

Application Note 1070-207

Using PPMS Superconducting Magnets at Low Fields

<u>Abstract</u>

The superconducting magnets used in PPMS cryostats are capable of generating fields on the order of 10⁵ gauss (10 tesla) with the value determined to a very high degree of accuracy over most of this range. The magnetic field value reported to the user is based only on the current flowing from the magnet power supply. At low fields (less than 1 tesla), the magnetic field experienced by the user's sample can deviate significantly from the reported magnetic field. For example, the magnitude of this field error can be up to 20 gauss in a 9 tesla magnet and up to 120 gauss in a 14 tesla magnet. The origin of this effect is described and methods for its mitigation are outlined.

Section I: Introduction

When using a superconducting solenoid to control the magnetic field in an experiment, the user must be aware of pinned magnetic flux lines and flux movement within the magnet in order to better know the magnetic field at the location of the user's experiment. There are additional effects to consider when sweeping the magnetic field such as current leakage through the persistent switch. This application note will discuss how these effects manifest in the magnets used for the Quantum Design PPMS. As the title implies, the effects are of greatest relative significance at low fields and are very important to keep in mind when conducting research on soft magnetic materials whose magnetic state is strongly affected by small changes in magnetic fields near zero field.

The first thing to point out is that at the time of writing this note (May 2009), Quantum Design does not incorporate a field sensor such as a Hall sensor in our instruments. The magnetic field reported by the PPMS is based on the current that is being generated in the magnet power supply (if the magnet is in driven mode), or the last current that was sourced from the power supply when it was connected to the magnet (if the magnet is in persistent mode). The magnet power supply current is translated into a magnetic field using a constant field-to-current conversion factor (or "B/I ratio") for that given magnet. This yields a magnetic field value that is very accurate at higher fields (above about 1 tesla).

There are four phenomena that cause the PPMS reported magnetic field to differ from the actual magnetic field at the location of the user's sample. Below is a brief summary of these effects. The remainder of this application note will describe each these phenomena in greater detail.

1) **Magnet remanence:** alloys such as those used in the windings of superconducting magnets will permit the magnet field to enter the superconducting material as threads of quantized magnetic flux when placed in sufficiently large magnetic fields. These flux lines are "pinned" and immobilized at defects in the material. Movement of flux lines causes energy dissipation and can destroy the superconducting state. When the magnetic

field is removed (for our case, when the magnet current is set back to zero from a high field), some of these pinned flux lines remain behind and create a small magnetic field seen at the sample location.

- 2) **Flux creep and escape:** over time, some of these pinned magnetic flux lines will redistribute and may even leave the wire material altogether. The term "flux creep" describes the diffusion of flux lines inside a superconductor. In persistent mode, escape of flux from the interior of the wire can lead to induced currents in the magnet due to the topology of the superconducting magnet.
- 3) **Current leakage in the persistent switch:** in the driven state of the magnet, the persistent switch is resistive and is in parallel with the magnet. When sweeping the magnetic field, the existence of an inductive voltage across the magnet implies that a small amount of current from the magnet power supply will go through the resistive switch instead of the magnet.
- 4) **Data synchronization:** the PPMS data acquisition software tries to synchronize the measurement of all the system variables including the magnetic field and the user's measurement (magnetic moment, resistance, etc.). This synchronization is accurate and precise to about 0.25 seconds. When measuring during a magnetic field sweep, this synchronization error limit can be observed.

An outline of this note is as follows: Section II will discuss the first two effects of remanence and flux motion. Section III will discuss the latter two effects of current leakage and synchronization as these are only relevant when charging the magnetic field. Section IV presents methods for mitigating the magnetic field artifacts that have been described.

