Procedure for Friction versus Load (FvL) data analysis

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I. Calibrating FvL data- Calibration experiments

A. Lateral calibration

(method according to Ogletree et al., Rev. Sci. Instrum. 67, 9 (1996))

1. **After performing FvL experiments, the tip should be scanned across a lateral calibration grating. This sample has long parallel ridges of well-defined crystallographic orientation, such as a $SrTiO_3$ sample or MikroMasch's TGG01. The TGG01 works best for all types of tips, even tips with a large radius.**
2. **Scanning an image with load variation so that at least 2 sloped facets (one up, one down) can be seen during the scan. Scanning at loads between -F_{PO} and $2F_{PO}$, where F_{PO} is the pull-off force, is sufficient.**

B. Normal calibration

(method according to Sader et al. Rev. Sci. Instrum. 70, 10 (1999))

1. **Determine the resonance frequency of the cantilever.**
   a) Measure amplitude of oscillation of the cantilever as a function of frequency of forced oscillation. Find the first significant resonance peak. This should correspond to the first resonance of the cantilever. Record the frequency at which the amplitude is a maximum within this resonance peak.
   b) Double check this frequency with that which is given by the manufacturer.
      (1) Your measured frequency should be in the same range as the manufacturer's value.
      (2) If not, you may have found a harmonic of the resonance frequency. Change the frequency range so that this resonance frequency may be found.
2. **Determine the Q factor of the cantilever.**
   a) Some software programs can calculate Q automatically.
   b) If it is not calculated automatically, record the resonance peak to be imported into a graphing software package (e.g. KaleidaGraph).
      (1) Determine the full width at half the maximum.
      (2) $Q = w/\delta w$, where w is the resonance frequency and δw is the full width at half maximum of the resonance peak.
3. **If possible, record ambient temperature and relative humidity, during the above steps.**
4. **Measure in-plane dimensions of the cantilever.**
   a) Use an optical microscope with a calibrated measuring device to determine the length and width of the cantilever.
   b) Be careful to measure the length from the appropriate base of the cantilever; this position is not always obvious, and it may be necessary to check with the manufacturer (or obtain a side-view image in an SEM or TEM).

II. Analyzing FvL data

A. Download the following files from http://mandm.engr.wisc.edu/faculty_pages/carpick/toolbox.htm :

friction_v_load.m, get_image_data.m, di_header_find.m, extract_num.m, batch_process.m

B. Normal calibration

1. Determine the normal spring constant of the cantilever.

a) Go to http://www.ampc.ms.unimelb.edu.au/afm/calibration.html

b) Input values for the resonance frequency, Q factor, and the in-plane dimensions of the cantilever.

c) The program will calculate the normal spring constant of your cantilever, in N/m. If you know the torsional resonance properties, the section at the bottom will calculate the torsional spring constant.

2. Determine the normal sensitivity of the cantilever.

a) From force distance curves measured on a stiff surface, such as silicon or diamond, calculate the normal sensitivity of the cantilever (i.e., the slope of deflection voltage vs. z displacement). Some AFMs calculate this slope for you from a force distance plot.

b) The normal sensitivity, S_N, is basically the conversion factor between the number of vertical nanometers traveled by the piezo and the change in volts measured by the movement of the laser spot on the photodiode. Units: [nm/V]

C. Lateral calibration

1. Take all friction vs load files, including friction calibration images, and place them in the same file folder as *friction_v_load.m*, *get_image_data.m*, *extract_num.m*, and *di_header_find.m*.

2. Open up MatLab and make the current directory the same as the one that holds the above files.

3. Run *friction_v_load.m* to calibrate the lateral sensitivity of the cantilever. (The prompts for the program should be self-explanatory, but here are the step-by-step actions. Italics here indicate what you see in the MatLab command window, or what you should type.)

a) Type the following at the MatLab prompt, where *tiplatcal.001* is the DI file that you wish to analyze: *friction_v_load('tiplatcal.001');*

b) *Input offset value to be subtracted from norm (none = 0):*

(1) A window will appear showing a force distance curve obtained from the variation of the load. Forces should be given here in volts and there will be an offset due to the initial alignment of the photodiode. The out-of-contact region of the force curve should be at 0V but may be offset due to this alignment issue. Therefore the data needs to be shifted accordingly.

(2) Enter the value in volts that this line is away from 0. You can use the zoom-in tool to help with this.

