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**)

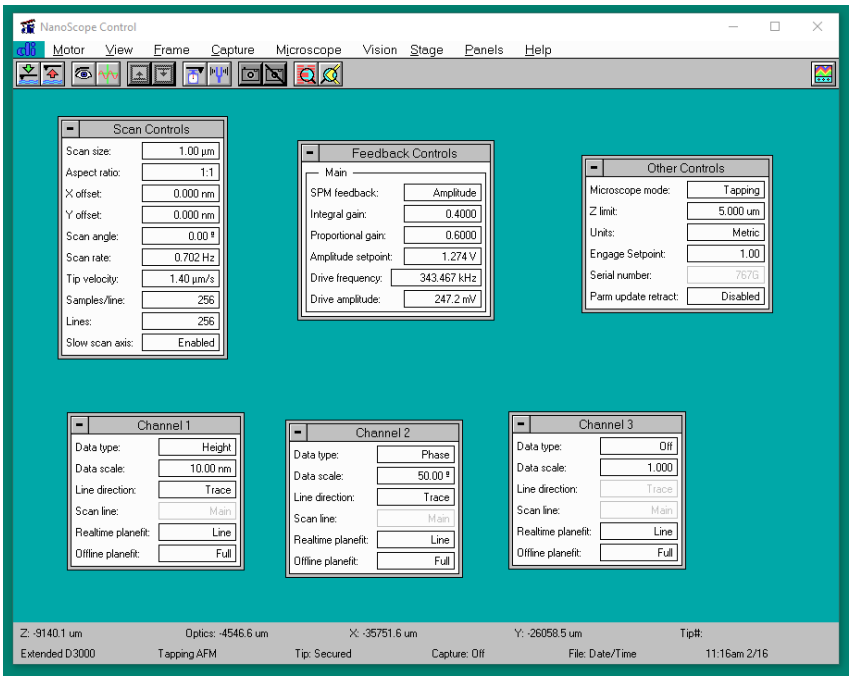

Figure 1: Software Control

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.

Figure 2: AFM cantilever holder with cantilever inserted

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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**)

Figure 4: Scanner lock screw

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).

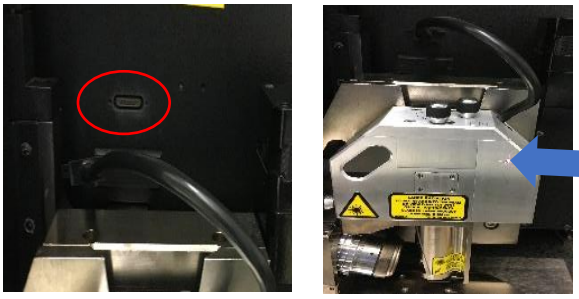
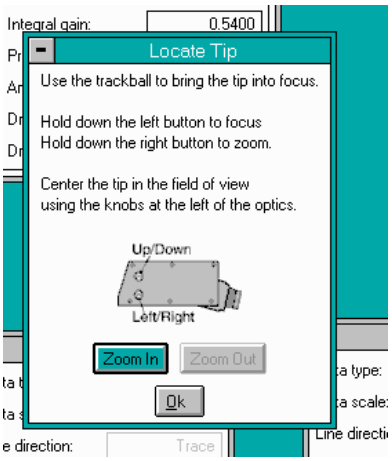
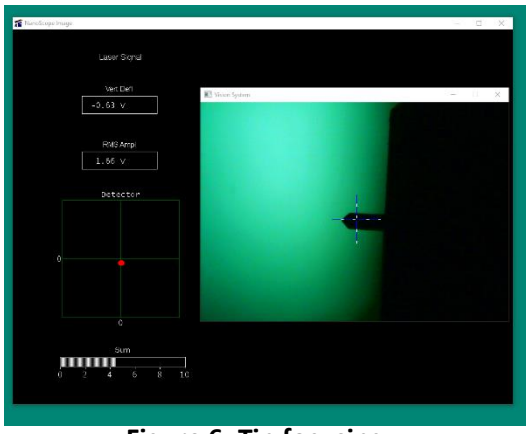


