

Instructions for easyScan Scanning Tunneling Microscope

(revision 1 April 2015)

NOTE: Instructions assume software is pre-configured per "UVA Instructor Guide for easyScan 2 STM and AFM"

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1) Prepare the sample and sample holder (if not already done by instructor):

- Magnetically attach the HOPG sample holder to sample cylinder

Be careful to handle the cylinder by the black plastic handle ONLY. Any time the cylinder is touched, the temperature will increase enough to make repeated measurements difficult.

Rule of thumb: a change of 0.1°C will expand the cylinder several nanometers!

- Clean sample cylinder by wiping LENGTHWISE along bottom of sample mounting cylinder with clean DRY cloth.

VERY IMPORTANT: Should the sample mounting cylinder later not advance properly, wipe with a fresh cloth dampened with isopropyl alcohol followed by a clean DRY cloth. Use a small swab wetted with alcohol to carefully clean the coarse-z drive cradle. More severe contamination is removed by a cloth wetted with TCE followed by a cloth wetted with methanol. Avoid getting TCE onto the plastic handle.

- Holding only by its black handle, pull sample cylinder back away from the probe position

- Verify that any previous probe tip has been removed from the STM

2) Start up the electronics (if not already done by instructor):

- Turn on the control electronics (long hexagonal black box)

All indicator lights on controller should briefly blink on and then off

Leaving only "STM Scan Head" (and possibly "Probe Status") lights on

- After controller lights have settled, click PC's "Nanosurf easyScan2" icon.

WAIT for "Controller startup in progress" pop-up box to complete the connection with the STM

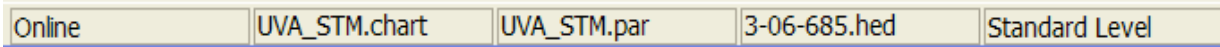
If ANY error messages appear, notify the instructor or TA!

Also inform the TA if the "Probe Status" light on the controller is bright red

3) Start up the software and verify its proper setup (ABSOLUTLEY CRITICAL!):

4a) Click Menu: File / Workspace / Load and select file "UVA_STM.gui"


By this point the bottom margin of the program must have these entries:



The numbers in the 4th entry (##-##-###.hed) MUST match the label on top of the STM

If these entries are wrong, the software is confused as to whether it is controlling an STM or AFM, or it lacks vital calibration and control data - ASK THE INSTRUCTOR TO FIX!!

4b) Verify these settings: Set point = 3 nA P gain = 1000
I gain = 200 Tip voltage = 50 mV

(On screen, at lower left - If not visible click the "Z-controller Panel" icon: )

4c) Click on top right graph in "Imaging Window" (icon: )

Verify that it is labeled "Topography - Scan Forward" AND that the program's top menu bar now includes "Color Map" / "Line Fit" / "Topography"

4d) Click on bottom right graph in "Imaging Window"

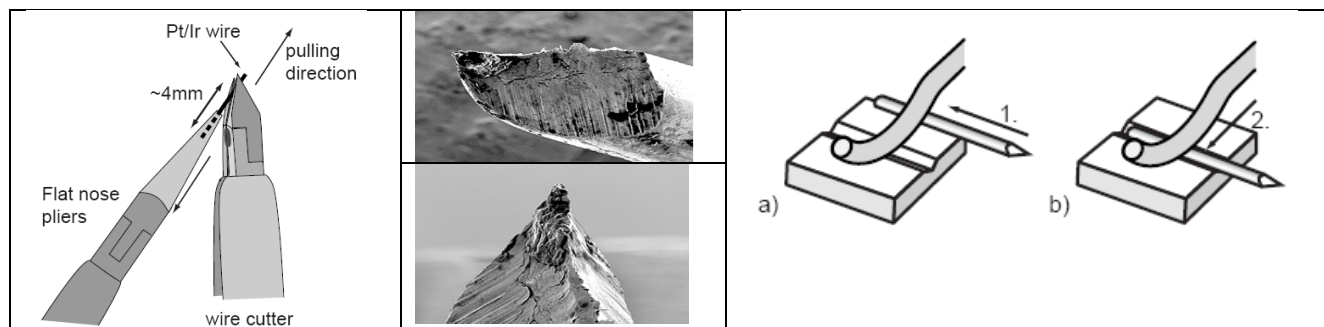
Verify that the program's top menu bar now includes "Line graph" / "Mean Fit" / "Topography"

4) Prepare the iridium wire probe tip (manual pages 23-25):

3a) Cut fresh Iridium wire probe tip - THE SINGLE MOST CRITICAL STEP

- Use technique illustrated below and in manual.
- If you get a "Batman" dual pointed tip, try using it: one tip is almost certainly longer.
- Final tip should have a total length of **5mm long or slightly less**

3b) Load tip into STM, pulling SIDEWISE under gold wire spring (below, right) GENTLY!!



