Fine Calibration Procedures for the SPM— a User's Guide

This support note provides detailed instructions for the fine calibration of Digital Instruments SPMs. It is intended as a supplement to procedures already available in your product *Instruction Manual*, and assumes some prior user experience with NanoScope software and calibration procedures.

Operators should check the SPM's measuring accuracy periodically to ensure that images are dimensionally represented within acceptable limits of error. If measuring accuracy is critical, or if environmental factors (e.g., humidity, temperature) impact the SPM significantly, this may require a quick check at the start of each imaging session.

217.1   SPM Calibration Theory

Digital Instruments employs a software-guided calibration procedure for all its microscopes. The procedural particulars of how calibration is executed using NanoScope software are beyond the scope of this document and include proprietary methods exclusive to Digital Instruments. The theory, however, is straight forward and consists of the steps listed below.

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| Rev.	Date	Sections	Ref. DCR	Approval
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- Scan a calibration reference having surface features of precisely known dimensions.

- Compare known dimensions on the calibration reference's surface with those estimated by the SPM software.

- Adjust calibration parameters until the SPM's dimensions accord with the true dimensions of the reference.

The **Capture Calibration** and **Autocalibration** routines are designed to optimize measuring accuracy over the entire measuring range of the scanner. In order to obtain the finest accuracy possible for a given scan size, the scanner calibration parameters can be manually fine-tuned. This may prove useful in applications where measuring accuracy must be better than 1 percent.

217.1.1 Theory behind calibration

For purposes of calibration, Digital Instruments employs various derating and coupling parameters to model scanners' nonlinear characteristics. By precisely determining points along the scanner's sensitivity curve, then applying a rigorous mathematical model, full-range measuring capabilities can be made accurate to better than 1 percent.

Consider the sensitivity curve represented here:
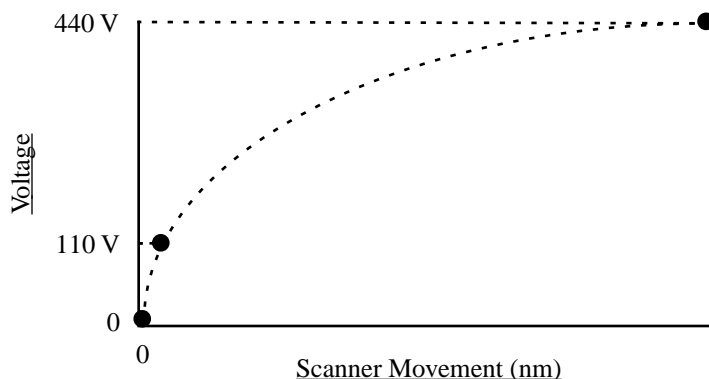
This curve typifies scanner sensitivity across the full range of movement. The vertical axis denotes voltage applied to the scanner. The horizontal axis denotes scanner movement. At higher voltages, the scanner's sensitivity increases (i.e., more movement per voltage applied). At zero volts, the scanner is "motionless." Plotting each point along the curve describes a second-order, exponential relationship which provides a rough approximation of scanner sensitivity.

Unfortunately, things are more complicated. Because piezo materials exhibit hysteresis, their response to increasing voltage is not the same as their response to decreasing voltage. That is, piezo materials exhibit "memory," which causes the scanner to behave differently as voltages recede toward zero. The graph below represents this relationship.
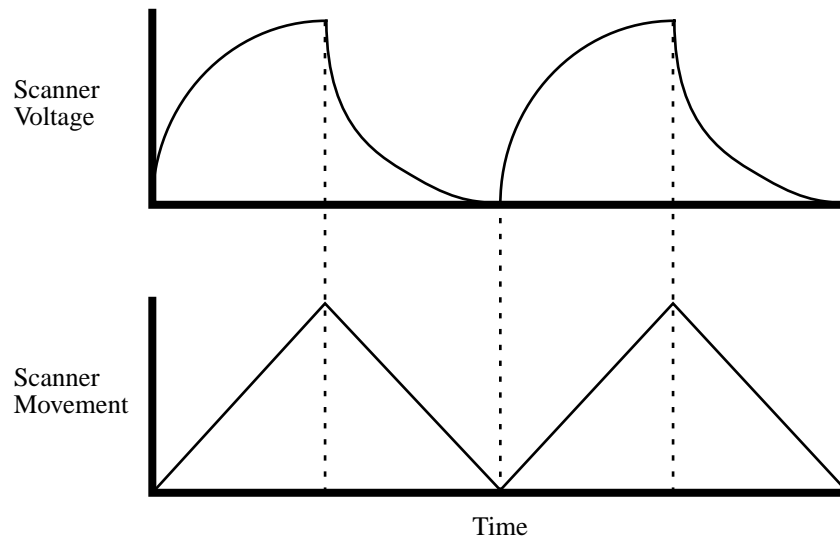
To produce the sharp, linear movements (triangular waveform) required for accurate back-and-forth scanning, it is necessary to shape the applied voltage as shown on the top graph above. Moreover, the applied voltage must compensate for scan rate and scan size. As scan rate slows, the applied voltage must compensate for increased memory effects in the piezo material. As scan size is decreased, the piezo exhibits more linearity. These effects are further complicated by X-Y-Z coupling effects (the tendency for one axis to affect movement in other axes).

Through rigorous quality control of its scanner piezos, Digital Instruments has achieved excellent modeling of scanner characteristics. Two calibration points are typically used for fine-tuning: at 150 and 440 volts. (A third point is assumed at 0 nm/volts.) These three points yield a second-order sensitivity curve to ensure accurate measurements throughout a broad range of scanner movements.

Because scanner sensitivities vary according to how much voltage is applied to them, the reference must be thoroughly scanned at a variety of sizes and angles. The user then tells the software the distance between known features on the reference's surface, and a parameter is recorded to compensate the scanner's movements. The X, Y and Z axes may be calibrated in any sequential order; however, the

linearization adjustments (Section 217-4) must be performed before any calibrations are attempted (otherwise, they will be undone by the linearity adjustments).
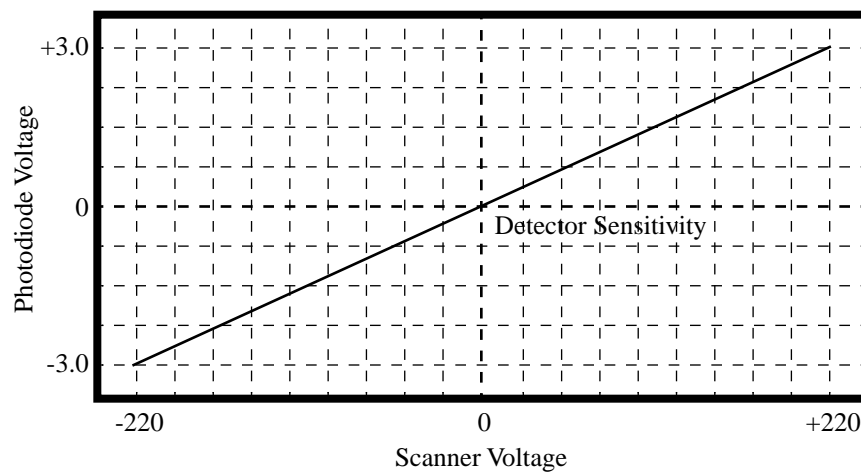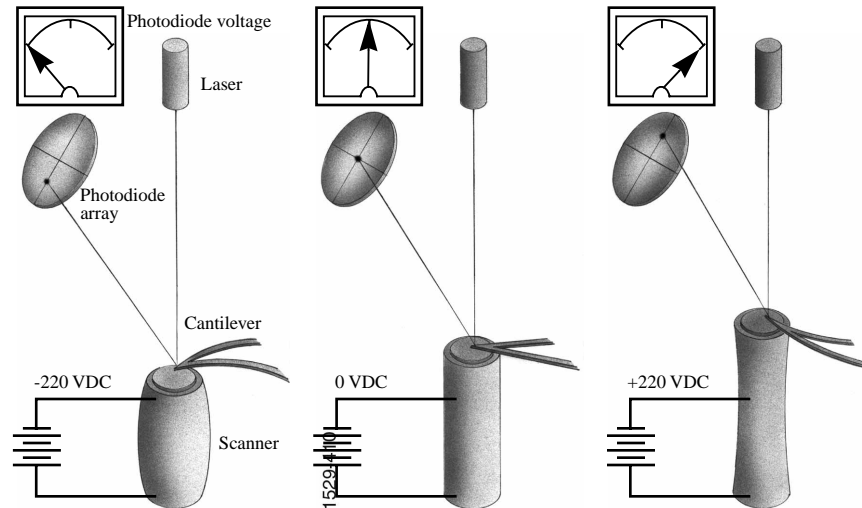
217.2 Sensitivity and Scanner Calibration —Background Information

