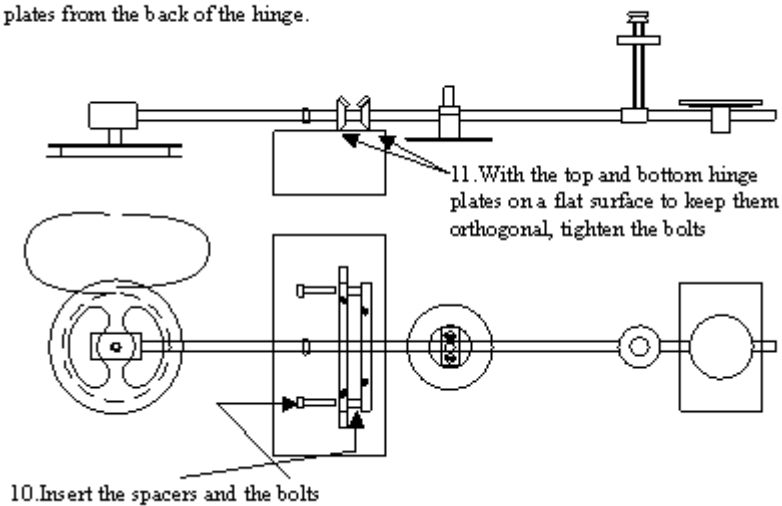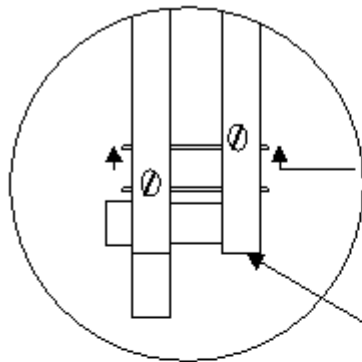
12.Replace the plates and the screws (loose).

13.Slide the springs between the retaining plates and the hinge plates then sideways until they touch the screws. Tighten the screws.

14.In turn loosen the screws on each retaining plate and align the ends to the pendulum hinge plate.
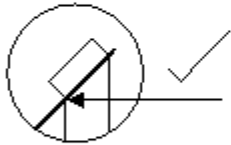
15.Ensure spring is pinched between the retaining plate and the hinge plates at one point.
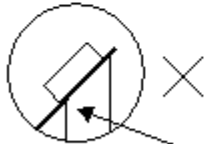
DO NOT have the retaining plate overlapping the hinge plate or the hinge plate overlapping the retaining plate
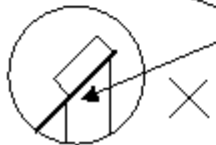
16. Check all the screws are tight.