Figure 5: Scanner plug and laser check

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





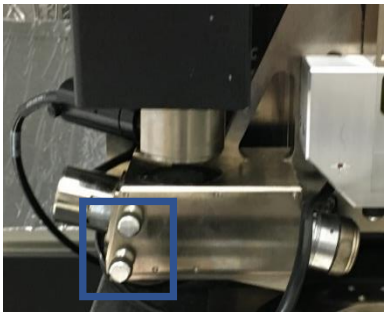


Figure 6: Tip focusing

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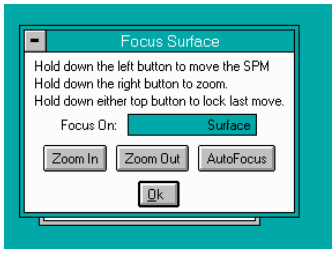
screen using the two knobs on the side of microscope (**Figure 6** blue rectangle). Click **OK.**

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

Figure 7: Laser Alignment

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)**.



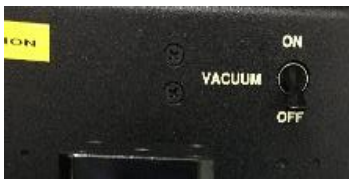
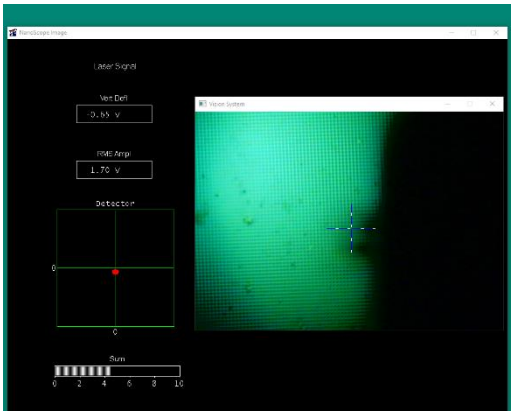


Figure 8: Focus surface option

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**)



