

## Design of a multifunctional sample probe for transport measurements

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**Abstract:** Herein, we describe a multifunctional sample probe equipped with a two-axis goniometer for transport measurements. The two-axis goniometer enables both computer-controlled 360° out-of-plane and manual 360° in-plane rotations. The multifunctional sample probe was successfully implemented to a host dewar and an electromagnet system. The developed probe is capable of performing transport measurements while fully controlling the parameters such as the magnetic and electrical fields, the angle, the temperature, and the current/voltage parameters in a wide range. The design of the multifunctional sample probe allows easy installation into different dewar and magnet systems and connection of a wide range of external devices. A software interface based on NI Labview visual programming language was developed to control the system. The setup was tested in the hysteresis loop measurements of perpendicularly magnetized Co/Pt/CoO ultrathin films with the anomalous Hall effect (AHE) method. We also performed temperature-dependent exchange bias measurements on the same ultrathin films where the developed probe revealed much better resolution compared to a standard vibrating sample magnetometry system.

**Key words:** Multifunctional sample probe, magneto-transport, anomalous Hall effect, I-V characterization, perpendicular exchange bias

### 1. Introduction

Magneto-transport experiments are based on the measurements of resistance, current or voltage characteristics of materials under the effect of a magnetic field [1–6]. Recent developments in data processing and storage technologies involve further progress in magneto-transport methods. Furthermore, many trend topics like skyrmions [7,8], magneto-caloric effect [9–12], spin Hall effect [10,13,14], and planar Hall effect for biosensor applications [15] are also studied by transport measurements. More energy efficient, much faster, smaller, and nonvolatile devices are strongly desirable for many technological applications. On the other hand, in order to develop these new generation devices, practical, fast, and flexible experimental instrumentation is required.

Furthermore, the type of the material under investigation and the measurement conditions are important factors for planning a transport experiment. In many cases, it is required to rotate the sample at a fixed temperature without taking the sample out and stopping the experiment. Besides the magnetic field, electrical field is also widely used to characterize technological devices and to manipulate the magnetization of ultrathin films and bulk materials [16,17]. For this reason, applying an electrical field is a desirable option for a transport setup. Depending on the material type and other needs, it is highly practical to have a wide range of controllable temperature, magnetic field, electrical field, angle, current, and voltage parameters in a single setup.

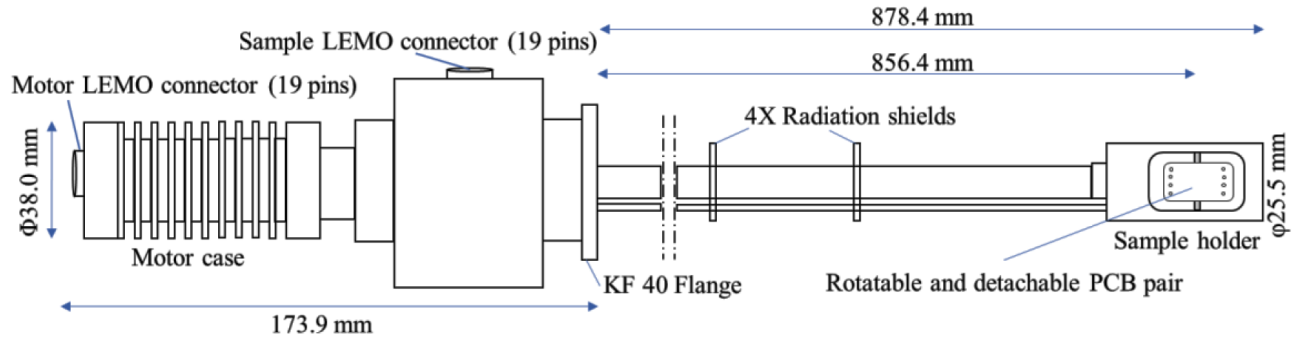
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Herein, we report the design and construction of a multifunctional sample probe with a rotatable sample holder. We successfully tested this probe in combination with a host dewar and a magnet system. The developed setup allows the manipulation of electrical and magnetic fields, sample angle, temperature, and current/voltage values in a variety of transport measurements using a single system. In order to test our designed system, anomalous Hall effect (AHE) measurements for perpendicularly magnetized ultrathin films were carried out and hysteresis loops for perpendicularly magnetized Co (4 Å)/Pt/CoO and Co (5 Å)/Pt/CoO test samples were obtained. The temperature dependence of exchange bias effect in the samples was also investigated by the AHE measurements at different temperatures.

