MPMS3 Application Note 1500-023

Background subtraction using the MPMS3

Abstract

While the experimenter should always strive to use/engineer sample holders that provide a magnetically uniform and low moment background as possible, for dilute magnetic samples, and/or when using the rotator, ETO, pressure cell, or FOSH sample holders, this may not be possible. In these select cases, background subtraction may become necessary. The MPMS3 is the most sensitive commercial SQUID-based magnetometer, and furthermore, when performing measurements using magnetic fields up to 7 Tesla, even seemingly miniscule impurities and inhomogeneities can produce a sizeable background signal compared to that of the sample.

The MPMS3 utilizes two different DC moment measurement modes, namely the classic DC scan and SQUID-VSM modes. Most importantly, background subtraction is treated differently for each measurement mode. This Application Note serves as an update to an older Note (1014-213), which describes background subtraction using the DC scan mode in the legacy MPMS and MPMS-XL magnetometers. As will be demonstrated, background subtraction using the SQUID-VSM measurement mode is relatively straightforward, whereas background subtraction using the DC scan mode requires substantial post-processing by the end-user.

Test Sample and Sample Holder

The test sample, for the purposes of this example, is a 2 mm x 2 mm $Ni_{80}Fe_{20}$ (10 nm)/Al (5 nm) bilayer film that was sputter deposited on a 0.5 mm thick thermally oxidized Si substrate.

A common challenge faced by the experimenter is how to easily mount and measure thin film samples such that the applied field is perpendicular to the film plane. If the sample can be cut into a 4 mm x 4 mm chip, then the sample can be easily wedged inside of a straw. Straw sample holders typically are often preferred as they have a low/uniform background moment. However, for small samples this is not possible and other sample mounting means, *e.g.* the brass half-tube along with quartz braces, must be utilized.

This Application Note will demonstrate background subtraction using the MPMS3 brass half tube sample holder (4500-608) along with the quartz braces (4096-399) for the aforementioned test sample. While this sample mounting technique is ideal for measuring thin film samples, unfortunately, it also presents a relatively non-uniform background where background subtraction is typically required. This sample mounting also allows for both DC scans and SQUID-VSM measurements to be performed and therefore easily compared, as will be done in this Application Note.

Below is a picture of the mounted sample. Note, two additional quartz braces are inserted on either side of the sample to promote a more magnetically uniform background for DC scan measurements. This arrangement also allows for easily removing the sample for the necessary background-only measurement.

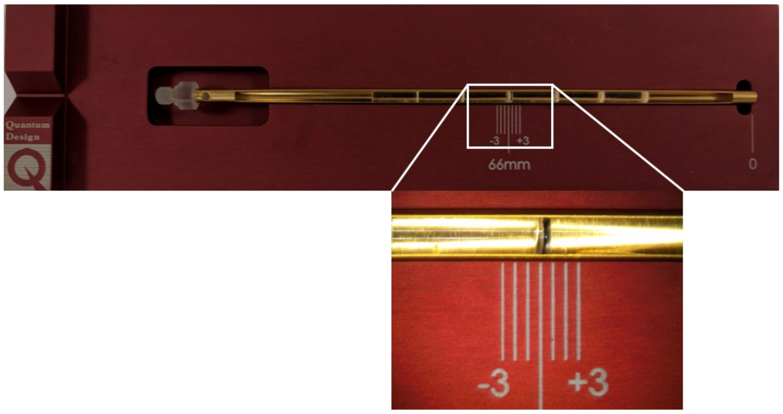


Figure 1. Sample mounting.

Background Subtraction: SQUID-VSM Mode

As the SQUID-VSM measurement mode does not require fitting a $V(z)$ waveform to calculate the moment, background subtraction is accomplished by a simple point-by-point subtraction of the $Moment_{background+sample}$ and $Moment_{background}$ measurements. It is critical that the *background+sample* and *background* measurements are as identical as possible, with the exception of course being the presence of the sample. Important considerations when performing a background subtraction using SQUID-VSM and DC scans include:

(1) Measure the *background+sample* and *background* datasets using the exact same sequence and stabilize for each field and/or temperature.

(2) Ensure the *background*-only measurement preserves the sample holder geometry as close as possible as that used during the *background+sample* measurement. For example, in regard to the sample holder shown in Fig. 1, the same gap between the two quartz braces securing the sample should be maintained for the *background*-only measurement.