17. Remove bolts.

18. Remove spacers. (From now on be careful as the springs can easily be bent.)
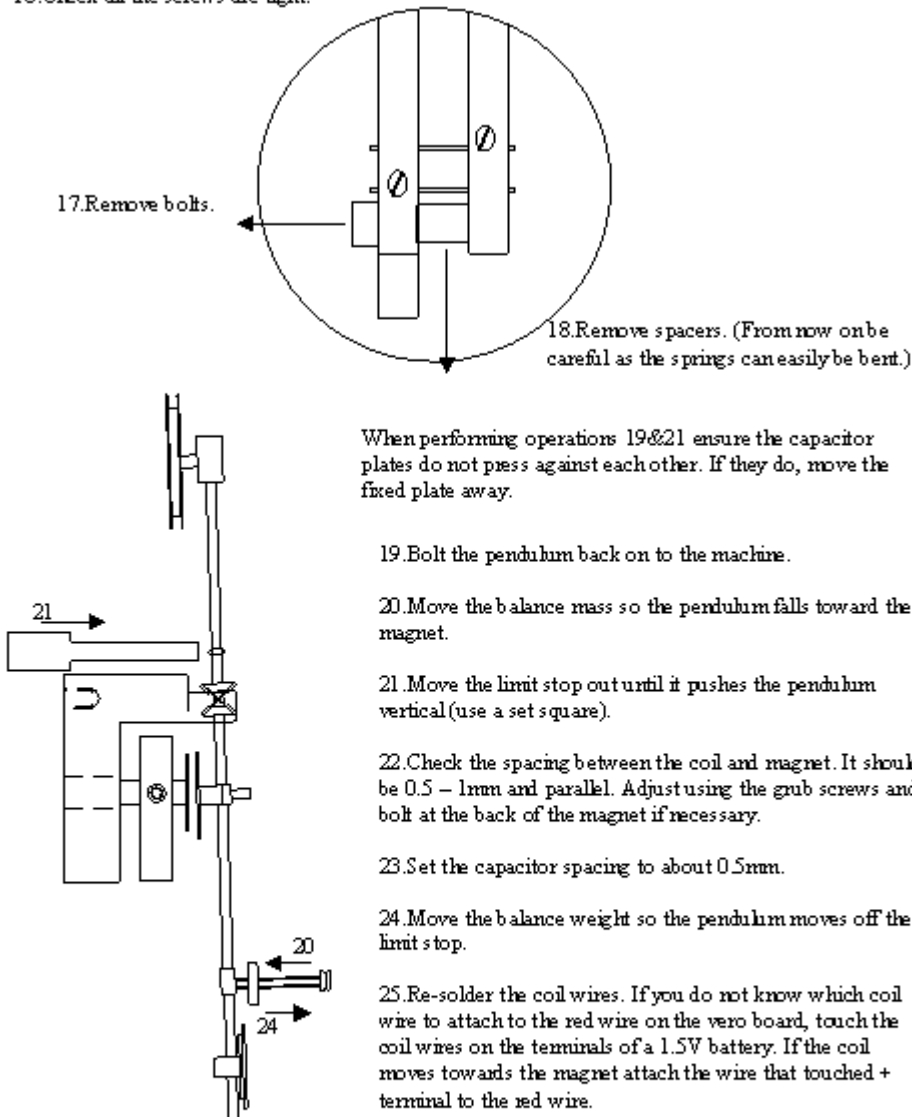
When performing operations 19&21 ensure the capacitor plates do not press against each other. If they do, move the fixed plate away.

19. Bolt the pendulum back on to the machine.

20. Move the balance mass so the pendulum falls toward the magnet.

21. Move the limit stop out until it pushes the pendulum vertical (use a set square).

22. Check the spacing between the coil and magnet. It should be 0.5 – 1mm and parallel. Adjust using the grub screws and bolt at the back of the magnet if necessary.

23. Set the capacitor spacing to about 0.5mm.

24. Move the balance weight so the pendulum moves off the limit stop.

25. Re-solder the coil wires. If you do not know which coil wire to attach to the red wire on the vero board, touch the coil wires on the terminals of a 1.5V battery. If the coil moves towards the magnet attach the wire that touched + terminal to the red wire.

26. Turn on electronics. Establish the capacitor spacing through the usual method. Perform a load calibration then a depth calibration.

Note: If fitting a replacement pendulum then always check the coil wire orientation with a battery as described in step 25

14.2 Vacuum Wafer Holder

Wafers below a thickness of 0.8 mm can exhibit stress-related non-uniformity in flatness created by the processes involved in forming them. In order to reliably measure hardness or carry out scanning experiments on thin wafers, it is necessary

to ensure the wafer is held firmly and to remove the air gap between the wafer and holder.

<u>How to use the Vacuum Wafer Holder</u>

14.2.1 Procedure for using the Vacuum Wafer Holder

1. Thoroughly clean (with isopropyl alcohol and clean gas) the back surface of the wafer and the top surface of the wafer holder assembly. Any dust or debris may stop full contact.
2. Place the wafer onto the ring's gasket and then place the wafer plate on to the ring, lining up the clamping holes as this is done.
3. Carefully turn the assembly over and clamp the ring onto the plate with the twelve screws provided.
4. Gradually clamp the assembly fully by turning each screw, in turn, bit by bit until the ring is in contact with the plate.
5. Attach the wafer plate to the sample holder plate.
6. Connect up the pump and reservoir (with the tap at the pump end) to the holder and switch on the pump.
7. Install the holder on the machine, taking care not to contact the diamond or the brass assembly at the bottom of the pendulum.

NOTE: Measurements should be made with the securing stub directly behind the area of interest.

