# Noise Floor Measurement, Dimensional Calibration, & Drift Correction

Calibration Procedures for the Dimension Series (3100 & Icon/Fast Scan) AFMs

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#### Introduction

The noise floor of an AFM is the summation of all the unwanted or spurious electrical signals and acoustic and mechanical noise sources within the system. (Here noise is defined as any signal other than the desired one being monitored.) Some noise sources may be intrinsic to the system, while others may be environmental in nature/origin. The noise floor sets a lower limit on the magnitude of real signals (e.g., changes in height/topography) that can reliably be measured. Thus the lower the noise floor, the greater the ultimate sensitivity of the AFM. Bruker Dimension series AFM systems are extremely stable, and the noise floor should remain within specification if the AFM is properly sited and maintained. The reason for measuring the noise floor on a regular basis is to verify that the AFM's sensitivity limit is within specification. If not, this is indicative of a problem with the AFM or its environment (e.g., a failing power supply that leads to 60 or 120 Hz electrical noise, a passive vibration isolation table lacking sufficient air, excessive mechanical vibrations due to HVAC systems or construction, etc.) that needs to be addressed for the system to function properly.

In addition to the noise floor, the AFM's vertical and lateral dimensional calibrations should be checked on a regular basis to ensure they are also within specification ( $\leq$ 1-2%), as the response of the piezos to an applied bias voltage will change over time, particularly during the first few months of use for new piezos. The vertical and lateral dimensional calibrations can be verified simultaneously using a VGRP-15M calibration reference from Bruker. This reference consists of an array of 180 nm deep 5  $\mu$ m x 5  $\mu$ m square pits etched on a 10  $\mu$ m pitch into a platinum-coated silicon wafer mounted on a 15 mm diameter steel puck. If the system is calibrated correctly, conversion values saved in the scanner calibration file allow the AFM to properly convert measured piezo voltages and/or strain gauge signals into the corresponding XYZ metric length scales for images. Since the conversion values are piezo-specific, each AFM head has its own separate scanner calibration file, named according to the serial number(s) associated with it. If one of the AFM heads is found to be out of specification, the NanoScope software has a built-in Auto Calibration routine that can be run as detailed in the Appendix and the corresponding AFM help files.

The following standard operating procedure (SOP) provides details about the noise floor and dimensional calibration procedures and specifications for the Dimension series AFMs (3100 and Icon/FastScan), along with information about the VGRP-15M calibration reference.

# **Specifications**

## **Dimension FastScan<sup>1</sup>**

- Vertical Noise Floor: <40 pm RMS (height sensor)
- X-Y position noise (closed loop): ≤0.20 nm RMS
- Z sensor noise level (closed loop): 30 pm RMS

## **Dimension Icon**<sup>2</sup>

- Vertical Noise Floor: <30 pm RMS
- X-Y position noise
  - o Closed Loop: ≤0.15 nm RMS
  - o Open Loop: ≤0.10 nm RMS
- Z sensor noise level (closed loop)
  - o 35 pm RMS
  - 50 pm RMS, force curve bandwidth (0.1 Hz to 5 kHz)

## VGRP-15M Calibration Reference<sup>3</sup>

Substrate 🧹							
	Units	Target 🥢	Minimum	<b>Maximum</b>	Comments		
Material	-	Silicon		- // -			
Туре	-	Square Chips	-	-			
Size	mm	8 x 8	-	-			
Resistivity	Ω-cm	0.018	0.010	0.025			
Dopant C		Antimony			VEDCITV		
Thickness	μm	300	295	305			
			Patterned A	rea			
	Units	Target	Minimum	Maximum	Comments		
Material	-	Single Crystal Si	-	-			
Size	mm	4 x 4	-	-			
Pit Length	μm	5	4	6			
Pit Width	μm	5	4	6			
Pit Depth	nm	180	165	195	To be measured and reported for every chip		
Pitch	μm	10	-	-	Value not measured		
			FS Coating	5			
	Units	Target	Minimum	Maximum	Comments		
Material	-	Ti	-	-	Titanium Adhesion Layer		
Thickness	nm	5	4	6			
Material	-	Pt	-	-	Protective Platinum Coating		
Thickness	nm	40	30	50			

<sup>&</sup>lt;sup>1</sup> Specifications are valid for an imaging bandwidth of 625 Hz, except for X-Y position noise, which is valid up to 2.5 kHz in Adaptive XY scanning mode for the FastScan system.

<sup>&</sup>lt;sup>2</sup> Except as noted, specifications are valid for an imaging bandwidth of 625 Hz for the Icon system.