Scanners serve as the "movers and shakers" of every scanning probe microscope. Typically, they consist of a hollow tube made of piezoelectric material such as PZT (lead zirconium titanate). Piezo materials contract and elongate when voltage is applied, according to whether the voltage is negative or positive, and depending upon the orientation of the material's polarized grain structure. Scanners are used to very precisely manipulate sample-tip movement in order to scan the sample surface. In some models (e.g., Small Sample MultiMode) the scanner tube moves the sample relative to a stationary tip. In other models (e.g., Dimension SPMs) the sample is stationary while the scanner moves the tip.

Not all scanners react exactly the same to a voltage. Because of slight variations in the orientation and size of the piezoelectric granular structure (polarity), material thickness, etc., each scanner has a unique "personality." This personality is conveniently measured in terms of *sensitivity*, a ratio of piezo voltage-to-piezo movement. Sensitivity is not a linear relationship, however. Because piezo scanners exhibit more sensitivity (i.e., more movement per volt) at higher voltages than they do at lower voltages, the sensitivity curve is just that—curved. This non-linear relationship is determined for each scanner crystal and follows it for the life of the scanner. As the scanner ages, its sensitivity will decrease somewhat, necessitating periodic recalibration.

The diagram below represents scanner crystal voltage versus photodiode voltage. In this instance, detector sensitivity is given as volt per volt, a parameter provided in the **Force Calibration** screen.

The **Microscope / Calibrate / Scanner** function displays the **Scanner Calibration** dialog box, allowing users to enter the sensitivity of their scanner's X-Y axes. Here, sensitivity is measured in terms of lateral displacement for a given voltage (nm/ volt). In addition, other parameters measure the scanner's X-Y coupling and other effects.

217.3 Calibration References

As described above, each scanner exhibits its own unique sensitivities; therefore, it is the task of the user and software to precisely measure these sensitivities, then establish software parameters for controlling the scanner. This task is accomplished with the use of a calibration reference.
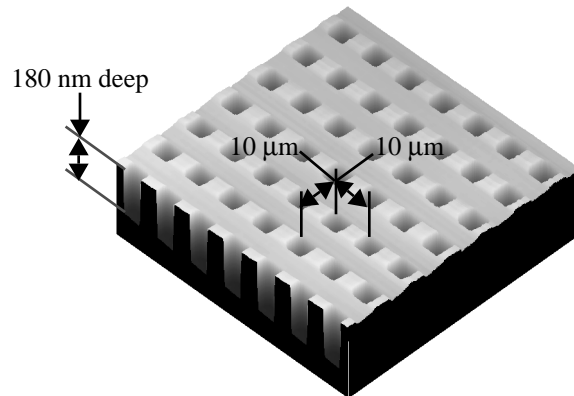
Figure 217-1   Section of Digital Instruments platinum-coated, silicon calibration reference.

One such calibration reference is shown in Figure 217-1 above. This calibration reference consists of a silicon substrate having a regular series of pits, each 180 nm deep, which is plated with platinum. Pits are spaced apart on 10 μm centers. Other, similar surfaces are available having different dimensions. Atomic-scale calibrations are generally carried out with mica or graphite, which exhibit very regular atomic lattices. Calibration references serve as the primary tool by which SPMs are calibrated. They serve as measuring sticks with which to gauge scanner displacement for a given voltage.

The SPM should be capable of measuring a calibration reference with an accuracy of 2 percent or better while scanning at the maximum **Scan size** setting. By using fine calibration techniques, it is possible to calibrate the SPM to much better than this.

217.4 Linearity Correction Procedure

For applications which demand good linearity, the following procedure can be used to optimize the linearity correction parameters for individual scanners. As discussed previously, linearity correction is especially important for long-range scanners (such as the "J" and "K").

217.4.1 Check scanner parameter values

If the system's original scanner parameters have been deleted, the scanner parameters can be copied from the software backup disk which is sent with every system. Individually purchased scanners are shipped with a head/scanner disk which contains the backup files, or a hard copy of the scanner parameters. In the event that files are not found, fax or call Digital Instruments for scanner calibration records.

217.4.2 Align calibration reference

Load the silicon calibration reference into the SPM. The reference will need to be aligned with the microscope scanner so that the tip scans parallel to the reference's features with the **Scan angle** set at **0** degrees.
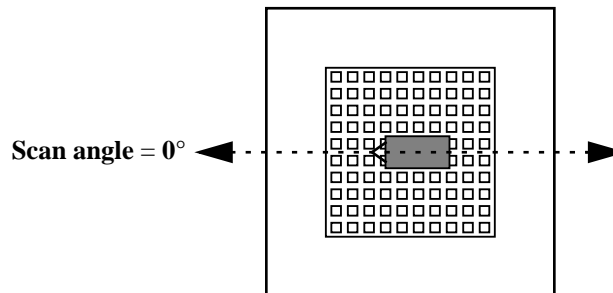
For "D" and "E" scanners, use a 1-micron pitch reference. For "G," "J," and "K" scanners, use a 10 micron pitch height reference. Align the reference within about 2 degrees of perpendicularity to the scan axes.

217.4.3 Set *Real Time* parameters

Set parameters in the control panels to the following values:

Panel	Parameter	Setting
Scan Controls	Scan Size	440 V
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	2.44 Hz
	Number of samples	256
	Slow scan axis	Enabled
	Z limit	440 V
Other Controls	Units	Volts
Channel 1	Data type	Height
	Z range	~ 20 V[a]

a. Adjust the **Z range** parameter to obtain the best contrast.

217.4.4 Set up SPM for contact AFM.

Set up the microscope for contact AFM imaging: set the **AFM mode** to **Contact**. (The microscope can also be calibrated using STM; however, this example will utilize contact AFM.) Set the **Scan angle** to **0** degrees. Adjust Real-time parameters to obtain a good-quality, maximum **Scan size** image. The **Scan rate** should be set to a value of **2.44 Hz** and the **Number of samples** parameter should be set at **256**.

With the sample engaged, check the scanning line relative to the reference's features; the scan should be orthogonal to the pits on the reference. If the reference requires rotation, **Withdraw** and rotate the sample to improve orthogonality between sample and scan line. Repeat until features are oriented orthogonally with the scan frame.

