Magnetic Force Microscope (MFM)
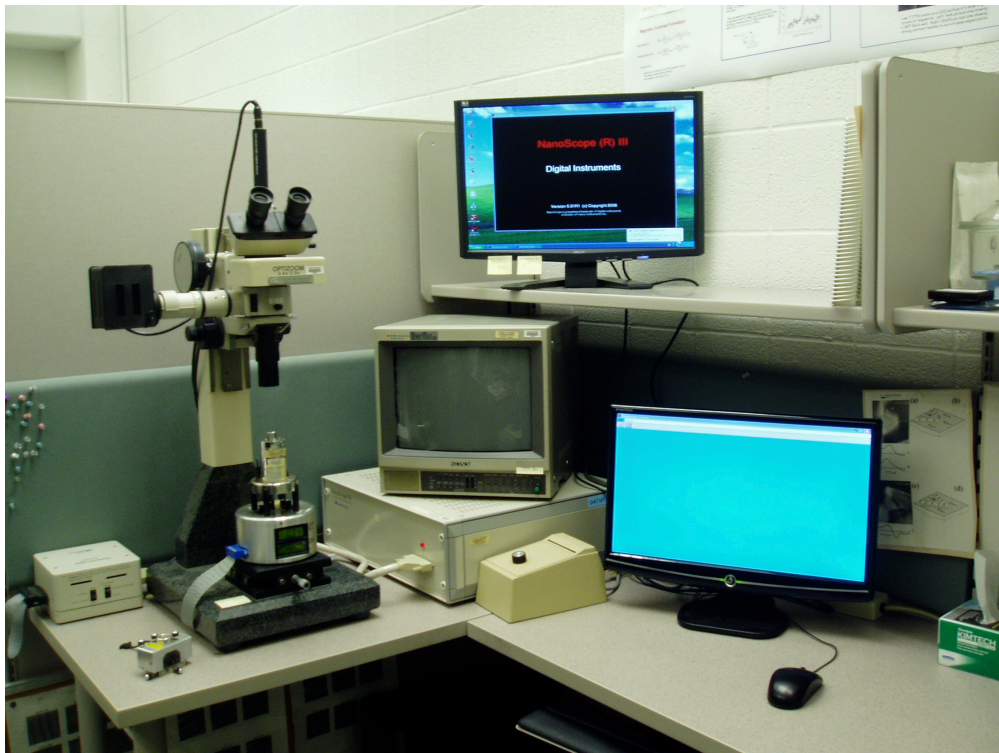
[Digital Instruments NanoScopeIII]

magnetic and topographic resolution ~20 nm [*at least* several hours/sample (1 cm² polished single grains, thin sections, or thin films)]
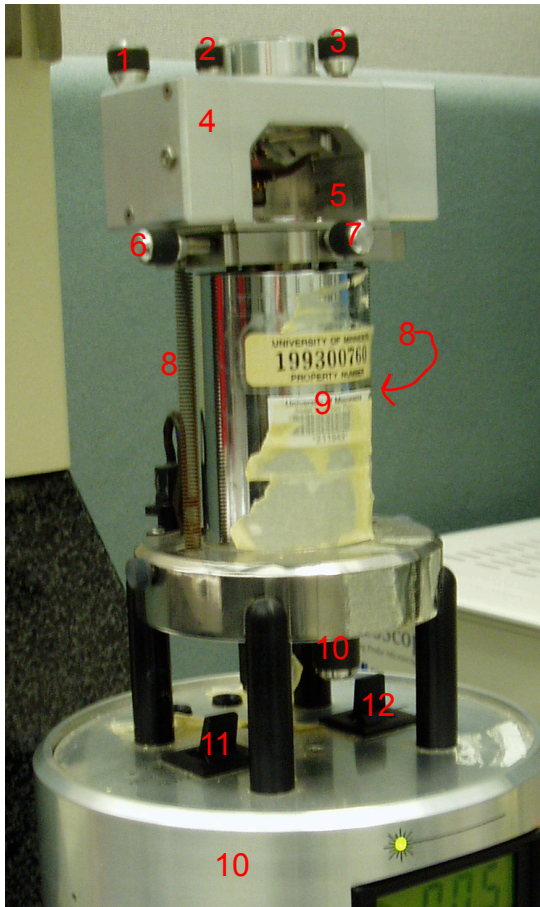
See *IRM Quarterly*, Winter 1992–93, Vol. 2, No. 4

See *IRM Quarterly*, Winter 1993–94, Vol. 3, No. 4

See *IRM Quarterly*, Spring 1994, Vol. 4, No. 1 (magnetic quantities and units)

The MFM consists of a microscope assembly with an attached video camera, digital image-processing electronics, external magnetic field coils (not shown), and a computer.