<sup>&</sup>lt;sup>3</sup> From Brukerafmprobes.com

# **Standard Operating Procedures**

# **Noise Floor Measurement**

### **Data Acquisition**

- 1) Load the appropriate probe into the corresponding probe holder.
  - a. Dimension FastScan system
    - i. Load a FastScan A probe into the FastScan Z probe holder and attach it to the FastScan head.
  - b. Dimension 3100 or Icon system
    - i. Load an OTESPA probe (or equivalent tapping mode probe with  $k \approx 40$  N/m) into the Icon probe holder and attach it to the Icon head.
- 2) Turn on the AFM.

<u>WARNING</u>: Do not power cycle the computer with the AFM on; this could cause severe damage to the piezo(s).

**NOTE:** Allow the laser to warm up for approximately 30 minutes prior to imaging for exceptionally precise measurements. This is crucial for accurate measurement of the AFM noise floor and checking the dimensional calibration and piezo drift properties of the system!

- 3) Login on the Excel sign in sheet located at the upper right of the AFM computer desktop.
- 4) Open the AFM control software by double clicking the NanoScope icon.
  - a. Dimension FastScan system
    - i. Ensure the Dimension FastScan head is both physically attached and displayed in the software (**Figure 1**). If not, use the *Change Scanner* option in the software to switch from the Icon to the FastScan head.
  - ii. Under Change Microscope Setup, select the Dimension FastScan from the Microscope Select menu.
    - iii. Under Select From select Tapping Mode, under Select Experiment Group select Tapping Mode in Air, and then under Select Experiment choose TappingMode in Air Soft Tapping (see Figure 1).
  - b. Dimension Icon system:
    - Ensure the Dimension Icon head is both physically attached and displayed in the software (Figure 2). If not, use the *Change Scanner* option in the software to switch from the FastScan to the Icon head.
    - ii. Under Change Microscope Setup, select the Dimension Icon from the Microscope Select menu.
    - iii. Under Select From select Tapping Mode, under Select Experiment Group select Tapping Mode in Air, and then under Select Experiment choose TappingMode in Air Soft Tapping (see Figure 2).

Select From:	Microscope: Dimension FastScan
Image: ScanAsyst in Fluid - 06/16/15   18:29         Or         Choose an Experiment Category:         ScanAsyst         Asyst         ScanAsyst         Tapping         Mode         Image: ScanAsyst         ScanAsyst         Tapping         Contact         Mode         Image: ScanAsyst	Forse the second s
Electrical & Mechanical Other SPM Magnetic Properties FastScan Bio	>> Change Scanner      >> Change Microscope Setup   Experiment Description
2 Select Experiment Group: Tapping Mode in Air Tapping Mode in Fluid	TappingMode is a standard Atomic Force Microscopy (AFM) technique where the sample topography (height) is measured by monitoring the amplitude of oscillation of the cantilever as the probe is scanned across the sample surface. A piezo stack excites the cantilever substrate vertically, causing the cantilever to move up and down with a constant sinusoidal amplitude at or
3 Select Experiment: TappingMode in Air TappingMode in Air - Soft Tapping	below its fundamental resonant frequency. When the probe interacts with the sample, the amplitude decreases. The feedback loop maintains a constant damped oscillation amplitude (typically 10 – 100 nm) by vertically moving the scanner at each (x,y) data point to maintain a constant "setpoint" amplitude. By keeping a constant oscillation amplitude, a constant tip-sample interaction is maintained during imaging.

**Figure 1.** The *Select Experiment* interface with the *Microscope Select* window opened indicating the Dimension FastScan head is selected. Red boxes highlight the proper experiment type and AFM selections.

Select From:	Microscope: Dimension Icon
Use previous experiment	
TappingMode in Air - Soft Tapping - 06/16/15   18:29	
Or	
Channe an Europian at Catagory	
choose an Experiment Category:	
Asyst 🍝 🌫	
ScanAsyst Tapping Contact	icon
Mode	
in the second se	
Electrical & Mechanical Other SPM	
Magnetic Properties	>> Change Scanner
	>> Change Microscope Setup
	Experiment Description
	TappingMode is a standard Atomic Force
Select Experiment Group:	sample topography (height) is measured by
Tapping Mode in Air Tapping Mode in Fluid	of the cantilever as the probe is scanned across
	the sample surface. A piezo stack excites the cantilever substrate vertically,
	causing the cantilever to move up and down with a constant sinusoidal "free" amplitude
	at or below its fundamental resonant
	sample, the amplitude probe interacts with the
3 Select Experiment:	amplitude (typically
TappingMode in Air - Soft Tapping	20 – 100 nm) by vertically moving the scanner at each (x,y) data point to maintain
TappingMode in Air - Standard Torsional Resonance (TR-Mode)	a constant "setpoint" amplitude. By keeping a constant oscillation amplitude. a
-	constant tip-sample interaction is maintained

**Figure 2.** The *Select Experiment* interface with the *Microscope Select* window opened indicating the Dimension Icon head is selected. Red boxes highlight the proper experiment type and AFM selections.

