MicroMag (Alternating Gradient Force Magnetometer) Procedure

Equipment

Fine-tipped tweezers
Alcohol in squeeze bottle
Dow silicone vacuum grease
Kim Wipes
pen or pencil
Log book

Starting Procedure

1. Sign into the MicroMag log book before starting.
2. Take a small amount of grease from the communal container using a clean toothpick or wooden end of a cotton swab and place it on a clean surface.
3. Before you start, clean off the probe holder (the black thing on which the probe rests while mounting a sample) with a little alcohol before you set the probe on it. Make sure that the alcohol has evaporated before you set down the probe.
4. Clean all of your tools with alcohol prior to using them. Make sure to clean the tools in between samples to minimize the chances of cross-contamination.
5. Allow the electronics to warm up for about an hour prior to starting your measurements.

Check the Probe for Contamination

Checking that the probe is not contaminated is absolutely critical to the reliability of the rest of your measurements!

Measure the probe to check for contamination

1. Load the blank probe onto the AGFM. Make sure that the probe is centered in all three directions. Use the flashlight, as you cannot see well enough without it.
2. Select Autofunctions from the main menu and press Enter
3. Select Initial Autotune; make sure Q is between 10-30 for the P1 phenolic probe. The resonance frequency should be between 580-629 Hz for the P1 phenolic probe.
4. Select *Incremental Autotune*, want Q about the same as above. Press Enter to accept results
5. Select *Incremental Autotune* again- same procedure as above step.
6. Select *Measurements*, then *Direct Moment*.
7. Check the following values
   a. Averaging time = 100 ms
   b. Maximum field = 10 kOe
   c. Set the gradient to whatever value you expect to do your measurements in. Always calibrate at the same conditions as the measurement you plan to do.
   d. Note exceptions: If you have a very weak signal and a lot of noise, you may need to increase the averaging time to 200 ms.
8. Select Hysteresis Loop; press Enter. There is no need to demagnetize or do an initial loop.
9. Evaluate results: Check the plot against the plot on the wall in the micromag room. You MUST expand the scale in x- and y-directions to see whether there are any features near H = 0. Simply looking at the output as it is plotted on the screen is not enough.
   a. If loop is strictly diamagnetic and the values match reasonably well with the reference, continue
   b. If there is ***any*** other signal (ferromagnetic, paramagnetic, etc.), you must clean the probe before beginning. Re-measure the empty probe before continuing.

Calibrate the Micromag

Calibration is critical to the accuracy of all the measurements you do. If you don't calibrate carefully, your results will not be meaningful.

1. Place the YIG standard (kept in the box on the desk) on the probe. It is critical that the standard be in the exact center of the probe. Keep the box closed to prevent dirt from getting in.
2. Insert the probe in the Micromag. Release the springs prior to lowering the sample. Lower the sample slowly and be very careful – the probe is very delicate.
3. Ensure that the sample is centered in all three directions. Use the flashlight to ensure that it is. If it is not exactly centered, use the translators in the x-, y- and z- directions to center the sample.
4. Check the following values
   a. Averaging time = 100 ms
   b. Maximum field = 10 kOe
   c. Field Increment = 50 Oe
   d. Gradient = whatever you plan to use. The program will not allow you to use gradient =1, so if your measurement requires this, calibrate using gradient = 0.1.
   e. Sensitivity = 100 memu
   f. Exceptions: If you have a very weak signal and a lot of noise, you may need to increase the averaging time to 200 ms. If you have a small coercivity or you are particularly interested in the behavior near H=0, go to 20 Oe.
5. Select *Autofunctions* from the main menu and press *Continue*
6. Select *Initial Autotune*, make sure Q is between 10-30 for the P1 phenolic probe. *Continue* to accept results.
7. Select *Incremental Autotune*, want Q about the same as above. Press *Continue* to accept results
8. Repeat the *Incremental Autotune* step above.
9. Select *Measurements* and *Direct Moment vs. Field*. There is no need to do an initial loop or to demagnetize the sample.
10. Set *Probe Factor* to 77.61 memu.
11. Measure another hysteresis loop to make sure that you get the same value (to within ±1%) for the saturation magnetization. If the value is not within this bracket, repeat steps 10 and 11 again.

