



## *Application Note 1500-012*

# SVSM User Training and Example Sequences

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## SQUID VSM Training – Example 1

### Zero Field Cooled – Field Cooled Measurement of a Superconducting Sample

This sequence and data file demonstrates the measuring of a superconducting sample in a nearly zero field environment (<5 Oe) and an environment with a finite field. The sample being measured is the provided standard of Indium and NbTi on a brass holder in the user kit. Indium has a known superconducting transition temperature ( $T_c$ ) of 3.40K at zero field. The sample is centered with an applied field prior to the start of the sequence.

#### Part 1: Sequence Parameters

- The magnetic field is first set to 2 Tesla and oscillated to zero to remove the remanent field in the system and achieve 0 Oe  $\pm$ 5 Oe.
- We cool to a temperature above  $T_c$  and then wait for an extended period to thermalize the system and sample.
- Measure while cooling to demonstrate that the standard magnet still has a remanent field. The MvsT sequence command is utilized because it handles all the correct timing, waiting and data collection built-in.
  - Start at a temperature above and stop below  $T_c$ .
  - Since we are interested in an effect that has a dependence on temperature, we sweep with a slow rate.
  - Sweep continuously so we do not have to wait to stabilize at each temperature for one minute. This decreases the temperature accuracy slightly in return for a much higher data collection density and shorter run times.
  - 6mm peak amplitude is chosen for the VSM measurements. The signal received for a sample scales with the square of the vibration amplitude. The peak amplitude is an excellent choice for measuring most moderate to low moment samples (less than  $10^{-3}$  emu). This ensures that we have good sensitivity in the normal region for the sample. Smaller amplitudes might be selected for samples with exceeding large moments or if problems are encountered with temperature stability at the lowest temperatures.
  - A two second averaging time increases the noise floor of our measurements in exchange for increasing the density of points in temperature so we have a more accurate picture of the transition temperature.

- Using a fixed range ensures that we do not lose any data during the transition due to autoranging. This can be especially important while sweeping a parameter or making a time dependent measurement with a moment that changes quite rapidly.
- Auto tracking is enabled since we are sweeping temperature.
- A small bias field, greater than the typical remanent field is applied.
- Using the same MvsT sequence command, we warm the sample through  $T_c$  to show the ZFC transition.
- Now field cool the sample using the same MvsT measurement.
- Finally, the system is returned to room temperature and zero-field in preparation for the next sample.