Scanner Assembly

1. photodetector displacement control
2, 3. laser beam displacement controls
4. scanner head
5. cantilever assembly (not shown)
6, 7. cantilever translation screws
8. scanning head hold-down spring
9. scanner body
10. scanner base
11. scanner mode switch
12. vertical displacement motor switch
13. vertical displacement manual knobs
14. microscope stage
15, 16. microscope stage translation screws
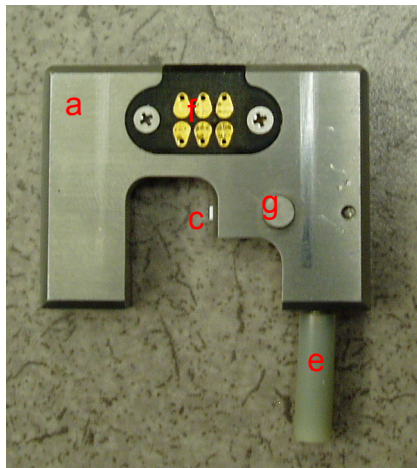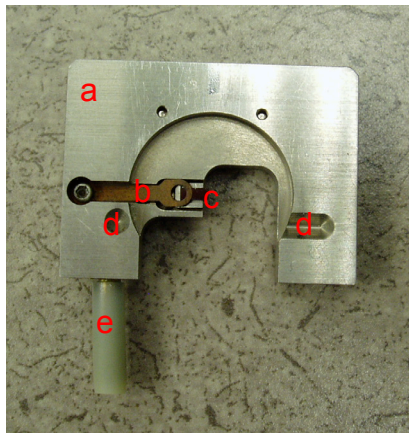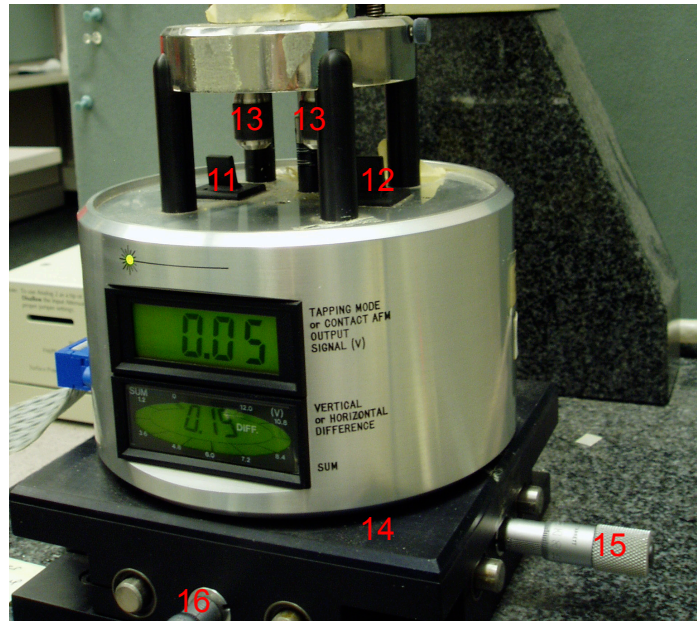
Cantilever assembly (5) details

a. mounting bracket
b. copper spring clamp
c. cantilever & tip
d. support divots
e. handle
f. contacts
g. clamp release

I. System startup

1. Turn on the NanoScopeIII. Switch is at the back of the large, tan box.
2. Turn on the MFM control computer and its TWO monitors.
3. Turn on the Sony video monitor, and turn up illumination to desired level.
4. Start and configure software:

    a. Click on the "Nanoscope v614r1" icon.

    b. Click on the microscope icon.

    c. A small box labeled "Add Views to RealTime1 box" will appear. Click "Select All", then "OK".

    d. A Single Scan window will be open. Click "Scan-Dual" on the left-hand side of the screen to open an additional Dual Scan window.

    e. Check that the data channels are setup as follows (Channel, Data Type, Scan Line, Line Direction*):

    >   1, Height, Main, Retrace
    >   2, Amplitude, Main, Retrace
   >   3, Phase, Interleave, Retrace

    > *Height*: topography generated by tip tapping across sample

    > *Amplitude*: variations in amplitude of tip oscillation; in tapping mode (Main) this is approimately the first derivitave of the height.

