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Scanning hall probe microscopy technique for investigation of magnetic properties

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ABSTRACT

Scanning Hall Probe Microscopy (SHPM) is a scanning probe microscopy technique developed to observe and image magnetic fields locally. This method is based on application of the Hall Effect, supplied by a micro hall probe attached to the end of cantilever as a sensor. SHPM provides direct quantitative information on the magnetic state of a material and can also image magnetic induction under applied fields up to ~1 tesla. This method is non-invasive with high spatial resolution and sensitivity. Furthermore, this microscopy technique can be operated in a wide range of temperatures while the magnetic field caused by hall probe is so minimal, which has negligible effect on the measuring process. Meanwhile, the sample does not need to be an electrical conductor, unless using Scanning Tunneling Microscope (STM) for height control. SHPM measurements can be performed in ultra-high vacuum (UHV) and are non-destructive for crystal lattice and complicated structures.

Keywords: *Scanning Hall Probe Microscopy; Hall effect; Hall sensor; Scanning probe microscopy; Magnetic field.*

INTRODUCTION

Study of magnetic materials has always been considered an interesting research area. Researchers have been trying to understand magnetism to make new magnetic materials and superconductors. Microscopy techniques employing sensitive magnetic sensors have enabled this idea. Various techniques such as scanning Hall probe microscopy, scanning quantum interference device (SQUID), magnetic force microscopy (MFM), magneto optical imaging and Faraday rotation can be used for this purpose. Although MFM has the highest spatial resolution among the above methods, the quantification of its signal is difficult. The magnetic field resolution is very high in SQUID, but its spatial resolution is limited. Therefore, SHPM technique with the ability of high magnetic and spatial resolution is known as a superior technique.

Moreover, this method is non-invasive and can be operated over a wide range of temperatures and external magnetic fields [1-3].

The Scanning Hall Probe Microscope is one of the SPM's techniques originated after the invention of Scanning Tunneling Microscope (STM) by Binnig and Rohrer in 1981.This microscope has essentially been designed to measure local physical properties at a surface by moving an appropriate sensor over the surface and taking an image. All these microscopes use scanning equipment, which is a three dimensional piezoelectric drive. The piezoelectric drive moves the probe precisely over the surface in three dimensions. In SHPM, the Hall probe is mounted on the scanning equipment to investigate the magnetic property. This idea was developed by Chang and *et al.* in 1992 [4] for the first time and then Oral and *et al.* [5] improved this instrument in 1996. Therefore, the sensitivity of modified SHPM was increased up to 3×10^{-6} T/√Hz at 300 K and it was enabled to take high resolution images. Measuring the Hall Voltage during this process directly yields the local magnetic field, which can be recorded and displayed in two dimensions using conventional scanning probe microscopy electronics and software [6-7].

In contrast to its companions, MFM and scanning-SQUID, scanning Hall effect sensors were already used in the late 1960s to image superconducting materials with a spatial resolution as high as 4 µm and a field sensitivity of 100 µT. It was not until the late 1970s, however, that semiconductor Hall sensors with a two dimensional electron gas layer (2DEG) were manufactured by modulation doping (Dingle 1978). This invention increased the mobility of electron carrier to values much larger than that of any of the other existing compounds, allowing the combination of high field sensitivity with high spatial resolution, even at low temperatures [6].

In this overview, scanning Hall probe microscopy method and its application and role in studying and designing new magnetic materials is presented.

Hall Effect

Scanning Hall probe Microscopy, as a member of SPMs family, is similar to other microscopes. However, the detection technique is different. The Hall probes operate based on the principle of Hall Effect.

According to Figure 1, establishing electric current causes electrons to pass through a straight line. Under these conditions, applying a magnetic field leads the electrons to deviate from the straight line, so that they will accumulate on one side and a potential difference is obtained. The magnitude of created voltage depends on the magnitude of electric current and magnetic field.