Before reading this application note, it is important to understand the basics of magnetic field control in the PPMS as described in section 3.5 of the *PPMS Hardware Manual*. Familiarity with superconducting phenomena, especially the mixed state of type II superconductors, is also highly recommended as this note will not provide that background (see ref. 1)

Section II: magnet remanence and flux motion

The effect of magnet remanence at low fields is an offset error in the reported magnetic field. This offset is dependent on the magnet history and is opposite in sign when coming from positive versus negative fields. Figure 1 below illustrates how this offset manifests in DC magnetometer data for a sample that is magnetically reversible. If one plots the magnetic moment vs. the reported PPMS magnetic field, the red curve results. Note that the hysteresis curve is "inverted" so that there is an apparently negative coercivity for the sample! On the other hand, if one were to plot the moment vs. the actual magnetic field at the sample location, the reversible blue M(H) curve results. While it is unphysical for the entire M(H) hysteresis loop to be inverted (it implies continuous heat absorption by the material as one cycles the field) it is possible to have a locally inverted region of the loop. The existence of locally inverted hysteresis loops is of interest to the research community, so it is important to discern real sample physics from instrument artifacts, as pointed out in ref. 2.

If we define the field error as:

field error = (real magnetic field at the sample) – (PPMS reported field)

and plot this quantity as a function of the field in a typical 9 tesla PPMS magnet (see Fig. 2), we see that the absolute error decreases at higher fields, the sign of the field error depends on the direction of magnet charging, and that switching the field charging direction results in the field

error traversing to the appropriate leg in a short field duration (see traversing lines at -3 and +5 tesla).



Figure 1. A magnetically reversible material (blue curve) can appear hysteretic (red curve) due to superconducting magnet remanence. Note the inverted (clockwise) nature of the loop.



Figure 2. Field error (defined in text) vs. the charge field in a typical 9 tesla PPMS magnet. Note the negative sign of the field error when decreasing from positive fields.

For Figs. 2 - 5, the magnetic field at the sample location was determined by measuring a paramagnetic Dy_2O_3 sample at 300 K using the VSM option.

Another important feature to notice in Fig. 2 is that the field error is negative when discharging from positive fields. That is, if setting zero field in linear charging mode from +1 tesla or above, the resulting field at the sample will be about -18 gauss in this example. So, if one wants to reach true zero field it means stopping the field discharge short at +18 Oe instead of overshooting zero field as one might intuitively think (see footnote 3 for a discussion of magnetism units). Finally, while Fig. 2 shows mirror-symmetric curves for field increasing and decreasing, this is not strictly true on closer inspection. It has been observed that the field error magnitude near zero field is different by several gauss (-18 vs. +22 gauss for one particular 9 T magnet) for the different charging directions.

Figure 3 compares the typical magnitude of the field error in 9, 14 and 16 tesla magnets currently in production for the PPMS. Note the log-normal plot style necessitated by the large variation in the field error as a function of applied field.

As with the other figures, this should not be used to generate a correction for data taken on a different system as the magnitude of the remanence varies from magnet to magnet. Rather, these figures should be used for their qualitative information.



Figure 3. Typical magnitude of field error as a function of the magnet field for PPMS 9, 14 and 16 tesla magnets.



Figure 4. Drift in the magnetic field at the sample location upon setting zero field from a large positive field value. Final magnet state is persistent in each case. Experiment was carried out on a 9, 14 and 16 tesla magnet. Inset shows detail for the 9 tesla magnet: note that the field value decays and even changes sign after only a few minutes.



Figure 5. Effect of using driven mode (D) vs. persistent mode (P) after reaching zero field. Data from 9 tesla magnet in Fig. 4 used for this illustration. The field decay effect is essentially removed by using driven mode from the start (red dashed line). Reconnecting the persistent magnet to the power supply after 60 minutes (D) resets the field at sample to a value near the original field. Thereafter, decay rate (dB/dt) in persistent mode follows the behavior seen before the jump.

The effect of magnetic flux motion on the magnetic field at the sample location is illustrated in Fig. 4. In all cases, the field was set from a large positive field to zero in linear mode and set persistent immediately. For the 9 tesla magnet, the initial field was +5 kOe and for the 14 and 16 tesla it was >1 tesla. The effects shown here are seen to be independent of the initial field providing it was larger than the value stated above.