14.3 NanoTest Health and Safety

Please be aware of the following points:

1.   **Mains Supply**
      The control unit is powered by a mains electricity supply. The power cord should always be disconnected before the control unit is opened.

2.   **Non User Serviceable Components**
      The control unit does not contain user serviceable components. You should always contact Micro Materials before working on the unit.

3.   **Ventilation Holes**
      In order to prevent overheating, the control units are fitted with ventilation holes. You should ensure that these holes are always free of obstruction and you should not insert any objects into them.

4.   **Earthing**
      The cases of all control units should be connected to the building earth (ground).

5.   **Cabinet Heaters**
      If mains powered cabinet heaters are used, you should check the cable insulation (particularly where it enters the heater) from time-to-time for any signs of ageing or deterioration. The temperature controller should be disconnected from the mains prior to inspection.

6.   **Electromagnetic Environment**
      The use of the apparatus outside of the laboratory invalidates conformity with the protection requirements of the Electromagnetic Compatibility Directive (89/336/EEC).

IMPORTANT: The contents of this notice should be bought to the attention of all users of the equipment.

14.4 Bridge Box adjustment - more on trouble-shooting

Examples of good (Plot 1) and bad (Plots 2-14) Pendulum Tests

PLOT 1- **an ideal result with a minimum value stated on the screen of less than 1%. (Values are often below 0.5 %.)**

The pendulum balance is adjusted so that the signal does not come onto scale for about 1/3 of the first screen. This allows for any difference in the electronic zero between low and high range that may be fractions of a mV. The final screen starts when the signal has fallen by 5% of that original level.

PLOT 2 - **a Bridge Box adjustment problem**. It is possible to obtain the 7.9 Volt reference level but be on the wrong side of the minimum value set. To correct this, turn the lowest knob on the Bridge Box in the direction that causes the signal to minimise. Move the signal through minimum and keep turning in the same direction until the signal again reaches the reference value and try the pendulum test again.
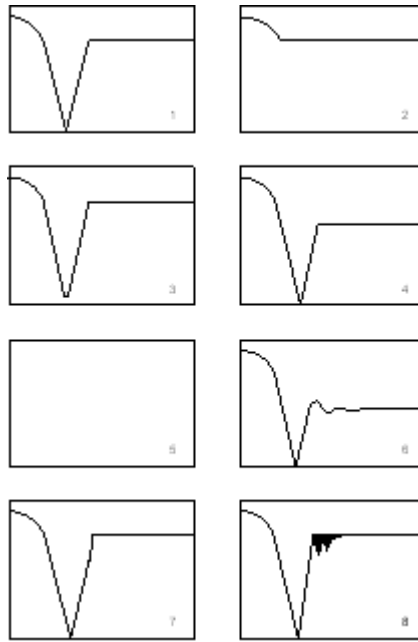
PLOT 3 - **above the 1% minimum value. To set the minimum value, a combination of tuning with all three knobs must be done, with the fine tuning for minimum achieved using the more sensitive resistor value (middle).** If, after tuning carefully and the minimum is still above the 1% acceptance level in the pendulum test, a very slight turn of the middle knob in the clockwise direction (to ~0.2V) may reduce the minimum further.

PLOT 4 - **the final constant value is too low**. The time adjustment of the 7.9V $\pm$ 1 value requires only use of the bottom control on the bridge unit.

Other possible causes of the change in amplitude are:-
(1) if the temperature of the cabinet is changing and affecting the thermal stability of the system.
(2) the pivot springs may be damaged and the change is brought about by the pendulum moving to a new position when different loads are applied i.e. the difference between the large load applied at the start of the Bridge Box adjustment program and the value that is applied after the first Pendulum Test (although it is important to note that even with a perfect pivot, a change in signal of ~0.01 V can be expected; larger deviations would need further investigation).