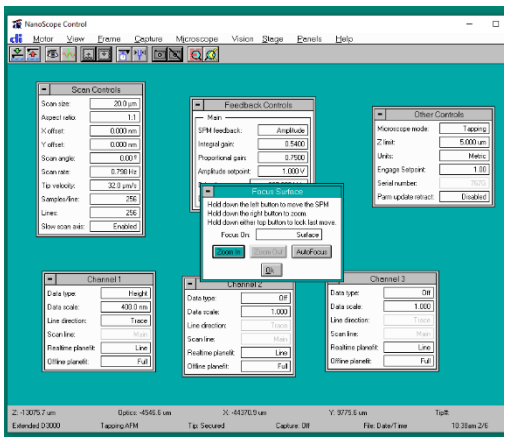


Figure 9: Focused Surface

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

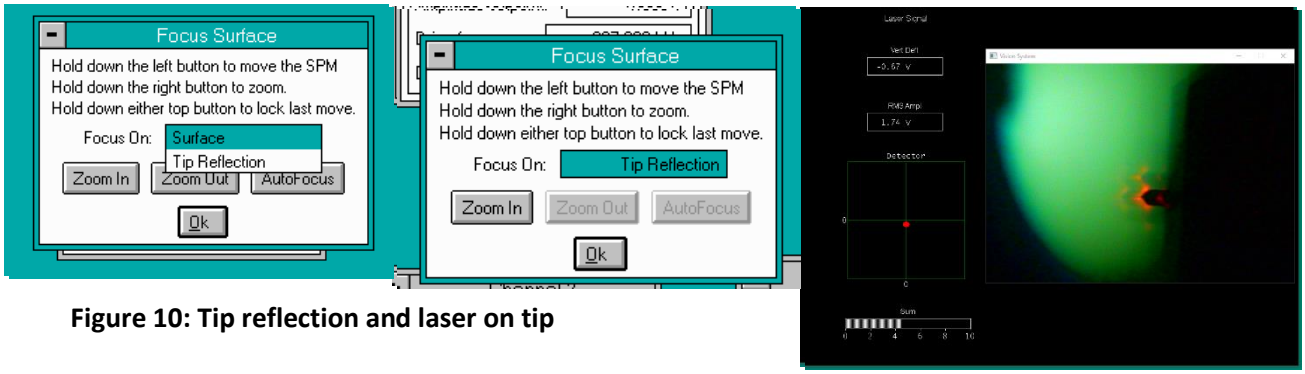


Figure 10: Tip reflection and laser on tip

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

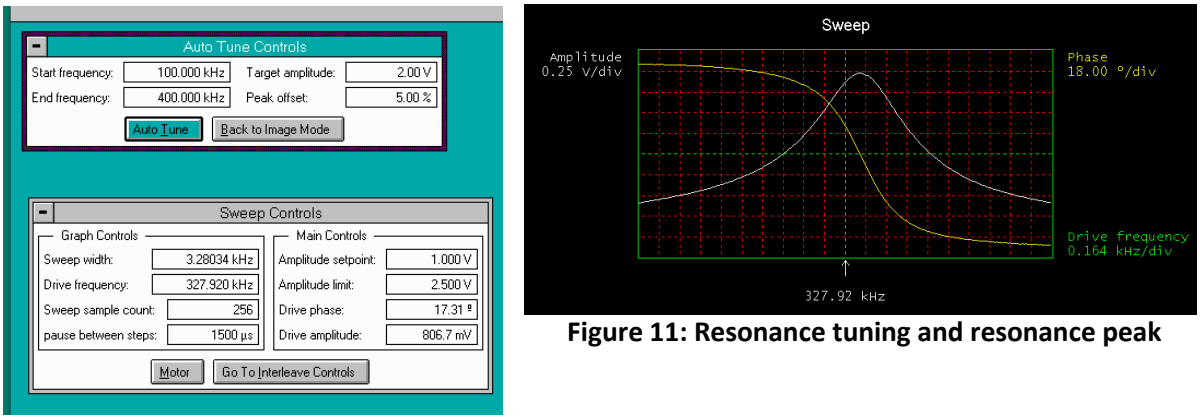


Figure 11: Resonance tuning and resonance peak

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)

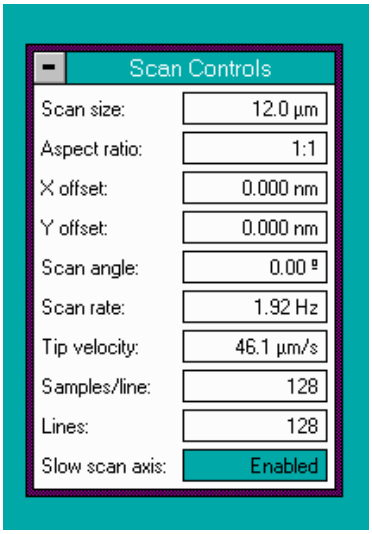


Figure 12: Scan Parameters

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"

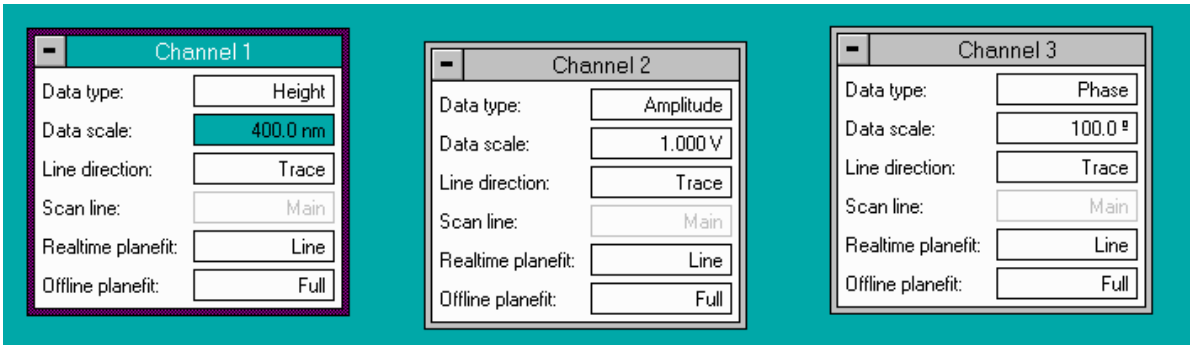


Figure 13: Three Chanel parameters

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

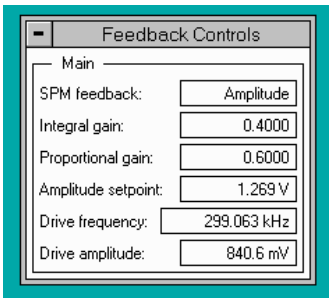


Figure 14: Feedback control

Other controls (**Figure 15**)