## 2. Instrument

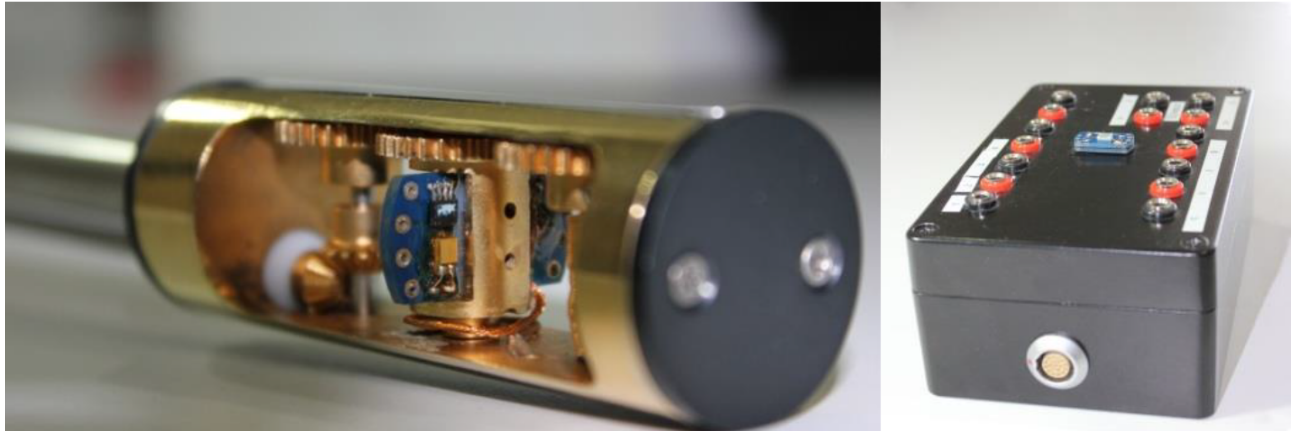
In order to carry out various transport measurements at different geometries in a single setup, we designed a rotatable sample holder that enabled computer-controlled  $360^\circ$  out-of-plane and manual  $360^\circ$  in-plane sample rotations in vacuum conditions for a wide temperature range.

Figure 1 shows the schematic drawing of the multifunctional sample probe. The total length of the sample probe and the diameter of the sample holder are 878.4 mm and 25.5 mm, respectively. The vertical and horizontal position of the sample is set to correspond to the center of the superconducting magnet within the host dewar. The computer controlled out-of-plane rotation of the sample holder is provided by a stepper motor located on top end of the sample probe, with a precision of  $0.125^\circ$ . Figure 2 (left) shows a picture of the sample holder attached to the bottom end of the probe. The sample holder includes a printed circuit board (PCB) located on the axis of a gear wheel. Samples with sizes up to  $12\text{ mm} \times 12\text{ mm}$  are mountable on a pluggable male PCB. The sample probe and the sample holder are made of nonmagnetic stainless steel and brass, respectively.