PLOT 5 - **the signal remains out of range during the pendulum test**. The mass of the pendulum countering the force applied by the coil may be very large (perhaps because of the use of a massive probe), so the counter-weight at the bottom of the pendulum may need screwing closer to the pendulum's centre. If this is not the cause of the problem it may be possible that the Depth in lead was not reconnected before attempting the pendulum test.
PLOT 6 - **the signal doesn't reach the 7.9V level**. A possible problem with the adjustment of the balance weight. The counter-weight may need screwing towards the centre.
PLOTS 7 and 8 - **acceleration to the limit stop and contact noise**. Both plots could be due to electro-static effects causing a build-up of charge on the limit stop. Plot 7 would then be the result of charge difference between the two metal surfaces resulting in attraction between them, leading to the pendulum accelerating as the two surfaces get closer. Plot 8 would be the result of repulsive forces between the two metal surfaces, causing them to bounce until the coil force is larger than the repulsive force. Another possible reason for plot 8 may be a lack of vibration isolation.
PLOTS 9 and 10 - **pendulum test result too wide or narrow**. Normally, this can be attributable to a capacitor plate spacing problem. Too wide, as in plot 9 and the depth measurement loses sensitivity, too narrow, as in plot 10 and not enough depth is available at maximum sensitivity. The typical plate spacing to set for the NanoTest is ~0.3 - 0.5 mm when the pendulum is on the limit-stop, which equates to a maximum available depth at full system sensitivity of ~1000 -1300nm. (Not having the spacing close to the expected setting may also have a knock-on effect when using other options.)

The capacitor spacing can be changed by adjusting the plate screws.
PLOT 11 - **disturbed signal while the pendulum is moving towards the limit stop**. A problem with the analogue signals coming from the A-D Board.
PLOT 12 - **hesitation in pendulum swing**. Not regarded as a problem, as the points at which the slope changes occur are random with each new pendulum test.
PLOT 13 - **rounding of the curve as contact is made**. This indicates a build-up of contamination on the surface of the limit-stop. Cleaning the contacts using paper soaked in alcohol followed immediately by the use an air spray to remove any liquid is all that is needed.

PLOT 14 - **movement of the pendulum obstructed well before limit-stop contact is made**. This could be caused by a number of possibilities:-

      a.   Coil leads touching the magnet or fouling around the coil.
      b.   Fibres caught between the coil and magnet.
      c.   Highly contaminated limit-stop.
      d.   Looseness of the pivot springs or the pivot not secured properly.
      e.   Limit-stop Ground lead fouling at some point.

PLOT 15 - **signal oscillating on start of the pendulum test**. Common reason for this type of plot is setting the counter-weight too close to the mechanical balance of the pendulum causing it to swing excessively as the coil voltage is applied. To cure this, **the counter-weight needs to be moved slightly further out.**
PLOT 16 - **signal remains at the reference level**. Again, a problem with the mechanical balance of the pendulum. Here, the pendulum stays on the limit-stop whether or not a coil current is applied, suggesting **the counter-weight is having no effect and so needs to be moved a lot further out.**
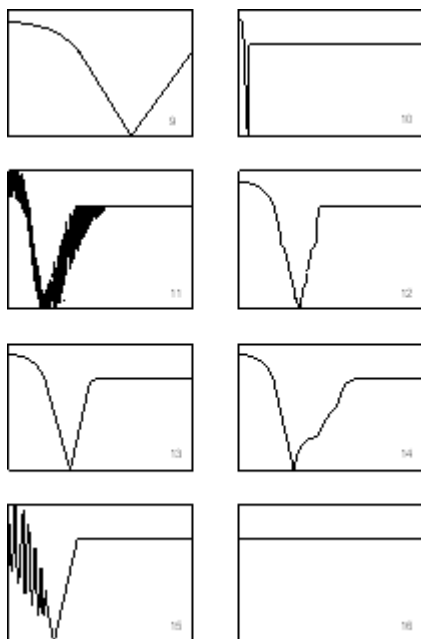
14.5 More on troubleshooting

[Cleaning the Capacitor Plates](#)

Cleaning the limit-stop

14.5.1 Cleaning the capacitor plates

When the capacitor plates are dirty they become "sticky" and tend to stick together affecting the performance of the instrument. To clean the capacitor plates, wet a piece of paper with isopropyl alcohol and use this to carefully and gently clean between the plates, then dry with an air blower.

14.5.2 Cleaning the limit-stop

Over time a build up of contamination on the limit stop can occur which can easily be removed by the same cleaning procedure. (also see the text for plot 13 in section 14.4 above.)

