HYSITRON™

Probe Calibration

Performing a probe calibration specifically for each probe will compensate for non-perfect probe shape. For example, the probe radius of curvature and normal dulling of the probe can both be corrected for by re-calibrating the probe.

During a nanoindentation test, the nanoindentation probe is driven into a sample and then withdrawn by decreasing the applied force. The applied load [P] and depth of penetration [h] into the sample are continuously monitored. A load vs. displacement curve can be generated from the collected data. Figure 1 is an example of a load vs. displacement curve in which the load is increased at a constant rate to some peak value (loading), held at that value for a set amount of time and then decreased to zero (unloading) at the same rate as loading. The sample hardness [H] and reduced modulus [E_r] can then be calculated.
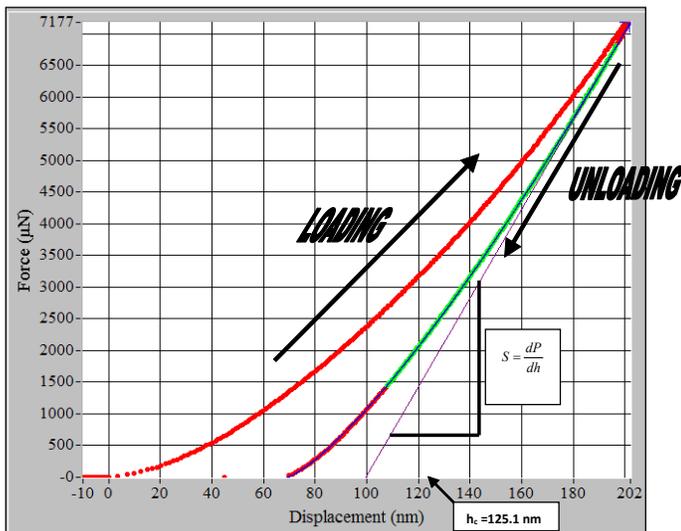

Figure 1: Force vs. Displacement curve for the standard fused quartz sample.

The contact area is determined from a probe area function [$A(h_c)$] where [h_c], the contact depth, is found with Equation 1.

Equation 1:
$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}$$

To account for edge effects, the deflection of the surface at the contact perimeter is estimated by taking the geometric constant ε as 0.75.

The cross-sectional area of an indentation (shown in Figure 2) illustrates the relationship of [P], [A], [h_c], and [h].
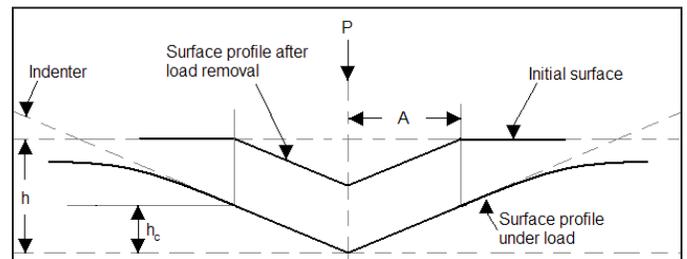

Figure 2: Schematic representation of an indentation showing quantities used for analysis.

The reduced modulus is related to the modulus of elasticity (E) through Equation 2:

Equation 2:
$$\frac{1}{E_r} = \left(\frac{1 - v^2}{E}\right)_{sample} + \left(\frac{1 - v^2}{E}\right)_{indenter}$$

For a standard diamond indentor probe, $E_{indenter}$ is 1140 GPa and $v_{indenter}$ is 0.07. Poisson's ratio varies between 0 and ½ for most materials.

The hardness has the normal definition:

Equation 3:
$$H = \frac{P_{max}}{A}$$

where [P_{max}] is the maximum indentation load and [A] is the resultant projected contact area at that load.

The reduced modulus is defined as Equation 4:

Equation 4: $\quad E_r = \dfrac{S\sqrt{\pi}}{2\sqrt{A}}$

Where [S] is the stiffness of the unloading curve and [A] is the projected contact area. The initial unloading contact stiffness (the slope of the initial portion of the unloading curve) is defined as Equation 5.