5) Roughly position the HOPG sample cylinder with respect to the probe tip:

5a) Rotate the sample cylinder to optimize your view of the sample's center region

On the graphite sample it's OK if edges of flakes stand up a bit. But such flakes must not obscure your ability to observe the reflection of the approaching iridium probe tip. (If they do, rotate the sample cylinder or have instructor prepare fresh graphite surface)

5b) Move the sample forward until its leading edge rests up on the piezoelectric drive cradle

DO NOT JUST PUSH! The cylinder must ALSO be tilted to get up onto the cradle

5c) Now move the cylinder forward until the probe ALMOST touches the sample surface

If your hands are steady enough, **try to get the tip within 1mm of the sample surface, at which point you should see the reflection of the tip in the sample.** BUT DON'T COME SO CLOSE THAT THE TIP EVER TOUCHES THE SAMPLE!

Watch the "Probe Status" light on the black controller box. **IF THIS LIGHT TURNS RED: You have contacted the sample, and must make a new probe tip!**

5c) Place plastic cover on STM, positioning lens so probe tip and sample are visible

6) Understanding the software "Positioning Window" (invoked by icon:):

- If necessary resize until you can read full wording of the buttons:



Meaning of these buttons:

Coarse manual adjustment (YOU moving the sample, fast, but extreme care required):

Advance = Fast movement of sample toward tip (as long as button is depressed).

Retract = Fast movement of sample away from tip (as long as button is depressed).

Final fine and slow automatic movement of the HOPG sample:

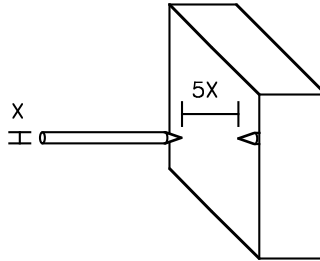
Approach = Slow final movement of sample to tip UNTIL electron tunneling is sensed.

Withdraw = Slow movement of sample away from tip by a fixed distance.

7) Bringing the STM probe to the graphite surface:

7a) Manually approach by holding down the  button:

Through the magnifying lens, get the probe tip and its reflection to within about 5 wire diameters:



BE CAREFUL to watch the tip and sample as you hold the button or you will “crash” into sample

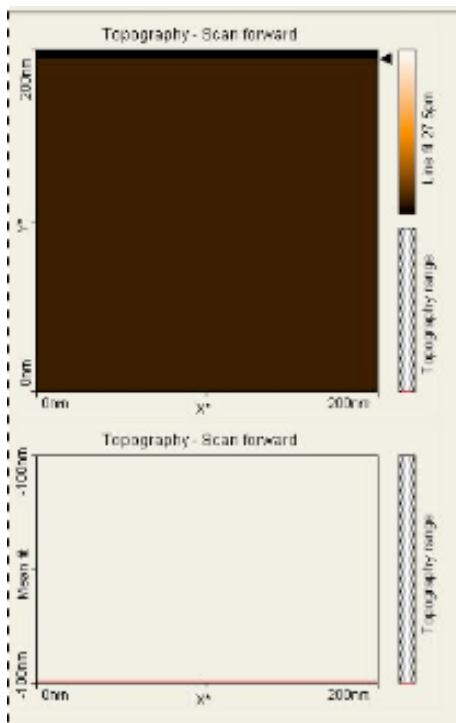
If the tip and sample do touch, the “Probe Status” LED on the controller will turn RED indicating you have crashed the tip. If this happens you MUST go back and cut a new tip

7b) Complete approach automatically by clicking  button

Approach takes 5 -10 minutes – BE PATIENT. Check occasionally to be sure sample has moved.

NO MOVEMENT: If absolutely no movement is seen over ~5 minutes, RETRACT the sample cylinder and ask the instructor to re-clean sample cylinder per step 1.

FALSE APPROACH: Software says approach is complete but you are getting only a uniform black or dark orange image with the scan line below at absolute top or bottom of scale:



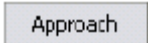
During approach, the STM is “listening very hard” for the beginning of electron tunneling to/from the sample.

Indeed, you told the STM to stop when it “thinks” it sensed 3nA of such current.

But 3 nA is an awfully small current and pops of electronic noise can sometimes be this large.

