

Vibrating Sample Magnetometry

Aims and Objectives

- Observe the changes in the magnetism of dysprosium as a function of temperature.
- Identify paramagnetism, ferromagnetism and antiferromagnetism.
- Understand the nature of a vibrating sample magnetometer.

Introduction

Some atoms, such as iron or nickel, have a net magnetic moment, meaning that the atom has a magnetisation, and can be envisaged as a small magnetic dipole. In a solid made up of these atoms, above a certain transition temperature, the magnetic moments act essentially independently and the material as a whole possesses no spontaneous magnetization - it is in a *paramagnetic* state. Below the transition temperature, the individual magnetic moments establish long range order, and the material as a whole displays order magnetic behaviour. In iron, there is a spontaneous magnetization, and all of the moments point in the same direction. This is called *ferromagnetism*. More exotic ordered states are also possible. To examine this here, you will use a *vibrating sample magnetometer* to investigate the magnetic response of a sample of dysprosium to changes in magnetic field and temperature.

Background

All atoms or ions can be placed into two categories: (i) paramagnetic, and (ii) diamagnetic. In the first case, when placed in a magnetic field, they will develop a magnetic moment parallel to the external magnetic field. In the second case, they will develop a magnetic moment anti-parallel to the external field. This experiment concentrates on an atom that is paramagnetic - dysprosium. Dysprosium is a rare earth metal; this means that the relevant valence electrons are from the $4f$ and $5d$ shells. At room temperature in a sample of dysprosium, there is no ordered magnetic moment, and the measured magnetization in zero field is $M = 0$.

What magnetization do you expect for a single dysprosium atom?

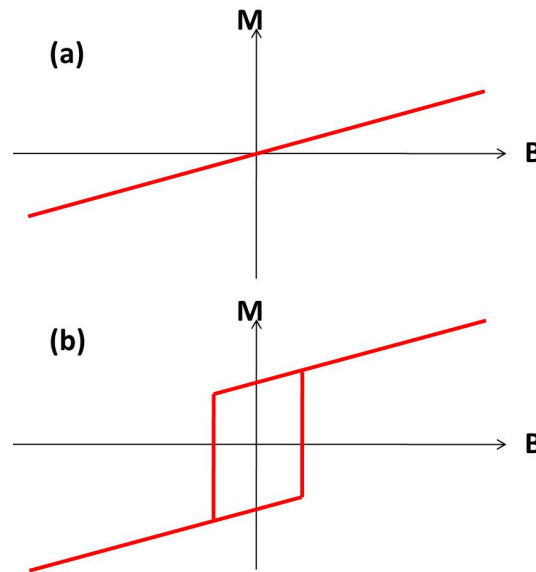


Figure 1: Magnetization as a function of external magnetic field for: (a) a paramagnetic material, (b) a ferromagnetic material (note the spontaneous magnetization in the absence of a magnetic field).

If such a material is placed in an external magnetic field, it develops a finite magnetization, which will vary linearly with applied field, disappearing if the field is removed (Figure 1a). This is characteristic of a *paramagnet*.

On cooling such a material down, it may develop into an ordered magnetic state. The simplest of this is ferromagnetism. Here, the ‘ferro’ indicates the development of spontaneous order, not the presence of iron. The curve in Figure 1b is an example of a *hysteresis loop*. The previous treatment of the sample is important in determining the condition of the sample at the current time. The loop shape arises in this case because of the energy cost associated with switching magnetization across the whole sample. This is accomplished by nucleation and growth of domains with a magnetization antiparallel to that in the bulk of the material. This is a lossy process, and the energy lost going round the loop can be calculated from the area contained by the loop.

Will this linear variation continue indefinitely?

There are many types of magnetic order. Figure 2 illustrates some of the types available using a 2D square lattice. This magnetic order arises due to quantum mechanical interactions between the atoms, known as *exchange* interactions. These are not the same as the dipolar interactions that one might expect between neighbouring magnetic dipoles. To see this, consider the energy associated with two neighbouring magnetic dipoles, μ_1 and μ_2 , separated by a distance \mathbf{r} . The energy varies as a function of the relative orientation of the dipoles.

$$E = \frac{\mu_0}{4\pi r^3} \left[\mu_1 \cdot \mu_2 - \frac{3}{r^2} (\mu_1 \cdot \mathbf{r})(\mu_2 \cdot \mathbf{r}) \right]$$

Estimate the energy that you would expect for the dysprosium, and convert this energy into a temperature. When you have made some measurements, compare your results with the energy scale calculated here.