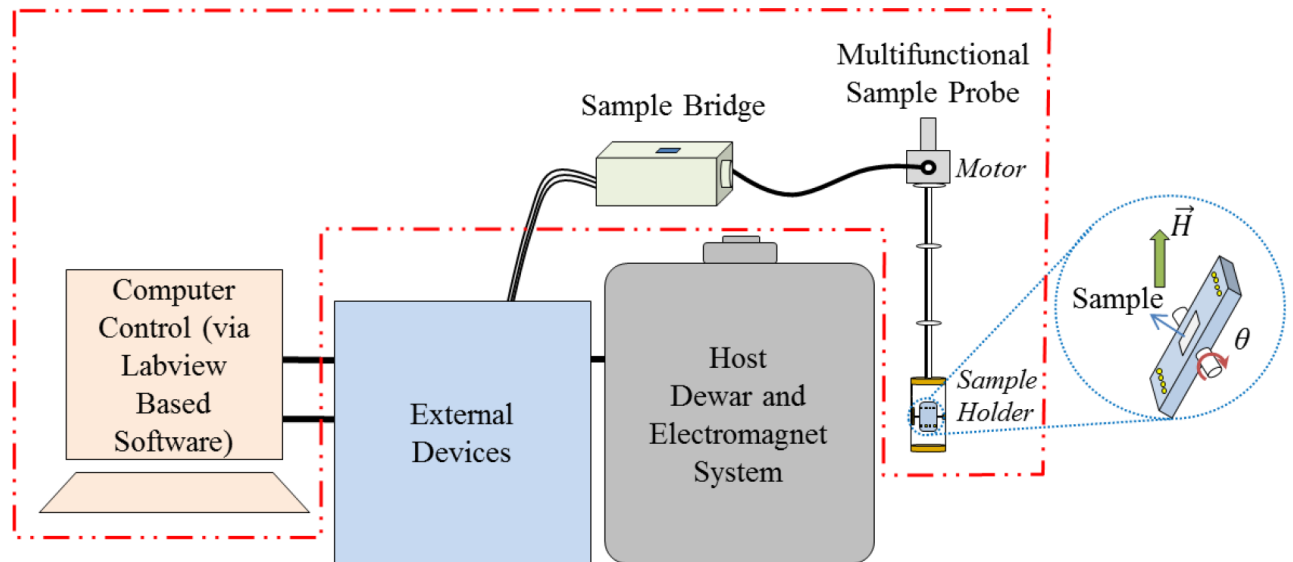


**Figure 1.** Schematic drawing of the multifunctional sample probe.

The multifunctional sample probe includes a vacuum-tight connector on top, which connects to a sample bridge via an insulated cable. The sample bridge is designed as a cable distribution and connection box and has 12 contact holes for the connections between the sample holder and all other external devices, as shown in Figure 2 (right). Four of the connection holes located on the sample bridge are reserved for the cernox thermometer and the other 8 holes are used for the measurement devices. The sample bridge may also be used as a test measurement tool since it has a female PCB which allows checking the connections of the sample before the measurements. A general scheme of the transport measurement setup consisting of a multifunctional sample probe, a sample bridge, external devices, a host dewar, and an electromagnet system is shown in Figure 3. With this design, installation and de-installation of the sample probe can be performed within seconds.