- Secure the VGRP-15M calibration reference from Brukerafmprobes.com to the sample chuck. The calibration reference that should be used has several black asterisks (\*) on the box, as shown in Figure 3 (left).
  - a. Align the reference so that it is as square as possible relative to the AFM head travel.

**NOTE:** It is important to have the patterned area as square as possible relative to the scan window. Rotate the sample chuck prior to engaging the surface to align the pits so they are square with respect to the AFM head travel within <2°. Alternatively, you can make small adjustments to the Scan Angle later so the patterns appear square in the height channel, but this can lead to coupling between the X and Y axes, which can be problematic when checking the dimensional calibration of the AFM head.

- b. The sample may have acetate cellulose replicating film covering it to keep it clean and protect it from dirt and other debris. The replicating tape film can be removed with tweezers, as shown at right in **Figure 3**.
- c. Although the purpose of the film is to keep the standard clean and prevent contamination, if the calibration reference needs further cleaning, the procedure is as follows:
  - i. Cut a piece of replicating tape film larger than the specimen surface.
  - ii. Use a dropper to place a drop of acetone on the surface of the sample.
  - iii. Gently lay the cut film over the sample surface, approaching at an angle so no bubbles form. This needs to be done before the acetone drop evaporates.
  - iv. Allow the acetone to completely evaporate (~5-10 minutes). UHP (99.999%) compressed nitrogen gas can be blown over the sample surface to speed this process up (~2-5 minutes) and help remove bubbles.
  - v. The replicating film can typically be removed using tweezers (alternatively, a removable adhesive tape such as Scotch tape may be necessary in rare circumstances).
  - vi. This process may need to be repeated 3-5 times in order to create a clean, contaminant-free surface.



**Figure 3.** The VGRP-15M calibration reference box marked with black asterisks (left), and VGRP-15M calibration reference covered with a thin film of cellulose replicating tape (right).

6) Complete *Setup*. Make sure to properly mark with the cursor where the probe tip is relative to the back of the cantilever.

- a. When performing the *Cantilever Tune*, be sure to use a *Target Amplitude* of 500 mV and *Peak* offset of 5.00% (Figure 4).
- b. After the Cantilever Tune, the Amplitude Setpoint may need to be set to 480 mV (Figure 4).
  - i. **Figure 5** illustrates where the *Amplitude Setpoint* (pink line) should lie with respect to the tuning curve (blue trace) and *Peak Offset* (green line).



**Figure 4.** *Cantilever Tune* default autotune settings. The *Target amplitude, Peak offset,* and *Amplitude Setpoint* are highlighted with red rectangles.



**Figure 5.** A typical tuning curve (blue trace) showing a 5% *Peak offset* (green line) and an *Amplitude Setpoint* (pink line) at the proper location for Soft Tapping.

7) Using *Navigate*, find a visually clean area to engage on the sample that is next to (but not on) the patterned area (red circle in **Figure 6**).



**Figure 6.** Optical image (0x zoom) of the VGRP-15M calibration reference showing the grid of pits for later dimensional calibration (red square) and clean surrounding area on which to engage and measure the noise floor (red circle).

- 8) Prior to engaging, verify that the scan parameters are set to the values shown in **Figure 7** (left for FastScan, right for Icon).
  - a. Click on the *Expanded Mode* red puzzle piece to show more scan parameters.



- b. Parameters to double check (those marked with an asterisk (\*) differ between the two systems):
  - i. Scan
    - 1. Scan Size
    - 2. Aspect Ratio
    - 3. X Offset
    - 4. Y Offset
    - 5. Scan Angle
    - 6. Scan Rate\*
    - 7. Samples/Line
  - ii. Feedback
    - 1. Integral Gain
    - 2. Proportional Gain
  - iii. Limits