217.4.5 Check sample orthogonality.

Check the sample scan for orthogonality along both the X- and Y-axes. If the scan is aligned along one axis of the scan but not another, it may be necessary to adjust the microscope's **Orthogonality** parameter in the **Scanner Calibration** panel. To measure a captured image's orthogonality, view it using the **Off-line / View / Top View** function. On the display monitor, click on the **Angle** command, then use the mouse to draw a cursor between the edges or centers of widely spaced pits (see left figure

below). The angle should be measured with the vertex near the center of the image and the vertices in the upper-right or lower-left quadrant. The angle will be displayed on the white status bar at the bottom of the display monitor. Use the mouse to drag the cursor until it is oriented correctly,
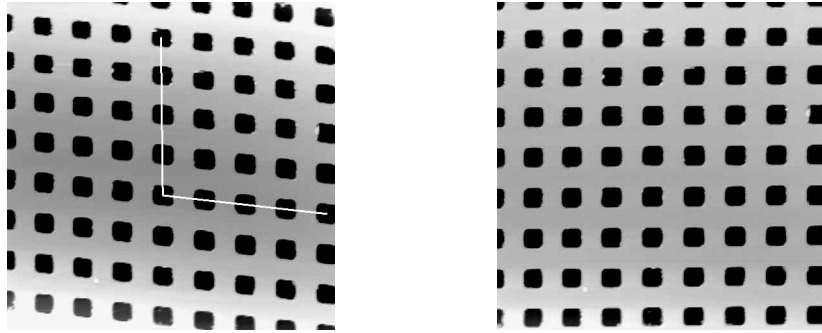
Left image: non-orthogonal image. Notice how pits are aligned with the vertical (slow) axis but skewed with the horizontal (fast) axis.
Right image: a corrected, orthogonal scan .

then read the angle off the status bar. If the angle differs by more than a half degree, a correction will be required; otherwise, skip ahead to the step below.

217.4.6 Adjust sample othogonality.

To adjust the orthogonality of the scan, click on **Real Time / Microscope / Calibrate / Scanner** to access the **Scanner Calibration** panel. The **Orthogonality** parameter is displayed on the bottom-right corner of the panel. Enter the difference between 90 degrees and the angle measured in the Top View image. (For example, if the angle measured in the Top View image was 92.5 degrees, enter a value of **-2.5** degrees into the **Orthogonality** parameter.) Click the **Ok** button to exit the **Scanner Calibration** panel, then capture another image and remeasure the angle. Repeat correction of **Orthogonality** until the scanned image shows less than 0.5 degrees of error. After a major change to the orthogonality parameter, it may be necessary to physically realign the calibration standard to the image frame.

217.4.7 Adjust *mag0* and *arg0*

Select **Microscope / Calibrate / Scanner** to display the **Scanner Calibration** dialog box. Set **mag0** and **arg** as follows:

When adjusting linearity, it is essential to keep in mind which part of the scan is the beginning and which is the ending. When the **Line direction** is set to **Trace**, the beginning of the fast scan is on the left, as indicated by the arrow base. When viewing up or down scans, be careful to check the image against itself, not the residual

image from the previous scan. Also, be careful to compare only the part of the scan drawn since the last parameter change.

Because the display monitor screen itself is not linear, the best way to check **mag0** and **arg** is with the Real-time **Zoom** box.

A. Adjusting **Fast mag0**

After engaging, click on **Microscope / Calibrate / Scanner**. This will invoke the **Scanner Calibration** window. As parameters values are changed, the effects will be seen on the display monitor. Now move the mouse cursor to the display monitor and select **Zoom Out**. This will produce a box whose size and position can be changed by alternate clicks on the left mouse button. Adjust the box until it is about one-third the size of the scan. Click *once* on the right mouse button to park the box and free the cursor, which will jump to Execute. (Clicking twice will execute a **Zoom**, which we don't want to do.)

Select (highlight) the scanner parameter to be modified. The first one to do is **Fast mag0**. Now move the zoom box to the start of the fast scan (on the left if the Line direction is set to Trace). Move and size the zoom box until the beginning third of the scan's features are exactly aligned with the zoom box. *The beginning third of the scan will be the standard for judging almost all of the linearity values.* It is a good technique to ignore the set of features near the edges of the scan since these may be distorted slightly. Now move the zoom box to the ending third of the scan. Align one side of the zoom box with some features and observe how well the other side aligns with the features under it.

Compared to the beginning third of the scan, if the features in the ending third of the scan are too big to fit the same number of them inside the zoom box, type a smaller number for **Fast mag0**. If the features are too small, increase **Fast mag0**. Change **mag0**s by about 0.1 to 0.3 units at a time. Remember, every time a parameter is changed, the entire scan axis is affected, so after every change it is necessary to resize the zoom box at the beginning third of the scan and recompare the ending.

B. Adjusting **Fast arg**

Now that the beginning third of the scan is equal to the ending third, the center may need adjusting. Just as before, if the center features are too big for the box, reduce the **Fast arg**. If the features are too small, increase the **Fast arg**. Change **arg**s by 0.2 to 0.5 units at a time. Remember, changes affect the whole scan, so keep resizing the zoom box after changes.

Center Compressed   Center Expanded
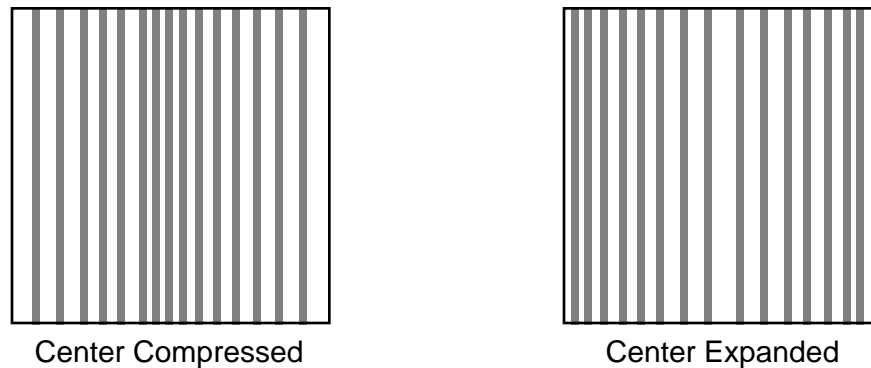
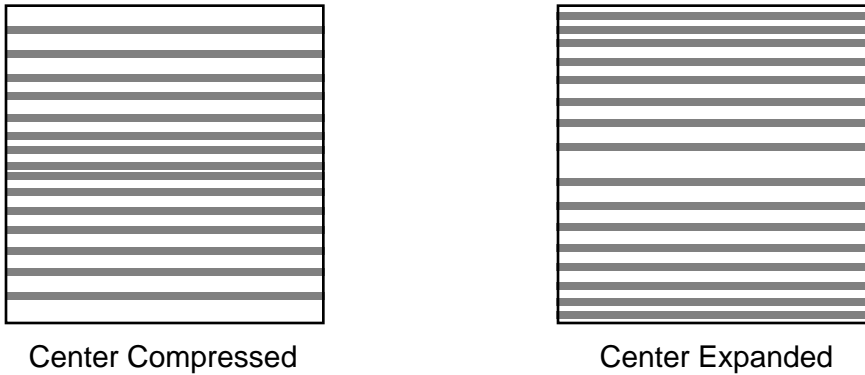**Figure 217-2 X scan linearization—compression vs. expansion.**

NOTE: After an adjustment in **Fast arg** is made, **Fast mag0** may need to be readjusted. Continue steps A and B until the rulings are evenly spaced across the Fast-axis. (If using a one-dimensional reference, it may be desirable to do step E {Adjusting **Fast mag1**} before proceeding; because adjusting the slow linearity will require the one-dimensional reference to be physically rotated 90 degrees. After finishing step E, return to here.)

After **Fast mag0** and **Fast arg** have been set, insert these values for **Slow mag0** and **Slow arg**. These will serve as close starting points before adjusting the slow linearities.

C. Adjusting **Slow mag0**

Follow the same instructions as for **Fast mag0**. Setting the slow linearities requires a lot of time because after making a parameter change it is necessary to wait until a fresh third of a scan has begun with the new parameters before the zoom box can be resized. Move the resized box to the ending of the scan and be prepared to measure the ending quickly. If the user is quick enough, a new parameter value can be typed in before the scan starts again. Be careful not to confuse scan top and bottom with beginning and ending, since the scan direction alternates. Adjust the zoom box to fit

the beginning third of the scan and check the ending third. If the features at the ending third are too big for the box, reduce the parameter. If the features are too small, increase. Be careful to compare only parts of the current scan, not the previous one.