One notices that the magnitude of the drift in the sample magnetic field (\sim 20-25 gauss) is similar for all magnets but that this effect is relatively more dramatic in the case of the 9 tesla magnet where the field changes from an initial value of -18 gauss to a final value of +7 gauss over the course of 1 hour (shown in inset).

Further information about this flux motion is gained from using driven mode (D in Fig. 5) instead of persistent mode (P) after reaching zero. The 9 tesla magnet data from Fig. 4 is used to illustrate the fact that if one keeps the magnet driven for a long enough time after discharging the magnet, the drift in the field is minimal. In addition, if one sets driven mode after settling the field in persistent mode for 1 hour, the field value jumps back to the original value of -18 gauss. After setting the field back to persistent mode (near the 70 minute mark), the field continues to decay by a small amount.

CAUTION: As driven mode causes elevated liquid helium boil-off from the dewar, only use driven mode or any field charging if the helium level will remain above 60% throughout the experiment.

These unusual observations can be explained by the fact that a small amount of pinned remanent magnetic flux in the magnet escapes from the magnet windings into the bore of the magnet. As illustrated in the table and the accompanying figure below, a magnet is originally in its virgin state (1) in which no flux is pinned inside it. Upon connecting the magnet to the power supply and passing current in the windings (2), a positive field is generated in the sample space at the center. After removing the current in the magnet and setting the magnet persistent (3), some flux remains pinned in the magnet and has the same sign as the original field because it resulted from field lines penetrating the magnet at positive fields.⁴ However, the stray fields from these "dipoles" are of course of the opposite sign, hence the negative initial remanent field at the sample location after discharging from positive fields. After an hour in the persistent state, a small fraction of the pinned flux lines have escaped from the magnet windings and are in the bore of the magnet (4). Since this flux is now in free space, it must be represented by currents flowing in the magnet and these are in the direction of the original magnetic field. Since the magnet is superconducting, these currents will not dissipate. The small amount of flux escape has a large effect on the field at the sample location due to a field amplification effect: the field (seen at the sample location) when the flux has moved into the bore of the magnet is much larger than the stray field when that same amount of flux was pinned in the magnet.⁵ Upon reconnecting the magnet to the power supply (5) in driven mode with a set point of zero field, the power supply removes the positive current in the magnet and the field that remains at the sample location is due to the stray fields from the remaining pinned vortices in the magnet. The magnetic field at the sample is nearly the same as in state (3) because the vast majority of the flux has remained pinned in the magnet.

	magnet	magnet			top view of magnet
Step	state	mode	I _{magnet}	B _{sample}	
1	virgin	persistent	0	0	
2	charged	driven	$I_2 > 0$	B ₂ > 0	
3	remanent	persistent	I ₃ = 0	B ₃ < 0	
4	remanent + 1 hour	persistent	$I_4 > 0$	B ₄ > B ₃	
5	remanent, reconnect ed	driven	$I_{5} = 0$	$B_5 \cong B_3$	

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Section III: current leakage and data synchronization

When charging the PPMS superconducting magnets, an inductive voltage appears across the magnet

$$V = -L\frac{dI}{dt} = -\frac{L}{B/I}\frac{dB}{dt}$$

where *L* is the inductance of the magnet (typically around 35 henries for a 9 T magnet), *B/I* is the field-to-current ratio constant (typically around 1950 Oe/A for a 9 T magnet), and *dB/dt* is the requested field sweep rate. This leads to a voltage across the magnet typically in the range of 0.2 to 4 V. From Fig. 6 it is evident that there will be a leakage current through the heated persistent switch $I_{leak} = V/R$ in this case. Converting this leakage current into a corresponding offset in the magnetic field, this implies an offset of $\Delta H = 10 - 200$ Oe for the case of a 9T system. If the user is performing measurements during a field sweep, the reported field values are subject to this offset. The firmware for the PPMS corrects for this effect using a stored value for *R* and

correcting the reported field value by the calculated ΔH . Note that this correction can be expected to remove ~90% of the field charging artifact due to the errors in determining *R* and the variation in *R* under different conditions. Some older systems (those shipped before 2005) might not have this switch leakage correction in place. To check if your system is using it, open the program Mon6000.exe in the C:\qdppms\tools folder and type magswtr? at the command prompt and hit the Enter key. If you see a value of zero reported for *R*, then the correction is turned off. Please contact your Quantum Design service representative for help if you would like to implement the correction.