1. Z Range\*

_		Dimension Fast	Scan		5	_		Dimension I	con	
F	∃ Scan		J	 1			Scan			
	- Sca	n Size	0.00 nm				- Scan Size	e	0.00 nm	1
UT.	- Asp	ect Ratio	1.00				- Aspect R	latio	1.00	
н.	- x o	ffset	0.000 nm				- X Offset		0.000 n	m
н.	- Y O	ffset	0.000 nm				- Y Offset		0.000 n	m
١.,	- Sca	n Angle	0.00 °				- Scan An	gle	0.00 °	
	- Sca	n Rate	1.99 Hz				- Scan Rat	e .	0.999 H	z
	— Tip	Velocity	0.00 µm/s				- Tip Velo	city	0.00 µm	n/s
н.	- Sam	ples/Line	256				- Samples	/Line	256	
н.	- Line	25	256				- Lines		256	
н.	- Slov	v Scan Axis	Enabled				- Slow Sca	an Axis	Enabled	t t
н.	- Sca	n Single Frame Number	1				- Scan Sin	gle Frame Number	1	
н.	- Rou	nding	0.100				L XY Close	ed Loop	On	
н.	⊢ XY (	Closed Loop	On			B.	Feedback			
E	∃ Feedba	ick					- Integral	Gain	1.000	
	- Inte	gral Gain	1.000				- Proporti	onal Gain	2.000	
	- Pro	portional Gain	2.000				- Amplitu	de Setpoint	480.0 m	١V
	- Am	plitude Setpoint	485.0 mV				- Analog2		0 V	
I.	- Ana	log2	0 V				- Drive Fre	equency	312.670	9 kHz
	- Driv	re Frequency	1376.009 kHz				- Drive An	nplitude	424.2 m	V
	- Driv	e Amplitude	76.29 mV				- Lock-In	Phase	161.6 °	
	Loc	K-In Phase	-108.1 *		5		- Lock-In	BW	21.99 k	Hz
U.		K-IN DW	2 500 LLL-		4		L LP TM D	eflection BW	2.500 k	Hz
١.	— LP ∃ Interle:		2.300 KH2				Interleave			
	∃ Limite	ive				B.	Limits			
11		ange	3.33 um				- Z Range		13.0 µm	1
	Red	uced Z Delav	2.00 s				- Reduced	I Z Delay	2.00 s	
	- Z A	uto Center Boundary	0 %				- Z Auto C	Center Boundary	0 %	
	- Am	plitude Range	2000 mV				- Amplitu	de Range	2000 m	v
	∟ тм	Deflection Limit	24.58 V				L TM Defl	ection Limit	24.58 V	
E E	∃ Other				_		Other			
P		ISE S	S T A	F		1.			25	

Figure 7. The engage settings for the Dimension FastScan (left) and Icon (right) systems.

- 9) Verify that the AFM is using *Standard Engage Mode*, **not** *Smart Engage Mode*.
  - a. Go to Microscope, then Engage Settings.
  - b. Under Smart Engage, make sure the Engage Mode selected is Standard (Figure 8).

💩 Engage Parameters	<b>×</b>
🖯 General Engage	*
└─ Sew tip	Triggered
Tapping Engage	
<ul> <li>Trigger safety</li> </ul>	15.0 µm
<ul> <li>Sewing trigger [%]</li> </ul>	96
<ul> <li>Pre engage setpoint [%]</li> </ul>	92
<ul> <li>Delta Setpoint</li> </ul>	-0.0500
<ul> <li>Final Delta Setpoint</li> </ul>	0.100
<ul> <li>Test threshold slope</li> </ul>	3.00
<ul> <li>Test threshold dZ</li> </ul>	0.100 nm
<ul> <li>Engage min setpt [%]</li> </ul>	25
<ul> <li>TM engage gain</li> </ul>	0.250
Engage Setpoint	1.10
Stage Engage	=
<ul> <li>Sample clearance</li> </ul>	1000 µm
<ul> <li>SPM safety</li> </ul>	100 µm
<ul> <li>SPM engage step</li> </ul>	0.729 μm
<ul> <li>Withdraw Z Pos</li> </ul>	Smart Lift
<ul> <li>Withdraw Focus Tip</li> </ul>	No
Load/Unload height	30000 µm
Auto Align	
Auto Align Photodetector	Yes
Smart Engage	
<ul> <li>Engage Mode</li> </ul>	Standard
<ul> <li>Fast engage velocity</li> </ul>	50.0 %
<ul> <li>Fast engage threshold</li> </ul>	100 %
- Fast engage height	-10000 µm
Height Engage	
	Cancel OK

Figure 8. AFM set up to use Standard Engage Mode. Red boxes indicate the relevant Engage Parameters.

10) *Engage* the AFM probe on the sample surface.

a. After engaging, set the scan size to 1  $\mu$ m.

- 11) When beginning to scan, the probe may not be contacting the sample surface. If this is the case, slowly decrease the *Amplitude Setpoint* in small increments using the left arrow key until you just begin to track the surface.
  - a. Once the probe comes into contact with and is properly tracking the surface, the trace and retrace of sample topography (height) will agree with each other.
- 12) Optimize the scan parameters and confirm the area is free of debris.
  - a. Dimension FastScan System
    - i. The scan rate should be  $\geq$ 2.0 Hz.
  - b. Dimension Icon System
    - i. The scan rate should be  $\geq$ 1.0 Hz.
- 13) **Gradually** lower the scan size and *Z limit* according to the series of values given in Table 1. Allow the AFM to trace and retrace a few lines each time before decreasing the values again. You may also lower the Integral and Proportional Gain values at each step.
  - a. Dimension FastScan System

- i. Only lower the Z limit by a maximum of 0.5  $\mu$ m per step until reaching the final minimum value of 0.5  $\mu$ m (500 nm).
- b. Dimension Icon System
  - i. After the initial large decrease to a Z limit of 6  $\mu$ m (i.e., approximately half the Icon's full Z range), only lower the Z limit by a maximum of 1.0  $\mu$ m per step until reaching the final minimum value of 1.0  $\mu$ m.

