Aim: Magnetic characterization of ferromagnetic samples by Vibrating Sample Magnetometry.

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1. Introduction

Vibrating Sample Magnetometry (VSM) is a measurement technique which allows to determine the magnetic moment of a sample with very high precision. The aim of this lab course M106 is to enlarge upon the use of this widespread technique introduced in the lab course B512, where different ferromagnetic samples were characterized concerning magnetic hysteresis and demagnetization. Here, we will gain a deeper understanding of the behavior of magnetic materials and its measurement. In order to lay the foundations, first the measurement principle and the properties of ferromagnetic materials will be summarized (magnetic domains, magnetic hysteresis, demagnetization) and then we will elaborate on the magnetic anisotropy of ferromagnetic materials.

1.1. Measurement principle

Vibrating Sample Magnetometry (VSM) is based on Faraday's law which states that an electromagnetic force is generated in a coil when there is a change in flux through the coil [1]. In the measurement setup, a magnetic sample is moving in the proximity of two pickup coils as indicated in Fig. 1.

![Schematic representation of a VSM](image)

Figure 1: Schematic representation of a VSM. Adapted from [1]

The oscillator provides a sinusoidal signal that is translated by the transducer assembly into a vertical vibration. The sample which is fixed to the sample rod vibrates with a given frequency and amplitude (60 to 80 Hz and 1 mm, respectively). It is centered between the two pole pieces of an electromagnet that generates a magnetic field \( \vec{H}_0 \) of high homogeneity. Field strengths in the range of \( 10^6 \text{ A/m} \) are commonly possible with...
laboratory VSM systems. Stationary pickup coils are mounted on the poles of the electromagnet. Their symmetry center coincides with the magnetic center of the static sample. Hence, the change in magnetic flux originating from the vertical movement of the magnetized sample induces a voltage $U_{\text{ind}}$ in the coils. $H_0$, being constant, has no effect on the voltage but is necessary only for magnetizing the sample. According to Faraday, the voltage in a single winding of the pickup coil can be written as

$$U_{\text{ind}} = -\frac{\partial \Phi}{\partial t}$$

where $\Phi$ is the magnetic flux. For $n_c$ pickup coils with a flat surface $A$ and $n_w$ windings, Eq. 1 gives

$$U_{\text{ind}} = \sum_{n_c} \sum_{n_w} \int_A \frac{\partial B}{\partial t} dA$$

When we bring the sample into the homogeneous field $H_0$ along the x axis, it will be magnetized along the direction of the field, resulting in a magnetic moment $\vec{m}$ of the sample. Then, the sample will be moved periodically relative to the pickup coils. Here, we will consider a sinusoidal movement $\delta(t)$ of the sample perpendicular to the direction of $H_0$ (along the z axis) with amplitude $\hat{z}$ and frequency $\omega$

$$\delta(t) = \frac{dz}{dt} = \hat{z} \cdot \omega \cdot \cos(\omega t)$$

A moving magnetic moment means a change in the magnetic flux density $\vec{B}$. For a point at a distance $\vec{r}$ from the sample, it is

$$\partial \vec{B} = \mu_0 \cdot \delta(t) \cdot \nabla\{H(\vec{r})\}.$$  

With the field strength at distance $\vec{r}$ from the sample being

$$H(\vec{r}) = \frac{1}{4\pi} \cdot \frac{\vec{m}}{r^3} - \frac{3 \cdot (\vec{m} \cdot \hat{r}) \hat{r}}{r^5}$$

and the magnetic moment being oriented along the x axis

$$\vec{m} = \begin{pmatrix} m_0 \\ 0 \\ 0 \end{pmatrix}$$

we get

$$\nabla H(\vec{r}) = -\frac{3 \cdot m}{4\pi r^5} \begin{pmatrix} x \cdot (3 \cdot r^2 - 5 \cdot x^2) \\ y \cdot (r^2 - 5 \cdot x^2) \\ z \cdot (r^2 - 5 \cdot x^2) \end{pmatrix} = -\frac{3 \cdot m}{4\pi r^5} \cdot \vec{G}$$

where $\vec{G}$ contains information concerning the position and orientation of the pickup coils, summarized as the so called geometric factors $g_l$. 