**Figure 2.** Pictures of the sample holder (left) and the sample bridge (right).

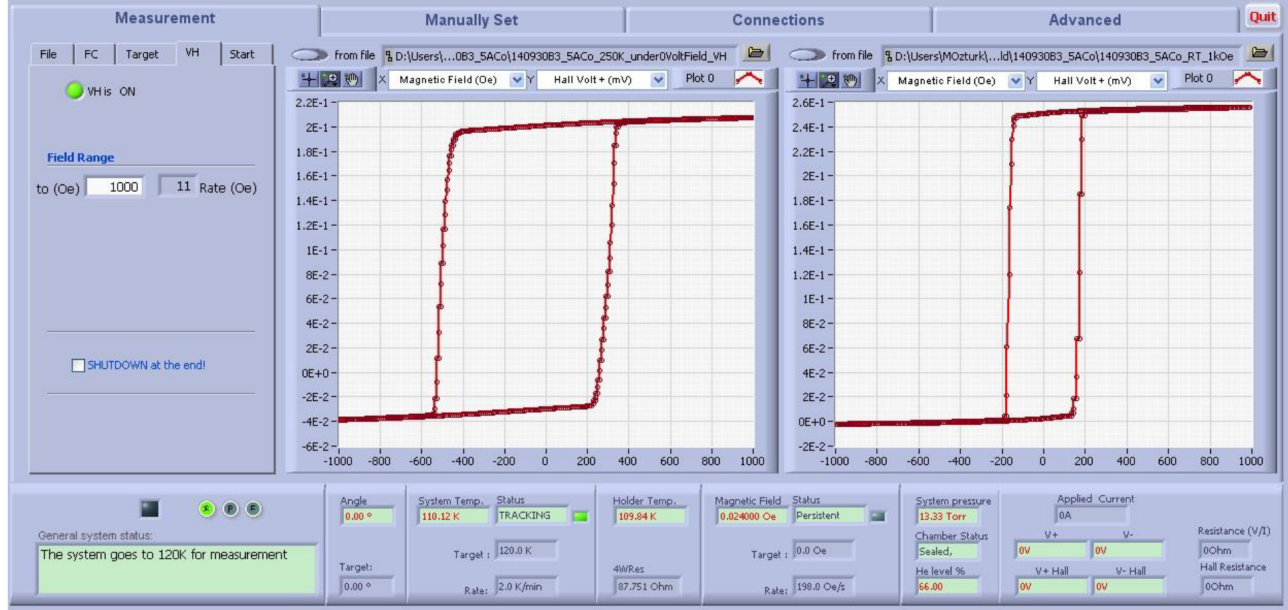


**Figure 3.** General view of the setup that includes the multifunctional sample probe, the host system, and any external devices.

In the test measurements, QD PPMS9T was used as a host dewar, which provided a range of magnetic field up to 9 T within the temperature range of 2 K to 400 K. A current source was combined with a voltmeter for the resistance measurements of low resistant materials. For samples with higher resistivity (i.e. semiconductors, insulators), a high resistance meter was employed in order to apply high voltages and measure low currents. It also served as a voltage source for the manipulation of the electrical field during the AHE measurements. Data acquisition was performed with a general purpose interface bus (GPIB) connected to the external devices. The magnetic field, vacuum, and temperature control of the whole setup was managed via the control unit of the host system. In addition to the temperature sensors of the host system, the sample probe included a calibrated cernox temperature sensor attached to the sample holder. This sensor provided a more accurate setting and thus prompted measurement of the temperature at the sample location.

Since the system has many variables (system and sample temperatures, sample angle, pressure control,

magnetic and electrical fields, current, and voltage), practical control of the devices and data acquisition needs a fully automated and computer-controlled interface. For this reason, we developed user-friendly software based on NI Labview. Figure 4 demonstrates the user interface of the software. Real-time information and the data from all devices of the setup are monitored at all times by the user interface. The software allows users to simultaneously plot the data of present and previous measurements.



**Figure 4.** The main user panel of the software that allows the control and the reading of all the parameters of the measurement setup.

### 3. Test measurements

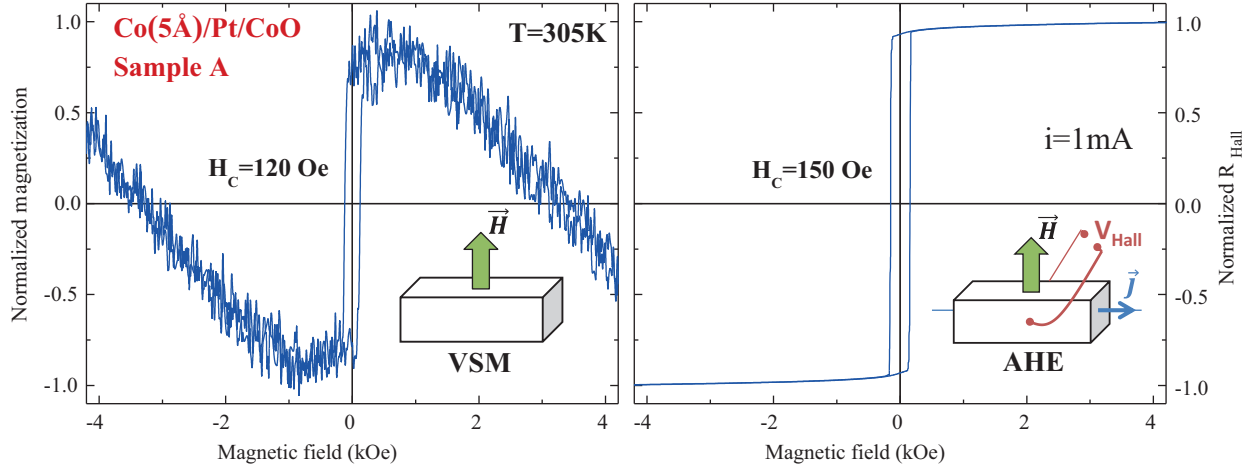
AHE measurements were carried out on perpendicularly magnetized Pt (5 Å)/Co (5 Å)/Pt (5 Å)/CoO (10 nm)/Pt (3 nm) (sample A) and Pt (5 Å)/Co (4 Å)/Pt (5 Å)/CoO (10 nm)/Pt (3 nm) (sample B) ultrathin films grown on fused silica substrates. For the observation of AHE contribution in thin films, the ferromagnetic layer should be perpendicularly magnetized or should have a perpendicular projection of magnetization. Hall resistivity ( $\rho_H$ ) of these kinds of thin films is the sum of two contributions, namely the contribution coming from the ordinary Hall effect ( $\rho_{HE}$ ) and the other from the anomalous Hall effect ( $\rho_{AHE}$ ) [6]. In ferromagnetic materials, since AHE is often much larger than the ordinary Hall effect, the Hall resistivity measurements reflect the magnetization changes and results in a hysteresis loop [6]. This can be expressed as