15 Glossary

Berkovich Diamond Three faced pyramidal indenter having the same depth to area ratio as a Vickers indenter.

Bridge box In the NanoTest instruments the displacement of the indenter is measured using a parallel plate capacitor such that the variation in the capacitance of the capacitor is proportional to the displacement of the indenter. The Bridge Box is used to convert the capacitance variation into the amplitude variation of a 5KHz sinusoidal voltage signal. Since the capacitance variations are extremely small the Bridge Box is mounted on the instrument close to the pendulum.

Damping plate The pendulum movement prior to measurement can be minimised or 'damped' by means to two parallel surfaces which are microns apart which create an air damping system. This reduces initial contact damage.

Depth + Offset signal Voltage signal produced by the depth board and used by the system to measure the displacement of the indenter. It can be monitored via a BNC connection on the back of the control unit or viewed in the system using Signal Display.

Depth Board The depth board converts the small amplitude variations in the 5KHz sinusoidal voltage produced by the Bridge Box into a D.C. voltage, the Depth + Offset signal. The Depth + Offset signal is measured by the system and used to determine the displacement of the indenter. At intermediate stages in the conversion process other electrical signals are produce by the Depth Board, namely the 'A/C Output' which is an amplified version of the Bridge Box signal and the 'Rectified Output' which is a D.C. voltage with a magnitude equal to the R.M.S. value of the 'A/C Output'.

Diamond area function This is the function that relates the cross sectional area of a plane through a diamond to the distance of the plane from the tip. Knowledge of the Area Function of the indenter used to produce an indentation to a measured contact depth allows the contact area and thus the hardness of the material to be calculated.

Dwell time The period for which the indenter is held in contact with the surface with the maximum load applied. The Dwell time can be varied in order to provide information about time dependent properties like creep.

Elastic modulus Also known as the Young's modulus or stiffness. The ratio of stress to strain.

Focal Plane The plane parallel to the Y-Z plane of the sample holder in which the surface of the sample is in focus 'under' the highest magnification lens.

Hardness The resistance to permanent deformation. For the purposes of nanoindentation work it is defined as the maximum applied load divided by the projected plastic area of contact.

Initial load This is a finite load applied in addition to the force required to just bring the pendulum into contact with the limit stop.

Linear fitting Data analysis by the Doerner and Nix method by which a linear fit is applied to the initial portion of the unloading curve.

Load-Partial-Unload A technique developed at Micro Materials for determining the variation of hardness and modulus with depth from indentations to different load at the same position.

Machine Compliance calibration A calibration procedure which works out the machine (frame) compliance so that subsequent indentation data can be adjusted to take account of this effect.

Measurement plane This is the plane parallel to the Y-Z plane of the sample holder in which the surface of the sample is normally 50 μm away from the tip of the indenter.

Multi-Point Repetitive Impact Testing An experiment in which the sample is caused to oscillate with the result that the pendulum recoils from the surface. The sample is slowly moved so that the next impact is adjacent to the previous one. Extremely useful technique for inducing film adhesion failure. Micro Materials have applied for patents for the technique.

Oliver and Pharr Authors of a key paper describing a popular method for analysing indentation load - displacement data. *J.Mater. Res. Vol 7 (1992) 1564.*

Pendulum test determines the correct adjustment to the bridge box unit.

Power law fit A function of the form $P = a(h-h_o)^m$ is fitted to the loading curve to determine the depth offset h_o, and adjust the data accordingly.

Rectified output is the depth signal after coming from the Bridge unit, being amplified and filtered, then rectified from the 5KHz sine wave to a DC level that can be handled by the A/D Board.

Reduced modulus is a measure of the elastic modulus of the sample. It can be thought of as an intermediate parameter accounting for the non-rigid effect of diamond indenters in contact with other materials. It can be easily converted to the Young's modulus if the sample's Poisson's ratio is known accurately (for example, υ is between 0.3 and 0.4 for most polymers, υ is 0.17 for fused silica).