3
Combining (2), (4) and (6) gives the induction voltage

\[
U_{\text{ind}}(t) = -\frac{32}{4\pi} \cdot m \cdot \mu_0 \cdot \cos(\omega t) \cdot \sum n_c \sum n_w \int_A \vec{dA} \cdot \left(\frac{1}{\bar{r}^7}\right) \cdot \vec{G}.
\] (7)

It is obvious that a change in time of magnetic flux originates from the vertical sample movement relative to the coils. The flux change detected by the coils is therefore proportional to a number of quantities: the frequency \(\omega\) of the vibration and its amplitude \(\bar{z}\), the magnetic moment of the sample \(\vec{m}\) and the distance to the pickup coils \(y_0\). Furthermore, \(U_{\text{ind}}\) can be increased by increasing the number of windings \(n_w\) and the number of pickup coils \(n_c\) used in the setup. Also, the geometry of the pickup coils influences the induction. [2]

The measurement setup is sensitive even to very low magnetic moments. Today’s vibrating sample magnetometers are able to detect magnetic moments of down to the \(\mu\)emu range, which corresponds to approx. \(10^{-9}\) g of iron [3].
2. Basic

2.1 Ferromagnetic materials

2.1.1 Magnetic domains

Magnetic materials are distinguished by their behavior in external magnetic fields. For ferromagnets, the $M(H)$ dependence is characterized by domain wall movement and magnetization rotation.

According to the theory of Weiss a non-saturated ferromagnet contains a number of small regions called domains, in which the local magnetization is homogeneous and reaches the saturation value. The direction of magnetization in different domains is not necessarily parallel. Two domains are separated by domain walls which are regions of approx. 100 nm where the magnetization rotates continuously (Fig. 2). They are classified by the angle of rotation ($90^\circ$ or $180^\circ$) and the mode of rotation (Bloch or Neel, see detail Fig. 2).

![Figure 2: Magnetic domain pattern of a Fe whisker, detail: Bloch and Néel wall.](Adapted from [4], [5])

When applying an external magnetic field, domains with magnetization parallel to the field will grow at the cost of domains with energetically more unfavorable magnetization alignment. As a consequence domain walls move through the sample and the overall magnetization increases in field direction. In magnetically soft materials, domain walls are broad and the movement of the walls requires small fields only whereas larger fields are necessary in magnetically hard materials.

A second mechanism that increases the magnetization is magnetization rotation within the domains. Due to magnetic anisotropies, certain directions are easier to magnetize than others. The spontaneous magnetization lies preferably along one of these easy directions. If the applied external field is not parallel to such an easy direction, the
magnetization rotates and aligns along the magnetic flux lines.
2.1.2 Hysteresis

With a VSM, the magnetic moment of a sample as a function of the external magnetic field strength can be measured. Fig. 3 shows (schematically) a typical result of such a measurement. The demagnetized sample ($\vec{M} = \vec{0}$) is firstly magnetized to saturation in an applied field (virgin curve, see Fig. 3a)).

![Image of hysteresis diagram]

Figure 3: a) Virgin curve, b) Hysteresis loop of a ferromagnetic material. [6]

Indicated are the two commonly used parameters initial permeability $\mu_i$ and maximum permeability $\mu_{max}$, defined in terms of a ratio of magnetic flux to field strength $\frac{B}{H}$. Permeability is not the slope of the virgin curve itself, but the slope of a line between the origin and a particular point on the curve (e.g. the upper inflection point before saturation for $\mu_{max}$).

Increasing and decreasing the external field from the demagnetized state to positive saturation to negative saturation and back gives a ferromagnetic hysteresis loop, which is shown in Fig. 3b). Here, the predominant, underlying mechanisms for each region are indicated. The region of highest permeability is governed by domain wall motion whereas magnetization rotation occurs rather at higher magnetic fields. When the applied field is reduced to zero the remanent magnetization $M_r$ remains. A magnetic field of opposite direction called coercivity field $H_c$ is needed to macroscopically demagnetize the sample.

Thus, the hysteresis loop contains important information about the magnetic properties of the sample. Characteristic quantities include the saturation magnetization $M_s$, the remanent magnetization $M_r$, the coercivity field $H_c$, permeability $\mu$ and the dissipated energy that is necessary to magnetize the sample in the opposite direction. The dissipated energy can be calculated from the area that is included by the hysteresis loop.
2.2 Demagnetization

In an external magnetic field, the presence of a magnetic material alters the magnetic induction $\vec{B}$ (Fig. 4a). If the sample has a finite length, magnetic poles are generated near the ends of the sample. These poles give rise to a magnetic field that opposes the external field and is therefore called demagnetizing field (Fig. 4b). The strength of the demagnetizing field depends on the magnetization $\vec{M}$ and the geometry of the sample. The field inside the magnetic material is now different from the external field and requires correction. The inner field can be calculated by

$$\vec{H}_{in} = \vec{H}_{ext} - N_d \vec{M}$$ (7)

where $\vec{M}$ is the magnetization and $N_d$ the demagnetizing factor which is only determined by the sample shape. For thin films, spheres and ellipsoids it is possible to calculate $N_d$, whereas for other shapes one needs to approximate.