**NOTE: Do not drop the Integral Gain or Proportional Gain below 0.1!** Gains this low will not give you an accurate representation of the true AFM noise floor.

**NOTE:** Slowly lowering the scan size and Z limit results in a more accurate measurement of the AFM's true noise floor. However, the Z limit could be lowered to 0.5  $\mu$ m (for the FastScan system) or 1.0  $\mu$ m (for lcon system) during the initial 1  $\mu$ m scan if there are no large topographical features in the image.

Scan Size	FastScan Z Limit (µm)	lcon: Ζ Limit (μm)
1 µm	3.3	13.0
500 nm	3.0	6.0
250 nm	2.5	5.0
100 nm	2.0	4.0
50 nm	1.5	3.0
25 nm	1.0	2.0
10 nm	0.5	1.0
1 nm	0.5	1.0

**Table 1.** Sequentially decreasing scan sizes and corresponding Z limit.

c. Make sure that *OL Plane Fit* (offline plane fit) is set to *None* for both Channel 1 (Height Sensor) and Channel 2 (Amplitude). This setting is located at the bottom of the Trace/Retrace window

RT Plane Fit	Line	-
OL Plane Fit	None	-

- 14) When you reach the final scan size of 1 nm, increase the Samples/line to 512.
- 15) Capture an image.
  - a. File path is \Capture\Lab Imaging\Noise Floor Measurement & Dimensional Calibration\FastScan or \Icon.
    - i. Create a new folder with the date for the image(s) being captured.
  - b. Capture file name should be NF\_VGRP-15M\_ScanRate\_Integral Gain\_Proportional Gain.000
    - i. Example: NF\_VGRP-15M\_2.0Hz\_IG1.0\_PG2.0.000
    - ii. It is important to include .000 at the end of the file name.

**NOTE: Do not use Capture Withdraw**, as you may have to capture several images in order to get a good measurement of the noise floor.

16) After capturing your first image, immediately begin to capture the next image with the filename extension auto incrementing to .001.

#### Analysis

- 17) While the second image is being acquired and captured, open and analyze the first image in NanoScope Analysis.
  - a. Perform a 1<sup>st</sup> order Flatten on the height channel.
  - b. Calculate the *Roughness*. Use the *Image Rq* to get the root mean squared (RMS) value for the surface roughness (**Figure 9**). Smaller values are better.
    - i. Dimension FastScan: The calculated Rq value should be  $\leq 0.040$  nm (0.4 Å or 40 pm).
    - ii. Dimension Icon: The calculated Rq value should be  $\leq 0.030$  nm (0.3 Å or 30 pm).



**Figure 9.** Example Noise Floor measurement showing an *Image Rq* of 43 pm or 0.43 Å. (The example shown is an image acquired on the Dimension FastScan.)

- 18) If your first image returns an appropriate noise floor value (≤0.040 nm for FastScan, ≤0.030 nm for Icon), then cancel the second capture. Otherwise, continue to capture images until you obtain an acceptable RMS value and/or determine the source/cause of the excess noise.
- 19) *Withdraw* the AFM probe from the sample surface.

- 20) Fill in the appropriate Icon or FastScan *Noise Floor Measurement & Dimensional Calibration Tracker* spreadsheet with the measured RMS *Roughness (Image Rq)* value for the noise floor.
  - a. The file path is <u>\\coen-cypress\Groups\Nano\SSLab\Paul Davis\MSE 596\Calibration Tracker\</u> <u>Dimension FastScan Noise Floor Measurement & Dimensional Calibration Tracker</u> or <u>\Dimension</u> <u>Icon Noise Floor Measurement & Dimensional Calibration Tracker</u>, where <u>\\coen-cypress\Groups\Nano\</u> is more commonly referred to as the L drive.
  - b. Fill in the date of the Noise Floor measurement along with the RMS roughness value. Also include any important comments related to the noise floor measurement (e.g., ongoing construction, etc.)

**NOTE:** The purpose of the Noise Floor Measurement & Dimensional Calibration Tracker spreadsheet is to enable longitudinal performance tracking of each AFM to see if environmental changes are negatively impacting performance and/or there is a problem with the instrument.