Center Compressed   Center Expanded

Figure 217-3 Y scan linearization—compression vs. expansion.

D. Adjusting **Slow arg**

Follow the same instructions as for **Fast mag0**. Adjust the zoom box to fit the beginning and ending of the scan, then check the center. If the features in the center are too big, reduce the **Slow arg**. If the features are too small, increase.

After an adjustment of **Slow arg**, **Slow mag0** may need to be readjusted. Continue to follow steps C and D until the rulings are evenly spaced along the slow axis.

E. Adjusting **Fast mag1**

Initial Adjustment

Close the **Scanner Calibration** window by selecting **OK**. (Selecting **Restore** will reset the parameters to the values when the box was opened.) Change the **Scan size** to **150 volts**. Select **View / Scope Mode**. On the display monitor, select **Dual Trace**. If the two scope traces do not overlap, **Fast mag$_1$** needs to be adjusted. On the control panel, select **Slow scan axis**. When some tall features appear on the scope trace, tap the keyboard's right or left arrow key to toggle **Slow scan axis** to **Disabled**. Select **Microscope / Calibrate / Scanner** to open the **Scanner Calibration** box. Select **Fast mag1**. Use the left and right arrow keys to change the value until the two traces are aligned. (The yellow retrace line will shift in the same direction as the arrow.) When done, select **OK** to close the **Scanner Calibration** box. Set **Slow scan axi**s back to **Enabled**. Select **View / Image Mode**.

Fine Adjustment

Initial adjustment is usually adequate; however, if more precision is desired, adjust **Fast mag1** using the same procedure as in step A (see "Adjusting **Fast mag0**"). As before, set the **Zoom** box for the beginning of the scan and then check the ending. (Because the scan is small, it may be necessary to use a **Zoom** box up to one-half as large as the scan). If the ending of the scan is bigger than the beginning, reduce **Fast mag1**. If the ending is too small, increase the value.

F. Adjusting **Slow mag1**

With the **Scan size** still set to **150 volts**, select **Microscope / Calibrate / Scanner**. Select **Slow mag1** and input the value from **Fast mag1**. For medium-sized scanners (C to F), it is probably worth stopping here, because the **Slow mag1** value is usually 100-120 percent of the **Fast mag1** value. However, if more precision is desired, adjust **Slow mag1** using the same procedure as in step C (see "Adjusting **Slow mag0**"). As before, set the **Zoom** box for the beginning of the scan and then check the ending. (Because the scan is small, it may be necessary to use a **Zoom** box up to one-half as large as the scan). If the ending of the scan is bigger than the beginning, reduce **Slow mag1**. If the ending is too small, increase the value.
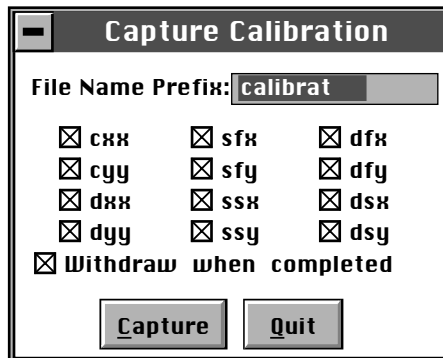
•Wait one complete frame with the new value before readjusting the **Slow mag1** value.

This completes the linearity adjustments. Check the final result by capturing an image and checking it with the **Off-line / Modify / Zoom** window. The scanner is now ready for calibration of the X and Y parameters.

217.5 X-Y Calibration using **Capture Calibration**

Scanners are calibrated by setting parameters in the NanoScope software using the **Capture Calibration** command. This basic step must be performed before fine calibration. The basic procedure using version 4.XX software and a 10-micron calibration reference (fig. 217.1) is described in this section. Earlier versions of NanoScope software are similar. Note that "D" and "E" scanners require a 1-micron cross-ruling.
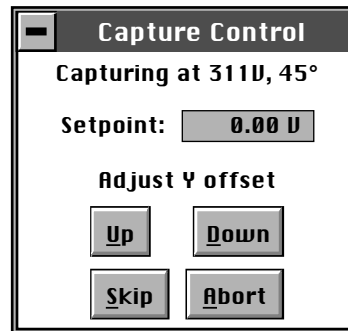
1. With the **Scan rate** set at **2.44 Hz** and **Number of samples** parameter at **256**, a full **Capture Calibration** will require about 70 minutes. Increasing the **Number of samples**, or decreasing the **Scan rate** will significantly increase the required time.

2. Using the mouse, click on **Real-time / Capture / Calibration**. The **Capture Calibration** dialog box will be displayed:

```
┌──────────────────────────────────────┐
│ ▬   Capture Calibration              │
├──────────────────────────────────────┤
│  File Name Prefix: calibrat          │
│                                       │
│    ⊠ cxx     ⊠ sfx     ⊠ dfx         │
│    ⊠ cyy     ⊠ sfy     ⊠ dfy         │
│    ⊠ dxx     ⊠ ssx     ⊠ dsx         │
│    ⊠ dyy     ⊠ ssy     ⊠ dsy         │
│  ⊠ Withdraw  when  completed          │
│                                       │
│      ┌─────────┐   ┌──────┐          │
│      │ Capture │   │ Quit │          │
│      └─────────┘   └──────┘          │
└──────────────────────────────────────┘
```

The **Capture Calibration** dialog box lists twelve parameters used in the calibration procedure. If this is a first-time calibration, or if the microscope's calibration has not been checked within the last three months, verify that all parameters are selected. Click on the **Capture** button to initiate the automatic calibration routine.

**NOTE:** The microscope will begin an automatic series of scans on the reference which require about one hour total to complete. During each scan, the scanner moves the piezo using carefully calculated movements. Many of these movements are unusual, giving rise to a variety of images which do not "look" like the normal reference. (E.g., pits may resemble trenches, features may be presented at various angles.)

3. As each routine is executed, the user may need to adjust the scan slightly to optimize the calibration image. This is accomplished using the **Capture Control** dialog box, which is displayed on the control monitor throughout the calibration routines.

```
┌─────────────────────────────┐
│ ▬   Capture Control         │
├─────────────────────────────┤
│    Capturing at 311U, 45°   │
│                             │
│   Setpoint:  [   0.00 U  ]  │
│                             │
│       Adjust Y offset       │
│                             │
│    [  Up  ]    [ Down ]     │
│                             │
│    [ Skip ]    [ Abort ]    │
│                             │
└─────────────────────────────┘
```

Note the status bar at the bottom of the control monitor. The "capture" status will start at skip 2—meaning that the program will skip the current scan and one more before capturing an image for later calibration. (This allows hysteresis and drift to settle out when the scan changes direction and size between images.) If the scan has not settled out by the time the capture status changes to "On," click on the **Skip** button to increment the capture to skip 1, skip 2, or skip 3. Do not click on **Abort** unless you want to stop the entire **Capture Calibration** program.

Features should extend across as much of the displayed image as possible. If portions of features are missing, or if the image is blank, click repeatedly on the **Adjust Y offset / Up** or **Down** button to adjust the scan until more of the features are imaged. When the image looks optimized, allow the software to capture the image without disturbing it. The software will then automatically index to the next image.
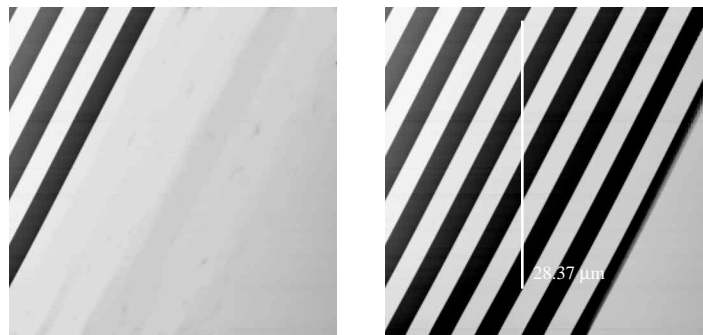
Figure 217-4 Use the *Adjust Y offset / Up* or / *Down* buttons to improve a partial calibration image (left). Calibration image at right is improved and may be used to measure distances between features.