If such a pop does occur, following your orders, the STM will stop its approach.

If this is what happened (“Cause #1”), the solution is easy:

- Hit the “STOP” button (above the image) to stop the scan
- Only a few seconds later by hitting  button

The STM should then proceed until true electron tunneling begins.

LESS LIKELY CAUSES OF A FALSE APPROACH (and their solutions):

Cause #2) If you get this screen almost IMMEDIATELY after you START the approach, it may be due to current leakage through a dust particle. Try blasting it away by hitting "cleaning pulse" button in Z-Controller Panel.

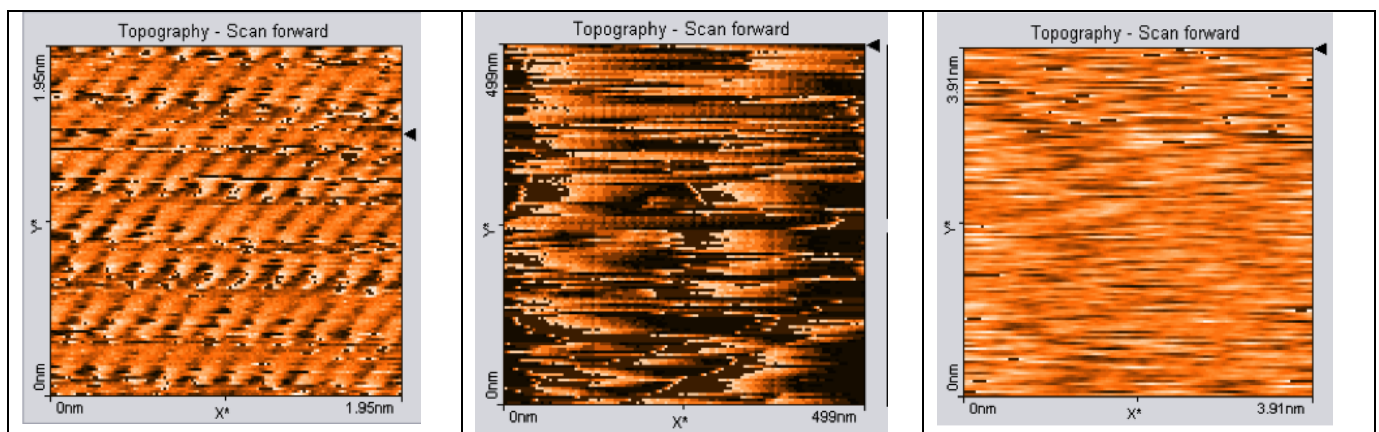
Cause #3) The current sensing software works more poorly at higher approach speeds: To slow the approach, click on the **Approach Options** bar. In the drop down box, reduce the approach speed setting by 5%. If this does not work, reduce the speed by 5% one or two more times. This will generally solve the problem (but require a longer wait for the probe to make contact with the sample).

Cause #4) A final possibility is that STM has the wrong calibration files or corrupted calibration files: Go back to check step 3.

NOTE: EVEN AFTER BEGINNING GOOD SCANS, THE STM CAN JUST LOOSE CONTACT WITH THE SAMPLE. Solution is same as above: Hit "Stop" / Hit "Approach"

STOP: Images like those below - You are in trouble

Persistent streaky low quality images resembling:



This can happen if electrons are simultaneously tunneling from multiple places on the tip. It can occur when there are multiple active tips (e.g. after a good tip was blunted by crashing into the HOPG surface), or when a single tip has accumulated some sort of contamination.

Rare solutions (last ditch efforts to save a tip by burning off contamination / secondary tips):

- In the **Tip Properties** Panel: Click the "Cleaning Pulse" Button.
- In the **Z-Controller** Panel, briefly change the Set Point to 20 nA.