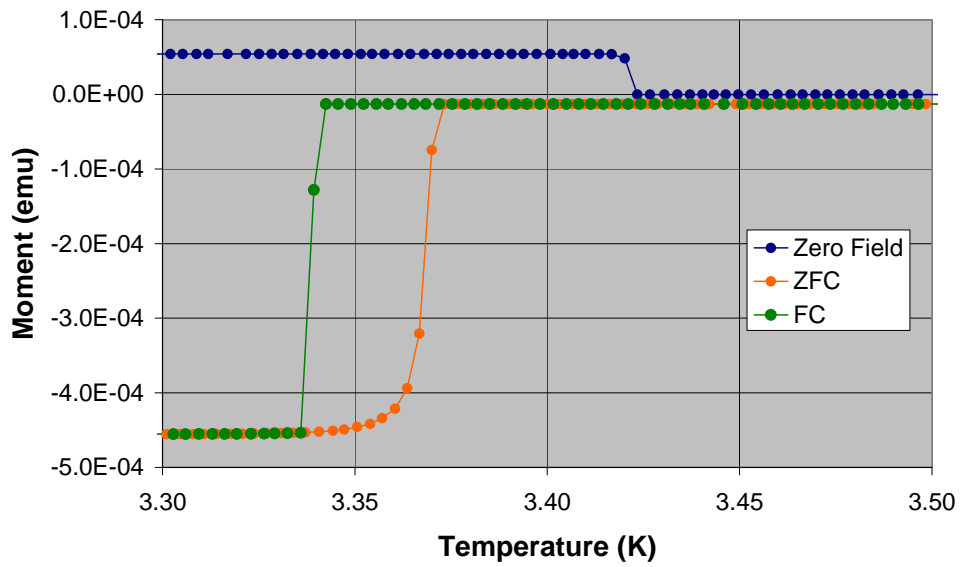
## Part 2: Sample Data

The attached figure is data from the example sequence. Several features are noticeable and we will go through them each in turn. First and most noticeable for the results of a zero field cooled measurement, the system field is not zero. The remanent field on the system oscillating to zero from 2T is -1.0 Oe for this system. This small negative field is the cause for the observed positive moment in the superconducting state. If smaller fields are needed, there is an option for the SVSM which uses the ULF module and can achieve  $0 \pm 20$  mOe. Applying a small positive field that is larger than the specified remanent fields for our system shows the superconducting temperature being suppressed.

Another piece of information that is evident about the system is that even though we have milliKelvin resolution on temperature of the system, our temperature accuracy is still 1% of the reported temperature. The transition temperature of indium is 3.40K and we can see that the transition temperature in the example data set is  $3.423\text{K} \pm 0.003\text{K}$ , but the accuracy of the temperature is  $\sim 0.034\text{K}$  in this region.

The difference in the transition temperatures between the field cooled curve and the zero field curve is from a supercooling of the sample. This effect is dependent on the actual applied magnetic field and is well documented. For more information on the effect please consult Feder and McLachlan paper from 1969 on the topic.

# ZFC and FC Indium



Feder, Jens and David S. McLachlan. "Superheating and Supercooling in Single Spheres of Tin, Indium, and Gold-Plated Indium." *Phys. Rev.* Jan 1969: 763—776.

## SQUID VSM Training – Example 2

### Magnet Remanent Field

This sequence and data file demonstrates how to determine the remanent field left in the magnet after different operations. The sample used for the measurements can either be the palladium mounted on the brass holder or the Er-YAG mounted on the quartz. The magnitude of the moments received in the data file will change, however the overall analysis made will not.

**Note:** The provided example sequences need to be run with MultiVu version 1.0.5 or higher. They might not work prior versions of MultiVu.

### Part 1: Sequence Parameters

- Set 7 Tesla and center the sample to make sure that we are properly centered on the sample.
- Set zero field from 7 Tesla linearly to maximize the remanent field left in the magnet.
- Measure a four quadrant MvsH measurement from 100Oe to -100Oe.
  - To prevent artifacts in the measurement, a sweep rate is used that does not cause the magnet to change between fields in time scales that are less than 1 second.
  - Unless very slow ramp rates are used and for specific purposes, we always recommend stabilizing at field rather than sweeping the field for measuring MvsH loops.
  - These are moderately sized moments and 5 second averaging with 5mm peak amplitude provides excellent measurement quality.
  - Sticky auto ranging and auto tracking are usually always enabled outside of specific applications.
- Set a 7 Tesla field and oscillate the field in the magnet to zero.
- Measure a four quadrant MvsH measurement from 100Oe to -100Oe.
  - The measurement parameters are the same as the previous MvsH measurement
  - This measurement loop is an example of joining together a high resolution section centered on zero with a faster low resolution section. This allows for collection of more data in the region where we are interested mostly.
- Reset the magnet to remove the remanent field from magnet.
- Measure a final four quadrant MvsH measurement from 100Oe to -100Oe.
  - The MvsH loop used is exactly the same as the previous loop.
- Return to room temperature and zero field in preparation for use by the next user.