    > *Phase*: Shift in phase of tip oscillation from the driving signal. In Interleave mode this is mostly related to the magnetic signal.

    > *Scan line*: Main or Interleave. The Main scan is the first scan when the tip goes over and back, tapping the surface to get topograpy. The Interleave scan is the second scan, when the tip flys over the surface at constant elevation. Typically, Height is derrived from the Main scan, Phase is derrived from the Interleave scan, and Amplitude can be taken from either, depending on whether you want to use it more for topography or more for magnetic information.

    > *Line direction*: Trace or re-trace. Selects which part of the scan line (the over or the back) will be used. Typically, the re-trace gives better data, but not always
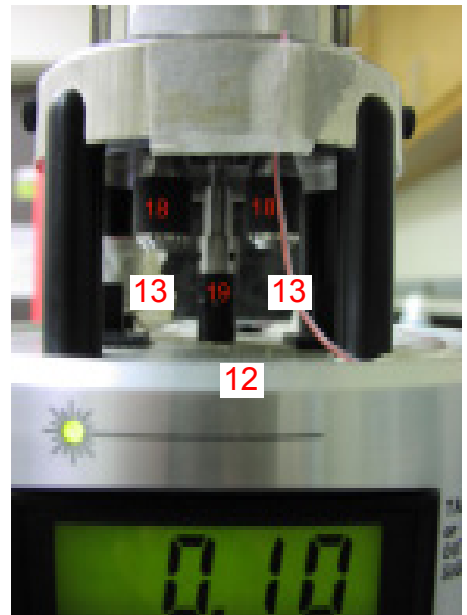
II. Mounting Samples

1. Samples must be well polished and no greater than 1-cm diameter.
2. Attach sample to disk mount with double-sided tape.
3. Install disk mount onto scanner.

III. Installing Scanner Head

1. Leave cantilever assembly (5) out for now.
2. Place head (4) on scanner body (9).
3. Hold firmly while attaching the two hold-down springs (8; one on each side).
4. Plug laser in -- wire attached to the scanner head should be plugged into socket on the scanner body (not shown).
5. Adjust vertical position using two manual (13) and one motor-controlled (12) adjustment screws, to allow room for installation of cantilever assembly.
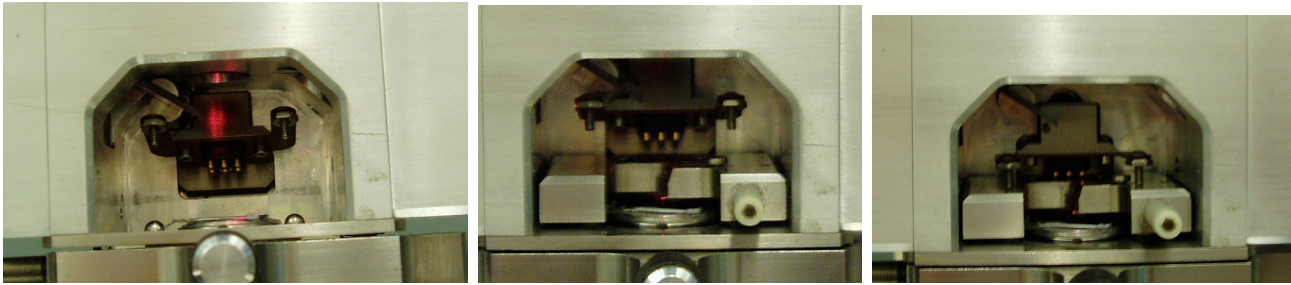

IV. Installing Cantilever Assembly (see above images)

1. Verify that sample surface is no higher than the tops of the three vertical adjustment screws.
2. CAREFULLY place cantilever assembly in scanner head, cantilever side down, so that the divots rest on the tops of the vertical adjustment screws. Do not allow the cantilever tip to contact the sample surface. Readjust vertical position screws if necessary.
3. Tighten the hold-down clamp using the knob (not shown) on the back of the scanner head, so the cantilever is held firmly in place.