Figure 4: a) Distortion of homogenous magnetic field by magnetic sample (bar magnet); b) Demagnetization of a bar magnet. [8]
2.3 Magnetic Anisotropy

If the magnetic properties of a sample are dependent on directions, it is magnetically anisotropic. Magnetic anisotropy arises from dipole-dipole interactions and spin-orbit coupling. Macroscopic shape anisotropy has its origin in long range dipole interactions arising from free poles at surfaces. It can be represented in terms of the stray fields created external to the sample. In thin films stray field energy is minimized if the magnetization is in-plane and maximized if the magnetization is out-of-plane.

Another form of magnetic anisotropy is the magnetocrystalline anisotropy. When investigating single crystalline Fe, Ni, or Co samples, one notices that the magnetization curves along certain crystallographic directions differ from each other. In iron, the magnetization curve along the ⟨100⟩ direction is steeper and reaches saturation at lower external fields than along ⟨110⟩ or ⟨111⟩ and is therefore the easy axis of magnetization (see Fig. 5). In case of nickel, ⟨111⟩ is the easy axis and ⟨100⟩ the hard axis of magnetization. In absence of an external field, the spontaneous magnetization lies preferably along one of the easy axes. The origin of the magnetocrystalline anisotropy lies both in the coupling of the spin part of the magnetic moment to the electronic orbital shape and orientation (spin-orbit coupling) and the chemical bonding of the orbitals on a given atom with their local environment [8].

![Magnetization curves along the crystallographic axes of body centered cubic iron; inset: Crystal structure of bcc iron, indicated are easy, medium, and hard axis of magnetization. [8]](image)

In polycrystalline materials, the contribution of magnetocrystalline anisotropy depends on whether there is a preferred grain orientation (texture) or if the grain orientation is randomly distributed such that the local anisotropies average out to zero across the sample volume.
Additionally to the sources mentioned above, **stress induced magnetic anisotropy** may figure into the anisotropic magnetization behavior. The magnetization direction and mechanical strain in a material are closely connected due to spin-orbit coupling, resulting in a contribution to the magnetic anisotropy energy. This can be observed as a change of a sample’s dimensions when it is exposed to a magnetic field, which is described by the magnetically induced or anisotropic strain \( \lambda = \frac{\Delta l}{l} \) (in analogy to the mechanical strain \( \epsilon \)). The process is depicted schematically in Fig. 6, showing the rotation of magnetic dipoles of a row of atoms with the externally applied field and the resulting strain as the atoms’ nuclei are forced apart.

\[
\vec{\lambda} = 0 \\
\vec{\lambda} \uparrow
\]

\( \lambda \) is either positive or negative or changes its sign with increasing field strength, depending on the material and, for single crystals (Fig. 7a)), on the crystallographic direction or, for polycrystalline materials (Fig 7b)), on the texture. Materials are usually described by their saturation magnetostriction \( \lambda(M = M_s) = \lambda_s = \frac{l_s - l_0}{l_0} \). Here, \( l_s \) is the sample length in the saturated state and \( l_0 \) is the length of the demagnetized sample. For positive magnetostriction (\( \lambda_s > 0 \)), the application of a magnetic field leads to an expansion (tensile stress, \( \sigma > 0 \)) along the field direction, while negative magnetostriction (\( \lambda_s < 0 \)) results in a contraction (compressive stress, \( \sigma < 0 \)) of the sample.

\[
\begin{align*}
\text{a) Ni single crystal} \\
\text{b) polycrystalline Fe, Co and Ni}
\end{align*}
\]

Inversely, if a magnetic sample is exposed to mechanical stress, the domain structure is influenced and thus, the preferred direction of magnetization (easy axis) is changed.
This relation between mechanical strain and the low field magnetic sample properties is called Villari effect or magnetomechanical effect and can be observed by comparing the magnetic hysteresis with and without applied stress. As a sample with $\lambda_s > 0$ exposed to a magnetic field will experience tensile stress along the field direction, the same sample will, when exposed to tensile stress, be easier to magnetize in this direction (Fig. 8a). Accordingly, the opposite is true for materials with $\lambda_s < 0$, application of tensile stress will decrease the permeability, while compressive stress will increase it (Fig. 8b). This can be summarized by distinguishing between the cases of $\lambda_s \sigma > 0$ and $\lambda_s \sigma < 0$, as depicted in fig. 8c).