After the first four images with the diagonal stripe pattern have been captured, the user can usually leave the system unattended while the program continues to completion. Some of the next images may seem stretched in one dimension; however, this is normal.

When all calibration images have been obtained, the software will prompt the user that it is finished.

4. Go to the **Capture** directory (!:) where all **Capture Calibration** files have been saved. Click on **Off-line / File / Browse** (or the **Browse** icon) to review all **Capture Calibration** files.

5. Verify that all calibration images contain features spanning the full width and height of the image frame. (See right Figure 217-4 above as an example.)

NOTE: All images unsuitable for calibration must be recaptured. Record the file name extensions of any unusable files (e.g., .cxy, .dyy), then delete them. Reengage the reference surface and select **Capture Calibration** again. Verify that the file name prefix is identical to that of the usable files, then deselect all usable file name extensions from the last capture (click on their box to remove the "X"). Finally, click on the **Capture** button to recapture the selected files.
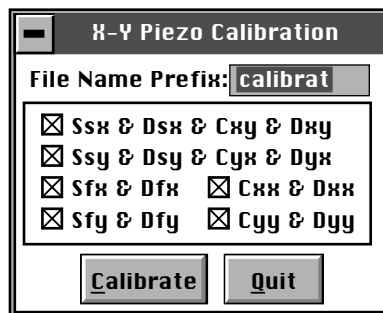
217.6 Off-line / Utility / Autocalibration

After the **Capture Calibration** routine is completed, the user measures surface features contained within each image and enters their dimensions into the software. The software compares its estimates with the actual (user-entered) dimensions to make final corrections. This portion of the calibration is carried out using the **Off-line / Utility / Autocalibration** command.

To utilize the **Off-line / Utility / Autocalibration** command, do the following:

1. Select any of the captured calibration images in the Capture (!:) directory. Click on the **Off-line / Utility / Autocalibration** command. The control monitor will display the **X-Y Piezo Calibration** dialog box:

```
┌─────────────────────────────────────┐
│ ■    X-Y Piezo Calibration          │
├─────────────────────────────────────┤
│ File Name Prefix: calibrat          │
│ ┌─────────────────────────────────┐ │
│ │ ⊠ Ssx & Dsx & Cxy & Dxy         │ │
│ │ ⊠ Ssy & Dsy & Cyx & Dyx         │ │
│ │ ⊠ Sfx & Dfx   ⊠ Cxx & Dxx       │ │
│ │ ⊠ Sfy & Dfy   ⊠ Cyy & Dyy       │ │
│ └─────────────────────────────────┘ │
│       ┌──────────┐  ┌──────┐         │
│       │ Calibrate│  │ Quit │         │
│       └──────────┘  └──────┘         │
└─────────────────────────────────────┘
```

Verify that the file name prefix assigned to the captured files from the **Capture Calibration** routine described in section 217-5 above is correct.

2. For normal calibration, verify that all parameters are selected in the dialog box, then click on **Calibrate** to execute the routine.

The software will sequentially present various calibration images on the display monitor while prompting the user to draw either a vertical line or a horizontal line. The control monitor will simultaneously display various dialog boxes—one for each image—requesting that a distance be entered.

3. Use the mouse to draw a line on the image. The line should be drawn to span as many features as possible, preferably connecting like edges. For example, consider the following:

Autocalibration
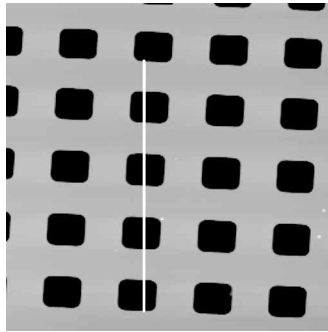
Draw a vertical line

In this example, a line is drawn from the bottom edge of one feature to the bottom edge of another feature four rows away—a distance of 40 microns. The control monitor simultaneously displays a dialog box for entering the distance indicated by the white line:
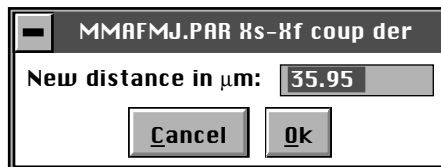
The distance displayed in the box (in this example, 35.95 μm) is the software's estimate (based on current calibration values) of the length of the line drawn on the image. If the line length is different from the value shown, it will have to be changed. (In this example, the user would enter a value of **40**.)

4. Enter the distance covered by the white line drawn on the image. If a 10-micron reference is being employed, like portions of features are spaced 10 microns apart (e.g., between bottom edges, left sides, etc.).

IMPORTANT! Features must be measured without regard to how they appear in calibration images. Remember, features may be represented as having stretched, distorted, or angled appearances due to the unusual movements employed during **Capture Calibration** scanning. Regardless, features are separated by the same (e.g., 10-micron) spacings.

5. Continue drawing lines and entering measured distances until all **Capture Calibration** images have been measured. When the software is finished, it will prompt the user that it is done.

If **Capture Calibration** and **Autocalibration** routines have been completed correctly, the SPM should be calibrated within 1-2 percent accuracy over most of the scanner's measuring range. Should it be necessary to obtain still better accuracy, the SPM can be fine-tuned to obtain maximum measuring accuracy. This is accomplished through the use of calibration parameters discussed in the next section.

217.7 Fine-tuning for X-Y Measuring Accuracy

Fine-tuning is usually performed at two **Scan size** settings: **150** and **440** volts. Both horizontal and vertical measurements of sample features are made, then compared with actual distances. Based upon this comparison, computer parameters are fine tuned. To fine tune your SPM for maximum X-Y measuring accuracy, review each of the steps below.

NOTE: If you are using an "A" scanner to image atomic-scale features, it will necessary to substitute graphite or mica for the silicon calibration reference. See section 217.9 below for directions in atomic-scale calibration of the X- and Y-axes.

217.7.1 Prepare system for fine-tuning.

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (440 volts). Verify that the **Scan angle** is set at **0.00** degrees. Mount a calibration reference into the SPM and begin imaging. This may consist of a generic (e.g., 10-micron, silicon) reference, or a sample having features of known dimensions (e.g., grating, etc.). Optimize the image quality.

IMPORTANT! Your calibration and fine-tuning procedures are no better than the procedures and references used. Choose both carefully!

217.7.2 Measure horizontally at 440 V scan size.

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (**440** volts). Verify that the **Scan angle** is set to **0.00** degrees. **Engage** the surface.

2. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them. (For example, on a 10-micron, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The screen will display the measured distance between pits next to the line.

Now check to see whether the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to section 217.7.3.

3. Based upon the results in step #2 above, perform the following calculation:

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

That is, divide the known distance by the distance displayed next to the line drawn in step #2 above. Record this quotient.