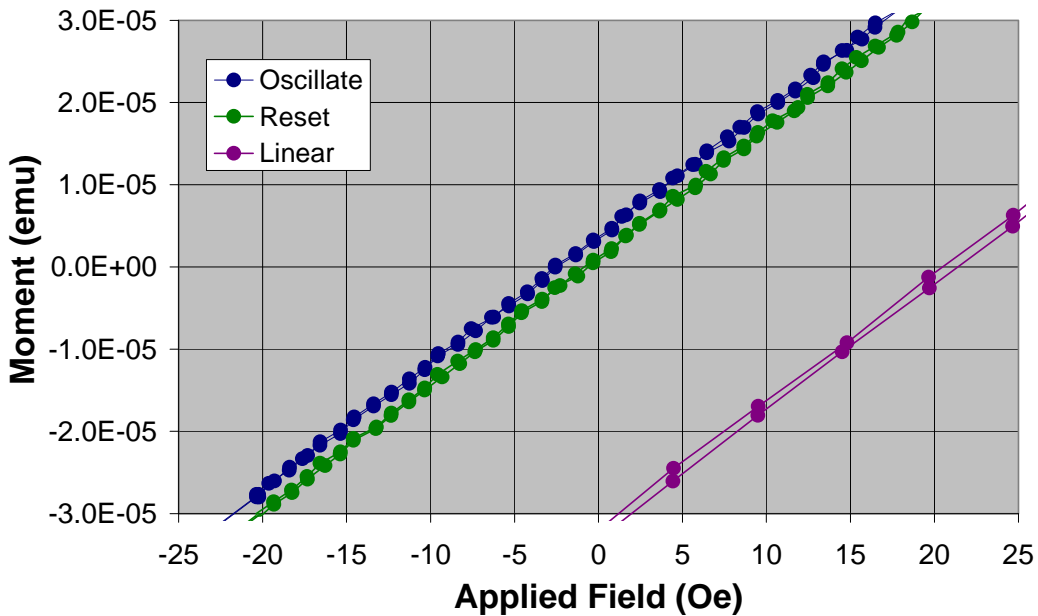
### Part 2: Sample Data

Attached is a section of the sample data set that is received from running the sequence. The first two curves are from oscillating the field to zero to remove the remanent field and resetting the magnet. Based on the zero moment intercepts from the curve, we can see that this SVSM typically has 1.0 Oe remanent field from resetting the magnet and 2.5Oe remanent field from oscillating to zero from several Tesla. The remanent field

that is left in the magnet is unique to each system and is left as an exercise to the user to determine the remanent field in their SVSM. To truly reduce the field in the sample space to the milli-Oersted range is to use the additional Ultra Low Field Option for the SVSM. This uses separate coils to remove the field and make the field more homogenous over the sample region.

The final curve show in the data set is taking the magnet to 7 Tesla and then going linearly to zero. The remanent field in the system is -22Oe for this system, even though the system reports zero field. This high of a remanent field is common when doing MvsH measurements to large fields (> 5000 Oe) and going linearly to each field. Unfortunately this is the nature of measuring with a superconducting magnet. On the SVSM, subsequent runs of the same sequence should produce the same fields and field history. Thus we can recommend that if you need to know the actual field at each point during a measurement, run your sequence with a known standard such as Pd and use that to calculate the correction to the field at each point. More information regarding the behavior of the superconducting magnet on the SVSM can be found in the application note on Magnetic Fields (1500-011).

## Magnet Remnant Field Using Pd



## SQUID VSM Training – Example 3

### Moment versus Field Loops

This sequence and data file demonstrates using the built in Moment versus Field command in MultiVu. It shows how to combine several MvsH commands into one loop to achieve a greater data density in the region of interest and less data in regions that are less important to speed data collection. The sample used for this measurement is the indium and NbTi on a brass holder.

**Note:** The provided example sequences need to be run with MultiVu version 1.0.5 or higher. They might not work prior versions of MultiVu.