Microscope mode: select "tapping"

Z limit: make sure it is larger than 5um

Units: metric

Engage setpoint: 1.00

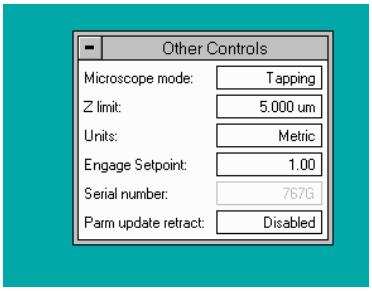


Figure 15: Other Control

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

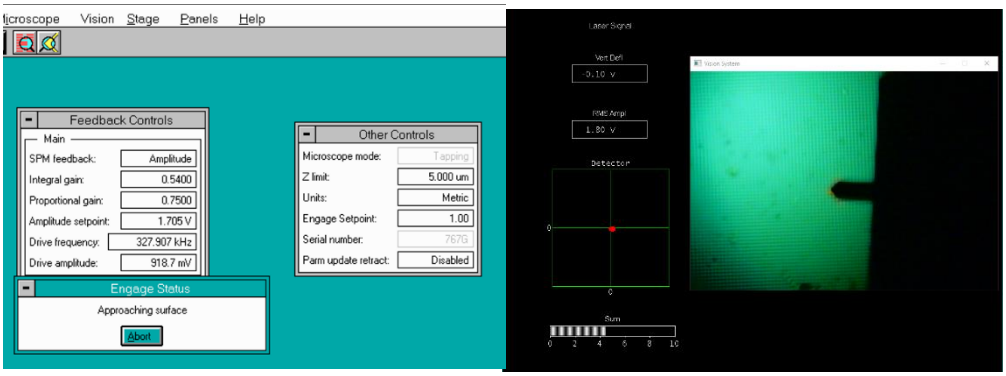


Figure 16: Tip Engaging and Approaching to the Surface

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

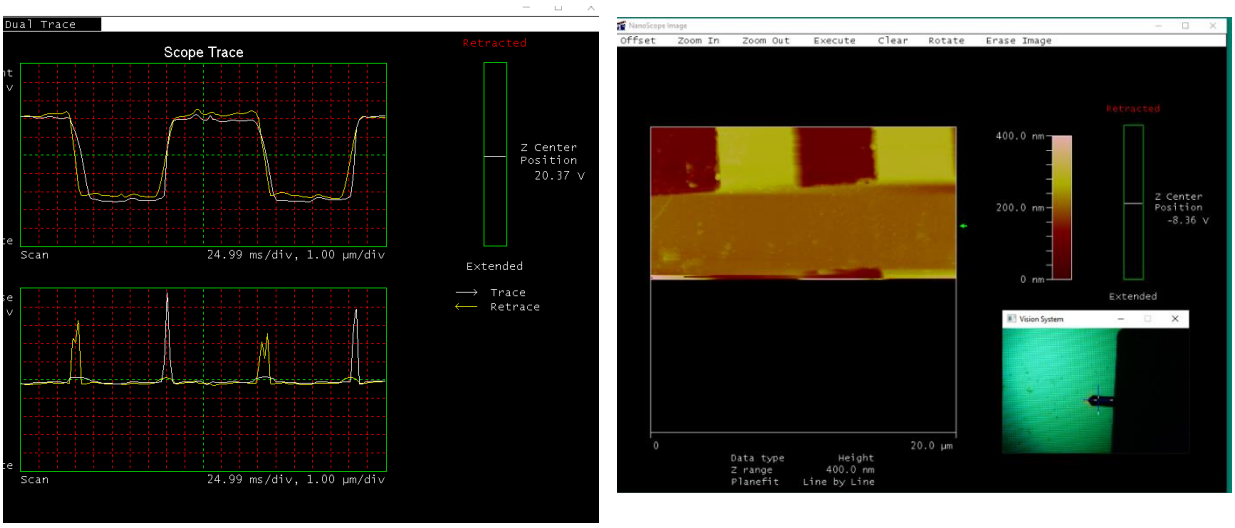
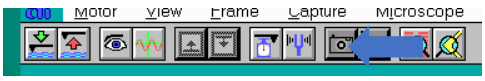