(3) For example, if the out-of-contact line is at –1.2V, type in –1.2 at the prompt.

c) *Do you want to refine the offset? (yes = 1)*

(1) Type *0* or hit *ENTER* if offset value is correct.

(2) If you type the number incorrectly or wish to modify the offset, type *1* at this prompt. Then repeat process as in b2, according to the offset that you now see in the plot.

d) *Perform lateral calibration using this image? (yes = 1)*

Type *1*

e) *Normal and lateral forces will remain in volts for lateral calibration routine*.

pull_off_force_V =-2.2084

Your image should contain 2 sloped features/facets parallel to the edge of the image.

image_size =512 (pixels)

scan_size_nm =100 (nm)

Specify facet 1 See plot for (lateral offset) image (rotate the image for a top view.) Input the range of pixels to include (smallest value first.) Select the first edge (integer between 1 and image pixel size):

(1) A window will appear showing in 3D the trace minus retrace friction image. Rotate the image using the rotate tool to see the image as 2D with colors giving the 3rd dimension.

(2) The colors should show you the friction image captured on the faceted surface. You should have two facets visible. Make sure you know which region the tip is going up on during the trace (this is the UP slope) and which the tip is going down on during the trace (DOWN slope).

(3) Also the edges of the image are slightly lower than the rest of the image. You want to exclude these regions of the image from the analysis.

(4) Determine the region you would like to analyze for the first facet. It should be a region of relatively consistent color values in the fast scan direction. Note the pixel numbers that enclose this region.

(5) Type in the corresponding pixel numbers to eliminate data before the first edge facet. E.g. if there is about 20 pixels unwanted data on the edge, then type *20* at first prompt.

f) *Select the second edge (integer between 1 and image pixel size):*

(1) Again determine what pixel number you would like to cut the facet off at.

(2) E.g. if the data is good from pixel 20 to pixel 190, type *190*.

g) *Do you want to revise this region? (yes = 1)*

(1) This prompt allows you to go back and fix things is you made an error in choosing the region.

(2) With this prompt will appear the region you have chosen, with the graph axes modified so that the beginning edge starts at pixel 1.

(3) If you hit *ENTER* or type anything but 1, the program will continue.

(4) If you type *1*, care needs to be taken to redo the region. You will either need to remember what you think it should be in

relation to the original pixel numbers, or you can type *1* and *512* to see the original image with its original numbers, and then redo the region once more.

h) *Calculated lateral offset and halfwidth from each of the two sloped features.*

The program will output a text file with the name *tiplatcal_001.txt*. This will have the space-delimited data in 5 columns and a row of data for every line of the image (512 for this example), where the columns are: Deflection[V] Halfwidth(1)[V] LatOffset(1)[V] Halfwidth(2)[V] LatOffset(2)[V]. These columns are cantilever deflection, friction loop halfwidth on facet 1, friction loop lateral offset on facet 1, friction loop halfwidth on facet 2, and friction loop lateral offset on facet 2, respectively. This file will be created in the same folder as the original file.

4. Repeat process (step 3) for additional friction calibration files.

5. For large batches of friction calibration files, use *batch_process.m* This procedure works only for files with extensions in order, i.e. .001, .002, .003, etc.

a) Type *batch_process(friction_v_load,'tiplatcal.002', 4)*;

for example, to process without break, the files *tiplatcal.002*, *tiplatcal.003*, and *tiplatcal.004*.

b) The prompts and inputs will be exactly the same as when running *friction_v_load.m* on its own, just it keeps you from having to type in the different file names every time.

6. Using the output files from *friction_v_load.m*, plot deflection vs the other variables. Determine the slopes of each of these curves. Use Ogletree et. al RSI (1996) to determine the lateral sensitivity factor of the cantilever/AFM system for the experiment. This will be S (non dim.).

D. Analyzing FvL data

1. Once the normal stiffness and normal and lateral sensitivities are calculated, you are ready to analyze data to create fully calibrated FvL plots. You may also go ahead an analyze FvL data without the calibration numbers.

2. Run *friction_v_load.m* on actual FvL experiment files.

a) Follow steps in C1,2,3a-c

b) *Perform lateral calibration using this image? (yes = 1)*

Type *0* or hit *ENTER*.

c) *Enter the normal force calibration in nN/V (none = 1 or return):*

(1) Enter the value of the spring constant of the cantilever in N/m and multiply it by the normal sensitivity of the cantilever in nm/V. Enter this value at this prompt. You may multiply the numbers together at the prompt if you like.