### Part 1: Sequence Parameters

- First we reset the magnet to remove any remanent field left in the system and wait for this process to finish.
- The MvsH measurement is enclosed in a scan temperature loop to demonstrate how this is done and gather the MvsH data at several temperatures.
- Inside the loop an extra wait for thermalization of the system used.
- A series of MvsH commands are used in series to create a 6 quadrant MvsH measurement from 1000Oe to -1000Oe with more dense data collection from 100Oe to -100Oe.
  - To prevent artifacts in the measurement, a sweep rate is used that does not cause the magnet to change between fields in time scales that are less than 1 second.
  - Unless very slow ramp rates are used and for specific purposes, we always recommend stabilizing at field rather than sweeping the field for measuring MvsH loops.
  - These are moderately sized moments and 5 second averaging with 5mm peak amplitude provides excellent measurement quality.
  - Sticky auto ranging and auto tracking are usually always enabled outside of specific applications.
  - $H_0$  is the center point of the sweeps and the  $H_{max}$  and  $H_{min}$  are the end points. I use those to line up the sequence commands to create seamless measurement loops.
- After completing the MvsH measurement at several temperatures the system is returned to room temperature and zero-field in preparation for use by the next user.

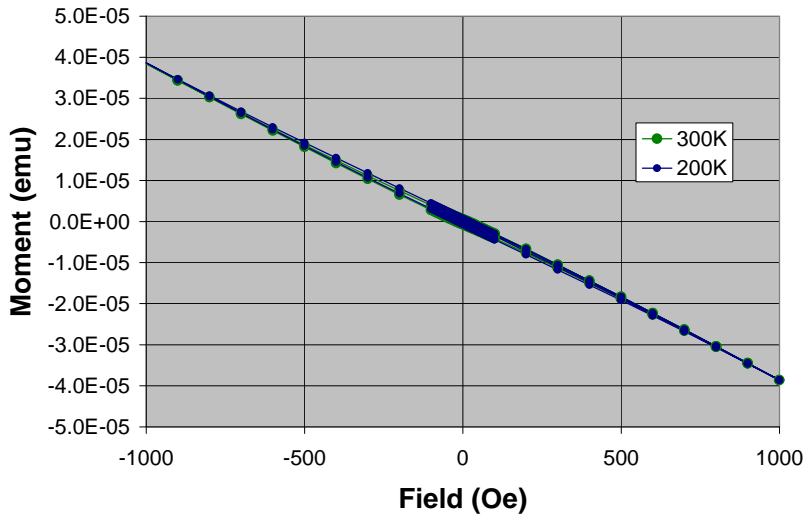
### Part 2: Sample Data

Attached is the section of sample data from running the sequence. The NbTi and In sample were chosen for this sequence for several reasons. The first is that this sample is the lowest moment sample that we provide at room temperatures so we can explore using an MvsH measurement for that. The second is that it illustrates some of the issues typically seen when using a brass holder for low moment samples.

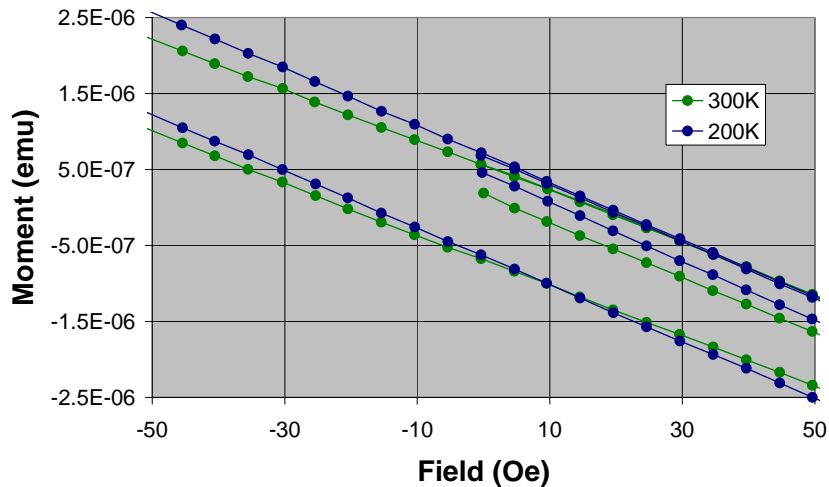
The moment versus field measurements are conducted below 5000Oe which helps prevent large remanent fields from building in the magnet. Each one of the separate MvsH commands connects to the next MvsH command to perform a seamless magnetization loop.

