

Magnetometry by means of Hall micro-probes in the Quantum Design PPMS*

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Abstract

This application note will describe how to perform Hall micro-probe magnetometry experiments with the Quantum Design Physical Properties Measurement System (PPMS). This technique allows detection of magnetic signals from very tiny crystals, typically 10 – 100 μm in size, and extends the temperature range for magnetization measurements down to $\sim 0.3\text{K}$ when Hall probes are used in combination with the Model P825 Helium-3 insert. This application note covers general issues about Hall magnetometry, as well as detailed instructions on how to employ the Hall probes with different inserts for the PPMS. Examples of possible measurements will also be described.

*This study was performed by Dr. Andrea Candini and Prof. Marco Affronte at the S3 National Center on Nanostructures and Biosystems on surface of INFM-CNR and Università degli Studi di Modena e Reggio Emilia, Via Campi 213/A, 41100 Modena, Italy and it was partially supported by the European Network of Excellence MAGMANet contract N.515767.

I. GENERAL

A. Principle of Operations

Discovered by Edwin H. Hall in 1879, the Hall effect is the appearance of a transverse voltage (known as the Hall voltage V_H) when a charge current is placed in a perpendicular magnetic field. This effect is depicted in Figure 1.

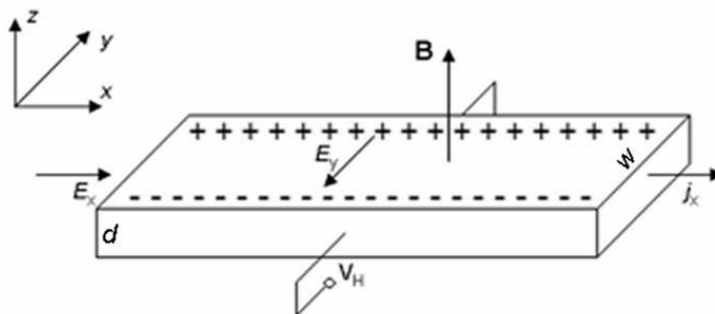


FIG. 1: Schematic view of Hall's experiment

It turns out that

$$V_H = \frac{R_H I B_z}{d} \quad (1)$$

where I is the driving current, B_z the magnetic field component along the z axis and d is the film thickness. In the free charge -Drude- model the Hall coefficient R_H (material dependent) can be simply expressed as:

$$R_H = \frac{1}{ne} \quad (2)$$

n being the charge carrier density and e the electron charge (equal to 1.6×10^{-19} C). The Hall voltage V_H is thus proportional to the magnetic field and to the current and it is inversely proportional to the carrier density n of the conductor.

When the density of charge carriers n and the geometrical factors are known, a Hall probe can be used to measure the magnetic field. If a magnetic sample is placed near the Hall sensor, as shown in Figure 2, then the Hall voltage will be proportional to the sum of the magnetic **flux** originated by external magnetic field \mathbf{B} and by the *stray field* coming from the sample that enters in the *active area*. Since the stray field is related to the magnetization of the sample, the device works as a magnetometer, based on the Hall effect.

Interested readers can find an extended literature on Hall probe magnetometry and its possible applications in References [1–10]. In addition, References [11–14] give further details on how to relate the Hall signals with the magnetic field that enters in the active area.

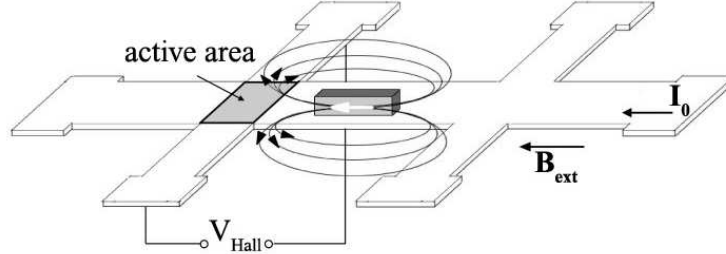


FIG. 2: Principle of Hall probe magnetometry. A magnetic sample is placed close to the active area of the Hall sensor and magnetized by an external field. The output signal will be proportional to the stray field generated by the sample’s magnetization that enters in the probe’s sensitive region.