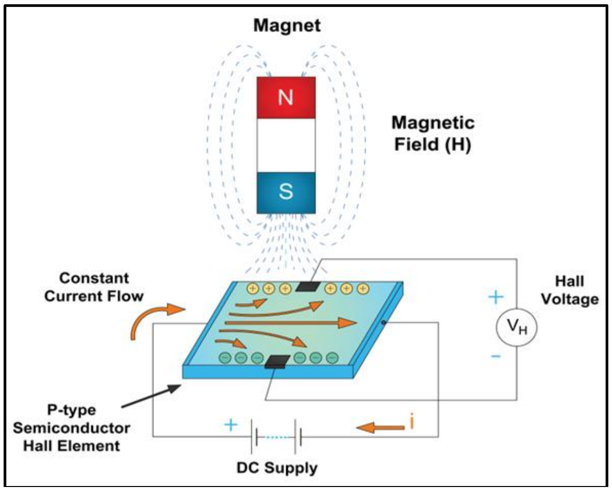

Fig.1. Effect of magnetic field over the current electricity flow

Due to this effect, which was discovered by Edward H. Hall (1878), when a conductor is placed in an external magnetic field perpendicular to the current flow. The transverse voltage, which is called Hall voltage, appears across the material and can be measured as follows:

$$V_x = -\frac{I_y B_z}{n_{2d} e}$$

Where, I_y is the current passing through the Hall bar, B_z is the magnetic induction perpendicular to the plane of the Hall bar and n_{2d} is the carrier density per unit area of the Hall bar.

Therefore, materials with small carrier densities such as semi-metals and semi-conductors show larger Hall voltages. Recently, evaporated films of bismuth, InSb and GaAs have been used in the structure of Hall bar systems. One of the most sensitive Hall probes with extra high spatial resolution is constructed by $GaAs/Al_x Ga_{1-x}$ as hetero-junction structures [8].

As observed in Figure 2, the Hall voltage and the developed electric field are perpendicular to the direction of applied magnetic field and current. According to the well-known Lorenz force, when a charge carrier moves along a direction perpendicular to the magnetic field, the Lorenz force is applied to the current perpendicular to the both magnetic fields and current direction and finally causes the carrier charges to accumulate towards one edge of conductor.

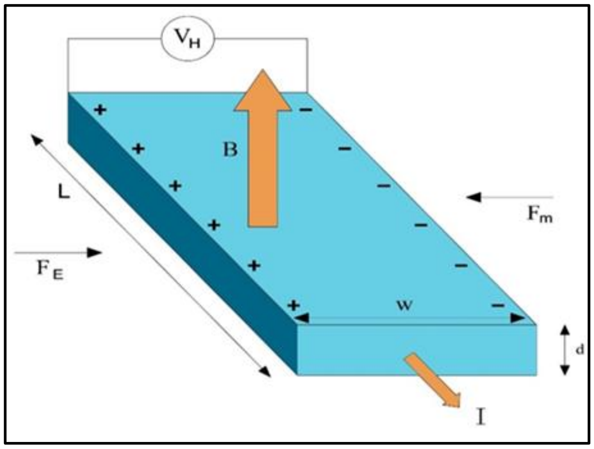

Fig. 2. Hall Effect schematic illustration [1]

Hall voltage has different polarities for positive and negative charge carriers, named holes and electrons in semiconductors, respectively. In the semiconductors, at temperatures above absolute zero, electrons can be excited across the band gap into the conduction band, which creates an electric current. When an electron is transferred to the upper band, a hole remains in a regular lattice and when an external voltage is applied to the semiconductor, the hole and electron will move through the material in opposite directions. According to this point, the dopant produces extra electrons in n-type semiconductors and makes extra holes in p-type semiconductors. It can be concluded that depending on the type of semiconductor, the sign of voltage applied will be different and the unknown materials can be investigated.

The velocity of drift charge carriers can be also measured by moving the Hall probe with different speeds until the Hall voltage vanishes. In this case, the moving charge carrier in magnetic field has been stopped and thus the average drift velocity, which is proportional to the mobility, can be obtained [1].

SHPM body

The schematic illustration of Scanning Hall Probe Microscope is shown in Figure 3 and its main components are:

- Hall probe sensor to gather data
- A system for placing the probe close to the sample surface
- A piezoelectric to move the probe over the sample surface with high precision
- An electric feedback system to control the moving parts of system
- A computer for analyzing the measured data and converting the data to the image

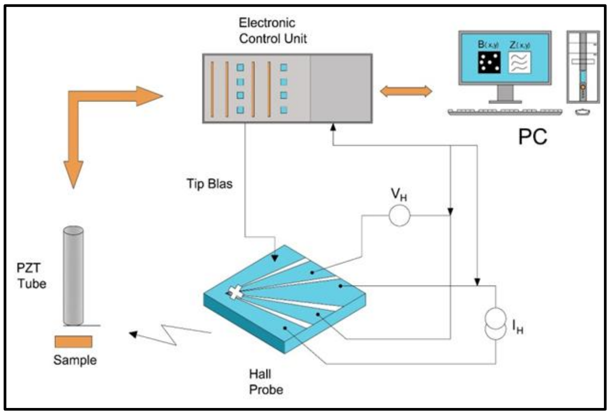

Fig. 3. Schematic illustration of SHPM [1]

As in other SPM's microscopes, in SHPM, the probe is one of the most important components of microscope because of its responsibility for gathering data from the surface sample. The resolution of microscope is dependent on the size of active area and height of the probe. The alignment of probe against sample is also important to achieve good result.