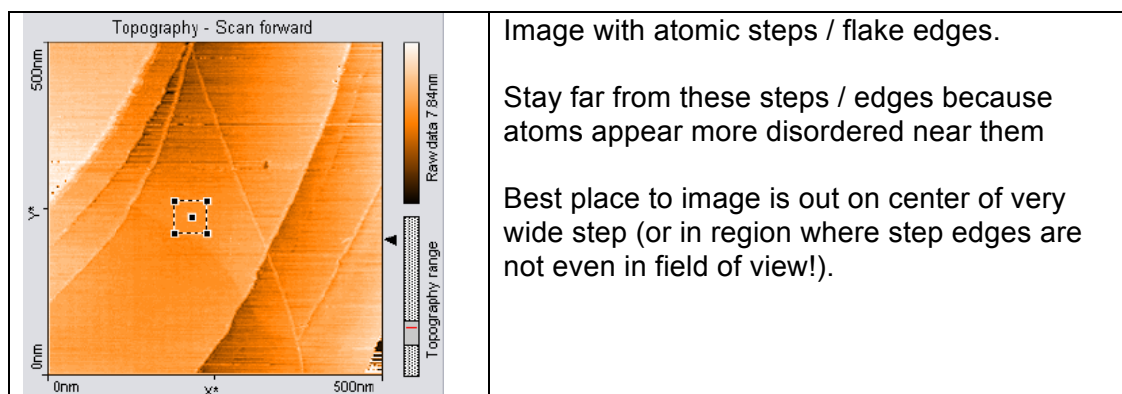
The more common solution: Cut and install a new tip (!@#@\$@!#!#@\$!!!!)!

GO: Images like that below - Find an atomically smooth region

8) **CHANGE TUNNELING CURRENT TO WORKING VALUE:** Setpoint = 1 nA

9) **Seek out an atomically smooth region of the HOPG surface:**

At low magnification (e.g. image size of 50 - 100 nm), look for straight lines that might be flake edges, or very large steps of many graphite layers. These edges are usually surrounded by imperfect HOPG surface. To clearly image atoms, you must avoid these regions.



ALTERNATIVE A - The center region of your image is free of steps: Jump to the next page

ALTERNATIVE B - Your image is FULL of unavoidable steps:

MOVE by clicking the developing image in the “Topography – Scan Forward” pane.

- Then click the MOVE button immediately above the image.
- Click on image and draw an arrow giving the direction and distance you wish to move
- Finally, under the “Tool Results Panel” on the left, click the MOVE button

ALTERNATIVE C - One part of your image is free of steps (e.g. lower left corner):

ZOOM into a portion of your current image that is flat and free of steps

- Click on the developing image in the “Topography – Scan Forward” window pane
- Click the ZOOM button immediately above the image
- Click and drag a box around the area of interest
- Under the “Tool Results Panel” on the left, click the ZOOM button

Looking for atoms in an atomically smooth region

10) Examine your image and line scans:

To the left of the image is a box labeled "**Image Size**"

It starts with value of 0.2 microns = 2000 nm. A rough rule of thumb is that 1 nm ~ 10 atoms.

Thus your starting image is about 2000 atoms across, so you will not be able to see atoms yet.

But you do want to keep verifying that no steps or particles have crept into your imaging area

The line scan (below the image) also tells you a LOT about the operation of your tip.

Ideally, at this point those scans should be steady, straight (but possibly sloped) lines

10) Begin to magnify your image:

- To the right of the "**Image Size**" box, click its down arrow
- This decreases the width of the image by two, thus doubling your magnification
- WAIT UNTIL AT LEAST ONE UP AND ONE DOWN SCAN ARE COMPLETED

11) Magnify in slow steps down to a 1-5 nm image size (~ 10-50 atoms across)

With EXTREME LUCK atoms will now show as bumps about 1/10 th nanometer across

If atoms appear, proceed to the page on "Capturing and Saving Images" below.

ALTERNATIVE TECHNIQUES FOR HIGHLIGHTING ATOMS

IMPORTANT:

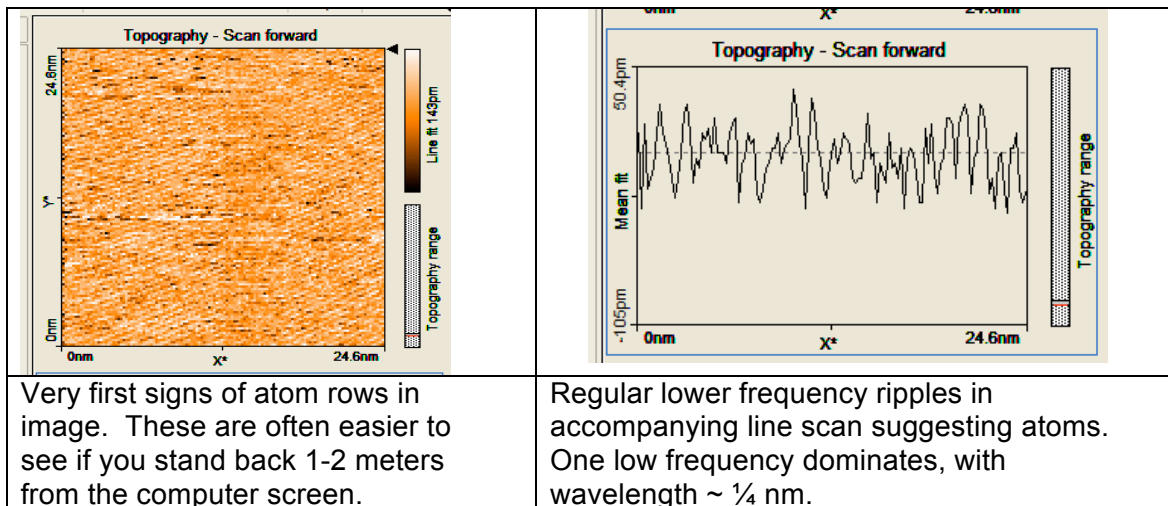
- Sometimes the techniques below are spectacularly effective; sometimes they seem to do nothing.
- Frantically applying multiple techniques will usually get you nowhere