Figure 6. Circuit diagram for the PPMS magnet in driven mode. The persistent switch is heated into the normal state and has a typical resistance of $R \sim 40$ Ohm. Typical inductance of the magnet is $L \sim 35$ H. The small amount of leakage during charging is corrected in the firmware of the PPMS using a value for R determined at the factory.

The last issue that leads to artifacts in the reported field arises from the fact that the magnetic field and the user measurement (magnetic moment, resistance, etc.) do not happen at precisely the same instant. Their relative timing is locked to within 0.25 sec, as mentioned earlier. This timing specification can be compromised, however, if the processor of the PC is loaded heavily. This can occur when using an internet browser or other processor-intensive programs concurrently with

MultiVu. Hence, it is recommended to avoid other activities on the PC at the PPMS, especially when making high-throughput measurements such as M(H) loops with the VSM option at a 1 second logging interval.

Section IV: mitigating magnetic field artifacts in measurements

The magnitude of the remanence and flux creep in the magnet can typically be reduced down to 1-2 gauss by setting the field to zero (from an initial field above 1 tesla) in oscillate mode, or can be further reduced down to <0.1 gauss by using the Low Field option for the PPMS (P700 or P701) which employs a fluxgate magnetometer and a low field coil in the magnet to remove trapped flux from the magnet.

Once a low-remanence state has been achieved in the magnet, you can make measurements at low fields (-100 Oe < H < +100 Oe) without inducing a significant amount of remanence. However, a field of 1 kOe will already introduce a lot of pinned flux in the magnet. Some users have expressed interest in using the low field coil (provides up to +/- 15 Oe) for obtaining highly accurate and precise M(H) loops with the VSM option. Please contact Quantum Design if you are interested in automating such a custom measurement.

Some measurement situations (most commonly M(H) loops from high fields through zero field) do not permit the use of low field techniques discussed above. In this case there are some guidelines we recommend in order to achieve best results:

- Determine the magnet remanence and flux creep properties of your magnet (see Figs. 2 5) by using
 - a Hall sensor: 3084-180 is a mounted Hall sensor provided by Quantum Design, please read PPMS application note 1084-309 on our website <u>www.qdusa.com</u>; a pre-calibrated Hall sensor can be purchased from other vendors which will provide an absolute measure of the magnetic field while the provided QD Hall sensor calibration is sufficient only for relative measures of the magnetic field.
 - (if there is a VSM or ACMS option on your PPMS) a pure paramagnetic sample such as the Pd standard sample or Dy_2O_3 powder that is free of any measureable amount of ferromagnetic impurities (very important a ferromagnetic signature will lead to an underestimate of the real magnet remanence value); this option is good since in principle the M(H) slope for these materials is very nearly constant so one can determine the absolute field error at each field contact apps@qdusa.com for more information.
- The remanence properties of the magnet are very reproducible as long as the same field charging recipe is followed each time; thus, once the field error table is determined by using a standard sample, you can run the same field charging sequence on your sample and apply this field correction; alternately, you can modify the field set points of the measurement protocol so that the actual field is the desired one (in the example of the 9T magnet from Fig. 2, setting +18 Oe from positive high fields will produce a field at the sample of ~0 Oe).
- To avoid flux creep effects (Figs. 4 and 5) when making measurements such as remanent magnetization relaxation, keep the magnet in driven mode for one hour before setting it persistent sufficient liquid helium (>60%) must be present in the PPMS throughout the experiment.