Hall probe fabrication

The Hall sensor is the most important part in the collecting data step. Therefore, it must be made with extreme precision. The positions of tip and Hall probe are shown in Figure 4.

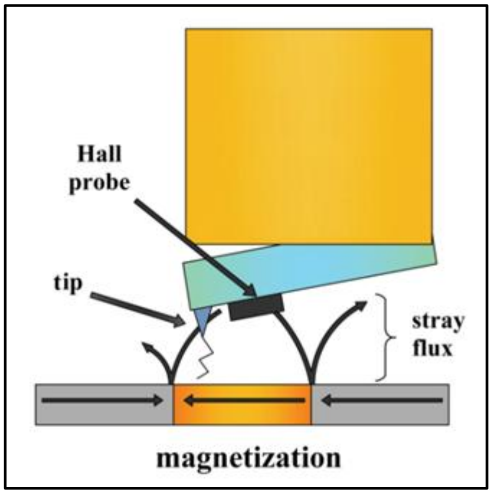

Fig. 4. Hall probe and collecting magnetic data by Hall probe

To produce the Hall probe, a typical cleaned wafer is needed, although accumulated photo-resist will usually exist at the edge and corners of wafer surface after cleaning step. This phenomena leads to unexpected diffraction of light during pattern process and decreases the quality performance of probe. This problem will be reduced by using the whole wafer or a piece of wafer with a round edge. The pattern is made by photolithography and wet chemical etch process over the wafer. For this purpose, the chip is immersed in HCl solution (H_2O: H_2O_2: HCl, 4:10:55) for one hour and is then coated by resist to prevent the back side from etching and increase the quality of wafer.

A typical standard method to clean the surface is as follows:

First, the sample is boiled in trichloroethane for 2 min, followed by immersion in acetone for 5 min. In the next step, the chip is boiled in isopropyl alcohol for 3 min, cleaned with deionized water and dried in nitrogen flow.

The chip is now ready for lithography process. A photo-resist layer is spun over the surface and the pattern is then transformed by exposing UV light to the photo-resist layer. The suitable photo-resist thickness is about 1 μm, which can be achieved by spinning the AZ5214E photo-resist at 10000 rpm for 40 s. Before exposure, the chip should be hot baked at 110°C for 55 s to evaporate the resist solvent and harden the photo-resist.

The etching solution (H_2O: H_2O_2:H_2SO_4, 320:8:1) is used to develop the pattern. The typical etch rate is 2 nm/s at 21°C. The chip is then hot baked at 120 °C for 2 min to prevent from etching the resist. The etching process can also be done by plasma. However in dry etching process, the plasma ions can easily damage the photo-resist. Therefore, depending on the gas used in plasma, a suitable metal such as aluminum must be used to protect the sample. Lithography and etching characteristics such as spin and time rate, expose power, development time and other parameters affect the quality of Hall bar's shape.

Approximately four individual Hall probe scans are created on a chip. Another etching step is applied to separate and cleave these probes by using the etching solution (H_2O:H_2O_2:H_2SO_4, 40:8:1). The typical rate of this step is about 13 nm/s and the probes are etched down for 1-1.2 μm. At the end, the chip is etched down again to remove any part of chip, which is higher than Hall bar. This prevents bonding wire.

The wafer material is semiconductor and unable to conduct the electricity well. Thus, it is necessary that a metallic layer be penetrated into the region to lower the noise. For this purpose, the coating area is defined by optical lithography and the sample is immersed into chlorobenzene for 15 min. Germanium, gold and nickel are then evaporated on the sample through the mask in a box coater with a base pressure of 10^{-7} mbar. Acetone is used to lift off process and then the chip is annealed at 450 °C for 45 s with a Rapid Thermal Process (RTP) system. Metallization process is repeated by using Ti and Au as 10 nm titanium layer and 150 nm gold layers are created over the surface. The created Hall probes are cleaved from each other and mounted on a chip holder by a gold wire with a diameter of 12 μm.