Measuring Hysteresis Loops

1. Mount your sample on the probe. Use the minimum amount of grease possible, as grease attracts dirt.
2. Check to make sure sample is centered on probe and probe is centered in the magnetometer in all three directions (x, y and z)
3. ***Unless there is some reason you do not want your sample exposed at all to the field prior to measurement***, Apply a field large enough so that you know your sample is saturated. If you don't know what to expect from your sample, use the maximum field. Note the value of the magnetic moment in units of emu.
4. Check the following values
   a. Set the sensitivity according to the maximum value you found in the above step.
   b. Averaging time = 100 ms
   c. Maximum field = 20 kOe. (Unless you have a specific reason to not use the highest field possible, always use this field.)
   d. Field Increment = 25 Oe
   e. Gradient = 1
   f. Note exceptions: If you have a very weak signal and a lot of noise, you may need to increase the averaging time. If your sample has low coercivity, you may get a more accurate measurement of the coercivity by using a field increment of 10-15 Oe.
5. Autotune the probe and sample
   a. Select *Autofunctions* from the main menu and press *Continue*
   b. Select *Initial Autotune*, make sure Q is between 5-20. *Continue* to accept results.
   c. Select *Incremental Autotune*, want Q about the same as above. Press *Continue* to accept results.
   d. Repeat the *Incremental Autotune* step.
6. If you want to measure the initial part of the loop, Select *Include Initial Moment*. You can demagnetize by selecting the *Demagnetize before measuring* option on the hysteresis loop window or Alt. field demagnetize from the tools option mention on the main window. The initial field for the demagnetization must be significantly larger than the coercivity of your sample. If the field is not sufficient to demagnetize the sample, you will have to increase the initial field. The larger the initial field, the more time it will take to demagnetize your sample, so try to use the lowest field you can. If the magnetization is not close to zero when no field is applied, repeat the demagnetization until it is.
7. Select *Direct Moment vs. Field*.
   a. Press *Initial + loop* if you want to measure both the initial part of the hysteresis loop and the loop itself
   b. Press *Initial* if you only want the initial part of the curve
   c. Press L*oop* if you only want the hysteresis loop per se.
8. When the loop is done...
   a. Evaluate the results before continuing
      i. Does your data take up the entire vertical range? For example, if the graph shows a maximum M value of 1×10^{-5} emu, but your saturation magnetization is 4×10^{-6} emu, you will need to change the sensitivity to 5×10^{-6} emu and take the data again.
      ii. If you took an initial curve, did the data at H = 0 correspond to M = 0? If not, you may need to demagnetize your sample again and repeat.
   b. On the screen that shows the hysteresis loop, select *Description*. Type the sample description into the box, click OK
   c. Save the file
      i. Click on *File*
      ii. Select *Save File*

iii. Save the file using a descriptive name in the appropriate directory: C:\data\users\folder with your data files

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d. On the *Direct Moment vs. Field* window
   i. Select the Tools menu
   ii. Select *Normalization not in effect*
   iii. Select *Normalize by mass*. A window will appear.
   iv. Enter sample mass – make sure your units are correct
   v. The sample mass should appear on the list of parameters shown on the graph generated by the micromag program.

Cleaning the Probe

1. Never use anything other than alcohol to clean the probe. **Never** use acetone, as it will dissolve the glue that holds the probe face to the probe.
2. Ultrasonicate the probe for two minutes in a beaker of alcohol.
   a. You will have to stand over the ultrasonicator and hold the probe.
   b. Make sure that no more than the glass part of the probe plus maybe 5mm above that is immersed in the alcohol.
   c. Do not dip any other part of the sample probe into the alcohol.
3. After ultrasonication, use a very gentle stream of alcohol from a squeeze bottle to rinse the probe. The ultrasonification shakes the dirt loose, but then the dirt is swirling around in the beaker full of alcohol. This step assures that the dirt isn't re-deposited on the probe.
4. Don't try to dry the probe, as you may break it.
   a. Put a small piece of KimWipe® on the probe holder
   b. Set the probe so that it is resting on the KimWipe. The KimWipe will wick away the alcohol by capillary action.
   c. You may need to rotate the holder slightly to bring the edges of the probe holder in contact with the KimWipe
   d. The probe must be entirely dry before you start using it. Otherwise, the grease will trap the alcohol and then it may cause drifts in your measurement.

When You're Done

1. Clean up the area around the micromag.
   a. Don't leave KimWipes or other debris.
   b. Throw away any grease that you didn't use. Never return grease to the box.
2. Sign out of the logbook.

General Notes

- If the Autotune procedure doesn't work or Q varies widely, check to make sure that the sample is centered. You may have to change the gradient if the sample has a large magnetization.
- Don't let your sample touch the pole pieces or other parts of the micromag when loading it. This is bad for the probe, but also could contaminate your sample.
- Never put anything but a brand new toothpick in the grease. It is very easy to contaminate the grease and then everyone else will be measuring junk.
- Do NOT save your files on the micromag computer. Make sure that you transfer them to a flash drive and then to one of the lab computers as soon as possible. Make a backup.

Isothermal Remanent Magnetization and Demagnetization Remanence

(NOTE: this still needs to be revised to match the rest)

1. Select Autofunctions; press Enter, then hit...
   ---**Intitial Autotune**, make sure Q is between 5-20. Enter to accept results.
   ---**Incremental Autotune**, want Q about the same as above. press Enter.
   (you will have to wait for the new Q, the machine is on at this point so being quiet is helpful)
2. Select Demagnetize...
   F1=>by Field

or

   F2=>by Magnetization
   Initial Field: 5000 Oe; Decrement: 1~10%.
   If the field is not sufficient to reverse the remanent magnetization, try a larger field (10KOe).
3. Select **Direct** Measurement Menu; press Enter
4. Select Remanence, then chose: Isothermal Remanet Magnetization + DC Demeg Remanence. (field=3K Oe)
5. When IRM+ DCR is done...

- F1, Enter description
- F2, Save
- Hit Spacebar, F4 to normalize by mass, enter mass of sample.
- Hit Spacebar until menu with Plot appears, Make sure plotter has paper loaded, Hit F3, then Plot all.