4. Click on the **Real time / Microscope / Calibrate / Scanner** option. The **Scanner Calibration** dialog box will be displayed:

Scanner Calibration			
X fast sens:	230 nm/U	**Y fast sens:**	228 nm/U
X fast derate:	0.230 nm/U^2	**Y fast derate:**	0.218 nm/U^2
X slow sens:	267 nm/U	**Y slow sens:**	257 nm/U
X slow derate:	0.286 nm/U^2	**Y slow derate:**	0.270 nm/U^2
Xs−Xf coupling:	0.548 nm/U^2	**Ys−Yf coupling:**	0.569 nm/U^2
Xs−Xf coup der:	0.027 pm/U^3	**Ys−Yf coup der:**	0.222 pm/U^3
Xs−Yf coupling:	0.078 nm U^2	**Ys−Yf coupling:**	0.134 nm U^2
Xs−Yf coup der:	−0.04 pm/U^3	**Ys−Yf coup der:**	0.0668 pm/U^3
Fast mag0:	1.40	**Slow mag0:**	1.47
Fast mag1:	1.05	**Slow mag1:**	0.860
Fast arg:	3.00	**Slow arg:**	3.00
Fast cal freq:	2.44 Hz	**Slow cal freq:**	4.77 Hz
Piezo cal:	440 U	**Rounding:**	0.00
Allow rotation:	Allow	**Orthogonality:**	0.00 deg

<center>[Ok]   [Cancel]</center>

Multiply the quotient obtained in step #3 above by the **X fast sens** value shown on the **Scanner Calibration** panel, then enter the new value. The new value will adjust the scanner's fast axis to more closely match calculated distances with actual feature distances. The new sensitivity setting will take effect as soon as it is entered. To save it to the computer's hard disk, click on the **Ok** button. This will also close the Scanner Calibration panel.

217.7.3 Measure vertically at 440 V scan size

1. Return to the image of the calibration reference. Clear the horizontal line drawn in section 217.7.2 above (click the right mouse button, or click on **Clear**), then draw a vertical line between features. After waiting for at least three full scans (to allow the piezo to stabilize), select two widely spaced features and draw the line from like portions of features (top edge-to-top edge, etc.). The SPM will display the calculated distance between features.

2. Check to see whether the microscope's calculated distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to section 217-7.4 below.

3. Based upon the results in step #2 above, perform the following calculation:

$$\frac{\text{known distance between features}}{\text{SPM-calculated distance between features}}$$

That is, divide the known distance by the distance displayed next to the line drawn in step #2 above. Write this value down.

4. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **Y slow sens** parameter.

5. Write down the **Y slow sens** value shown on the **Scanner Calibration** panel. Multiply the quotient obtained in step #3 above by the **Y slow sens** value shown on the **Scanner Calibration** panel, then enter the new value. This should adjust the scanner's slow axis to more closely match calculated distances with actual feature distances. To save the new parameter value, click on the **Ok** button.

217.7.4 Measure horizontally at 150 V scan size

1. Set the **Scan size** parameter on the **Scan Controls** panel to one-third the maximum (**150** volts). Verify that the **Scan angle** is set to **0.00** degrees, and that **Units** (**Other Conrols** panel) is set to **Volts**.2. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them. (For example, on a 10-micron, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The microscope will display the measured distance next to the line.

Now check to see whether the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to section 217-7.5.

3. Adjustments may be made in one of two ways. The first method uses to trial and error to "dial in" in the correct value. The second method calculates a precise correction.

- *Trial and error method*— Go to the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **X fast derate** parameter (or **Y fast derate** for Y-axis adjustment). If the measured distance is less than the actual distance, decrease the **X fast derate** parameter slightly (or **Y fast derate** for Y-axis adjustment) and remeasure image features. Adjust deratings up or down until measurements accord with known feature distances.

- *Calculation method*— Perform the following calculation:

$$\frac{s - \left(\frac{a}{m} \cdot [s - d(440 - v)]\right)}{440 - v}$$

Where *s* is the **Sens** value; *a* is the actual distance; *d* is the derating value; *m* is the measured distance; and, *v* is the **Scan size** in volts.

4. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **X fast derate** parameter.

5. Write down the **X fast derate** value shown on the **Scanner Calibration** panel. Multiply the value obtained in step #3 above by the **X fast derate** value shown on the **Scanner Calibration** panel, then reenter the new **X fast derate** value. This should adjust the scanner's fast axis to more closely match calculated distances with actual feature distances. To set the new parameter value, click on the **Ok** button.

217.7.5 Measure vertically at 150 V scan size

1. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a vertical line between them. (For example, on a 10-micron, silicon reference, draw the line from the top edge of one pit to the top edge of another pit as far away as possible.) The microscope will display the measured distance next to the line.

Now check to see whether the microscope's measured distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, no further calibration is required.

2. If the displayed distance does not agree with the known distance, perform the calculation shown in Step #3 above to make a correction.

3. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **Y slow derate** parameter.

4. Write down the **Y slow derate** value shown on the **Scanner Calibration** panel. Multiply the value obtained in step #2 above by the **Y slow derate** value shown on the **Scanner Calibration** panel, then enter the new value. This should adjust the scanner's slow axis to more closely match calculated distances with actual feature distances. To set the new parameter value, click on the **Ok** button.

217.7.6 Change *Scan angle* and repeat calibration routines

As a final step, change the **Scan angle** on the **Scan Controls** panel to **90** degrees, then repeat steps 217-7.2—217-7.5 above for the following parameters: **Y fast sens**, **X slow sens**, **Y fast der**, and **X slow der**. This will ensure the scanner is calibrated along both the X- and Y-axis.

217.8 Fine-tuning for Z Measuring Accuracy

With respect to obtaining accurate Z-axis measurements, users should be aware that while it is generally not difficult to obtain accurate X-Y calibration references, it is much more difficult to obtain accurate Z-axis results. High-quality Z-axis calibration standards may cost $2000 or more each. The platinum-coated, silicon calibration reference distributed by Digital Instruments have 180 nm vertical features which are accurate to within ± 3 percent and are used throughout the examples provided within this section. If greater accuracy is desired, an appropriate calibration standard must be selected, and/or a metrology head employed using a Digital Instruments Dimension Series microscope.

IMPORTANT! Your calibration and fine-tuning procedures are no better than the references and techniques employed. Choose both carefully!

Users should be aware that Z-axis calibration is very sample-dependent. It is not easy to control Z piezo dynamics because the Z-axis does not move at a constant rate, as the X- and Y-axes do during scans. Furthermore, offsets can effect the piezo over a period of minutes.

217.8.1 Engage surface and begin imaging

Set up the microscope for contact AFM imaging: set the **AFM mode** to **Contact**. (The microscope can also be calibrated using STM; however, this example will utilize contact AFM.) Engage the surface and adjust Real-time parameters to obtain a good-quality, maximum **Scan size** image. The **Scan rate** should be set to a value of **2.44 Hz** and the **Number of samples** parameter should be set at **256**.

Verify that the **Z Center Position** value shown next to the image display hovers around 0 volts. If the **Z Center Position** value is non-zero, use the **Real Time / Motor / Tip Up** and **Tip Down** buttons to adjust.

217.8.2 Adjust *Scan size*

Depending upon the type of scanner employed, adjust the **Scan size** parameter to include part of a pit, or up to 4 pits, within the image, along with portions of the surrounding flat area. The image should include Z-axis depths from the highest to the lowest elevations.

"A" scanners, when set at their maximum **Scan size** (440 volts), may image only a small portion of one pit. Adjust the sample and/or microscope stage until one side of a pit is visible, along with portions of the flat, "rim" area around the periphery of the pit.

"J" scanners are capable of imaging up to eighteen pits. Set the **Scan size** to a value which displays four pits and surrounding flat area (approximately **40.0** microns).

Intermediately sized scanners should be set at values between those of "A" and "J" scanners.

217.8.3 Capture and correct an image

Allow the scanner to stabilize by scanning 2-3 frames. If the scanner has been running at extreme voltages, this may require a few minutes.

Capture an image by clicking on **Capture** in the **Real Time** screen, or use the **Capture** icon. When the image is captured, go to the Off-line screen.