SO SLOW DOWN! MAKE SMALL CHANGES (factor of 2 at most in one step)!

WORK THROUGH ONE TECHNIQUE FULLY BEFORE TRYING ANOTHER!

Assuming you have not yet seen atoms, demagnify out to a 10-25 nm image size

You will be looking for very faint lines in the image OR regular oscillations in line scan below the image



TECHNIQUE 1) Help the software to electronically "level" the sample:

- 1a) Make sure the "**Rotation**" box has a value of 0°
- 1b) In the Imaging Window, click on the Line Scan pane and observe the vertical axis limits
- 1c) Figure out the total span of the vertical axis on the line scan

- In line scan above it is $105 \text{ pm} + 50.4 \text{ pm} = 155.4 \text{ pm}$

IF YOUR NUMBER IS LESS THAN 1000 pm, you are fine, skip ahead to another technique

- 1d) OTHERWISE: Adjust the "**X-Slope**" value by $\pm 0.25^\circ$ until the line scan becomes relatively flat OR the vertical limits are down to at least a picometer scale

- 1e) Then change the "**Rotation**" box value to 90°

1f) Adjust the "**Y-Slope**" value by $\pm 0.25^\circ$ until the line scan vertical limits are down to at least a picometer scale

1g) Then change the "**Rotation**" box value back to 0°

TECHNIQUE 2) Rotate the direction of your line scans

- Change the "**Rotation**" box value by 30° to 45° (exact value seldom matters)

TECHNIQUE 3) Optimize the line scan time

For image sizes of > 10 nm, keep the "**Time/Line**" box value at 0.2 sec

For image sizes of 10 nm to 3 nm, change the "**Time/Line**" box value to 0.3 sec

For image sizes of < 3 nm, change the "**Time/Line**" box value to 0.4 sec

TECHNIQUE 4) Collect different number of points: Try **Points/Line** values of 256 (or 64)

TECHNIQUE 5) Alter feedback loop values (seldom required for newest software):

- Change only one parameter at a time (by a factor of ~ 2) then wait approximately one half-scan up or down to observe image change

- If there is no positive change, try changing that parameter a factor of 2 in *other* direction. Keep that value if it improves the image, abandon it if no change or if image worsened

- Move to another parameter and cycle through its values

- I-gain (often the best parameter to play with) controls how the signal responds to longer changes in the surface such as a gradual incline. Too little and the scan may go black or white; too much and the tip may crash.

- P-gain controls how fast the signal responds to small/fast changes in the surface. Too low and the surface looks flat; too high and the surface looks the same at EVERY scale!

Typical settings for successful HOPG observation:

Image: Size 3 -10 nm (less than 3nm is possible if you have a heroic tip)

Number of Points = 64 - 256

Scan Time = 0.05 – 0.4 seconds

Z-controller: P- Gain = 100-2000

I – Gain = 200-2000

Current set point = 0.3 - 2 nA

Capturing and Saving Images:

DURING MEASUREMENT, CAPTURE THE IMAGES:

- 1) During any measurement, click on the “Topography – Scan Forward” window
- 2) Click on the “Photo” button immediately above/right of this image (it will turn blue)

When scan reaches the top or the bottom, a new window with that saved image will pop up

Program will continue saving images as long as "Photo" button is blue

For now, simply minimize these new windows (dropping them into the background)

AFTER MEASUREMENT, SAVE THE IMAGES TO FILES (TWICE!):


- 1) To save your data to a file, click on its minimized window:
- 2) First save ALL of the measurement information:

Go to FILE / Save (as an "nid" Nanoscience Inc. format file)

- 3) Then save a bitmap image (which **cannot** later be reprocessed, e.g. in 3D):