B. Setup Description

1. Devices

Various types of Hall sensors are currently available in the market [15]. Even if in principle any material will show the Hall effect, depending on the specific measurement, some materials show better characteristics than others. For instance, when working at low temperatures, GaAs/AlGaAs heterostructures, with a two-dimensional electron gas (2DEG) below the surface, are usually considered as one of the best choices. This is because of the special combination of small density n and high mobility μ of the charge carriers that results in a high signal to noise ratio. On the other hand, for room temperature applications, InSb is the material generally employed, since it has the highest mobility among the semiconductors in this temperature range.

Another important issue is the geometrical size of the active area of the probe, since it is directly involved in the coupling between the sensor and the field coming from the sample. Hence, this will roughly determine the size of the measurable sample. When working with magnetic samples with sizes down to the micrometer scale, Hall devices with active areas in

the range $10 \times 10 \mu\text{m}^2$ to $100 \times 100 \mu\text{m}^2$ are usually the preferable choice since they offer the best sensitivity.

2. Sample Mounting

- Glue the semiconductor chip containing the Hall sensors on the Resistance sample holder, possibly with silver paint to assure good thermal contact.
- Connect the electrical pads of the probe with those of the sample holder, following the scheme depicted in Figure 3, i.e. following the labels printed on the holder and remembering that the current and voltage leads must be perpendicular.
- Place the magnetic specimen over the probe surface with cryogenic grease (Apiezon N). In order to avoid damages of the sensor, a soft tip can be used to pick up the sample and position it. This must be placed nearby to the sensor active area, to optimize the flux coupling. The final result should look like the example shown in Figure 4.

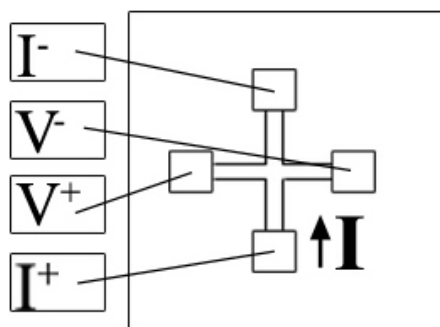


FIG. 3: Wiring the Hall probe

C. Performing Measurements

With the Hall probe wired as described in the previous paragraph, measurements can be simply performed with the dc-Resistivity option P-400 [18]. The system will measure the -Hall- resistance given by

$$Res = \frac{V_{\text{Hall}}}{I} = \frac{R_{\text{H}}}{d} B \quad (3)$$

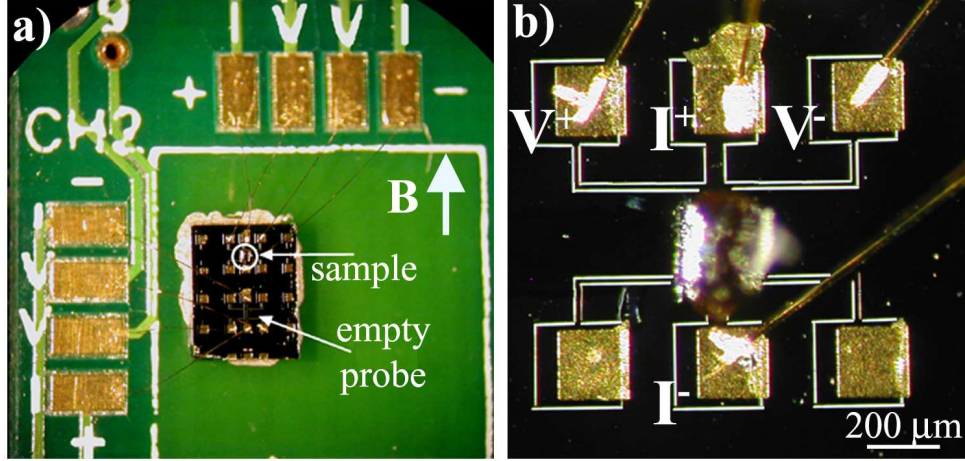


FIG. 4: Typical sample mounting on a Hall probe with a magnetic crystal on it. a): View of the sample holder (sample board for the horizontal rotator - part number 3084-371). Probes are wired following the scheme depicted in Figure 3. b): zoom of the sample region. One edge of the crystal is aligned with the active area of the probe. The probe is prepared for measurements in the parallel geometry (see Section II B). Contours of the Hall devices have been highlighted for clarity.

where R_H/d is the Hall coefficient of the probe, while B is the sum of the external field and the stray field due to the sample's magnetization.