21) Proceed to the Dimensional Calibration SOP below.

# **Dimensional Calibration**

#### **Data Acquisition**

- 1) If you did not do so previously, go to the *Setup* menu and make sure to properly mark where the probe tip should be relative to the back of the cantilever with the blue crosshair. If you are unsure about the tip setback amount, consult the Bruker AFM Probes catalog.
- Navigate to an optically clean patterned area of the VGRP-15M calibration reference (red square in Figure 6). Note that the patterned area is 4 mm x 4 mm, so if the origin is set at the bottom left corner of the pattern, the center will be at -2000, -2000 (in microns).
- a. Change the settings to the ones seen in **Figure 7** (left for FastScan, right for Icon), but decrease the Scan Rate to 0.5Hz.
  - b. To make it more likely that you will engage exactly where you want to on the sample, you may want to calibrate the optics to compensate for parallax error. This can be accomplished by going to the *Calibrate* menu, then selecting *Optical* and *Optics/SPM Axis Co-linearity*. This will open a Wizard that will guide you through the optics calibration process.

**NOTE:** The optics calibration is more important for the Icon head than the FastScan head because the Icon head employs a side-mounted camera and optics rather than integrated top-mounted camera and optics in the FastScan head.

**NOTE:** It is important to have the patterned area as square as possible relative to the scan window. Rotate the sample chuck prior to engaging the surface to align the pits so they are square with respect to the AFM head travel within <2°.

- 3) *Engage* the AFM probe in the center of four pits.
  - a. After engaging, increase the scan size to  $1 \, \mu m$ .

- 4) When beginning to scan, the probe may not be contacting the sample surface. If this is the case, slowly decrease the *Amplitude Setpoint* in 5-10 mV increments until you begin to track the surface.
  - a. Once the probe comes into contact with the surface, the trace and retrace of sample topography (height) will agree with each other.
- 5) Set the Scan Size to 22  $\mu$ m.
- 6) Use XY offsets to center the scan as closely as possible at the center of four pits.
- 7) Once the four pits are square with respect to the scan window and the trace and retrace are exhibiting good agreement, **increase** the *Samples/Line* to **512** and capture an image.
  - b. File path is \Capture\Lab Imaging\Noise Floor Measurement & Dimensional Calibration\FastScan or \Icon.
    - i. Use the date-specific folder created above in the Noise Floor Measurement SOP.
  - a. Capture name should be DC\_VGRP-15M\_ScanRate\_Integral Gain\_Proportional Gain\_.000
    - i. Example: DC\_VGRP-15M\_0.5Hz\_IG1.0\_PG2.0.000
    - ii. It is important to include .000 at the end of the file name.
- 8) After capturing your first image, immediately begin to capture the next image with the filename extension auto incrementing to .001.

#### Analysis (Vertical Dimension)

- 9) While the second image is being acquired and captured, open and analyze the first image in NanoScope Analysis.
  - a. Perform a 1<sup>st</sup> order XY Plane Fit.
  - b. Perform a 1<sup>st</sup> order Flatten, using boxes (Stop Bands) to exclude the pit regions.
  - c. Calculate the *Depth* of the pits. Use the *Peak to Peak Distance* with the *Depth* tool to find the average depth (**Figure 10**). This should return a depth of 180 ± 15 nm (and more typically, 180 ±





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Results	
<ul> <li>Peak to Peak Distance</li> </ul>	177.047 nm
<ul> <li>Minimum Peak Depth</li> </ul>	217.582 nm
<ul> <li>Maximum Peak Depth</li> </ul>	40.5 nm
<ul> <li>Depth at Histogram Maximum</li> </ul>	40.5352 nm
Number Peaks Found	143

**Figure 10.** Image of the VGRP-15M dimensional calibration reference (top left) and the corresponding histogram (top right). The *Peak to Peak Distance* calculated using the *Depth* tool can be used to measure the overall depth of the pits (bottom). The example shown is from a Dimension Icon image.

**NOTE:** The two peaks seen in the histogram at right in **Figure 10** correspond to the substrate surface and bottom of the pits, respectively. Thus if the two dashed line markers are set to the two peaks (the default setting), the peak to peak distance tool will calculate the depth of the pits. If there are more than two peaks, move to a new location and restart from Step 1) of the Dimensional Calibration SOP.

- 10) If your first image has a depth of ~180 nm (± 15 nm), then cancel the second capture. Otherwise, continue to capture images until you obtain a reasonable depth value.
  - a. If the machine is out of specification, please see the *Calibration* information in the Appendix, the Bruker help files, and/or talk to the SSL Manager.
    - i. The *Calibration* information in the Appendix was taken from the Bruker NanoScope help files, and can be found clicking on Help in the NanoScope software toolbar.