Figure 8: Magnetization behavior of a) a positive magnetostriction material (poly-Ni$_{68}$Fe$_{32}$), of b) a negative magnetostriction material (poly-Ni) under tensile and compressive stress. c) Case differentiation $\lambda_s \sigma$ for an isotropic material. [8,9]

Magnetostriction can also be observed in nanocrystalline and amorph ferromagnetic materials. In these cases, the magnetic anisotropy can be induced by a temperature treatment in a magnetic field.
2.4 Units

Table B-1. Conversion from CGS to SI Units

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Symbol</th>
<th>Gaussian &amp; CGS emu(^a)</th>
<th>Conversion Factor, (C)(^b)</th>
<th>SI &amp; Rationalized mks(^c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetic flux density, Magnetic induction</td>
<td>B</td>
<td>gauss (G)(^d)</td>
<td>(10^{-4})</td>
<td>tesla (T), Wb/m(^2)</td>
</tr>
<tr>
<td>Magnetic Flux</td>
<td>(\phi)</td>
<td>maxwell (Mx), G·cm(^2)</td>
<td>(10^{-8})</td>
<td>weber (Wb), volt second (V·s)</td>
</tr>
<tr>
<td>Magnetic field strength, magnetizing force</td>
<td>U, F</td>
<td>gilbert (Gb)</td>
<td>(10^4/4\pi)</td>
<td>ampere (A)</td>
</tr>
<tr>
<td>Magnetic field strength, magnetizing force</td>
<td>H</td>
<td>oersted (Oe), G·cm(^2)</td>
<td>(10^3/4\pi)</td>
<td>A/m(^1)</td>
</tr>
<tr>
<td>(Volume) magnetization (^\dagger)</td>
<td>M</td>
<td>emu/cm(^3)</td>
<td>(10^{9})</td>
<td>A/m(^1)</td>
</tr>
<tr>
<td>(Volume) magnetization (^\dagger)</td>
<td>G</td>
<td>emu/cm(^3)</td>
<td>(10^{9/4\pi})</td>
<td>A/m(^1)</td>
</tr>
<tr>
<td>Magnetic polarization, intensity of magnetization</td>
<td>J, I</td>
<td>emu/cm(^3)</td>
<td>(4\pi \times 10^{-4})</td>
<td>T, Wb/m(^2)</td>
</tr>
<tr>
<td>(Mass) magnetization</td>
<td>(\sigma, M)</td>
<td>emu/g</td>
<td>(4\pi \times 10^{-7})</td>
<td>A·m(^2)/kg, Wb·m/kg</td>
</tr>
<tr>
<td>Magnetic moment</td>
<td>(m)</td>
<td>emu, erg/G</td>
<td>(10^{-3})</td>
<td>A·m(^2), joule per tesla (J/T)</td>
</tr>
<tr>
<td>Magnetic dipole moment</td>
<td>(j)</td>
<td>emu, erg/G</td>
<td>(4\pi \times 10^{-10})</td>
<td>Wb·m(^1)</td>
</tr>
<tr>
<td>(Volume) susceptibility</td>
<td>(\chi, \kappa)</td>
<td>dimensionless emu/cm(^3)</td>
<td>((4\pi)^2 \times 10^{-7})</td>
<td>Henry per meter (H/m), Wb/(A·m)</td>
</tr>
<tr>
<td>(Mass) susceptibility</td>
<td>(\chi_p, \kappa_p)</td>
<td>cm(^3)/g, emu/g</td>
<td>(4\pi \times 10^{-10})</td>
<td>m(^3)/kg, H·m(^2)/kg</td>
</tr>
<tr>
<td>(Molar) susceptibility</td>
<td>(\chi_{mol}, \kappa_{mol})</td>
<td>cm(^3)/mol, emu/mol</td>
<td>(4\pi \times 10^{-13})</td>
<td>m(^3)/mol, H·m(^2)/mol</td>
</tr>
<tr>
<td>Permeability</td>
<td>(\mu)</td>
<td>dimensionless</td>
<td>(4\pi \times 10^{-7})</td>
<td>H/m, Wb/(A·m)</td>
</tr>
<tr>
<td>Relative permeability(^l)</td>
<td>(\mu_r)</td>
<td>not defined</td>
<td>-</td>
<td>dimensionless</td>
</tr>
<tr>
<td>(Volume) energy density, energy product(^k)</td>
<td>(W)</td>
<td>erg/cm(^3)</td>
<td>(10^{-1})</td>
<td>J/m(^2)</td>
</tr>
<tr>
<td>Demagnetization factor</td>
<td>(D, N)</td>
<td>dimensionless</td>
<td>(1/4\pi)</td>
<td>dimensionless</td>
</tr>
</tbody>
</table>

