Quantum Design

Application Note 1096-305

Sample Centering and Temperature-Dependent Magnetization Measurements Using the VSM

When making VSM measurements in the PPMS family of instruments (PPMS, DynaCool or VersaLab), the sample is positioned in the detection coil set by suspending it from a carbon fiber sample rod which is held in the VSM motor. The coil set is plugged into the sample chamber, which in the PPMS and DynaCool is an 87 cm long sealed thin-walled metal tube of which the upper 75 cm are type 304 stainless steel and the lower 12 cm are high purity copper. In the case of the shorter VersaLab chamber, the upper stainless section is only 17 cm long. This shorter chamber as well as the reduced temperature range (50-400 K) in VersaLab will reduce the effects discussed here, thus this application note will focus on the PPMS and DynaCool platforms.

The VSM motor position is fixed at the top of the sample chamber[1]. Thus, the centering of the VSM sample will be affected by relative length changes in the sample chamber versus the VSM sample rod. The carbon fiber of the sample rod has a very low thermal expansion coefficient, while the stainless steel of the (PPMS and DynaCool) sample chamber will contract in length by ~2mm when the sample region temperature changes from 300 K to 2 K. The VSM software provides a sample centering mechanism which is referred to as the "touchdown" operation, and compensates for this relative shift by periodically touching the end of the sample holder down to the VSM detection coil set base so that the current position of the sample relative to the coil set is determined (see the relevant *VSM Option User Manual* for your platform for more details on this).

Figure 1 shows a VSM data file for a moment vs. temperature sweep in which touchdown sample centering was performed every 10 K or 10 minutes (these are the default settings). The "Center Position (mm)" quantity refers to the motor's position where 65 mm is the top of travel and 0 mm is the bottom, thus any changes to this value will reflect relative length changes between the sample rod and sample chamber. An increase in the center position value means that the sample chamber has contracted relative to the sample rod. This temperature scan was done very fast (~ - 7.5 K/min) in order to demonstrate the effect of thermal contraction – note the logarithmic axis for temperature.

[1] The one exception to this is on the 16 tesla PPMS system in which the large magnetic field gradients cause the motor to be pulled down slightly at high magnetic fields, necessitating sample centering as a function of magnetic field.

The main point in this figure is that the sample position is moving for **4 hours after temperature is stabilized at the VSM coil set**. This is because of the large thermal mass of the middle portion of the stainless steel tube and the fact that the temperature control algorithm is optimized in order to stabilize the temperature at the sample location, not in the middle of the sample chamber. In other words, it takes several hours for the thermal profile of the sample chamber to stabilize after a new sample temperature is established. In this case, the chamber appears to be expanding slightly over these 4 hours, which could be due to the fact that the very fast cool down under-cooled the middle part of the sample chamber.