Figure 17: Waveform and Scanning on the Surface



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

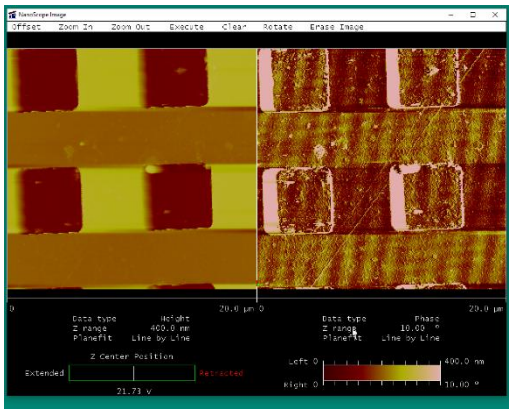


Figure 18: Scanned image of standard sample

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**).

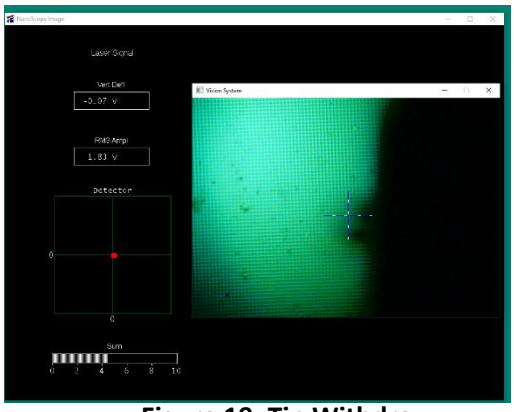


Figure 19: Tip Withdraw

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)**

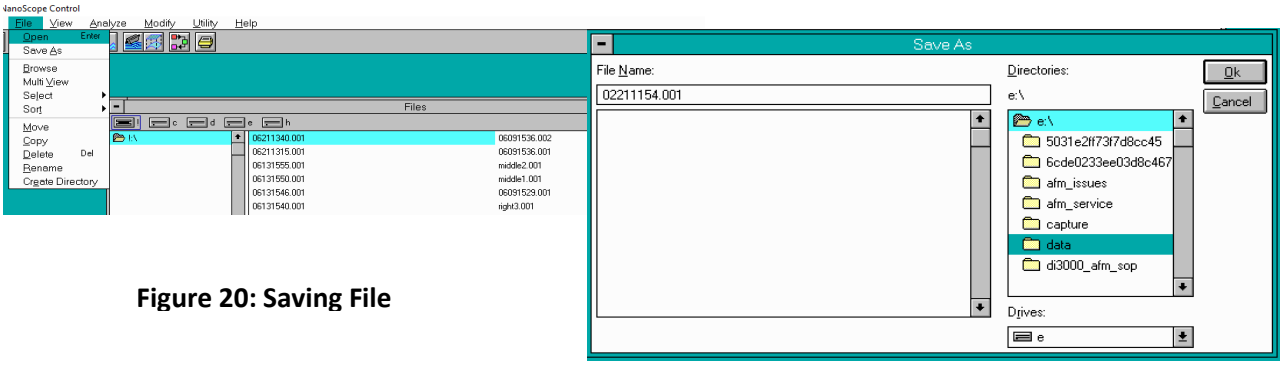


Figure 20: Saving File

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**)

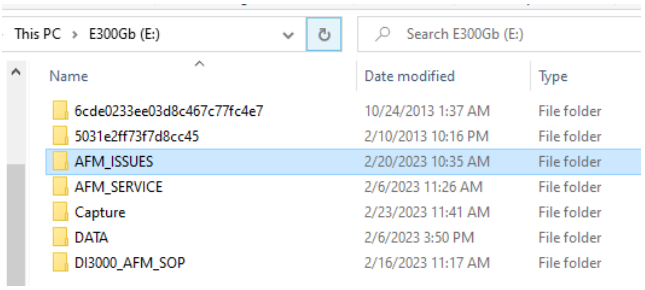


Figure 21: Saving error