In principle, exciting the probe with high values of current I will improve the measurements sensitivity. However, above certain material dependent limit high currents lead to heating effects that degrade the probe's performance. For low-temperature AlGaAs probes (with lateral size of the order of $\approx 10 \mu\text{m}$), optimal values for the excitation currents are usually found within $1 - 10 \mu\text{A}$. It is preferable to use the probes in the constant current mode, to avoid non-linearity effects during the measurements: through the **Bridge Setup** command in the Sequence Commands set the voltage and power limits at the maximum for each channel, and specify only the current value; select AC-driving mode and Standard-calibration mode. Under these conditions, at low temperature the sensitivity of the resistance bridge is usually $\approx 10^{-2} \Omega$.

In order to separate the sample's signal from the background due to the external field, the user may exploit several procedures:

i) align the plane of the sensor with the external field: this will strongly reduce the

background contribution since this is proportional to component of the external field *perpendicular* to the Hall probe plane; moreover this essentially allows to avoid Quantum Hall plateaus at low temperatures;

ii) wire and measure also an empty probe, that will work as a reference (the PPMS User Bridge board allows to measure up to three channels separately): best results will be obtained if all the probes are coming from the same substrate and consequently have very similar features. This method can actually be performed following two different routes: one is to employ the resistivity user bridge and make the subtraction between the signals *after* the measurements (any data analysis softwares can be used); the other is to make use of external instrumentation, as exemplified in Figure 5. A common configuration is to employ pre-amplifiers to measure the Hall signal of two active areas of the same device and a locked-phase instrument triggered with the current source to perform the final subtraction. In this case, the subtraction is performed analogically *during* data acquisition. This configuration also enhances the magnetic signal from the sample up to two times, when the sample fits exactly the distance between the two crosses;

iii) in case of magnetization measurements as a function of the external field, the background is a straight line ($V_H \approx R_H B_{\text{ext}}$) that can be easily evaluated and then subtracted.

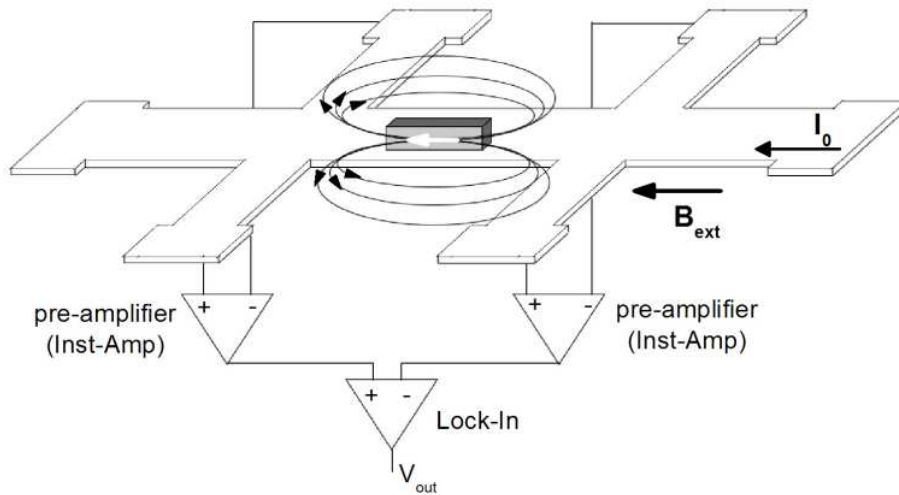


FIG. 5: Scheme of the experimental configuration when using external instrumentation. The Lock-In is triggered with the current source. Electrical noise is usually below $1 \mu\text{V}$.

It is generally accepted that, working with micrometric crystals, the absolute value of the magnetization can be hardly quantified: this is due to the difficulty in estimating the actual size and shape of the micro-crystal and - true for any magnetic flux detection technique- in the estimation of the geometrical factors that relate the stray field with the actual flux entering in the sensor. When possible, data taken with the Hall probe can be scaled by the user with the absolute values obtained using dc magnetometry such as the PPMS VSM option on bulk sample of the same material.

CAUTION: the user should be careful in manipulating the Hall probes, since they are very easy to damage, especially the 2DEG devices. Any type of shock, including thermal and electrical, must be avoided. Hence, the Hall probes should be cooled and warmed quite slowly (few Kelvin/min) and a wait time should be inserted at each new temperature before starting the measurements to allow the Hall probe to get in thermal and electrical equilibrium. To reduce the risk of electrical shock to the Hall sensors, have the Resistivity cable plugged into the PPMS and the bridge channels all turned OFF when inserting the probe into the PPMS sample chamber.