V. Initial Positioning of Cantilever Near Surface

1. Carefully lower the cantilever towards the sample, alternating between the two manual (13) and the motor-controlled (12) adjustment screws, until the tip is within about 2 mm of the surface. Use hand lens as necessary.
2. Focus camera on the sample surface.
3. Locate the area you wish to scan, using the cantilever translation screws (6,7) to position the tip, and the microscope stage translation screws (15, 16) as needed to keep the tip in the field of view.
4. Run the tip tuning procedure:
   a. Click on the tuning-fork icon.
   b. Click on "Auto-tune."
   c. The NanoScope Image screen should eventually display something approx-imating that shown at right. If it does
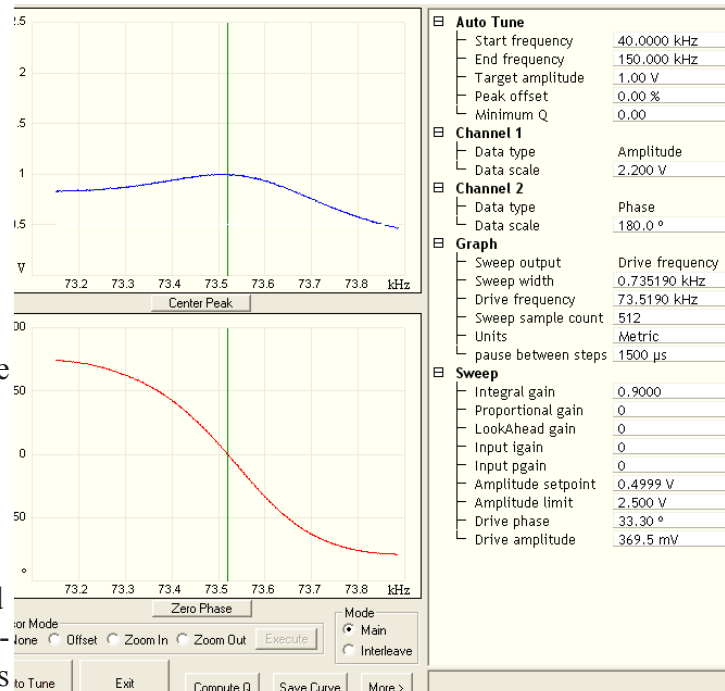
not, you may have a broken tip (See section IX on changing the tip).

    d. Click "Compute Q". Q is a quality factor related to tip resonance and should be ~200.

    e. Click "Exit". The Output Signal should be close to 1.0 V.

5. Continue lowering the cantilever toward the surface until the tip just begins coming into focus on the monitor. Try to keep the head parallel to the sample surface.

VI. Preparing to Scan Sample

1. You should now see the laser beam on the sample/cantilever, and the displays on the scanner base should be illuminated.

2. Use the laser beam displacement controls (2,3) to position the beam near the middle of the cantilever tip. You should be able to see it on the camera monitor (focus on the tip if necessary, then refocus on the sample surface).

3. Adjust the beam position (2, 3) and the photodetector (1) to get the maximum "sum" signal in the outer perimeter of the lower display of the scanner base, and the minimum absolute-value "difference signal in the center of the lower display.

4. Very slowly and carefully continue to manually lower the tip until the Output Signal Voltage decreases by ~5-10%.
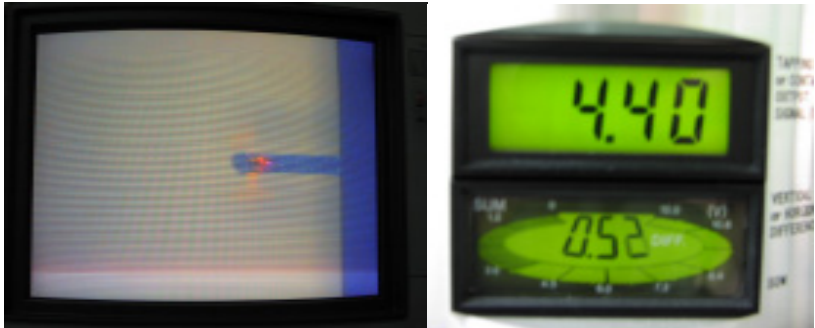
VII. Starting a Scan

1. Engage the tip by clicking on the green down arrow icon. The software will likely warn you: "Interleave mode is enabled - continue?" Click Yes.

2. The motorized vertical adjustment screw will lower the tip until contact is made, which may take a few minutes. If the initial height was too great, the software will issue a warning message ('motor transport exceeded normal range'); You can just click the engage button again, but the tip will likely land "north" of what you were aiming for.