As a result, it can be mentioned that the shape, thickness and probe material can be affective on the probe performance. Therefore, this manufacturing process requires a lot of attention [1].

Hall probe resolution and sensitivity

Spatial resolution is one of the most important factors in Hall probe performance, which is roughly determined by $\sqrt{H^2 + L^2}$.

Where H is the height of the probe above the sample and L is the size of the probe (Figure 5). It can be concluded that if the active Hall bar and distance between the probe and sample decrease, the spatial resolution will also increase [9, 10].

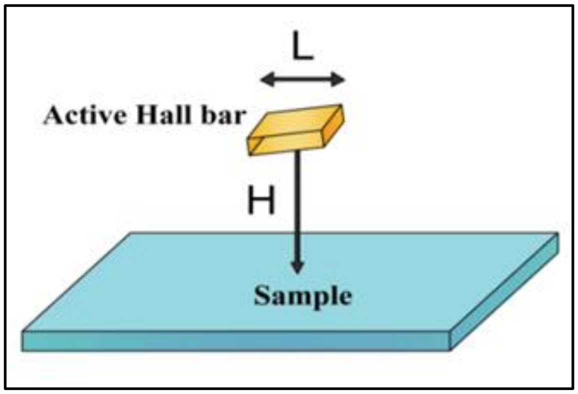

Fig. 5. Effective parameters determine the Hall probe spatial resolution [10]

Thus, to obtain a good sensitivity of Hall probe, the probe size should be reduced. When the sensor is large, most of the sample magnetic flux will pass up and down through the sample and thus the average magnetic field will decrease. While the smaller sensor is able to capture less of the field lines and the total received signal will increase by the probe.

However, decreasing the Hall probe size is limited because fabrication of submicron structures is difficult. In addition, the height of sensor is as effective as size in resolution. However, fabricating a small hetero-structure probe is essential to achieve acceptable sensitivity and resolution.

Meanwhile, the low frequency noise in large Hall probes and random telegraph noise, known as switching noise, are typical noises. These noises can change the resistance properties of Hall probe and thus may not be easily separated from measured magnetic signals. Scanning quickly is the only method to decrease the low frequency noise [2].

Another method to improve the Hall probe resolution is increasing its mobility; that is, suppressing the scattering carries. Nowadays, GaAs/Al$_{0.3}$Ga$_{0.7}$As is used in almost all Hall probes. It has identical lattice constant and can be commercially produced. Observations show that the GaAs/Al$_{0.3}$Ga$_{0.7}$As structure is a coherent single crystal with modulated conduction and valence band edges owing to the much larger band gap. The schematic diagram of the conduction band edge through GaAs/Al$_{0.3}$Ga$_{0.7}$As structure is shown in Figure 6.

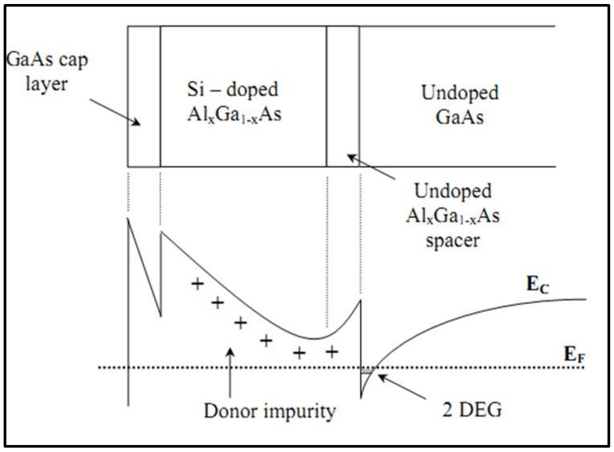

Fig. 6. Structure of GaAs/Al$_{0.3}$Ga$_{0.7}$As layer [7]

SHPM operational details

In order to investigate scanning Hall probe microscopy, the sample must be cleaned and placed on the sample holder and then the Hall probe brought co-linearly at the end of the microscope body. The sample position to the hall probe has about 1-15° tilt angle. In this position, Hall probe is placed near the sample as possible. A cryostat tube made of stainless steel is flushed by helium gas several times and is then placed in the SHPM and cooled to 77 K at a rate of 2-3 K/s. After that, the microscope is transferred to the liquid nitrogen dewar to maintain the temperature constant at 77 K. To remove the vibration of floor, it is better to place the dewar on a vibration isolation stage. A coil, which is able to create magnetic field up to ±140Oe with ±10V, is used to supply external magnetic field around the cryostat body. The sample is now ready for investigation by SHPM [1].

