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^2 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)

3) Inspect the Cantilever holder (Figure 2) using the stereo microscope. Clean it using toothpick and isopropanol and spray Nitrogen gun if required.
4) Mount the AFM cantilever probe into the groove.

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

8) **Focus the tip of the Cantilever:** Click the tip icon on the tool bar and bring the tip in focus using rolling ball (Figure 6). Bring the tip to center of

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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 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)

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

![Figure 10: Tip reflection and laser on tip](image)

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 $+0.1$ V 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 μm
- **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”

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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 control:**

SPM feedback: **amplitude** (Figure 14)

**Amplitude set point:** Start with 1.5 volt. The set point regulates the force on the probe

**Integral gain:** usually use low value <0.6

**Proportional gain:** Insert number by multiplying 1.5 to the value of integral gain

**Other controls** (Figure 15)

**Microscope mode:** select “tapping”

**Z limit:** make sure it is larger than 5um

**Units:** metric

**Engage setpoint:** 1.00

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

- When tip is engaging onto surface, the image of tip can be visible on the monitor (Figure 16). Do the following to maximize feedback control parameters:
• 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

17) Scan one frame prior to recording the image to adjust scan size, scan speed, and date scale (Figure 13, 14 and 17).

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

![Figure 19: Tip Withdraw](image)

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)

![Figure 20: Saving File](image)
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 e-drive AFM-Issues folder (Figure 21)

![Figure 21: Saving error]