Equation 5: $\quad S = \dfrac{dP}{dh}$

Rearranging Equation 4 yields:

Equation 6: $\quad A = \dfrac{\pi}{4}\left(\dfrac{S}{E_r}\right)^2$

To determine the area function, a series of indents at various contact depths (normal loads) are performed in a sample of known elastic modulus (typically fused quartz) and the contact area [A] is calculated using Equation 6. A plot of the calculated area as a function of contact depth is created and the TriboScan software fits the [A] vs. [h_c] curve to a sixth order polynomial of the form:

Equation 7: $A = C_0 h^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_4 h_c^{1/16}$

C_0 for an ideal Berkovich probe is 24.5 while for an ideal cube corner (90°) probe is 2.598 with C_1 through C_5 set equal to zero. An ideal cono-spherical probes is defined to have C_0 set to $[-\pi]$, C_1 set to $[2 \times \pi \times r]$, where [r] is in nanometers with C_2 through C_5 set equal to zero. To better fit the shape of a real probe, C_1 through C_5 will vary depending on the exact shape of the probe.

Procedure for the Probe Calibration

To begin the probe calibration, install the probe that is to be calibrated and the standard fused quartz sample provided with the instrument. Several indents (at least 20 recommended) will be performed

in the standard sample. In order to provide a relevant probe calibration some elastic deformation is required during the indentation tests. This may not be achievable with very blunt (typically large radius cono-spherical probes) and an alternate material may be required for the probe calibration.

Setting up the Load Function

The load function used for the probe calibration should be an open loop (or load control if available) load function with a 5 second load time, 2 second hold time and a 5 second unload time (Figure 3). For a generic probe area calibration the maximum load should be close to the maximum allowable of the transducer (around 10,000 µN). The minimum load should be small enough so that the contact depth will be lower than the lowest contact depth desired on other samples (typically around 100-500 µN for a standard Berkovich nanoindentation probe). Save this load function so that it can be opened from the automated methods window.
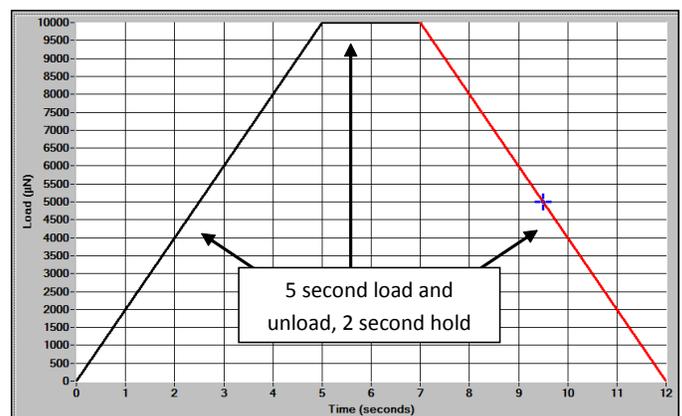

Figure 3: Open-loop load function for probe calibration.

Setting up the Method

Set up the instrument to run a method of indents on the fused quartz sample. A 10×10 array is recommended to increase the accuracy and reliability of the calibration. The method should cover the entire range of loads that the transducer is capable of, typically from about 10,000 µN down to around 100 µN. The exact loads used will depend on which type of probe is being used and where the user wants the calibration to be valid. Smaller loads and a smaller array of tests should be used for very sharp probes.

The peak load should be adjusted by percent while keeping segment times constant. These parameters are set by clicking on the *Load Function* button located on the *Automation* tab → *Methods* sub tab (Figure 4). By adjusting the peak load by percent the indentations will be more evenly distributed over the range of loads, which will yield a better probe area calibration.
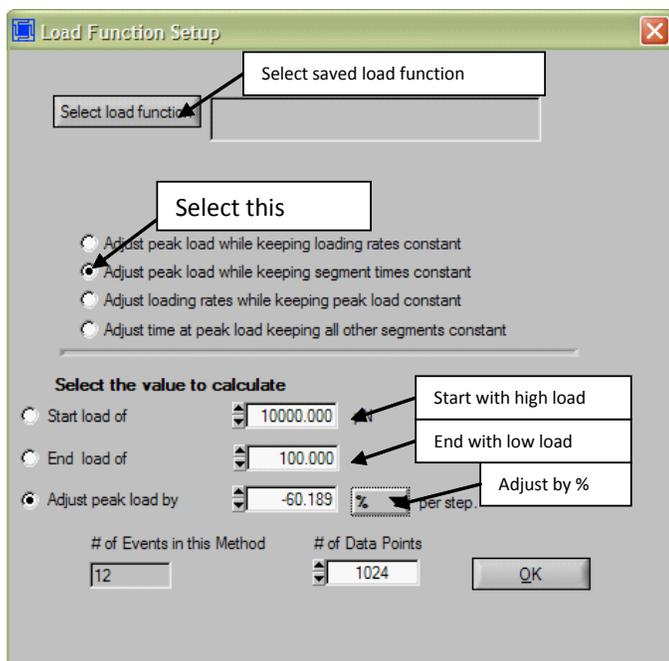

Figure 4: Methods load function setup window.

