# Standard operating procedure (SOP) for BRUKER DI 3000 AFM: tapping mode for topography on AFM reference sample Standard operating procedure (SOP) for Bruker (Digital Instruments) DI3000 AFM

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DI3000 AFM setup is used to collect small lateral size (0.25-25 micron) surface topography maps in tapping mode on nanoscale

**Sample requirements:** the samples for AFM measurements must be flat (with lateral sizes from 1 cm<sup>2</sup> to a 2-in wafer), clean of debris, dry, and topographically uniform on the micron to submicron scale.

**Compatible materials:** most dry solid samples, such as metals, semiconductors, oxides, 2D materials, solid thin films on substrates

**Incompatible materials:** liquids, soft materials (cells, photoresists, gels), structures with large size surface irregularities

Please inquire about other materials and applications.

#### **AFM** cantilever tips

**Standard:** single crystal silicon tips with gold or aluminum back coating, such as Bruker tips for tapping mode measurements

Force constant= 42 N/m and resonance frequency about 320 kHz

**Better:** Nanoprobe NCHR-10 Pointprobe – Silicon SPM sensor tips (Nanoworld)

Please note that nonstandard cantilevers are not compatible with the system configuration.

# Part A: find the resonance frequency of the cantilever

1) Start the SPM **software V 531r1** 



and click microscope icon

2) Preview software (Figure 1)

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3) Inspect the Cantilever holder (Figure 2) using the stereo microscope. Clean

it using toothpick and isopropanol and spray Nitrogen gun if required.

 Mount the AFM cantilever probe into the groove.



**Figure 2**: AFM cantilever holder with cantilever inserted



Figure 3: AFM head pin to insert cantilever holder, right panel: head after holder inserted

- 5) Gently mount the cantilever holder onto the AFM scanner head (Figure 3) by fitting the four prongs into the four pin holes on the scanner.
- 6) Upside down the AFM head onto the dovetail gently and guide it all the way to stop. Lock the scanner by turning the screw counterclockwise (Figure 4)
- 7) Connect the cable to electronics box (Figure 5 red





Figure 5: Scanner plug and laser check

8) Focus the tip of the Cantilever:

Click the tip icon on the tool bar and



Figure 4: Scanner lock screw

it is connected, the laser indicator on the scanner should turn red (Figure 5 blue arrow).



bring the tip in focus using rolling ball (Figure 6). Bring the tip to center of

circle). Once,





Figure 6: Tip focusing

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screen using the two knobs on the side of microscope (**Figure 6** blue rectangle). Click **OK**.

9) Find laser spot on the back of Probe:

First, find the laser on the stage. Use the top two screws of the scanner

(blue circle Figure 7) to adjust and locate the laser spot. Visually inspect that laser spot is on the cantilever. Observe the sum signal [2-4 in scale] and the red spot (elliptical or oval) appearing in the photodetector quad on display. Use side screws of the



Figure 7: Laser Alignment



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scanner (green circle Figure 7) to bring the red dot to the center in the photo detector and check in vision system Figure 6.

#### 10) Load sample:



Click focus surface icon: Focus Surface window appears (Figure 8) and use the trackball to move the stage. Place sample on vacuum stage and flip on vacuum switch (Figure 8).





11) Focus Surface: Click focus surface icon. Select surface to find the features on the surface of sample (Figure 8). Bring sample in focus using the rolling ball (Figure 9)





### 12) Tip reflection method for laser spot:



Figure 10: Tip reflection and laser on tip

Once the surface is in focus, select Tip Reflection on Focus Surface window (Figure 10), you could see the laser spot (diffusing red) reflecting off the probe on the video display (Figure 10). Center the red laser dot on the quad using two screws on the scanner. Make deflection value less than + or – 0.1 V. Go back to **Surface** mode.

13) Find resonant frequency of cantilever: Click **tuning fork** icon on the tool bar and



select 100-400 kHz range (Figure 11). Click auto tune to find the resonant



frequency. Target amplitude: 2 volt and peak offset: 0-5 %. You should see a sharp peak for amplitude (Figure 11). Click Back to Image Mode when done. The RMS amplitude value should be 90 -95 % of the target value.