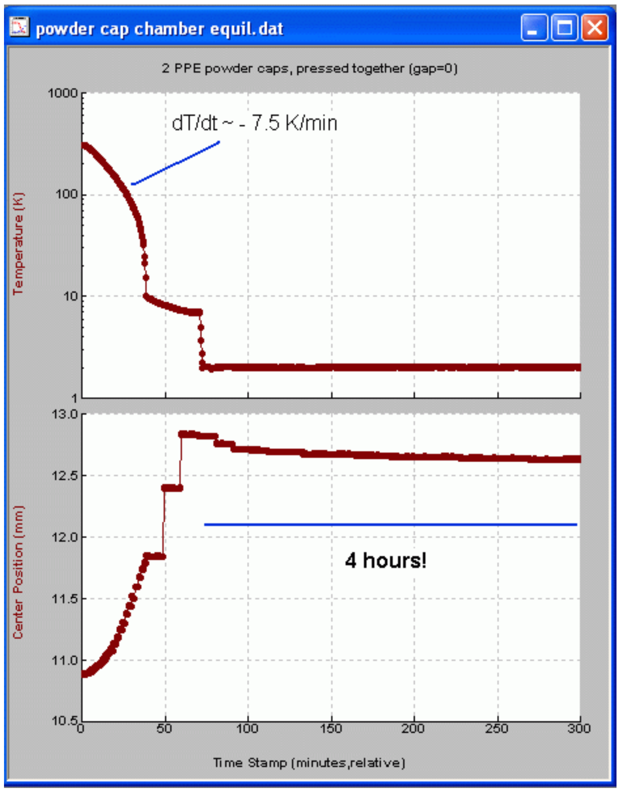

Figure 1: VSM data file showing change in sample position as temperature is lowered. Note log (T) axis.

The reason that sample centering is so important is that the reported magnetic moment depends on the vertical position of the sample relative the center of the coils. To understand how an error in vertical centering translates into an error in the reported moment, one must perform a centering scan on the sample as is typically done in the sample installation wizard of the VSM software. This data for the most recent scan is stored in the file `ScanData.dat` located in the `\VSM\LogFiles` folder. Figure 2 shows centering scans for the sample measured here (left) as well as an ideal sample (right). The vertical axis *Source* [2] corresponds to the magnetic moment in units of emu while the horizontal axis *Position* is the motor position in units of mm. The sample used for this investigation was two empty VSM powder sample holders pressed together, and

[2] in older versions of VSM software, one must plot *M.R.* instead of *Source* on the vertical axis.

was chosen because the reported moment was a strong function of position (change in reported moment vs. vertical position dM/dz ~ 20% per mm). When measuring small magnetic moments, this often occurs because the magnetic "end effect" of the sample holder or other nearby material (like the powder capsules) can dominate over the magnetic response due to the sample. In contrast, the ideal sample exhibits an extremum at the sample location so that the moment is independent of position to a first order approximation.