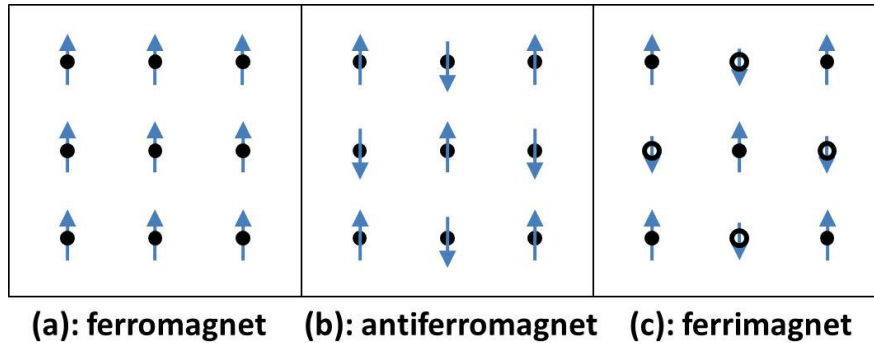


Figure 2: Cartoons of (a) ferromagnetic, (b) antiferromagnetic, and (c) ferrimagnetic order. The circles denote magnetic atoms, and the arrows indicate the orientation of the magnetic moment associated with each atom. In case (c), there are two types of magnetic atom, denoted by closed and open circles.

Experimental Method

The sample

The sample consists of a 6.6 mg sample of dysprosium. This is glued to a cantilever made of a piezoelectric ceramic (Figure 3). By applying a voltage, this is made to vibrate. The function generator supplying the oscillating voltage here is set to give a sine wave with ± 30 V peak-to-peak, at about 300 Hz. The peak-to-peak voltage should not exceed 60 V to avoid damaging the cantilever. The frequency set should match the mechanical resonance frequency of the cantilever. This resonant frequency changes as a function of temperature. At room temperature, the resonant frequency is ~ 270 Hz. The resonance is located by monitoring the response of the sample to the (amplified) input. At resonance at room temperature, this should be about 200 mV peak-to-peak, and is shown on the left-hand oscilloscope, with the voltage divided by 100 on screen. This signal is then passed to a lock-in amplifier and compared with the driving frequency. The out-of-phase component is monitored. This should be zero when on resonance. If it is not zero, the frequency will be automatically adjusted until

the resonance is found again. The time constant for this searching process is several seconds. This should all operate automatically, but you should periodically check the output on the oscilloscope to confirm that resonance has not been lost.

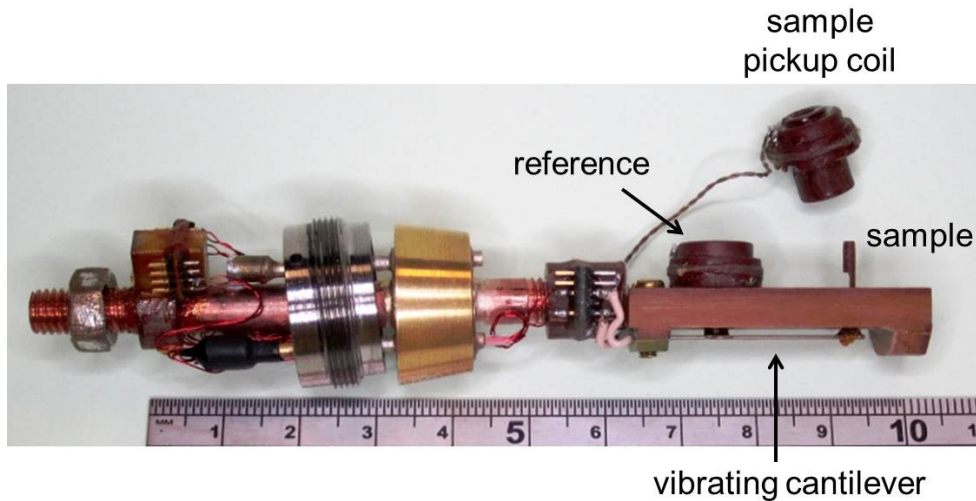


Figure 3: A close up view of the sample mounting stage, which is made out of Tufnol, a type of machinable plastic.

Figure 3 shows the sample stage. The sample is glued down at the end of the cantilever, and a pickup coil is then placed around it. As the magnetic sample vibrates, a voltage is induced in the pickup coil that is proportional to the magnetic moment. Further details on this principle can be found in the article by Foner listed in the recommended reading. A second reference sample is also present. This is a piece of NdFeB magnet. This is assumed to have a constant magnetization at all temperatures and fields, and therefore can be used to normalize the data measured on the dysprosium sample for any temperature or field-dependent behaviour of the cantilever and experimental setup. Measurements of the reference and the sample are made alternately; during a given experimental run, the computer will switch between the two using an optical relay, and report back measurements on both. The measurements of reference and sample pass through a lock-in amplifier, which has a particular *sensitivity* setting that determines the scale against which the measurements are made. It is possible for the measurements to overload the scale being used (as indicated by a red light indicating 'OVL'). In this case, the sensitivity will need to be altered to ensure accurate readings.