Setting up the Piezo Automation

For systems that are not equipped with the automated staging system, setting up a full method will not be an option. For these systems a piezo automation will be created for the probe calibration.

Create an *Array* piezo automation. The recommend amount of indentations is 25. To accomplish this, set the scan size to 30 µm and create an array that is 5×5 with a 5 µm spacing between each indent. Again, start with a high load (around 10,000 µN) and finish with a relatively low load (around 100-500 µN). The exact loads used will depend on which type of tip is being used and where the user wants the calibration to be valid. After the data has been saved the analysis in the following section is identical.

Using a TS 70/TS 75 TriboScope

For TriboScope systems that are not equipped with the Automated TriboScope feature, the nanoindentation tests required to perform the probe calibration must be performed individually.

Approximately 20-25 tests that cover a range of contact depths and loads is recommended. In general, more tests should be performed at lower peak normal force to better fit the lower displacements of the probe. The exact loads used will depend on which type of tip is being used and where the user wants the calibration to be valid. After the data has been saved the analysis in the following section is identical.

Data Analysis

After the indents have been completed click the *Analysis* tab → *Quasi* sub tab in the TriboScan software and click on the plot multiple curves icon (![icon]). From this window, click the *Add Curves* button (Figure 5) on the right hand side of the screen and select the curves from the probe calibration method. The resulting screen should look similar to Figure 5.
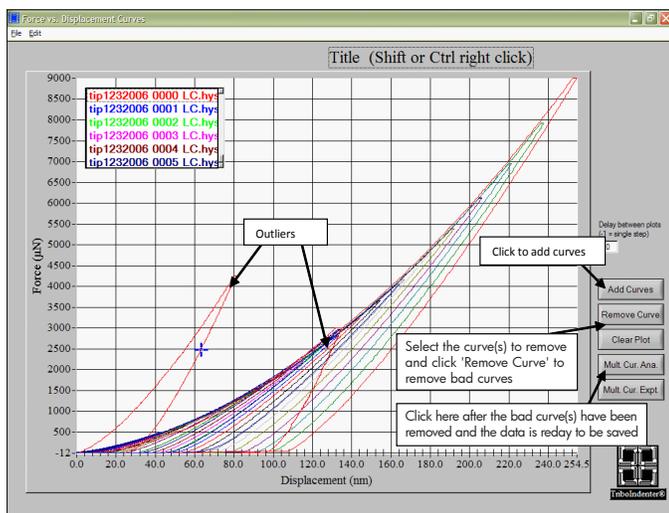

Figure 5: Plot of probe calibration curves.

Notice that in Figure 5 there are outlier curves that need to be removed before proceeding. A number of things including a dirty sample or a pre-existing scratch or indent on the sample could cause these outliers. If there are more than a few outlier curves from the method, check that the sample is clean and that it is the standard fused quartz sample provided with the instrument and then re-perform the indents.

After the outlier(s) have been removed from the data set click the *Mult. Cur. Ana.* button on the right hand side of the screen and save the text file. A screen showing hardness and modulus will pop up; close and disregard this screen. These values for hardness and modulus were calculated with a default probe

area function, not the function specific to the probe and are likely to be incorrect.