3. Once the tip is engaged, a scan will automatically begin. The tip may not properly engage, resulting in relatively flat, featureless scan lines, and trace and retrace lines that are not coherent. In this case, decrease the Amplitude Setpoint by very small amounts until you start to see features in the Height channel (topography) and the trace and retrace lines track well together.

4. Viewing Data. Data is displayed in two modes.

    a. *Image Mode.* Data from each Channel are displayed in an X-Y-Z plot (X and Y are distance, while Z is, e.g., the phase data represented by a color).

    b. *Oscilliscope Mode.* Individual scans displayed in X-Y charts (X is distance and Y is, e.g., phase). The charts constantly refresh with each scan. This mode can be useful for diagnosing problems.

VIII. Scan Controls and Channels
There a number of settings that may be changed while you are scanning. Most of these are located on two tabs, the Main tab and the Scan tab.

1. **Main tab options**:
    a. *Scan size*. The maximum scan size is ~100 µm, but the maximum practical scan size is often much smaller and can be limited by how flat your sample surface is. Samples that have rounded surfaces (typical of oxide grains which can be more resistant to polishing than the surrounding matrix) make it difficult for the tip to stay in contact with the sample across the entire scan.
    b. *Aspect ratio*. Typically 1:1, but this can be changed for non-square scans.
    c. *Scan rate*: Scan rate in Hz. The scan rate can typically be increased for smaller scan sizes. Image quality is often improved at lower scan rates, especially if the sample has significant topography.
    d. *Samples/line*: The number of samples (data points) per scan line (typically 256).
    e. *Amplitude setpoint* (described in VII.3.d.i) which can be found under Feedback Controls. If the Height data (Oscilliscape Mode) are featureless and don't track each other between trace and re-trace, the tip may not be making good contact with the surface. Gradually decrease the Amplitude Setpoint until you start to see features in the curves and the trace and re-trace look similar.

2. **Scan tab options.**
    a. *X and Y offsets:* Shifts the scan center away from the piezo zero position. These can be used to make small changes in scan position, but offsets >10µm are not recommended and cause increased wear on the piezos.
    b. *Scan angle*: 0° scans horizontally (along the x axis) and 90° scans vertically (along the y axis). If a samples has elongated topographic features, it is sometimes more favorable to scan along the axis of the topography. Also, if you are concerned an elongated feature in your image(s) may be an artifact, it is sometime helpful to re-scan the feature at a 90° angle to the original scan.
    c. *Slow scan axis*: set to Enabled.

IX. Changing Scan Location
If you need to move the center of your scan by <10µm, you can use the X and Y offset controls. If you need to move the scan farther than that, it is best to disengage the tip from the surface using the red up arrow button. If you are moving a small distance (a few 10s of microns), you can probably carefully move the tip and then re-engage by clicking the green down-arrow button. If you need to move a farther distance, you want to ensure that you don't drive the tip into the sample -- this is easy to do if your sample isn't completely flat or if the scanner body is at a slight angle with respect to the sample, it is possible to drive the tip into the sample; press the red up-arrow 2 or 3 more times, and then raise the tip manually so it is well above the sample.

X. Capuring Images
The images displayed are not saved until you tell the software to capture the image. Notice that at the bottom of the NanoScope Control screen it says "Capture: Off."   Under the main (top) menu, select RealTime > Filename. You can then enter a filename to write the data to. Then click "Capture." The comment at the bottom of the screen will either read "Capture: Next" or "Capture: On."

The Capture will begin when the scanning reaches the top or bottom of the scan area and starts over. If you do not choose a file name, the data will be saved with a date/time stamp. You can also use the camera icon to capture next, or the camera! icon to immediately capture what is on the screen.

XI. Shut-Down Proceedure

1. Disengage tip by clicking on the red up-arrow icon. Do this several times to get tip well above samples surface.
2. Continue to raise tip a few more mm, alternating between the two manual (13) and the motor-controlled (12) adjustment screws.
3. Remove the cantilever assembly by loosening the hold-down clamp and gently lifting it up and out of the scanner head. Place the assembly (cantilever side down) on a flat surface inside the dessicator.
4. Raise the scope/camera, and turn off the light to the microscope.
5. Unplug the laser.
6. Remove scanner head by holding gently but firmly in place while the hold-down springs (8) are released.
7. Remove sample.
8. Turn off power to the NanoScopeIII (switch on back of large tan box).