Go to FILE, then EXPORT, then CURRENT DOCUMENT AS bmp, png or tiff file

Ending a Measurement

- Click “Withdraw” button to automatically pull back a little ways from sample. Stop and then:
- Click  to then pull farther back

Final Notes

- Return all tweezers and tools to the box at the appropriate station before leaving
- Immediately notify an instructor of any damage to the equipment
- Under no circumstances are you to modify or enable hardware/software settings to access the Internet

Image Processing

Using the "NanoSurf EasyScan 2" software - for a crude 3D map of your image:

- 1) Click on the image window
- 2) On the top outermost program bar, click "Line Graph" icon and switch "3D View"
- 3) Rotate 3D view as desired
- 4) Under the Tools menu you can also apply "Glitch" and "Noise" filters to smooth the data
- 5) Go to FILE, then EXPORT, then CURRENT DOCUMENT AS in the format you want

Using the "NanoSurf Reports" software - for a FAR BETTER 3D map with noise filtering:

This is a very powerful program but it came with no instructions, its interface is unconventional, and that interface was apparently written in another language and then very clumsily translated into English (with often weird choices of English words). So it takes a bit of persistence to make it work for you.

In the program, you call up an STM data file (*.nid). You then select filters/operators to do things like level the image, filter out noise, even up rows or columns, cut out low or high points, convert to 3D, color ...

For most of the filters/operators, a large dialog box will appear offering you alternative adjustments and giving you a preview of what the filtered image will look like if you go ahead and click OK.

If you DO click OK, the program then generates one or two NEW images at the very bottom/right edge of the page.

If two images are created, one is the new corrected image. The second new image is what was subtracted from the original image to make the new image (e.g., if you filtered out noise, the second new image will be a map of the noise signal the program subtracted out).

Your NEXT choice of filter/operator will be applied to whatever image is now surrounded by a dashed box. MAKE SURE this is your new/improved image and not the second image with the subtracted/discarded data (if not, click on your improved image to surround it with the dashed box).

You can also use this dashed box selection technique to go back up on the page to an earlier image and process it. But if you do, know that its newly processed images still appear at the very bottom of the page.

To simplify tracking of all your images, click **Bank / Show the Bank of Studiables** and the program will list all of your images as icons in a vertical column at the right of the page.

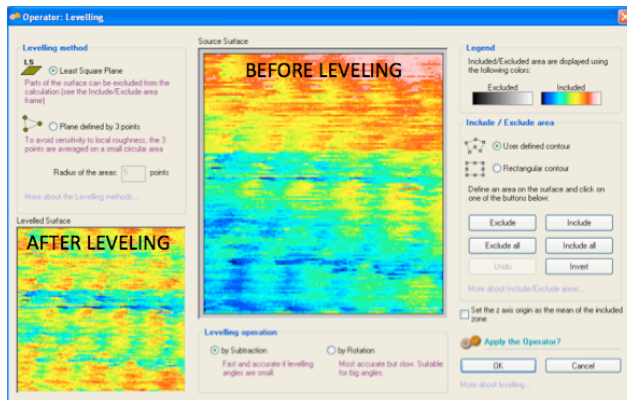
Nevertheless, to avoid confusion, **I strongly recommend that you work with only one data (*.nid) file at a time.** When you are completely done with it, and have saved everything you want, clear everything by exiting and then re-entering the program.

Suggested filter/operators are listed (in suggested order of application), starting on the next page:

Load your nid format file into program **Nanosurf Reports** by clicking **File / Open a Studiable**

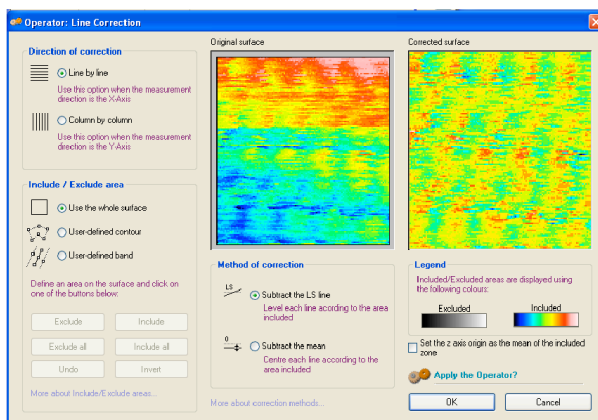
Clean up your image by eliminating features believed to be instrumental artifacts:

- Apply **Operators / Leveling**: Use this to level an image higher at one edge (i.e. different color / lighter)



- Apply **Operators / Line Correction**: Use this if some rows (or columns) of atoms appear higher or lower than others, or if there are vertical (or horizontal) streaks in you image.