Calculate the Probe Area Function

Access the area function calculation window by clicking *Calibration* tab → *Tip Area Function* sub tab → *Calculate* sub tab. Select the *Open File* button near the top left of the window. If the instrument is equipped with nanoDMA, a window will prompt the user to choose between quasistatic or nanoDMA depending on which type of area function is currently being used. When prompted, select the text file saved from the multiple curve analysis (in the previous section) and click *Load*. When prompted, enter the reduced modulus for fused quartz, which is 69.6 GPa. The screen should look similar to Figure 6.
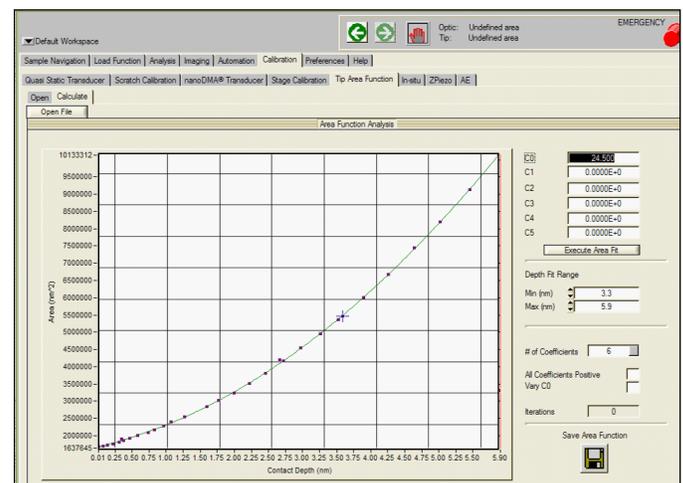

Figure 6: Area function analysis window.

Click *Execute Area Fit* and the software will fit a curved to the data. The fit should be a smooth parabolic shape with no inflections, similar to the fit shown in Figure 6. For a standard Berkovich probe the value of C_0 should be close to 24.5.

The user may choose to click on the plot and drag the red and blue lines to define an area to calibrate over if indentations will only be occurring at one

given depth. The user may also choose how many coefficients to use and if C_0 should vary or remain fixed.

Deep Indentations vs. Shallow Indentations

It is important to know when computing the area function whether the probe will be used primarily for deep indentations or shallow indentations.

When planning to perform deep indents, it is desirable to keep C_0 at 24.5 because this is the ideal Berkovich constant, which the probe will approach as it goes deeper into the sample. As [h_C] increases [C_1] through [C_5] become less important and Equation 8 reduces to Equation 9.

Equation 8: $$A = C_0 h_c^2 + \cancel{C_1 h_c} + \cancel{C_2 h_c^{1/2}} + \cancel{C_3 h_c^{1/4}} + \cancel{C_4 h_c^{1/8}} + \cancel{C_4 h_c^{1/16}}$$
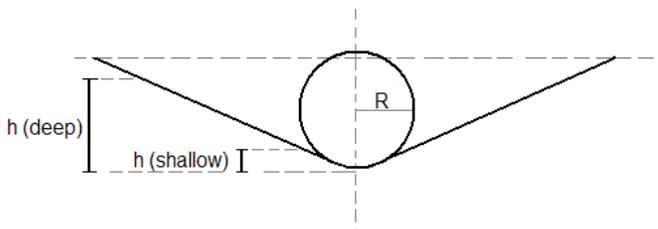
Equation 9: $$A = C_0 h_c^2$$

Figure 7: Probe radius effects with shallow indentations.

If planning to perform shallower indents, consider allowing C_0 to vary because at shallower depths the radius of curvature becomes the dominating effect in the area function. The probe will not go deep enough to approach the ideal Berkovich shape and the values in Equation 8 will not approach zero. Allowing C_0 to vary will help take the non-perfect probe shape into account at shallower depths. The value for C_0 should still be near 24.5 (or 2.598 for a cube corner probe) when C_0 is allowed to vary. If the value for C_0 is far ($\pm$ ~5) from the standard value (24.5 for a Berkovich or 2.598 for a cube corner probe) the probe may be damaged or the machine compliance value may be incorrectly set.

When finding the coefficients for the area function, it is also best to use as few coefficients as possible. When too many coefficients are used, the function will begin to fit to the noise in the data, and inflection points will develop. If the curve can fit well with only two coefficients, this is the best. However, if many data points are used, sometimes all 6 coefficients will need to be used to get a good area function. Typically, 3 or 4 coefficients works well.

Saving the Area Function

When the fit of the curve is satisfactory, click *Save Area Function* on the right hand side of the window in Figure 6 and the area function will automatically be applied to the system. To check that the area function was saved correctly (or load a previously defined area function), either select the *Calibration* tab → *Tip Area Function* sub tab → *Open* sub tab OR click define area function [🅰] from the *Analysis* tab → *Quasi* sub tab and the define area function window will open as shown in Figure 8.

The values for the constants on this screen should match the values for the constants calculated in the *Calibration* tab → *Tip Area Function* sub tab → *Calculate* sub tab (Figure 6). If the values do not match, click **File → Open** and select the file saved from Figure 6.