Finally, the small hysteresis present in the sample is due to magnetic impurities in the brass holder. This is not due to remanent field effects in the magnet which will cause a hysteresis in the opposite direction as explained in application note 1500-011. Brass sample holders should typically be used only for samples with moments greater than ( $10^{-6}$  emu).

### Moment versus Field - Indium NbTi



### Moment versus Field - Indium NbTi Zoomed In



## SQUID VSM Training – Example 4

### Moment versus Temperature Measurements

This sequence and data file demonstrates the built in Moment versus Temperature sequence command MultiVu. It demonstrates changing the parameters of the MvsT measurements to emphasize different features of the commands. The sample used for this measurement is the indium and NbTi sample on a brass holder. One important note about this sequence is that it assumes you know where the transition of the material is taking place and it takes a high density of points around the transition temperature. This sequence would not be appropriate for a sample with a transition temperature that is unknown or bounded by a large temperature range. It would be much more appropriate to perform a fast scan to determine roughly where the transition is and then modify this sequence to look at the transition in detail.

The other important detail about this sequence is that it shows how to tailor the MvsT measurement commands to look at specific characteristics in a transition. The loops looking at the two different transitions are slightly different, emphasizing different elements.

**Note:** The provided example sequences need to be run with MultiVu version 1.0.5 or higher. They might not work prior versions of MultiVu.

#### Part 1: Sequence Parameters

- Set room temperature and a large field to center on the indium and NbTi sample which has a weak signal at room temperature.
- Center on the sample within the sequence. Centering the sample within the sequence is not necessary, but more of an illustration of what is possible. The sample does need to be centered at some point, either from the sample installation wizard or within the sequence.
  - *To prevent bad data from being collected during a measurement due to measuring at the incorrect position, it is highly recommend to verify that the locate scan gives a good fit to the data. The verification of the fit should be in the form of a manual locate scan which should be done prior to running a sequence on a given sample.*
- Oscillate the magnetic field to zero to remove the remanent field in the magnet.
- Go to 10K and then wait for a while for the sample to thermalize properly. 10K is slightly above the  $T_c$  for NbTi.
- Using the MvsT measurement command we are interested in looking at the transition of NbTi in detail.
  - For the NbTi transition we are more interested in the signal to noise ratio and size of the transition above the noise floor rather than the actual transition temperature. We will gear the parameters for this type of measurement.
  - A slightly faster sweep rate is used for the data collection to speed the data collection process than for the Indium transition.
  - Sweep continuously is used for the temperature sweep since stabilizing in temperature will cause extremely long sequences and decreased data density.

