SHB Instruments, Inc.
19215 Parthenia St. Suite A
Northridge, CA 91324
(818) 773-2000
www.shbinstruments.com

Instrument Calibration

November 24, 1996

Revised April 7, 2004

Revised May 11, 2009

Unfortunately, there exist no thin-film flux standards that can be used to calibrate the vertical axis (total flux) of BH loop tracers like the SHB products. We have been working with NIST (National Institute of Standards) to make such standards available, but because of the varying wafer geometries used by our customers, as well as concerns about stability of standards, such standards are not yet available.

Our customers typically calibrate the vertical axis of their SHB instruments by setting the Hysteresis Looper readings to match reference samples they have calibrated on other instruments. We calibrate the instruments before shipment using an unofficial calibration standard provided to us by a knowledgeable customer.

Calibration really has two basic parts, Vertical (Bs), and Horizontal (Hc). Horizontal calibration is in principal easier, as one can purchase a gaussmeter (we recommend the Lakeshore Model 450), and place its probe in the center of the drive coils, and verify that the field produced agrees with the drive values we display.

Calibration steps:

Before beginning any calibration, make sure that the instrument internal adjustments have been done (under the Adjustments menu item – see Technical Note on Adjustments), and that the Earth's Field compensation has been adjusted.

1. Vertical Axis

Vertical (Bs) calibration is somewhat complex. Here we cannot use a simple instrument, as what we are measuring is the flux that the pickup coil captures from a particular sample. This is highly dependent on the pickup shape and number of turns, as well as the sample geometry. A calibration done for a 1" diameter sample in a given pickup will differ somewhat from that done for say a 4" diameter sample. Again, this is not an instrument limitation, but simply a result of the physics of the situation.

The flux captured by the pickup is to first order proportional to the magnetic thickness of the film being measured times the width of the sample within the pickup window. The distance the sample extends forward and backward (in the direction toward and away from the operator) is not critical, as long as it is least several times the dimension of the pickup windings in this direction, which is about ½ inch. Unfortunately the width of the sample within the pickup window is often poorly defined, as for example when dealing with round samples.

Here is an example of some calculations and calibration work done using a 3.2mm square piece of 4 mil Ni foil:

We were not able to fully saturate this material on the instrument (Model 109) used to measure the sample, but by extrapolating the loop we can estimate the value of Bs when saturation is reached as 210 nWb (good to perhaps 10%). To make the units consistent, we convert 210 nWb (nanoWebers) to 21 Mx (Maxwells).

What we want to calculate for the 4 mil thick, .32 cm wide Ni sample is the saturation induction, 4Pi Ms (in Gauss):

4PiMs = [21 Mx] / [(.32 cm) ((.004 in) (2.54 cm/in))] = 6459 G

Therefore Ms = 6459/(4Pi) = 514 emu/cm^3

The accepted value of saturation induction for Ni (per Bozorth) is 6084 G. These match to well within the approx. 10% certainty we have in the saturated Bs value. This shows that even with the 3.2mm square sample, which is "short" in the direction of the applied field, we are capturing essentially all of the flux with the pickup we used.

Therefore we can use the Bs measurements to calculate the product of the sample thickness in cm (t) and the saturation induction in Gauss (4PiMs):

Let Bs be the measured value for a given sample:

4PiMs t = (Bs/10) / (.3 cm) = Bs / 3

Customers who care about accurate vertical axis measurements often do this calibration by starting with two similar samples, and then carefully measuring the Bs value of each sample. One sample is cut into small pieces which are then measured on a VSM (Vibrating Sample Magnetometer). Given the VSM measurement and a knowledge of the cross-sectional area of the film on the sample tested in the VSM, one can extrapolate to the Bs value to be expected for a larger cross-sectional area sample measure on the Looper. If the user is willing to assume good deposition uniformity, only one small piece need be measured in the VSM.

Note that for both vertical and horizontal calibrations is preferable to use a thick sample if possible in order to achieve a higher signal to noise ratio, and therefore a more accurate calibration.

There is only one vertical calibration constant (vertical gain) settable on the instrument. At the user's choice the calibration can be done based on either the Bs or Br value of the material. There is no point in doing both – the second calibration will simply overwrite the first.

Note that the instrument has vertical attenuation modes to accommodate the measurement of very thick samples. These are called x10 and x100 modes, as opposed to the usual mode of operation, which is referred to as vertical x1 mode.

To perform a vertical calibration, the instrument should be set to the proper vertical sensitivity to display the desired reference loop, with a balance and a pattern memory store having been done before the sample is inserted, to assure a quality loop free of background noise.

The software must be at at least Engineer level before calibrating. The options for Cal Bs and Cal Br are identical, so only Cal Bs will be described here.

Select from the menu "Calibrate", then "Cal Bs". There will now be three options. The first, "Current Vertical Attenuation", will only adjust the calibration constant for the vertical attenuation mode the instrument is now in. In other words, if the instrument is in vertical x10 mode (because we are measuring a thick sample), only the constant for x10 mode will be adjusted. Or if we are in the more normal vertical x1 mode, only that gain constant will be set. Separate constants are kept for each of x1, x10 and x100 modes, so in theory each can be calibrated to a different standard if desired. When this option is selected, the user will be asked for the value in nanoWebers that this sample should read, and the measurements and adjustments needed to make the sample measure at this value will be done.