(2) For example, if the normal spring constant of the lever is 0.05 N/m and the normal sensitivity is 100nm/V, then you can either type in *5* or *0.05*100*.

(3) If the normal calibration is unknown, type *1* or hit *ENTER* at

the prompt.

d) *pull_off_force_V = -2.2084*

(1) The pull-off force is measured automatically and given in either nN or V, depending on if the normal forces were calibrated or not.

(2) This value is not recorded in the output file, so needs to be recorded manually.

e) *Eliminate edges from friction trace and retrace? (yes = 1)*

(1) Hit *ENTER* or type *0* if you do not wish to perform this operation. Note that this will need to be performed on most images, unless the scan size is so large that these edge regions can be ignored. Skip to part f).

(2) Type *1* if you wish the analysis of the image to take place on the sliding region of the images. This means that at the edges, where the tip initially sticks before sliding, will be eliminated from the analysis.

(a) *Calculating friction force (or energy dissipation) from region to be specified...*

image_size = 512 (in pixels)

scan_size_nm = 100 (nm)

See plot for trace-retrace image. Note: rotate the image for a top view.

Input the range of pixels to include (smallest value first.)

Select the first edge (>=1):

(i) A window will appear showing in 3D the trace minus retrace friction image. Rotate the image using the rotate tool to see the image as 2D with colors giving the 3rd dimension.

(ii) At the beginning and end of the image, the friction trace minus retrace values will be slightly smaller than the rest of the image. We want to eliminate these regions because this is where the tip has not yet begun sliding. Note the pixel numbers where this region begins.

(iii) Type in the corresponding pixel number to eliminate the first edge. For example if there are about 20 pixels of unwanted data along the edge, then type *20* at the first prompt.

(b) *Select the second edge (>=1):*

(i) Note the pixel numbers where the sliding region of the image begins.

(ii) Type in the pixel number corresponding to eliminate the second edge of the image. For example if there are about 20 pixels of unwanted data along the edge, then type *490* at this prompt.

(c) *Do you want to change the region? (yes = 1)*

(i) The modified image will appear with this prompt. Rotate the image again using the rotate tool to see if the edge regions have been eliminated.

(ii) If the edges have successfully been eliminated, type *0* or hit *ENTER*.

(iii) If the edges have not been completely eliminated, type *1*.

You will either need to remember what you think it should be in relation to the original pixel numbers, or you can type *1* and *512* to see the original image with its original numbers, and then redo the region once more.

f) *Choose friction or energy dissipation (energy = 1):*

Type *0* or hit *ENTER*.

g) *Enter the lateral calibration factor in nN/V (none = 1 or return):*

(1) Enter the value of the lateral calibration factor of the cantilever in nN/V. Use the value of S determined in C6 and multiply it by the normal calibration factor of the cantilever in nN/V. Enter this value at this prompt. You may multiply the numbers together at the prompt if you like.

(2) For example, if the normal spring constant of the lever is 0.05 N/m, the normal sensitivity is 100nm/V, and S=10, then you can either type in *50* or *0.05*100*10*.

(3) If the lateral calibration is unknown, type *1* or hit *ENTER* at the prompt.

h) The program will output a text file with the name *filename_001.txt*. This will have the space-delimited data in 2 columns: load data for every line (512 for this example) and friction data (trace-retrace) for every line. This file will be created in the same folder as the original file.

3. Repeat process (step 2) for additional friction calibration files.

4. For large batches of friction calibration files, use *batch_process.m*. This works best for files with extensions in order, i.e. .001, .002, .003, etc.

a) Type *batch_process(friction_v_load,'filename.002', 4)*;

for example, to process without break, the files *filename.002, filename.003*, and *filename.004*.

b) The prompts and inputs will be exactly the same as when running *friction_v_load.m* on its own, just it keeps you from having to type in the different file names every time.

E. Plotting FvL data

1. Now the data is ready to be plotted in a software analysis program such as KaliedaGraph.

2. When the data is plotted you may notice some stray data points clustered around 0 nN applied load. These stray points are due to a false frictional signal measured while the tip is out of contact with the surface.

a) To eliminate these stray points, plot the deflection or load versus an evenly spaced series of numbers. The plot will show you a force-distance curve during the sliding experiment.

b) From this plot, eliminate all the data points within the out-of-contact region of the curve. Then replot the FvL curve and you will have eliminated these stray points.