(3) Ensure the sample rod is in the same rotational position in the linear motor for both the *background+sample* and *background* measurements. If the sample holder and/or sample is not perfectly centered, then there could be radial offsets between the two datasets that could introduce error during background subtraction. One can mark one of the magnets at the top of the sample rod with a felt pen (red dot) and use an alignment mark on the linear motor to ensure the sample rod is inserted the same way for each measurement, as shown below in Fig. 2.

Figure 2. Alignment marks to ensure the same rotational position of the sample rod to minimize any radial offsets of the sample and sample holder within the gradiometer.

Fig. 3 shows the *background+sample* (gray squares) and *background* (red circles) SQUID-VSM measurements. Clearly, the background moment provides a significant contribution to the total measured moment. However, simply subtracting the two datasets yields the magnetic moment of the *sample* (blue triangles) alone. The sample data shows an out-of-plane saturation field of approximately 1 Tesla, consistent with a $Ni_{80}Fe_{20}$ thin film, and a high-field diamagnetic contribution consistent with the Si substrate. The SQUID-VSM data in Fig. 3 can be used as a comparison for background subtraction using DC scans, as discussed in the following section.

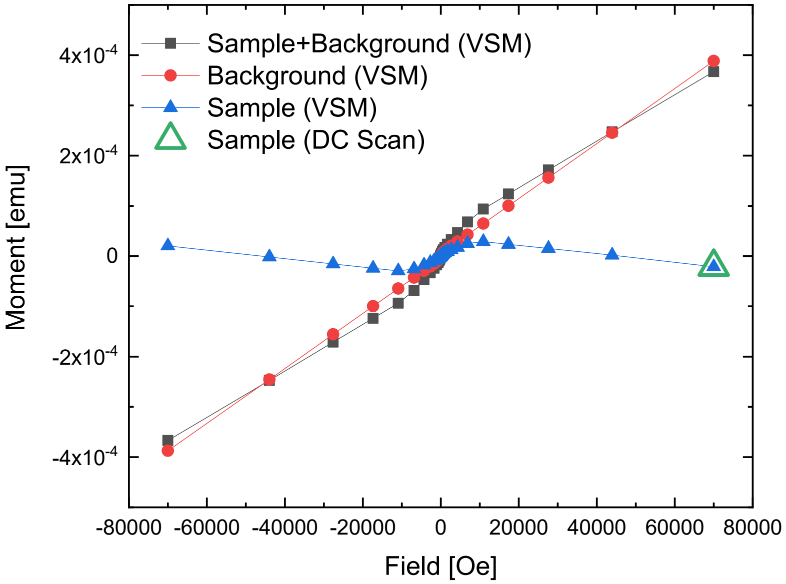


Figure 3. Thin film measurements using SQUID-VSM and DC scans.

Background Subtraction: DC Scan Mode

Background subtraction using DC scans is significantly more involved than for SQUID-VSM measurements as the moment is calculated by fitting an entire $V(z)$ waveform as the sample moves through the 2nd order gradiometer, where typical DC scan lengths in the MPMS3 are 30-35 mm. Therefore, proper background subtraction relies on first subtracting the $V(Z)$ waveforms of the *background+sample* and *background* measurements from each other, then re-fitting the resulting *sample* waveform. This must be done manually in postprocessing and requires first saving the raw (*.rw.dat*) datafile for the *background+sample* and *background* measurements. Refer to Application Note (1500-022) for a description of the *.rw.dat* data file format.