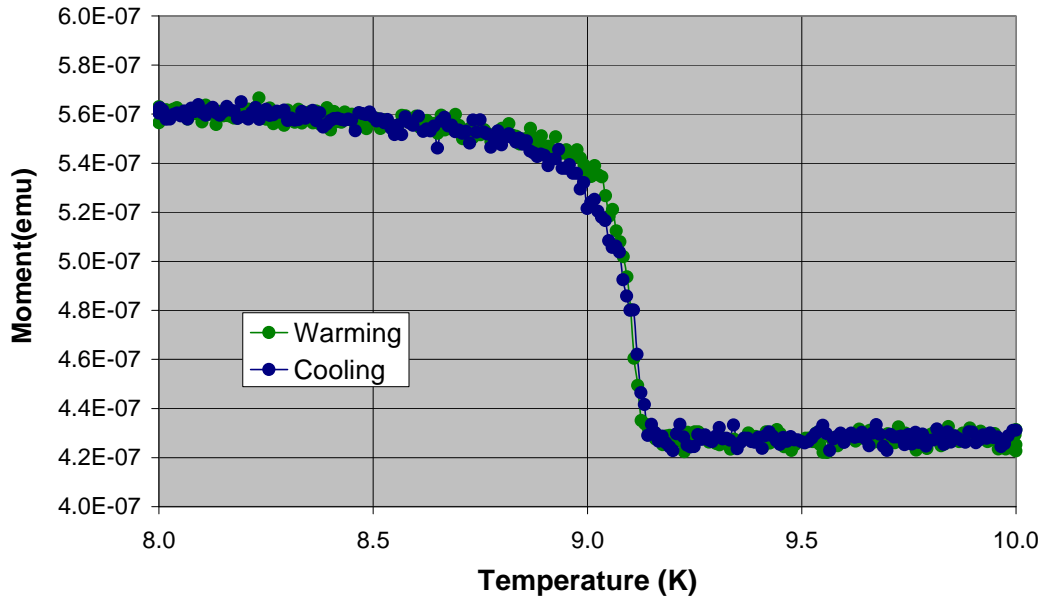


- We collect data continuously and keep all the measurements. We can post-process the data to remove the unneeded points. Due to the small amount of included NbTi, we have a very low moment transition. 6mm amplitude is chosen to provide excellent sensitivity and signal to noise ratio.
- Since the transition is small ( $\sim 10^{-7}$  emu) we use a 5 second averaging time to increase the sensitivity of our measurements at the expense of accuracy in the reported temperature.
- Sticky autoranging and autotracking are enabled as usual.
- Set 4K which is a temperature slightly above the  $T_c$  for Indium with a small wait to allow the sample to thermalize.
- Perform another MvsT command to look at the Indium transition between 4-3K
  - This time we are more interested in the temperature resolution of the transition, rather than the moment accuracy and the sequence command will reflect that.
  - A slightly slower cooling rate is used to help emphasize the transition temperature.
  - We still use sweeping continuously and continuous measuring to make sure that we capture as much data as possible.
  - We measure at 6mm again to reduce the background noise and emphasize our signal.
  - A faster averaging time of 2 seconds is used which will decrease the accuracy of the moment measurement, but increase our resolution on the temperature of the transition.
  - Sticky autoranging and autotracking are enabled as usual.
- We then perform the reverse MvsT from 3-4K to look at the temperature hysteresis present in the system to give us confidence that the sample is well thermalized during the process and to see if there is a difference between cooling and heating.
- 8K is then set which is slightly below the transition temperature of NbTi and a wait is used to ensure that the sample is equilibrated.
- A final MvsT measurement is performed from 8-10K to look at the NbTi transition. The same command, with just different temperature limits is used again.
- Finally the system is sent to room temperature and zero field in preparation for the next user.

## Part 2: Sample Data

Attached is a sample data set that is collected with this sequence. The first plot is the heating and cooling curve through the NbTi transition. The NbTi transition changes quite a bit depending on the composition of the alloy and so we do not have a firm number of what the transition should be. We do expect that the transition will be typically just below 9.2K. The transition itself is relatively small due to the small size of the sample ( $\sim 2 \times 10^{-7}$  emu in size) which is a nice demonstration of the noise floor of the machine. If we needed better resolution on the moment or better accuracy we would be better off mounting the sample to the quartz holder, but for our purposes brass is good enough. There are some small deviations in the transition from heating and cooling, but the over all temperature is constant which shows that we have a consistent thermalization for the two curves.