SHPM calibrating

A commercial hard disk drive can be used to calibrate SHPM. MFM data is also used for comparison at ambient temperature. For SHPM scanning, it is necessary that the disk surface becomes conductive to get tunneling current. Therefore, a 20 nm gold film is coated over its surface [7].

SHPM scanning modes

Usually, two operational modes are considered for SHPM, the STM tracking mode and the flying mode, which are illustrated in Figure 7. In the STM tracking mode, the sample must be brought near the sample until a tunneling current is created. Then, the Hall probe will scan the sample surface to measure the magnetic field and surface topography. However, the probe may become damaged in this mode. Consequently, the flying mode is much more usable. In this mode, the probe is brought near the sample surface again until the tunneling current is established. The distance between the sample and probe is then slightly increased to a few hundred nm and Hall probe will start scanning the surface in this new distance while the spatial resolution is slightly decreases. To compensate for this defect and achieve higher resolution, a small angle between the Hall probe and sample, as few as 1-2 degree, is needed [10].

However, there are other modes for SHPM operation: the simultaneous STM/SHPM mode, in which the Hall bar signals and STM topography are simultaneously monitored and the fast SHPM mode, in which Hall probe junction output is monitored line by line. In the real time SHPM mode, magnetic images are displayed after completing the measurement [1].

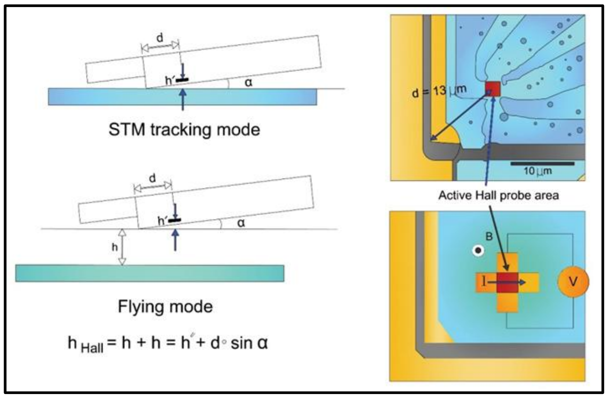

Fig. 7. SHPM scanning modes and an optical photograph of active Hall sensor area [10]

Applications

- **Superconductors**

One of the widest applications of SHPM is the investigation of vortices and flux distribution in super conductors [11, 12].The phenomenon of superconductivity is observed when the electrical resistance of certain materials completely vanishes at low temperatures. This was first discovered in 1911 by Kamerlingh Onnes, while they were studying the resistance of metals at low temperatures. After that, magnetization measurements of superconductors have been carried out using a Hall probe.

The full potential of the technique will be illustrated with results of vortex imaging studies of three distinct superconducting systems: (i) vortex chains in the "crossing lattices" regime of highly anisotropic cup rate superconductors, (ii) vortex–anti vortex pairs spontaneously nucleated in ferromagnetic superconductor hybrid structures, and (iii) vortices in the exotic wave superconductor Sr_2RuO_4at mk temperatures. The very strong crystalline anisotropy in the high temperature cup rate superconductor $Bi_2Sr_2CaCu_2O_{8+\delta}$ (BSCCO) is reflected in the vortex (and vortex lattice) structure as a function of direction of applied field.

The other applications, which are possible by SHPM include detecting magnetic domains and domain walls [13, 14] in magnetic systems such as floppy disk and Sr ferrite magnets. Bismuth is considered an appropriate alternative to GaAs/AlGaAs for the fabrication of nanometer sized Hall probes for the magnetic imaging of ferromagnetic domain structures at ambient temperature [15]. Detection of magnetic micro beads for biochemical applications [16, 17], imaging and development of new high density magnetic recording media [18, 19] , detection of magnetic resonance signals with a Hall sensor [20, 21], investigation of penetration depth in thin films [22] such as a high Tc$YBa_2Cu_3O_{7-\delta}$ thin film with single vortex resolution, studying magnetic film profiles using both topographic and thermo magnetic methods [23], and considering spatially varying flow in patterned microstructures are other possible applications of SHPM.