The fit function

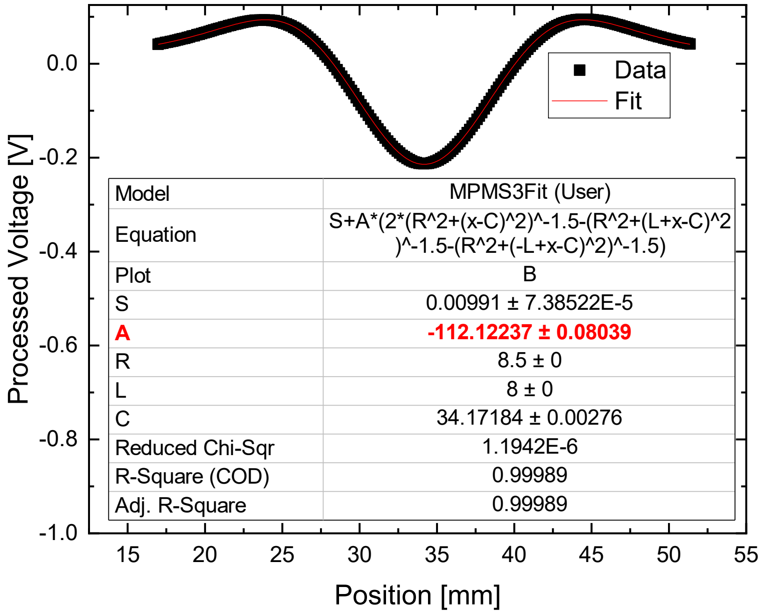
The basic functional form used to fit the $V(z)$ waveform is essentially unchanged from that presented in the original Application Note (1014-213). However, the dimensions of the 2nd order gradiometer are different, most notably the gradiometer is now much shorter. The basic functional form is:

$$V(z)=S+A\{2[R^2+(z-C)^2]^{-3/2}-[R^2+(L+z-C)^2]^{-3/2}-[R^2+(-L+z-C)^2]^{-3/2}\} \qquad \text{(Eqn. 1)}$$

where S is an offset voltage, A is the amplitude, R is the radius of the gradiometer (8.5 mm), L is half the length of the gradiometer (8 mm), and C is the sample center position. When performing fits it is recommended to always use the Processed Voltage saved in the *.rw.dat* file, which has already removed a linear drift and offset voltage. For this Application Note the *Origin* analysis software is used to perform fitting routines. When fitting the $V(z)$ waveform treat R=8.5 mm and L=8 mm as constants and S, C, and A as fit parameters. Note, when fitting the processed voltage data, S should be very small and for a properly mounted and centered sample C should typically be near 34 mm. The most important quantity extracted from the fit is the amplitude A, which via a system specific calibration factor can be used to calculate the magnetic moment.

Calculate the system-specific calibration factor

To calculate your system-specific calibration factor it is recommended to measure the Pd reference sample (4500-645) at room temperature for a given magnetic field and *Range* and save the *.rw.dat* file. Note also the reported moment (*e.g. DC Moment Free Ctr*) from the *.dat* file. Fit the Processed Voltage data using the above equation and note the amplitude A, as shown below.



Model	MPMS3Fit (User)
Equation	S+A*(2*(R^2+(x-C)^2)^-1.5-(R^2+(L+x-C)^2)^-1.5-(R^2+(-L+x-C)^2)^-1.5)
Plot	B
S	0.00991 ± 7.38522E-5
A	**-112.12237 ± 0.08039**
R	8.5 ± 0
L	8 ± 0
C	34.17184 ± 0.00276
Reduced Chi-Sqr	1.1942E-6
R-Square (COD)	0.99989
Adj. R-Square	0.99989

Figure 4. Fitting the Processed Voltage for the Pd standard measured at 50 Oe using *Range*=1.

The calibration factor for a given range is simply the reported moment divided by *A*. This exercise is repeated for the four available *Range* values (1, 10, 100, and 1000) and appropriate applied fields, as shown below in Table 1. The calibration factor values for a user system should be similar in magnitude and also have a negative sign. As expected, each calibration factor differs by an order of magnitude for each measurement range.

Table 1. System specific calibration factors.

Range (Field)	**Reported Moment [emu] DC Free Ctr**	**Amplitude from Fit [*V·mm³*] *A***	**Calibration Factor [emu/*V·mm³*] Reported Moment/*A***
1 (50 Oe)	7.0556E-5	-112.122	-0.000000629
10 (500 Oe)	7.0965E-4	-112.891	-0.000006289
100 (5000 Oe)	6.9612E-3	-110.711	-0.000062877
1000 (50,000 Oe)	6.9212E-2	-110.007	-0.000629159

Perform the background subtraction using DC scans

As a demonstration of background subtraction using DC scans the same sample/sample holder for the SQUID-VSM mode tests described above is used in order to provide an easy and direct comparison between the techniques. The processed voltage waveforms for the *sample+background* and *background* at 7 T and for Range=1 are shown below in Fig. 5.