NOTES:

a. Gaussian units and cgs emu are the same for magnetic properties. The defining relation is \(B = H + 4\pi M\).

b. Multiply a number in Gaussian units by \(C\) to convert it to SI (e.g. \(1 \mbox{ G} = 10^{4} \mbox{ T} = 4 \pi H\)).

c. SI (Système International d’Unités) has been adopted by the National Bureau of Standards. Where two conversion factors are given, the upper one is recognized under, or consistent with, SI and is based on the definition \(B = \mu_0 H + M\), where to \(\mu_0 = 4 \pi \times 10^{-7}\) H/m. The lower one is not recognized under SI and is based on the definition \(B = \mu_0 H + J\), where the symbol \(J\) is often used in place of \(J\).

d. \(1\) gauss = \(10^{5}\) gamma (\(\gamma\)).

e. Both oersted and gauss are expressed as \(cm^{-1} \cdot g^{-1} \cdot s^{-1}\) in terms of base units.

f. A/m was often expressed as "ampere-turn per meter" when used for magnetic field strength.

g. Magnetic moment per unit volume.

h. The designation "emu" is not a unit.

i. Recognized under SI, even though based on the definition \(B = \mu_0 H + J\). See footnote c.

j. \(\mu = \mu_0 \mu_r = 1 + \chi\), all in SI. \(\mu_r\) is equal to Gaussian \(\mu\).

k. \(H = H + \mu_0 M\cdot H\) have SI units \(J/m^2\), \(M\cdot H\) and \(B = H/4\pi\) have Gaussian units erg/cm\(^3\).

3. Experiment

3.1 Equipment

Components of the LakeShore VSM 7400 system are shown in Fig. 6: the electromagnet (a) with the pole pieces on which the pickup coils are mounted (b) and the vibrating/rotating head with positioning screws to center the vibrating rod (c). The VSM system also comprises a bipolar DC power supply (± 50V, ± 100A), a cooling system for both the electromagnet and the power supply, the VSM controller and a PC with LakeShore “IDEAS VSM” software to control the components and to collect the measured data.

Figure 6: LakeShore VSM 7400 system: a) overview of the setup, b) pick up coils in between pole pieces and c) VSM head with integrated transducer assembly.
3.2 Procedure

Before using the VSM, you must carry out a series of configuration steps.

- Insert the Ni standard into the VSM. The standard is ball-shaped, therefore magnetically isotropic, and has a magnetic moment of 6.92 emu at 5000 Oe.

- Find the exact position of the standard in respect to the center of the pickup coils. The vibrating rod can be adjusted by three screws on top of the VSM for \( x \), \( y \) and \( z \) direction. The pickup coils are connected in a way that, the sample being in the center of the coils, there will be a signal minimum along \( x \)-, a maximum along \( y \)- and a maximum again along \( z \)-direction.

- Run *Calibrations → Moment gain* to calibrate the instrument, i.e. to convert the measured voltage signal into the correct value of the magnetic moment.

After calibration of the VSM, the following measurements aim to address two topics. The first part covers basic magnetic characterization and the information that can be deduced from magnetization curves. The second part cope with demagnetization.

1. **Magnetocrystaline anisotropy energy:**
   Fix the Fe single crystal to the sample holder. Set \( H_0 \) to 3500 G and record the magnetic moment of the crystal during a 360° rotation of the sample. Find the angles corresponding to the different crystallographic / magnetic axes and record the magnetization curves of the easy axis and the hard axis.

2. **Stress induced magnetic anisotropy**
   Mount a sheet sample clamped in a sample holder provided by the supervisor into the VSM. Record the magnetization curves of the sample with and without applied stress along and perpendicular to the stress direction.

Determine the volume of each sample that you have measured.
4. Analysis

LakeShore software saves all data as ASCII text files (.txt). Copy these files to a USB flash drive of yours. The file format is as follows (in case of a magnetization curve):

[Header lines: time stamp etc.]

Field(G)           Moment(emu)
1234.56           7.89012

etc.

5. Report

Please answer to the following assignments:

1. Create a diagram showing the magnetic moment of the Fe single crystal in dependence of the rotation angle and indicate the angles which correspond to crystallographic directions. Create a diagram showing the magnetization curves for both the easy and the hard axis and calculate the magnetic anisotropy energy from the magnetization values.

2. Compare the magnetization curves of the sheet sample with and without applied stress for both measured directions. Please report on the observed effect and draw conclusions about the materials magnetomechanical properties based on these observations (qualitatively).

3. Give 3 common measurement errors of the VSM measurement principle and explain how they affect the measured magnetization curve.
6. References