1. Examples of Measurements

Since by measuring the resistance of the Hall probe it is quite straightforward to obtain the magnetization of the sample (even if in arbitrary units), many type of measurements can be performed. The most common will be reviewed here below.

Magnetization Measurements:

- fix the temperature and wait for the probe to stabilize (usual time ranges from 10 to 60 minutes, depending on the temperature and the cooling process).
- Select **Scan Field** from the Sequence Commands menu and measure the Resistivity of the channels wired to the Hall probe. An example is given in Figure 6.

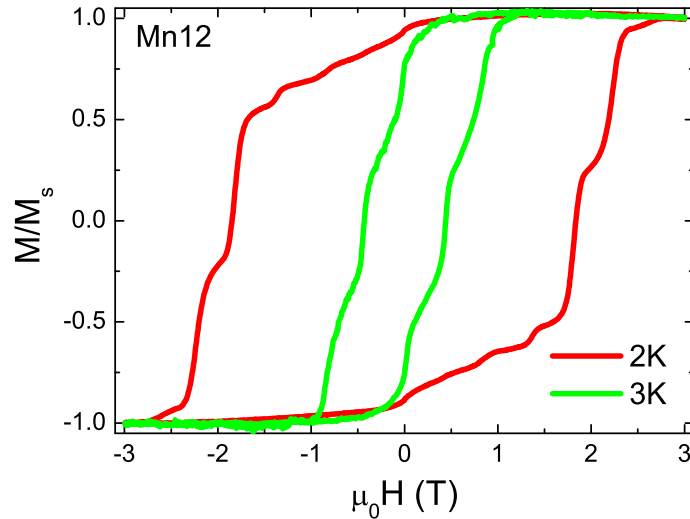


FIG. 6: Hall probe magnetometry on a Mn₁₂-ac [16] micro-crystal. The long side of the sample ($\approx 100 \mu\text{m}$ long), corresponding to the easy axis of magnetization of the molecule, was oriented parallel to the external field. Evidently, the signal is clean and the steps due to quantum tunnelling of magnetization are observable. Data have been acquired with an home-built pre-amplifier and an external Lock-In, using the experimental scheme depicted in Figure 5.

dc-Susceptibility Measurements:

- fix the external field and wait for the probe to stabilize (a few minutes are usually sufficient).
- Select **Scan Temperature** from the Sequence Commands menu and measure the Resistivity of the channels wired to the Hall probe. The user should let the system cool very smoothly to allow the Hall probe to be always at equilibrium. However, this type of measurement usually requires samples with strong magnetic signals, since the Hall coefficient of a probe may slightly vary with the temperature. Figure 7 shows an example of a temperature dependent dc-susceptibility taken with a Hall probe magnetometer.

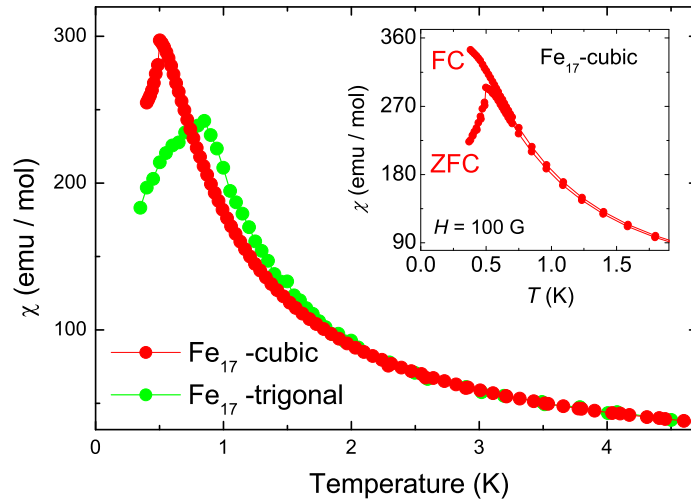


FIG. 7: Magnetic susceptibility of two derivatives of the Fe_{17} -magnetic molecule with the external field fixed at $H = 0.01$ T, taken with the Hall probe magnetometer. Inset: Zero-field cooled (ZFC) and field cooled (FC) magnetization data for Fe_{17} -cubic, elucidating the superparamagnetic blocking nature of the peak at $T_B \simeq 0.5$ K. To present data in absolute units, they have been properly scaled with those measured with the standard ACMS option on powder material down to 2 K. Data acquisition was made with the internal Resistance user-bridge. This Figure is taken from Reference [17].