As with all calibrations and adjustments, the results will be made permanent by being automatically saved to the "constant file" (c:\shbwin\constant.dat) on the hard disk, unless the option "No Constant automatic save" has been selected in the "File/Options" dialog box.

The second choice available under "Calibrate/Cal Bs" is "x1, x10 and x100…". This option when selected, will calibrate the vertical range of the instrument for all three of the three vertical attenuation settings. Obviously this selection can only be used if a very thick calibration standard is being used, as we must be able to get a usable loop even with the x100 attenuator in place. If this is done, samples will then read the same on all three vertical attenuation ranges.

The third choice "Manual Adjust (delta %)", is used when the Bs readings being obtained with the instrument are close to the desired value (within 10%), and the user wishes to simply adjust the vertical calibration up or down within the range of plus or minus 10%, without the need to perform a full calibration.

2. <u>Horizontal Axis</u>

There are two ways to calibrate the horizontal axis of the instrument. The older (and less reliable) method involves having a standard sample with known Hc value. The instrument is set up to display this loop under the same conditions as were in place when the sample was originally measured, and the command "Calibrate Hc/User Value" is selected. The user will be asked for the desired Hc reading, and the instrument will be adjusted to make the sample read that Hc value. But the user must be aware that the measured value for Hc will be a function of sweep frequency (the lower the more accurate – we often use 1 or 2 Hz on the Easy axis), as well as the drive level selected (this affects the dV/dT of the drive waveform, much as does changing frequency). Note that "Calibrate/Cal Hc" also has a "Manual Adjust" option that allows for small percentage "tweaks", just as for Bs. Calibration of the Transverse axis using the Hc method is only possible on older instruments that have Transverse pickups, using thick enough samples to be accurately measured using the Transverse pickups. The above process is simply repeated, using the Transverse pickup and Transverse field.

The second method of doing Horizontal calibration is more accurate, but require the use of an accurate DC gaussmeter (we recommend the Lakeshore Model 450 for both its AC and DC accuracy), as well as an axial (and ideally a transverse) probe. This method is selected from the menu as "Calibrate/Cal Field, and has two sub-options for "20 Oersteds" or "100 Oersteds" depending on the field range of the instrument being calibrated. An additional higher field choice is available on the high-field Model 110 instruments.

After selecting this option, the user will be taken through an automatic step-by-step process that involves measuring the DC field produced by the instrument using the gaussmeter, and having the instrument adjust its field to match the selected (20 Oe or 100 Oe) value. The process can be repeated several times if needed, and then "Canceled" when the calibration is adequately close.

This process should be repeated with the instrument set to Transverse field, using either a transverse reading probe on the gaussmeter, or an axial probe held in the transverse direction as close as possible to the center of the coils (not possible with solenoid drive coils).

Note that the Lakeshore 450 meter when switched from DC to Peak mode can be used to verify the field strength being produced by the instrument in normal AC operation, but its accuracy at 10 Hz is only 5%, and most other AC gaussmeters are worse.

3. R Calibration

Resistance mode calibration of the instrument can be based either on an internal 0.02% standard resistor ("Calibrate/Cal R/Internal"), or a sample of known resistance can be put in place with the tool in R mode, and the command "Calibrate/Cal R/User Value" selected to allow the tool to be calibrated to read the proper value for the sample being measured. Additionally, a manual adjustment feature is also available to allow percentage tweaks in the calibration constant.

4. <u>dR Calibration</u>
Calibration of dR/R can also be accomplished using internal precision resistors, by selecting the command "Calibrate/Cal dR/Internal").

Further options allow calibration of dR/R with a sample of known dR/R in place ("Calibrate/Cal dR/User Value"). This command has three sub-choices. The first does not assume that a dR/R measurement has been made (but it does assume the tool has been properly put in first R mode and the probes lowered, and then in dR mode). It will make a dR measurement, and then ask the user what the proper reading is, and recalibrate the tool.

The second option assumes that a dR measurement is already present in the Results window, and will use that measurement, along with the desired value specified by the user to do the recalibration. It is called a single point calibration because it assumes the presence of only one dR value, and only asks for one from the user. It adjusts only the gain in dR mode, with an assumption that there is no offset.

The two-point calibration method is more advanced, and assumes that two dR measurements have already been done (typically one on a high-resistance and one on a low-resistance sample), and will ask the user for the correct values for both of these samples. Both a gain and offset constant are then calculated, and used for all future dR measurements. Manual adjust options exist for both the single- and dual-point calibrations.

Please call or email if you have any problems or questions.

We are always interested in feedback on any problems you may be having with our products, or suggestions for changes that you would like us to make.

Contact:
Dr. Barry Megdal
President
SHB Instruments, Inc.
bmegdal@shbinstruments.com
(818) 773-2000