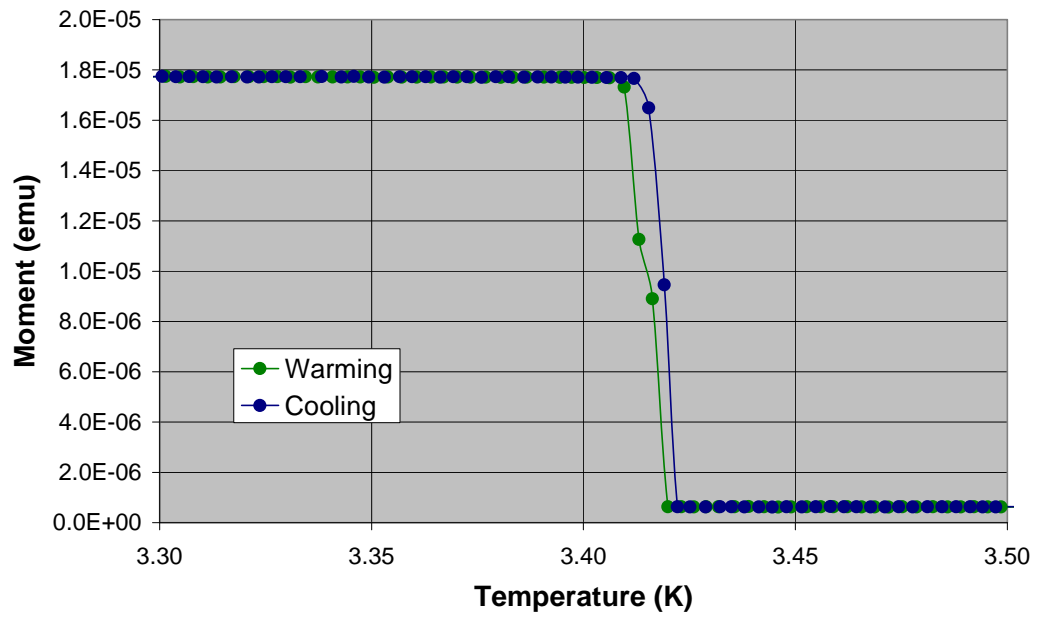
## M vs. T - Superconducting Transition For NbTi



The next sample is the Indium and since it is a pure sample (typically greater than 99.9% Indium), we know that the transition temperature is 3.40K. Using the remanent field in the system, we can induce a response from the superconducting transition that we can detect. This transition is very abrupt in temperature due to the purity and the fact that it is a type I superconductor. Since the remanent field in the SVSM is  $0 \pm 5$  Oe, the observed moment can either be positive or negative.

Another piece of information that is evident about the system is that even though we have milliKelvin resolution on temperature of the system, our temperature accuracy is still 1% of the reported temperature. The transition temperature of indium is 3.40K and we can see that the transition temperature in the example data set is  $3.423\text{K} \pm 0.003\text{K}$ , but the accuracy of the temperature is  $\sim 0.034\text{K}$  in this region.

# M vs. T - Superconducting Transition For Indium



## SQUID VSM Training – Example 5

### Isothermal Relaxation of the Magnetization for a Superconducting Sample – Above and Below the Critical Temperature $T_c$

This sequence and attached sample data demonstrate the measurement of the relaxation of the magnetization of a superconducting sample at a fixed temperature. We measure both above  $T_c$  and below  $T_c$  to show that the effect is coming from the sample and not from field decay within the magnet. The first measurement is a background measurement for comparison and the second is an actual measurement of the sample. The sample being measured is the provided standard of Indium and NbTi on a brass holder in the user kit. Each NbTi sample will have a slightly different transition temperature depending on the actual alloy in the wire, but it typically is around ( $T_c$ ) 9.2K at zero field. The sample is centered with an applied field (~2T) prior to the start of the sequence.

**Note:** The provided example sequences need to be run with MultiVu version 1.0.5 or higher. They might not work prior versions of MultiVu.

#### Part 1: Sequence Parameters

- Set the system to zero field to start the sequence and then cool the system to a temperature slightly above  $T_c$ . Wait for the sample to thermalize and then begin the relaxation measurement.
- While in the normal state for NbTi set a magnetic field of 3T from zero field going linearly to the field at the maximum charging rate that is possible for the SVSM.
- To measure at a fixed temperature and field for an extended period of time we use the Advanced Measurement Command, under the Measurement Commands, VSM heading.
  - To start the measurement we select start.
  - Continuous measuring allows the measurement to continue until we tell the system to stop measuring.
  - We are interested in measuring near the noise floor for the effect which typically takes several minutes to develop. 5 second averaging time gives a good compromise between providing a nice noise floor, not smearing the time dependence of the effect between successive points, and providing good resolution in time.
  - Autoranging and autotracking are enabled.
  - 6mm vibration amplitude provides a nice signal to noise ratio for samples with a moment smaller than 0.1 emu.
- To see the effect which is on the order of 1% per decade of time we measure for 40minutes. This is accomplished by waiting for 2400 seconds after starting a continuous measurement.
- The Advanced Measurement command is then stopped.
- Still in the normal state for NbTi, we set a magnetic field of 3T from the maximum field possible in the SVSM (7T).
- Repeat the same 40 minute decay measurement at 3T from 7T at 10K.