Why do we assume this?

To initiate a measurement, you will need to apply a magnetic field. *Do not go above 500 mT, as this may cause the magnet leads to melt.* To do this, power needs to be switched on the wall. The magnet power supplies (in the rack to the left) should then be powered up - switch things on from bottom to top. You can now start to make measurements using the software on the PC - *VSM*. This allows you to carry out

magnetic field sweeps and temperature sweeps. First of all, measure a field sweep at room temperature.

In your measurements, you obtain a voltage. This is proportional to a magnetic moment. Calibration measurements were performed earlier using a nickel sample instead of a dysprosium sample, as the magnetic moment of nickel is well documented. From this, it is known that the reference NdFeB magnet has a moment of 0.089 emu at zero field at room temperature. To calculate the final value for the dysprosium sample, you also need to consider the associated demagnetisation factor.

The next thing to do is to start cooling down the sample. Cooling power is provided here by helium, using a cryocooler. The helium compressor is located in another room, and will be turned on when you are ready to cool down. The compressor needs to be water cooled to work. Please ask a demonstrator to show you how to turn on the cryocooler. At this point, the sample will start to cool, unless you turn on the internal heater (by supplying a current to a resistor). To cool to base temperature takes approximately 1 hour. Apply a small field of 200 mT while the sample is cooling, and record the magnetization data as a function of temperature using the software.

Once below 100 K, try measuring another field sweep, and compare this with your previous field sweep at room temperature. At this point, you are ready to undertake further investigations as best seems appropriate.

To alter the temperature of the dysprosium sample, heat must either be supplied or taken away. In this experiment, there is a constant ‘cooling’ power supplied by the cryocooler. Heat is input by means of resistive heating. The temperature is controlled by an Oxford Instruments ITC4, or ‘Intelligent Temperature Controller’. The temperature is monitored by a silicon diode close to the samples. Through the PC, a desired setpoint is supplied to the ITC4, which can then actively control the heater by adjusting the power input to stabilize the temperature. The cooling power may or may not be constant as a function of temperature. To turn on the heater, the ITC4 must be put into LOCAL mode, using the LOC/REM button to switch to LOCAL mode. Then, turn on the heater, by putting it in AUTO mode. Finally, return the ITC4 to REMOTE mode so that it can communicate with the control software. The heater is not always on as a safety measure to avoid burning out the heater by constant application of power at high temperatures.

To control the temperature requires a feedback loop, and the method used for this is commonly referred to as ‘PID feedback’. In PID control, three terms are added to determine how much power should be applied to the heater. This approach is used for many other automation problems, not just temperature control. The P term is the proportional term. This calculates a heater output that is proportional to the difference between the setpoint and the actual temperature (the *error*). A large P value indicates that more heat will be put in. However, as one approaches the setpoint, the heater

output will tend to zero, and it is unlikely that a zero heat output will be able to maintain the desired temperature.

This leads us to the I , or integral, term. The integral constant I is multiplied by the error and added to the previous value of the integral heater output. As one approaches the setpoint, the rate of change goes to zero, leaving a constant heater output to maintain the desired temperature. In many cases, the P and I terms are sufficient to control the temperature. However, the approach can be improved by including a D , or derivative, feedback term, which gives a heater output equal to the rate of temperature change multiplied by D .

The values for P , I and D can vary quite considerably. At present, the PID settings are $P = 9$, $I = 0.3$ and $D = 0$. These values should be appropriate for the broad temperature range, but are unlikely to be optimized for every single temperature. The focus of this experiment is not on investigating PID feedback, but you may find it helpful to discuss this issue with people doing the Copper at Low Temperatures experiment, as this is more crucial to the success of their experiment. Further sources of information about PID control are given in the Recommended Reading.

Suggested investigations

- Investigate the effect of warming and cooling on the observed behaviour.
- Investigate the effect that the speed of temperature change has on the results that you obtain.
- Investigate hysteresis loops at a range of different temperatures.
- Investigate and identify all of the transitions shown by the dysprosium.

Recommended Reading

S. Foner, *Versatile and Sensitive Vibrating-Sample Magnetometer*, Review of Scientific Instruments **30**, 548 (1959).

D. R. Behrendt, S. Legvold and F. H. Spedding, *Magnetic Properties of Dysprosium Single Crystals*, Physical Review **109**, 1544 (1958).

F. Trombe, Comptes Rendus **236**, 591 (1953).

CTC100 User Manual, p.27, *Using PID feedback*, Stanford Research Systems, (2011).

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