Checking the Probe Area Function

The probe is now calibrated and saved to the system. All of the indents can be analyzed again using the new probe area function. The resulting values for hardness and reduced modulus should be correct for
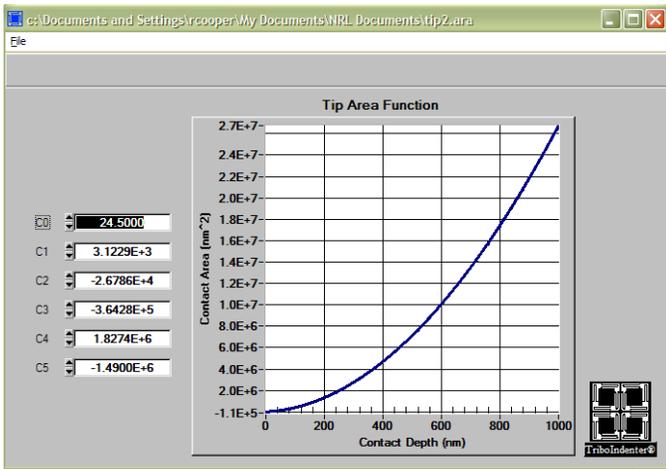
Figure 8: Define area function window.

fused quartz (hardness ≈9.25 GPa, reduced modulus ≈69.6 GPa). The hardness and reduced modulus vs. contact depth plot should now look similar to Figure 9. Notice that below around 40 nm the values for hardness become unreliable. This a normal effect related to the radius of curvature of the probe.

When the contact depth approaches the value R/3, with R being the radius of curvature of the probe, the hardness values become sporadic. The values for reduced modulus will stay relatively constant because the value was defined as 69.6 GPa when the area function was created. However, hardness is a computed value that is dependent on the contact area of the probe and is thusly related to the radius of curvature of the probe. Testing below the R/3 displacement value is not valid (illustrated with Figure 9 with a probe of radius of curvature of approximately 120 nm). To test in the displacement range less than this cutoff it is recommended to purchase a sharper probe with a smaller radius of curvature such as a cube corner or a NorthStar® probe available from Hysitron.

In fact, it is recommended that testing be performed only in the displacement range that the probe is calibrated for (from 40 nm up to 166 nm in this example). However, it can be assumed (given the probe has not been damaged) that the shape will continue as a Berkovich or cube corner beyond the calibrated depth. With that said, given that the correct value for C_0 has been entered, testing can be performed deeper than the calibrated depths assuming the probe will imitate its ideal shape at deeper depths.
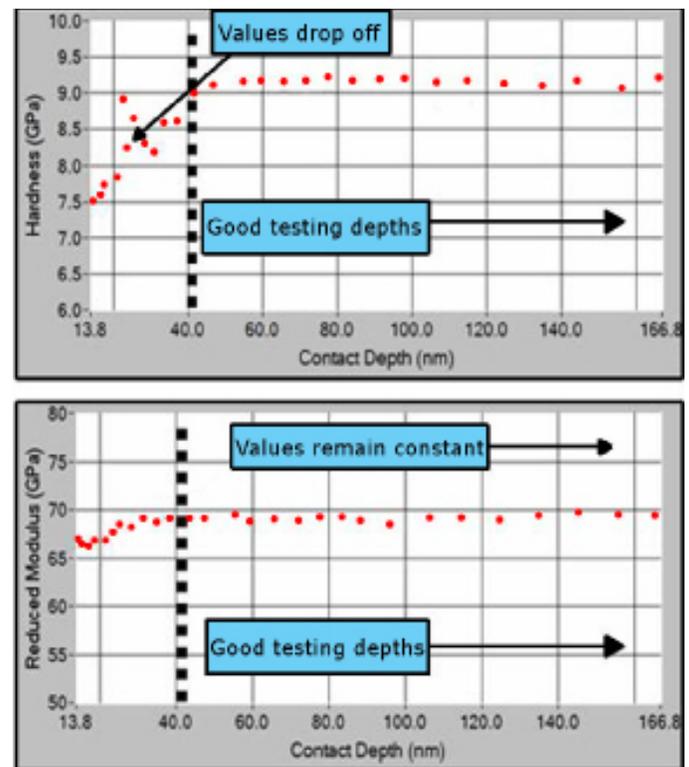

Figure 9: Hardness and reduced modulus plots for a probe with radius of curvature of approximately 120 nm.