#### **Analysis (Lateral Dimensions)**

- 11) After checking the depth (i.e., vertical or Z) calibration, the lateral (XY) dimensions need to be examined. There are two different ways to do this. Step 12) describes how to do this using the Section tool to measure the XY dimensions, while Step 13) describes using the Width tool to measure the XY
  - dimensions.
- 12) Starting with the previously processed *Depth* image, click on the *Section* tool.
  - a. For the lateral X dimension:
    - i. Right click on the Height Sensor image and select Rotating Box.
    - ii. Draw a box that goes through the center of two of the four pits without touching the upper and lower rounded corners of the pits. Select a pair of pits that are side by side (**Figure 11**, top left).
    - iii. Move the pair of vertical dashed lines in the *Section* window to the leading or trailing edges of the two pits (**Figure 11**, at bottom left, shows the leading edges selected).
    - iv. The lateral distance between pits (called the pitch) in the X direction is then displayed (Figure 11, bottom right).
      - 1. The value should be within <2% of 10  $\mu m$  (i.e., 10.0  $\pm$  0.2  $\mu m$ ).
      - 2. Record the value.
      - 3. Repeat steps 12)a. i. iv. for the remaining pair of pits.
  - b. For the lateral Y dimension:

- Draw a box and rotate it 90° (done by holding down the *Shift* key and moving the mouse) so that it passes through the center of two of the four pits without touching the upper and lower rounded corners of the pits. Select a pair of pits that are stacked above/below each other (Figure 11, top right).
- ii. Move the pair of vertical dashed lines in the *Section* window to the leading or trailing edges of the two pits (**Figure 11**, at bottom left).
- iii. The lateral distance between pits (called the pitch) in the Y direction is then displayed (Figure 11, top right).
  - 1. The value should be within <2% of 10  $\mu m$  (i.e., 10.0  $\pm$  0.2  $\mu m$ ).
  - 2. Record the value.
  - 3. Repeat steps 12)b. i. iii. for the remaining pair of pits.
- c. Skip to Step 14).





**Figure 11.** Height sensor image of the VGRP-15M dimensional calibration reference with a *Rotating Box* drawn through the desired central region of the pits for the lateral X (top left) and Y (top right) dimensions. The *Section* window showing the vertical dashed lines set to the leading edges of the two sectioned pits (bottom left). The *Section* tool calculates the Horizontal Distance between the vertical dashed lines (bottom right), which corresponds to the lateral pitch. The example shown is from a Dimension FastScan image.

13) Alternatively, starting with the previously processed *Depth* image, click on the *Width* tool.

- a. Make sure that in the *Inputs* section the *Feature Direction* is on *Below* (Figure 12, top right).
- b. Move the *Threshold Depth* (teal dashed line in the *Depth Histogram* window) to approximately halfway between the height of the wafer surface and the bottom of the pits (~128 nm in Figure 12, bottom)
  - i. The depth of the *Lowest Peak* (bottom of the pits) will be indicated by the pink dashed line in

the Depth Histogram window.

- ii. The height of the *Highest Peak* (wafer surface) will be indicated by the gold dashed line in the *Depth Histogram* window.
- c. Right click on the *Height Sensor* image and choose *Select Feature*.
- d. Click in the center of each of the four pits; default colors of the selected pits will be teal and blue (Figure 12, top left).
- e. Right click on the table with *X location*, go to the *Configure Columns* menu, and under *Columns to Display* check the box (☑) for *Y location*.
- f. The table will now update with the *X location* and *Y location* of each pit.

X location	Y location
6.227 (µm)	6.257 (µm)
6.098 (µm)	16.064 (µm)
16.058 (µm)	16.128 (µm)
16.171 (µm)	6.319 (µm)

- g. For each pair of pits, subtract the *X* location and *Y* location values in order to get the lateral pitch (distance between pits) in the X and Y dimensions for each pair of pits.
  - i. The subtracted value should be within <2% of 10  $\mu m$  (i.e., 10.0  $\pm$  0.2  $\mu m$ ), and more typically ~0.2%.