Retraction distance The distance which the pendulum is pulled back from the sample surface after completion of an indent. A short distance increases experimental time, however as the sample is also moved sideways on completion of the experiment care needs to be taken to ensure that the retraction distance exceeds the height of any surface features so as to ensure that the diamond does not make contact with the sample surface as the sample is moved to its new measurement position.

Continuous compliance The sample is oscillated (with a very small amplitude) using a piezoelectric element and signal generator, and the phase difference between the applied stress and the response of the diamond is monitored. The instantaneous contact compliance can be measured as the load is increased without the necessity of analysing unloading data. Hardness and modulus are determined from the contact compliance using standard methods. The technique can be used to determine the complex moduli (loss and storage moduli) of viscoelastic materials from measurements of their

contact compliance and the tangent of the phase angle.

Scanning mode NanoTesting term used to refer to the category of experiments in which the sample is moved whilst the indenter pendulum remains stationary. A constant or increasing force can be applied to the indenter. Scanning tests include scratch testing, topography, profilometry and friction measurement.

Single Point Repetitive Impact Testing An experiment in which the sample is caused to oscillate with the result that the pendulum recoils from the surface. The indenter makes contact repeatedly at the same point.

Strain The response (deformation) of the material to the applied stress.

Stress The effect of the applied load acting at a given point in the material. (stress=load/cross-sectional area)

Vickers Diamond Four faced 136° diamond pyramid used in conventional hardness testing. Not favoured for nanoindentation work due to the difficulty in machining four faces to a point. More commonly used for microindentation (MicroTest).

Zero load calibration This calibration determines the coil current required to produce zero load on contact, i.e. to place the pendulum in mechanical equilibrium just on the face of the limit stop.

16 Literature List

A full list of NanoTest application notes is available on request. The NanoTest has been used in scientific papers in a very wide range of application areas as shown in the following (by no means comprehensive!) list of published papers by NanoTest users:-

Categories:
- Testing Techniques
- General/theory
- Biomaterials
- Polymers
- Micro-electronics
- Particulates/ceramics
- Glass/silica
- Metals
- Tribology

- **Testing techniques**
Keywords: Scratch testing, micro impact-testing, high temperature measurements

Impact Testing – A repetitive contact technique
Micro Impact Testing: A new Technique for Investigating thin film toughness, Adhesion, erosive wear resistance and Dynamic hardness (B Beake, SR Goodes and JF Smith) - Surface Engineering 2001 187-193.

Micro Impact Testing Of Thin Films and Surfaces – JF Smith, F Riley & D Galusek
(Quality Testing, November 1998)

High Temperature Nanoscale Mechanical Property Measurements   0
JF Smith and S Zheng (Surface Engineering 2000)

Measurement Of the Mechanical Properties Of Multilayer Coatings by Nanoindentation
P. Hancock, M Fatima Vales Silva and JR Nicholls
Submiited to Mat. Sci. Tech.

Mechanical Properties of Multilayer Coatings by Nanoindentation
P. Hancock, M Fatima Vales Silva and JR Nicholls
15th Brazilian Congress of Mechanical Enginering, 22-26 Nov 1999, Soa Paulo, Brazil

- **General:**
Keywords: Finite element analysis, depth sensing indentation, modelling, hardness, load-displacement curves, compliance, calibration

An ultra-low penetration hardness tester, *D. Newey, M.A. Wilkins and H.M. Pollock,*
Journal of Physics E: Scientific Instrument, 15(1982), 119-122.

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Y. Sun, T. Bell and S. Zheng,
Thin Solid Films, 258(1995), 198-204.

Measurement of the single particle electrical clamping force
P. A. Arteaga, C.M.Martin, M. Ghadiri, U.Tuzun and B.Formisani,
Electrostatics 1995, University of York, 2-5 April 1995.

Surface mechanical property testing by depth sensing indentation.
 J.L. Meneve, J.F. Smith. N.M. Jennett and S.J. Saunders
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Three Dimensional Modelling of Indent Induced Plastic Zone at a Mesoscale
M.C. Fivel, C.F. Robertson, G.R.Canova and L.Boulanger
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Measurement of the single particle electrical clamping force
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Electrostatics 95, University of York, 2-5 April 1995

Prediction of nanoindentation hardness profile from a load-displacement curve
K.W.Xu, G.L.Hou, B.C.Hendirx, J.W.He, Y.Sun, S.Zheng, A.Bloyce, T.Bell
Journal of Materials Research, 13(1998)12, 3519-3526.