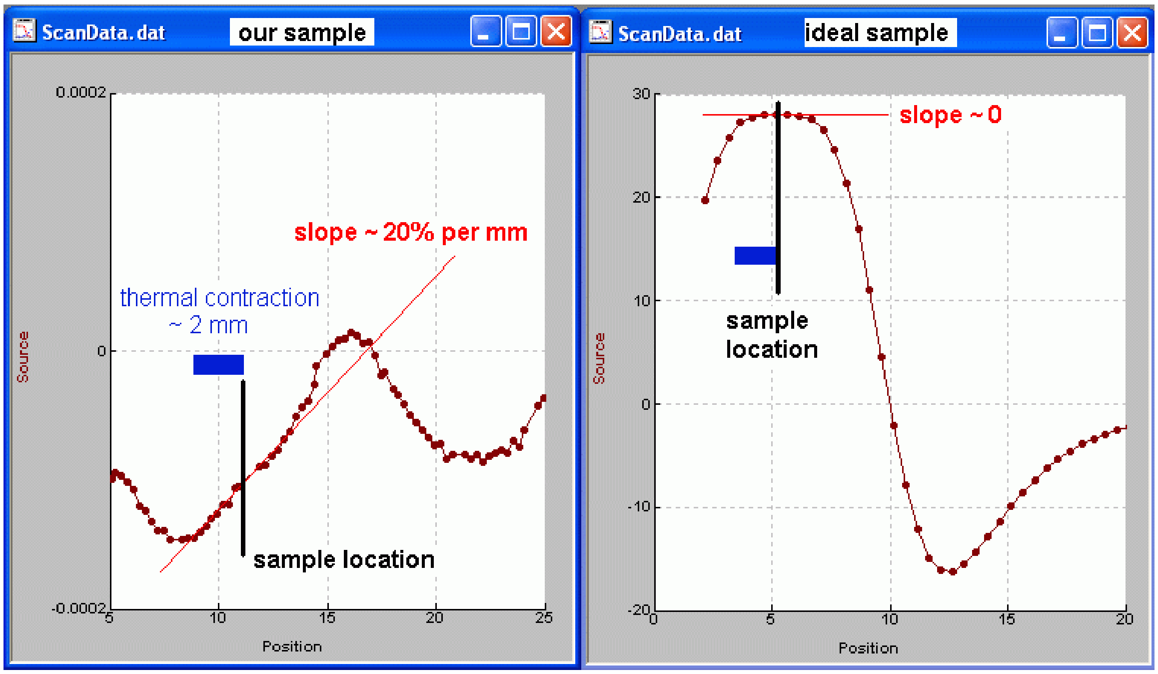

Figure 2: Centering scan data taken in 1 tesla applied field for the sample used in this investigation (left) as well as an ideal sample (right). Blue bar shows position shift expected when cooling from 300 K to 2 K without performing touchdowns.

An example of the artifact in the reported moment is shown in Figure 3, where the sample thermometer has reached a plateau near 10 K during cooling and hence the touchdown centering operations occur less frequently. That is, they occur only at 10 min. intervals, instead of the ~1.5 min. intervals which corresponded to 10 K changes at higher temperatures. However, it can be seen from the Center Position vs. time plot that **the sample chamber is still contracting at the same rate despite the fact that the VSM sample thermometer has reached a stable temperature**. This means that the sample walks off by as much as 0.6mm over the course of 10 minutes, and the reported moment drifts accordingly.

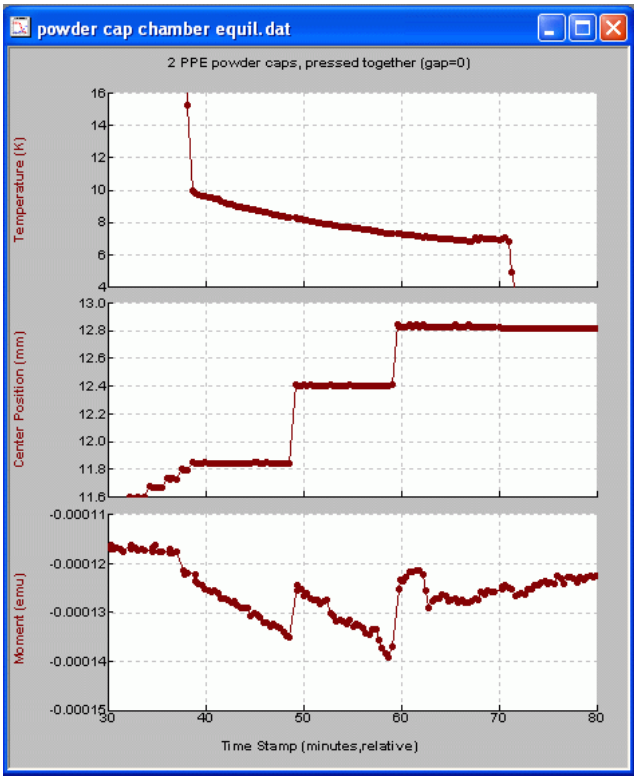

Figure 3: showing artifact in reported moment due to errors in vertical centering.

Thus, the following points should be kept in mind whenever making VSM measurements, and especially when temperature-dependent measurements are being performed:

1) When making temperature scans, touchdown every 10 K.
2) When at stable temperature, touchdown every 10 minutes unless you are confident that the sample chamber thermal profile is stable. Keep in mind that this will be affected by any temperature changes within the last 4 hours.
3) Try to mount your sample so that the reported moment is not a strong function of vertical position (see "ideal sample" centering scan in Figure 2).

If the touchdown centering operation is not appropriate to your sample, it is possible to activate an advanced centering option which allows you *within a measurement sequence* to re-center the sample by scanning or by setting an absolute motor position. Keep in mind that there are more risks involved when automating the centering scan or when setting the absolute motor position that can result in damage to the sample, sample rod, or even the motor. Please contact apps@qdusa.com for more information if you would like to access this advanced centering feature.