- Set zero field and 7K which is well below  $T_c$  for NbTi. Wait for the sample to thermalize at the new temperature.
- Perform the same series of field changes and decay measurements at the 7K in the superconducting state.
- Return the system to room temperature and zero field in preparation for the next user.

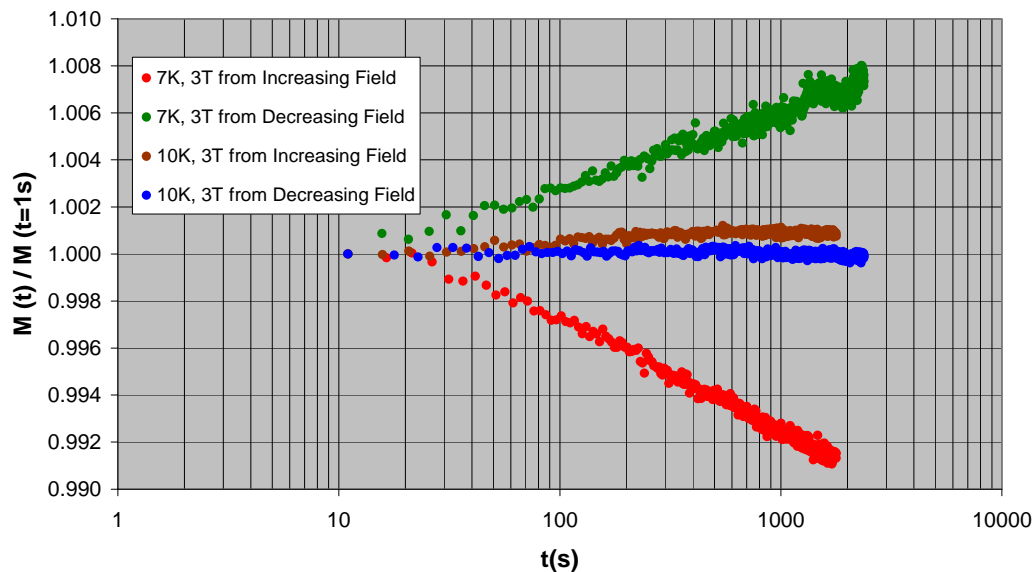
## Part 2: Sample Data

The attached figure is data from the example sequence. The noticeable features about the data are the distinction between the background measurement above  $T_c$  and then the measurement of the sample below  $T_c$ .

The reason for these two measurement sets is that we are interested in separating the effects of the magnet drift on the magnetization of the sample, from the actual decay of the magnetization. We can see there is a slight decay in the magnetic field as a function of time in the curve at 10K while in the normal state. This effect is examined more in the application note on magnetic fields (1500-011).

We can clearly see that there is logarithmic decay in the superconducting state of the magnetization as a function of time that has the opposite slope depending on the starting field as predicted by Bean's model for magnetization of a type II superconductor. To get the correct slopes, it would be appropriate to perform a background subtraction of the normal states overall magnetization from the superconducting state.

### Isothermal Relaxation of the Magnetization for NbTi; Above $T_c$ (10K) and Below $T_c$ (7K)



Bean, C. P. "Magnetization of Hard Superconductors." *Phys. Rev. Lett.* Mar 1962: 250—253.

Bean, C. P. "Magnetization of High-Field Superconductors." *Rev. Mod. Phys.* Jan 1964: 31—39.