It will be necessary to remove all tilt and scan line errata from the image before continuing. To remove scan line errata, select **Off-line / Modify / Flatten Manual**. The **Flatten order** parameter in the dialog box should be set to **1**. Go to the display screen, then draw stopbands over the pits along the Y-axis as shown here:
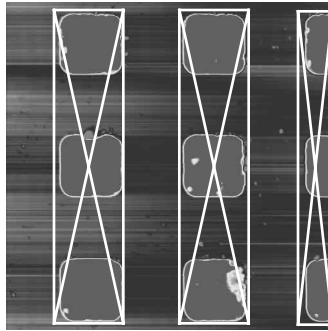
If the image encompasses only one pit, or a portion of one pit, cover as much of the pit as possible with a stopband while leaving the remainder of the top surface exposed. Click on **Execute** to complete the flattening procedure, then **Quit** the dialog box.

Next, it will be necessary to planefit the image to remove tilt. Select the **Off-line / Modify / Planefit Manual** option. Set the **Planefit order** parameter to **1**. Go to the display monitor, then select the X cursor from the top menu bar. Draw a horizontal line between pits as shown here:
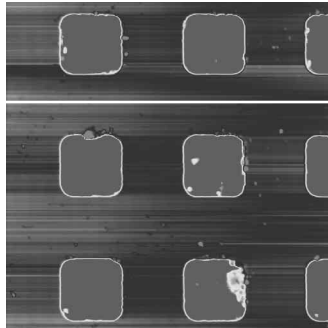

If the image encompasses only one pit, or a portion of one pit, draw the line on the top, flat portion of the reference, away from the pit. Click on **Execute** to complete the planefit, then **Quit** the dialog box. The image should now be corrected and ready for analysis.
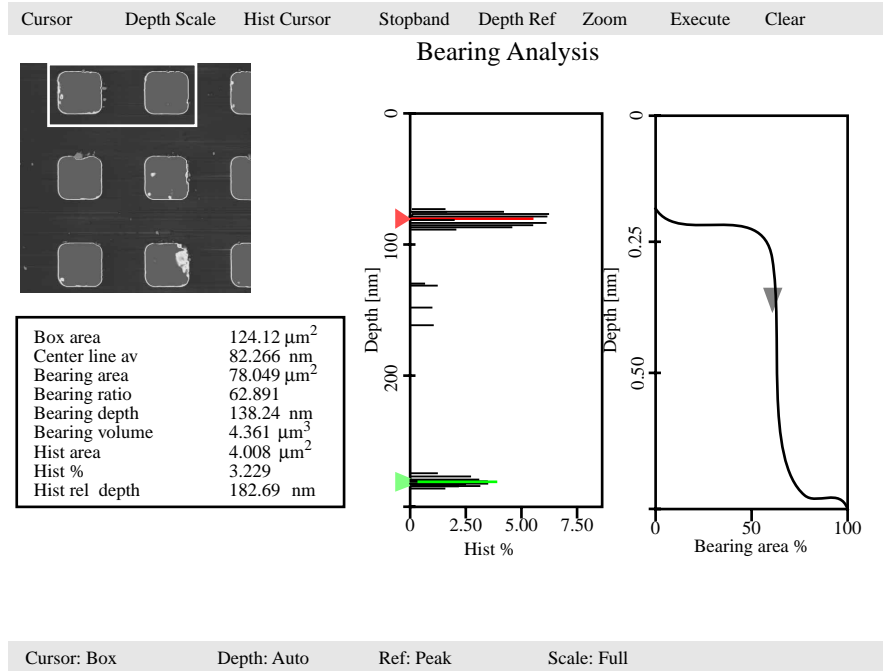
217.8.4 Measure vertical features

With the image corrected, its vertical features may now be measured. This is performed using **Bearing** analysis so that more data points are utilized.

Select the **Off-line / Analyze / Bearing** command. Go to the display screen and draw a cursor box around 1-4 pits, including a portion of the top, flat surface between the pits. Avoid including debris and pits with poorly defined features within the cursor box.

Draw cursor box around 1-4 pits.

Avoid pits having debris or poorly defined features.
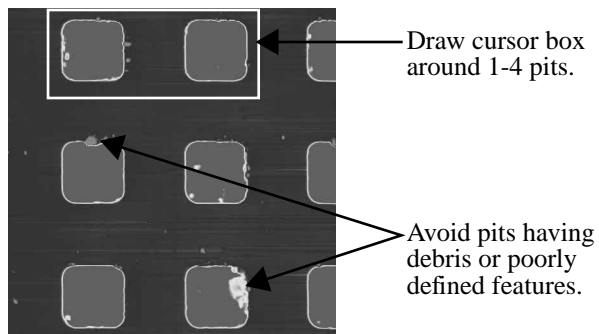
To commence **Bearing** analysis, click on **Execute** in the display monitor's top menu bar. Height data within the drawn cursor box will be graphed on the display monitor, and should show two, prominent spikes. These spikes correspond to two elevations on the surface: the bottom of each pit and the top surface.

Cursor	Depth Scale	Hist Cursor	Stopband	Depth Ref	Zoom	Execute	Clear

Bearing Analysis

Box area	124.12 μm²
Center line av	82.266 nm
Bearing area	78.049 μm²
Bearing ratio	62.891
Bearing depth	138.24 nm
Bearing volume	4.361 μm³
Hist area	4.008 μm²
Hist %	3.229
Hist rel depth	182.69 nm

Cursor: Box	Depth: Auto	Ref: Peak	Scale: Full

Use the mouse to slide the top (red) line cursor on the **Hist%** graph to the center-line of the top data spike as shown in the example above. Select **Hist Cursor / On** to generate another (green) line cursor; position this one on the centerline of the bottom data spike. The distance between the two cursors is given at the **Hist rel depth** field inside the data box. (Depending upon whether the red cursor is positioned over the top or bottom data spike, the **Hist rel depth** value may be either positive or negative. It doesn't matter; take the absolute value.)

If a 10-micron, silicon calibration reference from Digital Instruments has been used, the **Hist rel depth** value should be very close to **180 nm**. Its variance from this value gives a good indication of the microscope's measuring error in the Z-axis. If the value is too far from 180 nm, it will be necessary to make adjustments (see Section 217.8.5 below); write down the **Hist rel depth** value now.

To exit the **Bearing** dialog box, click on **Quit**.

217.8.5 Correct *Z sensitivity*

If the depth of pits on the 10-micron silicon calibration reference deviates significantly from 180 nm, it will be necessary to correct the **Z sensitivity** parameter in the **Z Calibration** dialog box. Corrections here do <u>not</u> change the voltage applied to the scanner's Z-axis piezo crystal, but affect scaling applied throughout the Z-axis measuring range; therefore, they should be made precisely and carefully.

Transfer to the **Z Calibration** dialog box by clicking on **Real Time / Microscope / Calibrate / Z**. The **Z sensitivity** parameter is listed at the top-left and should be highlighted. Write this value down now.

Perform the following calculation:

$$\frac{180 \text{ nm}}{\text{Hist rel depth value}}$$

That is, divide the actual depth of features (180 nm for the 10-micron calibration reference) by the measured depth (indicated in **Bearing** analysis by the **Hist rel depth** value). Multiply this quotient by the **Z sensitivity** value in the **Z Calibration** dialog box, then replace the value with the product. Click on the **Ok** button to enter the new **Z sensitivity** value.

NOTE: The numerator value above (180 nm) is for Digital Instruments' 10-micron silicon reference. For other calibration references, set the numerator equal to the depth of features measured by **Bearing** analysis. Ideally, calibration references used should have features with heights comparable to those being imaged and measured on samples.

217.8.6 Recheck Z-axis measuring accuracy and recorrect

After steps 217.8.1—217.8.6 above have been completed, it will prove useful to recheck the Z-axis measuring accuracy of the SPM. Repeat steps 217.8.4—217.8.5 above until the required accuracy is obtained.

NOTE: SPMs leaving Digital Instruments are normally calibrated to within 2 nm of accuracy using the 10-micron silicon reference. This corresponds to an accuracy of 1 percent. If greater accuracy is required, use a very high-precision calibration height standard with features comparable to those being imaged on samples.