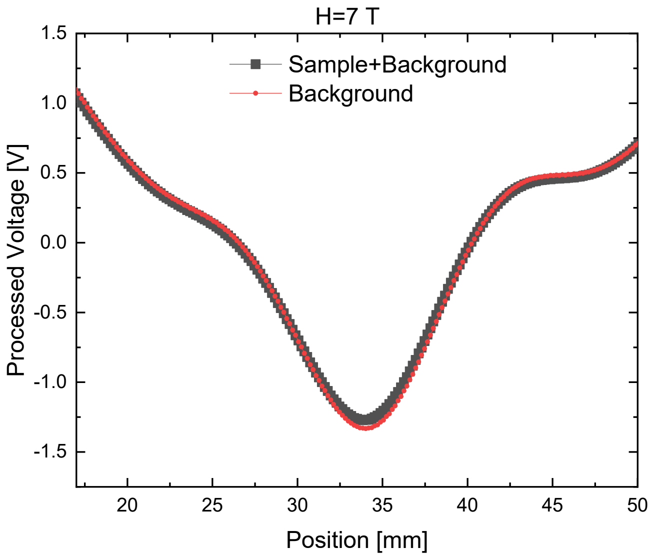
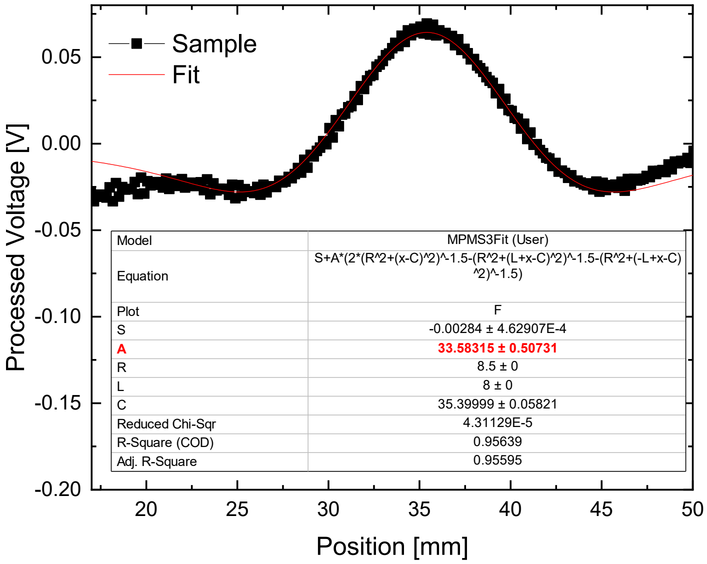


Figure 5. The Processed Voltage for the *sample+background* and *background* measurements collected at 7 T with *Range*=1.

Clearly, the *sample+background* and *background* $V(z)$ signals are comparable in magnitude, similar to what was observed in the SQUID-VSM measurements. The *sample-only* waveform can be found by simply subtracting the two waveforms, as shown in Fig. 6 below.



Model	MPMS3Fit (User)
Equation	S+A*(2*(R^2+(x-C)^2)^-1.5-(R^2+(L+x-C)^2)^-1.5-(R^2+(-L+x-C)^2)^-1.5)
Plot	F
S	-0.00284 ± 4.62907E-4
A	**33.58315 ± 0.50731**
R	8.5 ± 0
L	8 ± 0
C	35.39999 ± 0.05821
Reduced Chi-Sqr	4.31129E-5
R-Square (COD)	0.95639
Adj. R-Square	0.95595

Figure 6. Resulting *sample-only* signal in black, derived from subtracting the two waveforms shown in Fig. 5, with the resulting fit shown in red.

The *sample-only* waveform is well-fit by Eqn. 1 resulting in a moment of -21.12 μemu (33.583156 x -0.000000629) at 7 T. This agrees well with the value found from the VSM background subtraction procedure described earlier and is plotted alongside those results in Fig. 3 (open green triangle).

Additional notes regarding background subtraction using DC scans

It is recommended to measure the *background+sample* and *background* datasets using the same range. However, if this is not feasible, then make sure to multiply the processed voltage for the *background+sample* and *background* datasets by the range at which it was measured before performing the subtraction. One can then simply use the Range=1 Calibration Factor to convert the fitted amplitude *A* to magnetic moment.

For questions, contact apps@qdusa.com.