N I P I I A					
	45.6 nm	Grid Markers		120	
		- Threshold Depth		128 nm	
		- Lowest Peak		21/ nm	
		<ul> <li>Highest Peak</li> </ul>		40.4 nm	
		Inputs			
		<ul> <li>Feature Direction</li> </ul>		Below	
		<ul> <li>Threshold Plane</li> </ul>		-87.8 nm	
		<ul> <li>Number Histogram Bins</li> </ul>		512	
		<ul> <li>Reference</li> </ul>		Highest Peak	
		<ul> <li>Histogram Filter Cutoff</li> </ul>		0.00 nm	
		<ul> <li>Min Peak to Peak</li> </ul>		150 nm	
	-222.6 nm	<ul> <li>Distance From Peak Type</li> </ul>		Absolute Distance	
0 1: Height Sensor 220 pm		Distance From Peak		1.00 nm	
Deoth Histogram					
25					
20					
10					
5				A	
м <sup>_</sup>					
70 50		100	150	200	nm

**Figure 12.** Height sensor image of the VGRP-15M dimensional calibration reference showing the *Threshold Region* with the four pits highlighted in teal and blue (top left). The *Width* tool parameters (top right). The *Depth Histogram* showing the vertical gold, teal, and pink dashed lines (bottom). The example shown is from a Dimension FastScan image.

- 14) Once the AFM's noise floor and dimensional (XYZ) calibrations are within specification, *Withdraw* the AFM probe from the sample surface.
- 15) Fill in the appropriate *Noise Floor Measurement & Dimensional Calibration Tracker* with the depth value obtained from the *Peak to Peak Distance* and the lateral (XY) values of the pitch obtained using step 12) or 13) from the dimensional calibration measurement.
  - a. The file path is <a>\/coen-cypress</a>\Groups</a>\Nano</a>\SSLab</a>\Paul Davis</a>\MSE 596</a>\Calibration Tracker
  - <u>\Dimension FastScan Noise Floor Measurement & Dimensional Calibration Tracker</u> or <u>\Dimension</u> <u>Icon Noise Floor Measurement & Dimensional Calibration Tracker</u>, where <u>\\coencypress\Groups\Nano\</u> is more commonly referred to as the L drive.
  - b. Fill in the date of the Dimensional Calibration check, along with the depth (Z) and lateral (XY) values. Also include any important comments related to the noise floor measurement (e.g., ongoing construction, etc.)
- 16) Break down the AFM.
  - a. Move the AFM head all the way up and the stage forward.
  - b. Close the NanoScope software.
  - c. Log ending time into the Excel sign in sheet.
  - d. Cover the VGRP-15M calibration reference with replicating tape as described earlier in this SOP and return it to its box.
  - e. Return the used probe to the appropriate probe box.
  - f. Put away the probe holder.

# Appendix

The following potentially useful information has been taken directly from the Bruker Dimension Icon/FastScan help files.

#### Calibration

This section provides detailed instructions for the fine calibration of the Dimension FastScan SPM. In general, the scanner's calibration should be checked every three months. The main change in calibration will be the loss of piezo sensitivity in the first year of use. This is an expected behavior; loss of 10–15% of the initial scan size is common.

Bruker employs a software-guided calibration procedure for all microscopes. The procedural details of how calibration is executed using NanoScope software are beyond the scope of this User Guide and include proprietary methods exclusive to Bruker. The calibration is summarized in four basic steps outlined below.

- 1. **Orthogonality Adjustments:** The orthogonality adjustment compensates for a possible non-square scan pattern.
- 2. **Scanner Linearization:** Adjust parameters for correcting the capacitive nature of the piezo material and force the XY motion of the piezo to move at a constant velocity.
- 3. **XY Calibration:** Designate the software conversion values using one or both of the following methods.
  - Capture Calibration and Auto calibration: A built-in software routine automatically controls the scanner while capturing a series of data files. The data files are then used to "teach" the software the actual distance that the scanner moved. This process creates a customized set of conversion values that will be used to convert the known signals to a metric scale.

-OR-

- **Fine Tuning:** Manually test and adjust XY sensitivity and derating values to within 1% accuracy. The conversion values that scale the scanner motion are confirmed, or are adjusted if necessary to within 1%. These same values should be adjusted by the customer as the scanner ages due to use. A scanner will typically lose 10-20% of its scan range over the first year of typical use. The scanner will then become stable as long as it is not damaged.
- 4. **Calibrate Z:** Calibrate the Z scanner to within 2%. A separate calibration "look-up" table stores the conversion values for converting the control voltages applied to the Z tube. These values are determined by scanning over a sample with a known depth.

Bruker recommends that you adhere to the Calibration schedule shown in <u>Table 1</u>. For most applications it is sufficient to perform the Z calibration with the same frequency. Z calibration is now automated using

the <u>Z Auto Calibration</u> procedure. For critical height measurements Bruker recommends monthly Z calibration.

Calibration Routine	Time Frame	Frequency
7 Calibration (for General Applications)	First Year	Every 3 months
	Subsequent Years	Every 6 months
Critical Height Measurements	All Years	Monthly

Table 1: Recommended calibration schedule

Depending on your application or the amount of time your system has been in use, you may need to run through one or more of the various <u>Calibration Procedures</u> to ensure optimal functioning of your Dimension FastScan.



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