XII. Replacing the cantilever (tip)

1. Remove the cantilever assembly as described above in XI.3, and place it cantilever side UP on a flat surface.
2. If you are not sure whether or not the tip of the cantilever is broken completely off, examine it under the binocular microscope or with another magnifying scope.
3. Use your fingers to press opposite corners of the assembly down; this will depress the clamp release (g) and raise the spring clamp (b). Remove the cantilever with a pair of tweezers/forceps.
4. If the cantilever tip is broken off completely, it can be discarded. If it is not, it should be placed back in the container with the remaining tips. Do not flip it over when you set it down -- it is stored on its back with the tip pointing up. Used tips are stored at an angle to unused tips to warn other users that there may be a problem with it.
5. Inserting a new cantilever is often best done using a magnifying scope of some kind.
6. Select a new cantilever, and gently pick it up with tweezers. Cantilevers are stored on their "back", tip size up, so you will maintain that orientation as you insert it (i.e. don't flip it over).
7. Depress the cantilever assembly on a hard surface to raise the spring clamp (b) and slide the cantilever under the spring clamp.
8. Release the assembly to let the clamp tighten on the cantilever.
9. The cantilever should be flush against the back of the depression in which it sits. You may need to make slight adjustments to delicately push it into place.
10. Breathe.
11. Magnetize the tip by bringing it close to a strong permanent magnet.

XIII. Common Problems and Possible Solutions

1. *Trace and re-trace featureless and not tracking each other*. Tip probably not engaging with the surface. Try decreasing the amplitude setpoint until you start to see features in the topography.
2. *High-frequency "noise" in data*. Occasionally, regular, linear features will show up in the scans. If the period is ~1.5-2.5 μm and your sample is highly reflective, it may be due to opti-

cal interference. Try adjusting the laser alignment to minimize reflection of the sample surface (maximize reflection off cantilever). Occasionally, other sources of electrical noise may generate a similar artifact and can be difficult to eliminate. The artifact can often be minimized or eliminated during post-processing of the images with FFT tools (see below).

3. *Contaminated sample surface.* Dust or other contamination of the sample surface can result in a streaky image and/or loss of resolution. Clean the sample with methanol and rub continuously with a Kimwipe until the surface is dry. If the surface is characterized by many homogeneous small spheres of ~0.25 µm diameter, you probably have residual colloidal silica on the surface. It may be necessary to re-polish in order to remove it.

4. *Out of range.* If your sample has considerable topographic relief or if the scanner head is not level, as the scan progresses the cantilever may move too far or too close to the surface to work properly. Disengage and re-engage the tip with the software. (Note that if your scan is moving "upslope" on the sample surface, you may drive the cantilever into the surface and break the tip, so you should pay attention to the tip extension indicator at the bottom of the screen.)

5. *Static build up.* Especially in winter, static charge buildup in the scanner head assembly can cause numerous problems, including a tip that won't disengage properly at the end of each scan line. If you can't figure out what else is wrong, try using the specially-designed radioactive brush (remove cantelever assembly first) or the magic red ion gun (cantelever assembly can stay in place) to reduce static in and around the scanner head assembly.

XIV. Processing Images

Some post-processing of the images can be done using the NanoScope software. (Select File > Open... or double-click on an image/file shown along the right-hand side of the screen.) However, there are several freely-available image processing programs that have equal or greater funcionality and will allow you to continue working with the images after you have left the IRM. The one installed on IRM computers is called *WSxM* and is available from Nanotec (www.nanotec.es). This is only compatible with Windows machines. For the Macintosh, there is *Image SXM* by Steve Barrett at University of Liverpool. Both of these packages are designed for scanning microscopy. *ImageJ* is a more generic image-processing package from National Institutes of Health (NIH).

Common Processing:
1. *Flatten*. This is almost always needed to scale the data properly for display. It subtracts an average or background value from the image in either a very basic or a more sophisticated way.
2. *Equalize*. This will set your color scale so the maximum and minimum values are the max and min of your data. It essentially enhances contrast.
3. *Smoothing*. There are various smoothing or noise-reduction algorithms.
4. *FFT*. Noise that is periodic (perhaps resulting from optical interference -- see above) can sometimes be removed by using the Fourier Transform options.
5. *Cross-correlation*. This may be useful when trying to compare features between images (e.g. between height and phase)

XV. Most Important Thing to Have for MFM Work

Patience.