Indenter tip radius and load frame compliance calibration using nanoindentation loading Curves -
Y.Sun, S.Zheng, T.Bell and J.Smith Phil Mag Lett 79 1999 P649 - 658
ASM Handbook: "Nanoindentation",
H.M. Pollock, The Materials Information Society Handbook,
Volume 18 Friction, Lubrication and Wear Technology,1992.

Computational Modelling of the Forward and Reverse Problems in Instrumented Sharp Indentation
M. Dao, N. Chollacoop, K. J. Van Vliet, T. A. Venkatesh and S. Suresh
Acta. Materiallia 49, 3899-3918, 2001

- **Biomaterials**
Keywords: Ti6Al4V, Lactose

Polishing wear behaviour and surface hardness of ion-beam modified Ti6Al4V,
I.L. Singer, R.N. Bolster, H.M. Pollock and J.D.J. Ross,
Surface & Coatings Technology, 36(1988), 531-540.

Comparative study of mechanical behaviour of lactose from measurements on compacted beam specimens, controlled powder compression and single crystals.
P.G. Fagan, V.D. Harding, G.T. Nornan, P.A. Artega and M. Ghadiri
World Congress of Chemical Engineering, San Diego, CA, 14-18 July 1996.

- **Polymers**
Keywords: PET, polyimides, embrittlement, plasma deposition,

Micro-scale indentation of amorphous and drawn PET surfaces,
R.H. Ion, H.M. Pollock and C. Roques-Carmes,
Journal of Materials Science, 25(1990), 1444-1454.

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Nuclear Instruments & Methods in Physics Research, Section B -Beam Interactions
with Materials and Atoms, Vol.84, No.4, 1994, 484-490

Directional indentation of transcrystalline polypropylene
E.Amitay-Sadovsky, S. Zheng, J.Smith and H.D. Wagner
Acta Polymerica, 49(1998), 588-593.
Mechanical properties of plasma deposited polymer coatings
F. Benitez, E Martinez, M Galan, J Serrat, J Esteve
Surface & Coatings Technology, 125 (2000) p383

Near-surface mechanical properties of amorphous polymers
H. van Melick, A. van Dijken, J. den Toonder, L. Govaert, H. Meijer
Phil. Mag, A 2002 2093.

Craze initiation in glassy polymers: influence of strain rate, thermal history, and network density
 H.G.H. van Melick, O.F.J.T. Bressers, J.M.J. den Toonder, L.E. Govaert, H.E.H. Meijer:.
Polymer, submitted 2002.

- **Micro-electronics:**
Keywords: TiN, Si, Ge, Diamond Like Carbon (DLC), stress, silicon thin film

Effect Of Ion Bombardment on The Properties Of B_4C Thin Films Deposited by RF Sputtering
A Lousa, E Martinez, J Esteve and E Pascual
Thin Solid Films (1999) 210-213

Mechanical properties microprobing of TiN coatings produced by different techniques,
S. Zheng, Y. Sun, T. Bell and J. Smith,
Proc. 4th European Conference on Advanced Materials and Processes

Composition dependence of hardness and elastic modulus in Si-Ge measured by nanoindentation - possible consequences for elasto-plastic relaxation and diffusion
B. Roos, H. Richter and J. Wollweber,
Solid State Phenomena, Vols.47-48, 1996, 509 -516.
(EuroMat 95), Padua/Venice, Italy, 25-28 Sept. 1995, Symp. D&E, 177-184.

Impact of dopants and oxygen precipitation on silicon wafer hardness,
F.G. Kirscht, B. Roos, H. Richter, S.B. Kim and I. Suzuki,
Proceedings of the 2nd International Symposium on Advanced Science and
Technology of Silicon Materials, Hawaii, 25-29 November 1996.