- An alternate method which saves liquid helium is to set the field to zero in persistent mode, wait 1 hour, reconnect the magnet by setting zero field again, and at that point start measurements; this requires a wait of 1 hour before measurements ensue and may not be appropriate for all experiments.
- If the small field artifacts due to magnet charging are an issue, measure at fixed field values instead of during a field sweep; magnet can be in driven mode.
- Ensure that the correct value for the magnet persistent switch "normal" state resistance is being used.
 - Compare the measurement results (e.g., VSM M(H) on the Pd standard) when stepping the field vs. sweeping the field at various rates; they should be coincident to within the errors discussed in section III.

In conclusion, one must be cautious when using a magnet used for generating $\sim 10^5$ gauss to also accurately generate fields on the order of 10 gauss. By understanding the basic operating principles of a superconducting magnet and by following the guidelines laid out above, it is possible to greatly reduce artifacts in the reported magnetic field.

While we have focused here on the instance of the Quantum Design PPMS, the effects of remanence, flux creep and switch leakage currents discussed in this application note are relevant to any superconducting magnet with a persistent switch.

³ Note on the magnetic units convention: we refer here to the magnetic field **B** in units of gauss (where 10^4 gauss = 1 tesla) and it represents the real magnetic field at the sample. In contrast, the magnetic induction **H** is expressed in oersted (Oe) and, being defined as the field due to free currents, this is the one that the user controls when setting a current at the magnet power supply. In SI units, $\mathbf{B} = \mu_0(\mathbf{H} + \mathbf{M})$ where **M** is the magnetization density and is defined as arising from bound currents. For more information, see the Magnetic Units Conversion Table under Technical Resources at the Quantum Design website www.qdusa.com .

⁴ For viewing clarity in the figure, the magnetic field lines in the superconductor do not show the supercurrent vortices that generate the magnetic field.

⁵ Consider the persistent magnet as a superconducting ring of inner dimension of *R* and the pinned flux line in the superconductor with a radius *r*, where R >> r. Referring to the states in Table 1, we must compare the magnetic field at the sample location (taken as the origin) between the initial remanent state (3) and when the vortex moves out of the ring into the middle (4).

We know that flux is conserved: $\Phi = B_3(R) \cdot r^2 = B_4(0) \cdot R^2$

That is, the flux enclosed on vortex at position R (at the inner bore of the magnet) is compared with field in the bore of the magnet after the vortex moves out.

The equation above approximates the magnetic field B as constant over the area of the loops in both cases. While this is not strictly true, the error is small compared with the amplification effect we are observing and hence is neglected.

magnetic moment of vortex: $m = \pi r^2 \cdot I$

where I is the current flowing around perimeter.

magnetic field at center of the vortex (a current ring): $B_3(R) = \frac{\mu_0 \cdot I}{2r}$

put this value of *I* into above expression for moment: $m = \frac{2\pi}{\mu_0} B_3(R) r^3$

magnetic field at sample location due to pinned flux "dipole": $B_3(0) = -\frac{\mu_0}{4\pi} \cdot \frac{m}{r^3}$

insert expression for *m* from above: $B_3(0) = -\frac{B_3(R)}{2} \cdot \left(\frac{r}{R}\right)^3$

Use flux conservation equation to compute ratio of field seen at sample in state 4 vs. state 3:

$$\frac{B_4(0)}{B_3(0)} = -2\frac{R}{r}$$

Since $R \sim 2.5$ cm and $r \sim 10^{-3}$ cm, this amplification is over x1000.

This is why a tiny amount of flux creep out of magnet will have a large effect on the field seen at the sample location.

¹ M. Tinkham, Introduction to Superconductivity (McGraw-Hill, 1996).

² "Soft-magnetic materials characterized using a superconducting solenoid as magnetic source" Giovanni Mastrogiacomo, Jorg F. Löffler, and Neil R. Dilley, *Appl. Phys. Lett.* **92**, 082501 (2008), DOI:10.1063/1.2838733