Relaxation Measurements:

- Select **Scan Time** from the Sequence Commands menu and measure the Resistivity of the channel wired to the Hall probe.
- Temperature and field must remain fixed during the measurements. Due to relaxation of pinned magnetic flux in the superconducting magnet, the field at the sample location can drift significantly in persistent mode. Thus, keeping the magnet in driven mode is suggested when making sample relaxation measurements as this effectively eliminates any such field relaxation effects seen at the sample. (CAUTION: make sure to set the magnet persistent at the end of the experiment for magnet protection and helium conservation). For more information on such magnetic field artifacts, please refer to the Quantum Design application note titled “Magnetic Field Management Using Superconducting Solenoid Field Sources”.

Also the Hall probes have their own intrinsic relaxation time (usually in the micro-seconds range); however, Hall probe relaxation effects can be eliminated if an empty probe is measured as a reference. An example of this type of measurements is given in Figure 8.

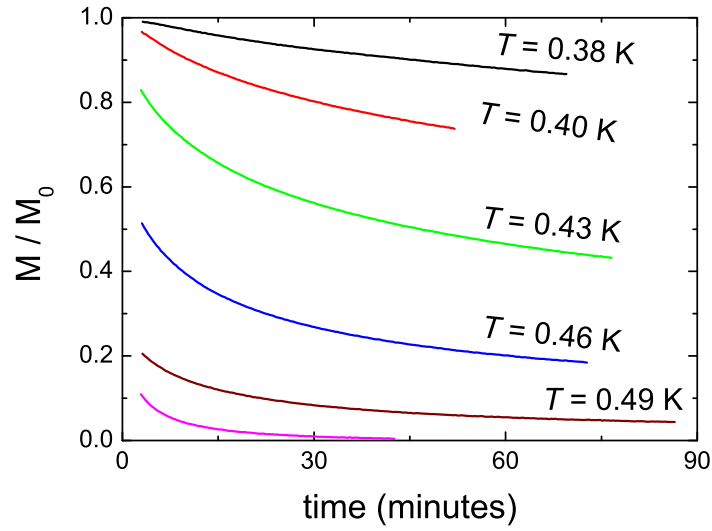


FIG. 8: Time decay of M for Fe_{17} -cubic, measured at zero-applied-field after saturation for the indicated temperatures. Fitting the decay with an exponential law, τ can be calculated for every temperature. This is found to follow an Arrhenius behavior from which the activation energy of the system can be calculated. Data acquisition was made with the internal Resistance user-bridge (subtraction of the empty reference probe has been performed in post-processing data analysis). This Figure is taken from Reference [17].

II. SHORT NOTES ON THE USE WITH DIFFERENT INSERTS

While all the procedures described in the previous sections are generally valid for all the inserts available for the PPMS, there are some precautions that will be addressed specifically.

A. Standard Resistivity Puck

Using this puck (part number 4084-109) the probe will be perpendicular to the external field and the background signal from the magnet field will be very large. When working at low temperature and in fields higher than ≈ 1 T, quantum Hall features may also affect the measurements.

B. Horizontal Rotator

This insert allows the user to make measurements as a function of the sample's orientation with respect to the external field. However, since also the background signal is changing, it may be difficult to compare measurements taken at different angles.

The user should employ the Resistivity Sample Board (part number 3084-371) as a sample holder. When the probe is aligned with the external field (position at 90°), the coupling between the magnetic flux and the sensor is maximized if the current channel is aligned with the field, and the magnetic specimen is placed as close as possible to the probe's active area, as shown in Figure 4.

C. He3 Insert

For the He3-insert the Resistivity Sample Mount is the part number 4092-610, that will fix the probe in the parallel geometry, as described in the previous paragraph. Usually the Hall sensors do not show any problem when cooled at very low temperature and current as high as $10 \mu\text{A}$ can be used. Actually, due to the low dissipation on the Hall probes ($< 0.1 \mu\text{W}$), this technique can be used also with Model P850 Dilution Refrigerator (min. temperature ~ 50 mK).

Note: since newer He3 insert have the horizontal sample mount, it is recommended to buy the vertical sample mount geometry from the Quantum Design (part number 4092-301). To

exchange these parts on the He3 probe, please contact your local Quantum Design service representative.

III. CONCLUSIONS

This application note is intended to provide to the PPMS user an introductory guide on how to perform Hall probe magnetometry, employing the internal Resistivity bridge. This technique is sensitive, inexpensive, flexible and reliable and it expands the dc magnetometry facilities of the PPMS to a wider range of temperatures and applications.

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