217.8.7 Calculate *Retracted* and *Extended offset deratings*

Piezoelectric materials exhibit greater sensitivity at higher voltages. In steps 217.8.1–217.8.6 above, the Z-axis was calibrated while scanning near the middle of its voltage range (i.e., **Z Center Position ~ 0 V**). In this section, the Z-axis piezo will be calibrated while extended and retracted to offset the increased sensitivity.

1. Engage the surface as described in sections 217.8.1—217.8.2.

2. Use the **Real Time / Motor / Tip Up** and **Tip Down** buttons to ensure the **Z Center Position** reads **100 V**.

EXPLANATION: By using the motor to move the tip up, the feedback loop forces the Z-axis piezo to extend to continue its tracking of the surface.

3. Refer to steps 217.8.3—217.8.4 above to determine the measured depth of the calibration standard with a 100 volt **Z Center Position**.

Record the measured depth; it should read 180 nm on a Digital Instruments 10 μm silicon calibration reference (model STS-1800). If the depth measured by the extended piezo is in error by more than two percent, an adjustment will be required.

4. Click on the **Real Time / Microscope / Calibrate / Z** option to display the **Z Calibration** panel, then click on the **Extended offset der** parameter.

Perform the following calculation:

$$(1 + \text{current offset der}) \ \frac{180\text{nm}}{\text{meas. depth}} \ — \ 1$$

5. Enter the result into the **Extended offset der**ating menu item.

6. The procedure for calculating and setting the **Retracted offset der** is exactly the same as for the Extended offset der; however, the piezo must be *retracted* by 100 V. Repeat steps #2-6 above in this section; however, start by using the **Motor Control** panel and the **Tip Down** button to retract the piezo to a **Z Center Position** of **-100 Volts**.

217.9 Calibration of "A" Scanners for Atomic-scale Measurement

The "A" scanner is the smallest scanner, with a total travel of approximately 0.4 µm along each axis. Its compact design lends excellent stability for atomic scans, and requires slightly modified X-Y calibration procedures. These are treated in this section. The procedure for X-Y calibration described below is essentially the same as those described in sections 217.7.2–217.7.3 of this support note; however, they substitute graphite or mica atoms for the pits seen on silicon calibration references. A similar procedure is outlined in section 9.4 of this manual for STM imaging of graphite. Please note that this procedure applies only to X-Y calibration of atomic-scale imaging. The Z-axis is calibrated in the normal way using a silicon calibration reference as described in section 217.8 above.

217.9.1 Prepare sample.

The calibration may be conducted with either highly ordered pyrolytic graphite (HOPG) or mica. Mica should be used for contact AFM; HOPG for STM. Both mica and HOPG need to be cleaved to obtain a good flat surface. Cleaving is accomplished by adhering tape to the surface and pulling it off; this produces a fresh surface of atoms having a regular lattice.

Place the sample to be used on a puck, then attach to the scanner cap.

2. To obtain contact AFM atomic-scale images, try the following **Real Time** parameter settings:

Panel	Parameter	Setting
Scan Controls	Scan Size	12 nm
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	61.00 Hz
	Number of samples	512
	Slow scan axis	Enabled
	Z limit	440 V
Feedback Controls	Integral gain	12.00
	Proportional gain	4.00
	Look ahead gain	0.00
	Setpoint	0 V

Panel	Parameter	Setting
Other Controls	AFM mode	Contact
	Input attenuation	1x
Interleave Controls	Interleave mode	Disabled
Channel 1	Data type	Height
	Highpass filter	3–4
	Lowpass filter	1

3. Engage the surface and adjust the **Integral gain** and **Setpoint** to obtain a good image. The **Setpoint** should be kept low if possible, and the **Z Center Position** should be close to **0 V**. Notice that the **Scan rate** is set much higher (~ **61 Hz**) for atomic-scale images, this to defeat some of the noise due to thermal drift.
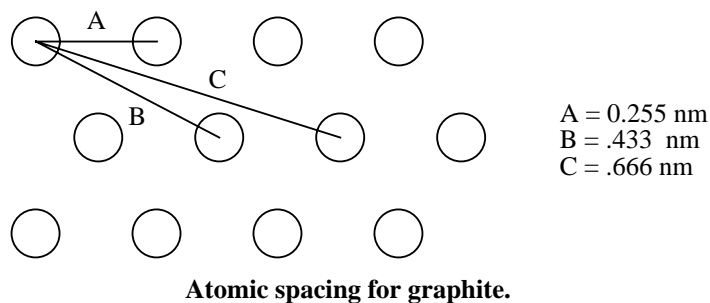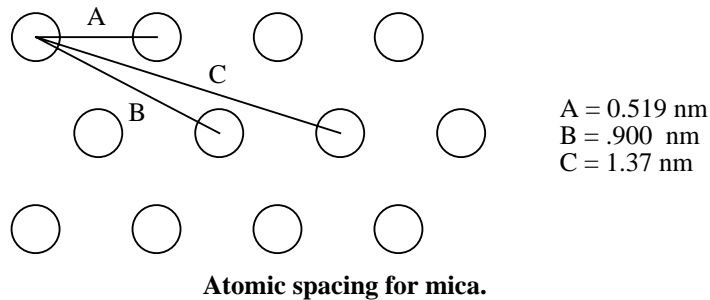
If difficulty is experienced obtaining an image, **Withdraw** and try a different site on the surface, then **Engage** again. Many users find it easiest to obtain good images measurements if the sample is rotated until atoms are oriented vertically.

4. Once a satisfactory image is obtained, **Capture** it. The image should appear similar to the image of graphite shown below. The captured image will be used for the calibration procedure described in the next several steps.
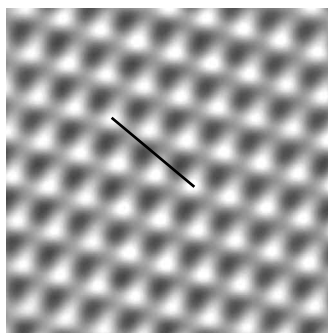
Typical atomic scan of graphite. Note the highly regular lattice of the atoms. The cursor line describes a distance of 6.66 Å.

5. Go to the **Off-line / View / Top View** option and measure the spacings between atoms using the mouse. Depending upon whether the sample is graphite or mica, the spacings should measure as shown below.

A = 0.519 nm
B = .900 nm
C = 1.37 nm

Atomic spacing for mica.

A = 0.255 nm
B = .433 nm
C = .666 nm

Atomic spacing for graphite.

Record the spacings between at least ten atoms observed in the captured image. It will prove useful to take several samples of each measurement and average them to ensure accurate results. (This can be done quickly by alternatively "walking" the cursor line from atom to atom; the average distance will be shown on the bottom-right corner of the display monitor's status bar.) If the measurements vary by more than 2 percent from the dimensions shown above, a correction should be made.

6. Correction of the X- and Y-axis is essentially the same procedure as described in sections 217-7.2—217-7.3 of this support note and will not be repeated here. The only significant difference is that the known distances must be adjusted for the smaller, atomic spacings of the atoms. Furthermore, only the <u>sensitivity</u> parameters are adjusted for atomic-scale imaging; namely:

- X fast sens at 0° **Scan angle**

- X slow sens at 0° **Scan angle**

- Y fast sens at 90° **Scan angle**

- Y slow sens at 90° **Scan angle**

The derating parameters are *not* adjusted for atomic-scale imaging, including:

- X fast derate
- X slow derate
- Y fast derate
- Y slow derate
- Retracted offset der
- Extended offset der

As referenced in section 217.7.9, the sensitivity parameters must be calibrated with the **Scan angle** set at both **0** degrees and at **90** degrees.

7. Z-axis calibration is done in the normal way, using a silicon calibration reference. Refer to section 217-8 for instructions on calibrating the Z-axis.