14) Close hood slowly

## Part-B: Scanning and Imaging

15) Check and set the parameters in real time-windows

Scan control (Figure 12)

Scan size: 5-20 um Aspect ratio: 1:1 Scan rate: 2 Hz (to start with) X offset:0 Y offset:0 Scan angle: 0 (to start with) Samples/ line: 128 (to start with) Slow scan axis: enabled (use "disabled" to maximize feedback loop parameters when engaged)

#### Channel 1:

Data type: height (Figure 13)

Data scale: set value to highest feature in scanned area (for example: if the highest feature is 100nm, set the data scale to 200nm to start with, this could be changed during scan) Line direction: trace Realtime plane fit: select "line" Offline plane fit: select "full" Channel 2 (2 and 3 are optional): Data type: amplitude Data scale: in volts Line direction: trace Realtime plane fit: select "line"

= Scar	Scan Controls				
Scan size:	12.0 μm				
Aspect ratio:	1:1				
X offset:	0.000 nm				
Y offset:	0.000 nm				
Scan angle:	0.00 9				
Scan rate:	1.92 Hz				
Tip velocity:	46.1 μm/s				
Samples/line:	128				
Lines:	128				
Slow scan axis:	Enabled				



Height J.Onm	Data type: [	Amplitude 1.000 V	Data type: Data scale:	Phase 100.0 <sup>g</sup>
1.0 nm	Data scale:	1.000 V	Data scale:	100.0 9
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Figure 13: Three Chanel parameters

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Offline plane fit: select "fill" **Channel 3** (only use one variable different from Channel 1, for comparison): Data type: phase Data scale: in volts Line direction: trace Realtime plane fit: select "line" Feedback Controls Main Feedback control: SPM feedback: Amplitude SPM feedback: amplitude (Figure 14) 0.4000 Integral gain: 0.6000 Proportional gain: Amplitude set point: Start with 1.5 volt. The set point 1.269 V Amplitude setpoint: regulates the force on the probe Drive frequency: 299.063 kHz Drive amplitude: 840.6 mV Integral gain: usually use low value <0.6 Figure 14: Feedback control Proportional gain: Insert number by multiplying 1.5 to the value of integral gain Other controls (Figure 15) Other Controls Microscope mode Tapping Microscope mode: select "tapping" Z limit: 5.000 um Units: Metric Z limit: make sure it is larger than 5um Engage Setpoint: 1.00 Units: metric Serial number: Parm update retract: Disabled Engage setpoint: 1.00

**Figure 15: Other Control** 

16) Click "engage" icon on tool bar. The computer then approach the tip to sample surface (Figure 16)

icroscope Vision <u>Stage P</u> anels <u>H</u> elp	Later Sign	
Feedback Controls     Main     Main     SPM feedback: Amplitude     Integral gain: 0.5500     Proportional gain: 0.7500     Angitude selpoint: 1.705 V     Drive frequency: 0.227.907 Hz     Drive angitude: 918.7 mV     Engage Status     Approaching suface     Approaching suface     Approaching suface	r Controls 1.0 V 1.0 V 1.0 V 1.0 V 0 Control S 1.0 V 0 Control S 0 Control S 1.0 V 0 Control S 0	Figure 16: Tip Engaging and Approaching to the Surface

When tip is engaging onto surface, the image of tip can be visible on the monitor (Figure 16). Do the following to maximize feedback Motor view ⊢rame <u>∪</u>apture Microscope control parameters: 4 6 i di katalari kata  $\mathbf{Q} \mid \mathbf{Q}$ 

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- Click "scope" icon
- Select "disable" slow scan axis in scan control window
- Maximize the "amplitude setpoint so the trace and retrace curve look matched but not necessarily overlapping each other (Figure 17).
- Select "enable" Slow Scan Axis
- Select "eye" to bring the imaging mode
- Motor
   Yiew
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   Image: State Stat
- 17) Scan one frame prior to recording the image to adjust scan size, scan speed, and date scale (**Figure 13,14 and 17**).



Figure 17: Waveform and Scanning on the Surface

18) Taking Image: Click "camera" in tool bar to the selected area and record an image (e.g.: Figure 18)





Figure 18: Scanned image of standard sample

19) **To move to another location**: Click "**the withdrawn icon**" once, and the tip will be withdrawn by 1 mm, the area should be still in focus (**Figure 19**).

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Figure 19: Tip Withdraw

20) To save fie:

After capture,



go to **image** icon. Data must be saved in **e- drive** 

only. Data path is: save as an select! e-drive and data (Figure 20)



# Part-C: Unload Sample

- 21) Withdraw the scanner at least 4 mm
- 22) move the stage and take out the sample carefully
- 23) Turn off the stage vacuum
- 24) Disconnect the scanner cable, disconnect the **tip holder** from scanner
- 25) Take out the tip out carefully
- 26) Slide the AFM scanner "**upside down**" to the dovetail gently and guide it all the way to stop and lock it.
- 27) Close the hood
- 28) Exit the software and the window.
- 29) Make sure you have entered your sample, tip information and any error you encountered in the logbook.

In case you encounter any error, report any error as:

- Write in logbook
- Take screen shot and save into edrive AFM-Issues folder (Figure 21)

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AFM_ISSU	ES		2/20/2023 10:35 AM	File folder
AFM_SERV	ICE		2/6/2023 11:26 AM	File folder
Capture			2/23/2023 11:41 AM	File folder
DATA			2/6/2023 3:50 PM	File folder
DI3000_AF	M_SOP		2/16/2023 11:17 AM	File folder

Figure 21: Saving error