- **Biology and medicine**

Scanning Hall Probe Microscopy (SHPM) imaging is a particular interest in biological and medical research because of being non-contact, no need for electrical conductor and non-destructive techniques. Meanwhile, this technique can be operated at ambient temperature. In neuroscience, SHPM with time varying magnetic fields is used to investigate certain psychological disorders as a non-invasive therapy and it is necessary to achieve precise and selective manipulation or stimulation in a controllable manner. SHPM techniques coupled with Hall magnetic sensing devices were employed for the characterization of stray magnetic field emanating from magnetic force microscope tips equipped with a Hall probe (with a small sensing aperture) and a high resolution piezoelectric stage. A scanning Hall probe microscope (SHPM) system can achieve the needed sensitivity and spatial resolution for micro scale magnetic field measurements and it can thus be adapted for the experimental characterization of micro electromagnetic probes. Therefore, fabrication of SHPM probe can be readily adapted to various biological manipulation and stimulation applications. For this application, a micro electromagnetic probe is needed consisting of a protruding (out of chip), sharp Perm alloy needle embedded into a three dimensional gold conducting coil. The probe fabrication is carried out using traditional surface micromachining processes coupled with assembly techniques. This hybrid approach significantly reduces fabrication difficulties [4, 24-28]. Figure 8 shows micro electromagnetic probe developed for biological samples. The probe consists of a supporting substrate, a solenoid conducting coil and a protruding permalloy magnetic core with a sharp tip, which allows interaction with biological samples [8].

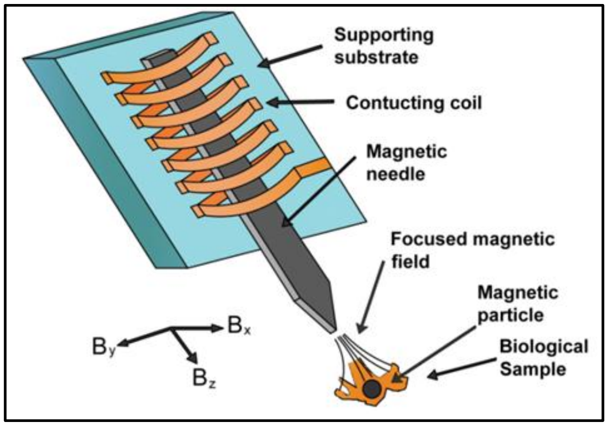

Fig. 8. Schematic illustration of developed micro electromagnetic probe for biological samples [11]

The Advantages of SHPM to other magnetic raster scanning methods

SHPM is a superior magnetic imaging technique due to many reasons such as [10]:

- Unlike the MFM technique, the Hall probe exerts negligible force on the underlying magnetic structure and is thus non-invasive.
- Unlike the magnetic decoration technique, the same area can be scanned frequently.
- Magnetic field caused by hall probe is so minimal that it has a negligible effect on measuring process.
- Sample does not need to be an electrical conductor, unless using STM for height control.
- Measurement can be performed at 5-500 K.
- Measurement can be performed in ultra high vacuum (UHV).
- Measurement is non-destructive for crystal lattice.
- Tests require no special surface preparation or coating.
- Detectable magnetic field sensitivity is approximately 0.1 µT- 10 T.
- SHPM can be combined with other scanning methods like STM.

The Limitations of SHPM

There are some shortcomings or difficulties when working with an SHPM, which can be described as follows [10]:

- High resolution scans become difficult due to the thermal noise of extremely small hall probes.
- There is a minimum scanning height distance due to the construction of the hall probe. This is especially significant with 2DEG semi-conductor probes due to the multi layer design.
- The distance between probe and sample can affect obtained image.
- Scanning large areas needs a long time.
- Relatively short practical scanning range (order of 1000's micrometer) along any direction.
- Housing is important to shield electromagnetic noise (Faraday cage), acoustic noise (anti-vibrating tables), air flow (air isolation cupboard), and static charge on the sample (ionizing units).

CONCLUSIONS

Scanning Hall probe microscope (SHPM) is a type of a scanning probe microscope, which incorporates accurate sample approach and position of scanning tunneling microscope with a semiconductor Hall sensor. This combination allows mapping the magnetic induction associated with the sample. Current state of the art SHPM systems utilize 2-D electron gas materials (e.g. GaAs/AlGaAs) to provide high spatial resolution (~300 nm) imaging with high magnetic field sensitivity. Unlike the magnetic force microscope, the SHPM provides direct quantitative information on the magnetic state of a material. The SHPM can also image magnetic induction under applied fields up to ~1 tesla and over a wide range of temperatures (mK to 300 K) [29].

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