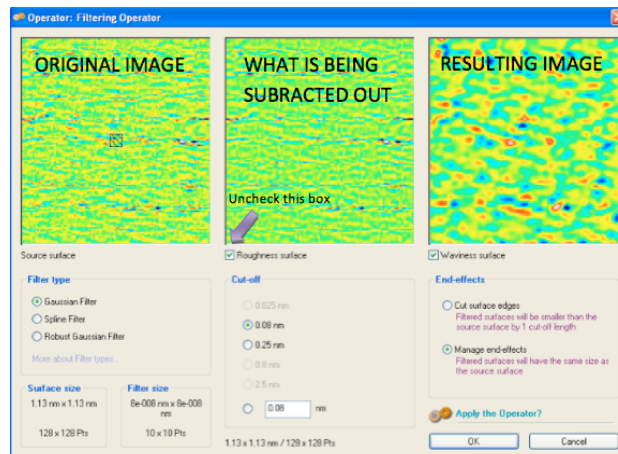
You have to select horizontal (“line by line”) or vertical filtering (“column by column”):



You can also (sort of) filter out diagonal variations by using both line correction options. First select the line by line option. Then, after applying that filter and producing a new image, on that new image apply the column by column option.

- Apply **Operators / Filtering**->**Waviness + Roughness**: Use this if (as is likely) there are a lot of bumps in your image that are much smaller than atoms (0.1nm). You can apply what amounts to a noise filter to smooth out those bumps:

When you select this filter, a window with three images will appear. The far LEFT image is your current image, the far RIGHT image will be the final image. The middle image is what will be subtracted from the starting image. As we will not need to keep the subtracted image, uncheck the box below it.



NOTE: This filter has a programming glitch: After you enter different numbers in the boxes (see above) DO NOT THEN HIT ENTER/RETURN as this mimics the "OK" button and applies the filter. Instead, type in the number and just wait a few seconds and the images will adjust themselves.

USING THIS FILTER: We expect atoms to be about 0.1 nm in size. So anything smaller should be noise, and can be thrown away. To do this we want to set the cut-off for 0.08 nm. You can do this in two ways:

If the .08 nm option is available, select it.

If not, enter .08 in the box below and select its option. DO NOT HIT RETURN

CHECKING YOUR RESULT: Note the real world width of your image – it is called out at the lower left of the window (above). It will usually be given in nanometers, so multiply it by 10 to estimate how many atoms you expect to see in your final image. For example, with a 6.5 nm (wide) image, you expect to see approximately 65 atoms across.

Look at the far right image to see the effects of the selected cut off filter and see how many bumps there are across the image.

If you count FEWER atoms than you expected, decrease the cut off filter value

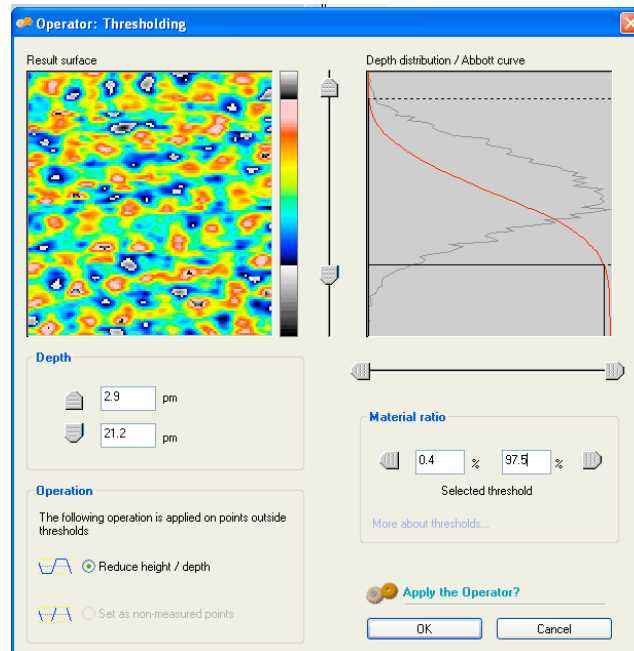
If you count MORE atoms than you expected, increase the cut off filter value.

When you are happy with your image, make sure the Waviness surface box is checked, then hit OK.