$$\rho_H = \rho_{HE} + \rho_{AHE} = R_0 H + R_A \mu_0 M \quad (1)$$

where  $R_0$ ,  $H$ ,  $R_A$ ,  $\mu_0$ , and  $M$  stand for the Hall coefficient, applied magnetic field, anomalous Hall coefficient, permeability of vacuum, and the material magnetization, respectively.

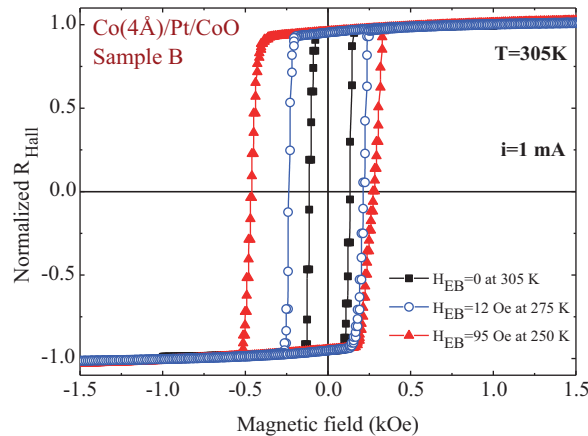
Figure 5 shows the hysteresis loop measurements of sample A obtained by both VSM and AHE methods. It is clearly seen from the figure that the hysteresis curve measured by VSM has a lower signal-to-noise ratio (SNR) and the diamagnetic contribution of the substrate is very dominant. On the other hand, the measurement

with AHE has much better SNR and the square shape of the loop is more obvious. This is due to inherently better sensitivity of AHE method for ultrathin ferromagnetic materials compared to the VSM method. Since methods such as VSM [18] and superconducting quantum interference device (SQUID) [19] are bulk measurement techniques, ferromagnetic properties of ultrathin films are usually measured against a strong background due to diamagnetic or paramagnetic contributions from the substrate, holder, or other layers.



**Figure 5.** Perpendicular magnetization measurements of Co(5 Å)/Pt/CoO ultrathin film (sample A) obtained by VSM (left) and AHE (right) techniques.

Figure 6 presents the temperature-dependent AHE hysteresis loop measurements of sample B. The loops were measured at temperatures of 250 K, 275 K, and 300 K. Since the sample had both ferromagnetic (FM) Co and antiferromagnetic (AF) CoO layers, a shift in the hysteresis loop of the FM layer was observed at the temperatures below the Néel temperature of CoO ( $\sim 290$  K). This shift is called the exchange bias (EB) effect [20,21] and has great importance for data storage and magnetic sensor technologies. These results prove the capability and the sensitivity of the developed probe for the magneto-transport measurements of ultrathin films.



**Figure 6.** Exchange bias effect measurements of Co (4 Å)/Pt/CoO ultrathin film were carried out by AHE method at different temperatures.

#### 4. Conclusion

In summary, a multipurpose sample probe with a rotatable sample holder was constructed and successfully integrated into a host electromagnet system. The setup allows magneto-transport measurements at various magnetic and electrical fields, angle, temperature, and current/voltages with full control of the experimental conditions. The setup is flexible in configuration and allows addition or removal of any external device according to the user demands. The multifunctional sample probe can also be used with other dewar and magnet systems for a variety of applications. We also developed user-friendly software that provided full control of the whole system and real-time data acquisition. We successfully tested the system in AHE measurements of ultrathin films. Square-like hysteresis loops and the EB effect were observed in perpendicularly magnetized Co (4 Å)/Pt/CoO and Co (5 Å)/Pt/CoO ultrathin films with very good sensitivity.

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