Stress in Hydrogenated Microcrystalline Silicon Thin Films
D Piero, C Voz, J Bertomeu, J Andreu, E Martinez and J Esteve
Mat. Res. Sym. Proc. 557 (1999)

Difficulties in estimating the rheological properties of diamond-like films by means of nanoindentation tests - *J.C.Pivin*
Thin solid films, 229 (1993) 83-92

Mechanical Properties of Nanometric structures of Si/SiC and C/SiN produced by PECVD.
E. Bertran, E Martinez. G Viera, J Farjas and P Roura
11th European Conference on Diamond, Diamond-Like Materials, Carbon Nanotubes, Nitrides and Silicon Carbide

- **Particulates/ceramics**
Keywords: nanoparticles, cement,

Use of depth sensing indentation systems for assessment of characteristics of bond in glass fibre inforced cement',
FRC 99 - 2nd Asia pacific specialty conference on fibre reinforced concrete, Singapore, August 99, - *Trtik, P.; Bartos, P.J.M* pp 235-243; ISBN 981-04-1541-9

Development of the Tensioned Push out Test for Study of Fibre-Matrix Interfaces,
Kalton, A.F., Ward-Close, C.M. and Clyne, T.W.
Composites, vol 25, (1994) p.637 -644.

Surface Analysis Of Nanostructred Ceramic Coatings Containing Silicon Carbide Nanoparticles produced by plasma modulation chemical vapour deposition
E. Bertran, G Viera, E Martinez, J Esteve, Y Maniette, J Farjas, P Roura
Thin Solid Films 2000, 495-500

Use of nanoindentation to assess potential attrition of particulate solids,
P.A. Artega, M. Ghadiri, N.S. Lawson. and H.M. Pollock,
Tribology International, 26(1993)5, 305.

Multilayer coating techniques to optimise the properties of TiB_2 – based coatings
Maria Fatima Vales Silva, Peter Hancock and John R Nicholls
Advanced Engineering Materials 2000, 2, No 10, p1666-1676

Nanoindentation Behaviour Of Clay/Poly(ethylene oxide) Nanocomposites
BD Beake, S. Chen, JB Hull and F Gao
Journal of Nanoscience and Nanotechnology (2002) Vol 2 73-79

Micro-impact testing: a technique for investigating fracture toughness
BD Beake, Maria Jesus Ibanez Garcia, James F Smith
Thin Solid Films 398-399 (2001) p. 438-443

- **Glass/silica**

Effects of ion irradiations on properties of polyphosphazene -silica composite films,
J.C. Pivin, G. Brusatin, M. Guglielmi, G. Facchin and M. Gleria,
Nuclear Instruments & Methods in Physics Research, Section B - Beam Interactions with Materials and Atoms, Vol.112, No.1-4, 1996, 294-297.

Fracture toughness and adhesion energy of sol-gel coatings on glass
J. den Toonder, J. Malzbender, G. de With, and R. Balkenende
J. Mater. Res., 17, 163 224-233, 2002.

- **Metals**
Keywords: Iron, steel, alloy, nanocrystallisation, polycrystalline metal films

Characterisation of plasma nitrided and PVD-TiN duplex treated armco iron and En40B steel by nanoindentation, S. *Zheng, Y. Sun, A. Bloyce and T. Bell,*
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C. Robertson, S .Poissonnet and L. Boulanger
J.Materials Research, Vol 13, No 8, August 1998

Size Effects on the Mechanical Properties of Thin Polycrystalline Metal Films on Substrates
Y. Choi and S. Suresh
Acta Materiallia, submitted 2002

Nanocrystallization during nanoindentation of a bulk amorphous metal alloy at room temperature
J.J. Kim, Y. Choi, S. Suresh and A. Argon
Science 295 (5555): 654-657, 2002.

- **Tribology**
Keywords: Wear resistance, coatings, tungsten carbide

Measurement, testing and optimisation of wear resistant coatings: the NanoTest 550,
J.F. Smith, TriboTest Journal, 1-4, June 1995

Mechanical and Tribological Properties of Tungsten Carbide Sputtered Properties
J Esteve, C Zambrano, C Rincon, E Martinez, H Galindo & P.Prieto
Thin Solid Films 2000 282-286

If you would like to add a paper using the NanoTest to this list please contact MML at the address below or email help@micromaterials.co.uk and we will be happy to include it.

17 Contact Details

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