- Apply **Operators / Thresholding**: Use this to chop off noisy (spiky) peaks or valleys in the data:

To chop off peaks, move the left horizontal slider inward (rightward) a small amount (up to 1.5%)

To chop off valleys, move the right horizontal slider inward (leftward) a small amount (up to 3%)



Having now cleaned up your image, apply tools to accentuate its features:

- Click **Studies/ Continuous Axonometric** to create a rotatable/zoomable 3D version of your image:

- Click OK in the window that pops up

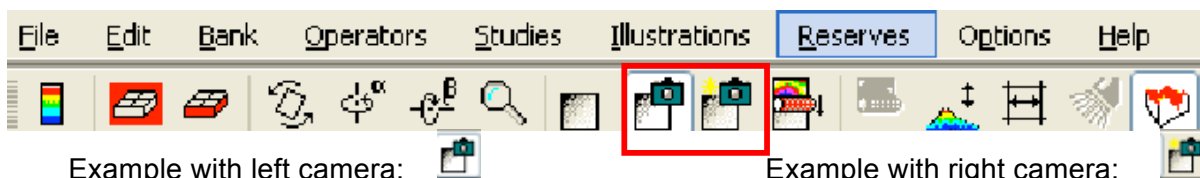
- Rotate 3D plot as desired

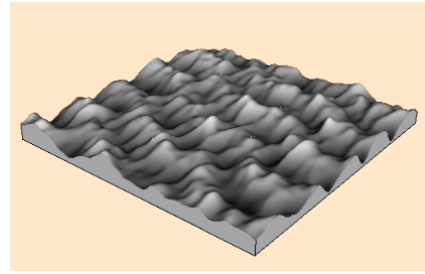
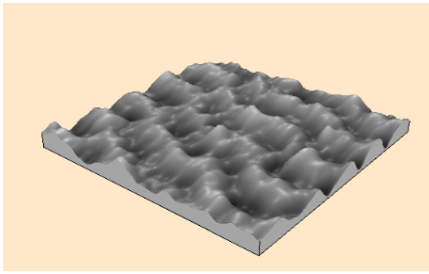
- To control the heights in this 3D image, adjust **Height Amplification** (rainbow colored volcano icon, below "Reserves" and "Options")

- To select alternate color pallets for your sample, click far left icon (under File on the menu bar)

- To change the background color click the 2nd to the far left icon (under File on the men bar)

- The two photo camera buttons will add or subtract black highlights of the elevation and contours. Choose the one you prefer.

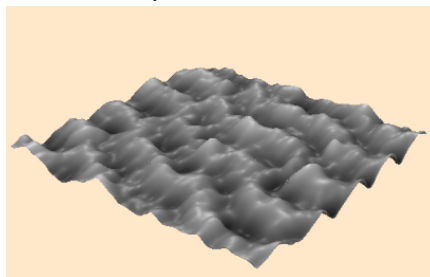




- In order to remove the sides of the axonometric view, making it a free standing surface, unclick the button with the red topography on top of the black box, titled “Show bulk”



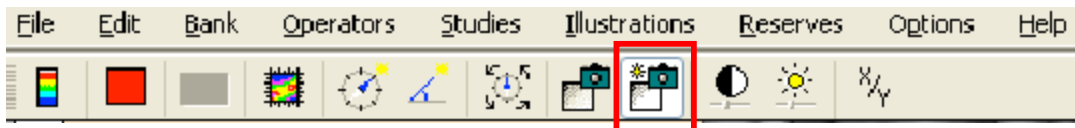
Example with  OFF:



- Click **Studies / Photo Simulation** to create a microscope-like image (which emphasizes the hexagonal carbon structure).

In order to make something that acts like an “animated GIF” (but which, unfortunately, cannot be saved):

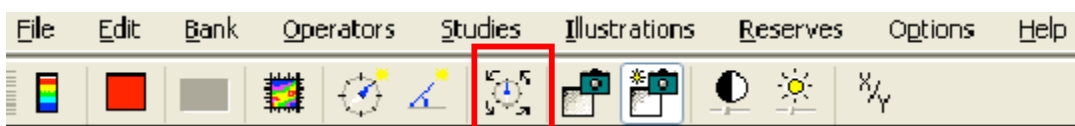
-Select the second photo button with the sun-like image in the top left corner



-Change the color palette to whatever you desire



-Select the icon resembling a timer with arrows around it



SAVING YOUR IMAGES (FOR YOUR REPORT):

Then to save EVERYTHING (in a complete but proprietary format):

- Click **File / Save the Document** (mmt format file)

Finally, to export you favorite images in a common graphics format, for each of them:

- Click on the image (to box it in with a dashed line)
- Click **Export Reserve Image** (choose tiff, gif, jpeg . . .)



UVA's Hands-on
Introduction to
Nanoscience