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An ultra-low temperature scanning Hall probe microscope for magnetic imaging below 40 mK

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We describe the design of a low temperature scanning Hall probe microscope (SHPM) for a dilution refrigerator system. A detachable SHPM head with 25.4 mm OD and 200 mm length is integrated at the end of the mixing chamber base plate of the dilution refrigerator insert (Oxford Instruments, Kelvinox MX−400) by means of a dedicated docking station. It is also possible to use this detachable SHPM head with a variable temperature insert (VTI) for 2 K–300 K operations. A microfabricated 1μm size Hall sensor (GaAs/AlGaAs) with integrated scanning tunneling microscopy tip was used for magnetic imaging. The field sensitivity of the Hall sensor was better than 1 mG/$\sqrt{\text{Hz}}$ at 1 kHz bandwidth at 4 K. Both the domain structure and topography of $LiHoF_4$, which is a transverse-field Ising model ferromagnet which orders below $T_C = 1.53$ K, were imaged simultaneously below 40 mK. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4897145]

I. INTRODUCTION

Scanning Hall Probe Microscopy (SHPM) is a well known quantitative and non-invasive magnetic imaging technique developed over two decades ago.[1] It has been used for investigating many magnetic phenomena in physics and material science using advantage of high magnetic field sensitivity[2] and spatial resolutions down to 50 nm[3] in a broad temperature range and high magnetic field. It is possible to image both conducting and insulating specimens using scanning tunneling microscope (STM) and atomic force microscope (AFM) feedback mechanisms,[4] respectively.

Hall sensors are made of various materials like GaAs/AlGaAs 2DEG heterostructures, epitaxial InSb thin films, Bismuth, etc., for different applications with different field sensitivities and spatial resolutions. Single layer graphene has recently been shown to be one of the alternative materials to increase the spatial resolutions of the current Hall sensors.[5]

SHPM sensors have a wide range of operation temperatures for scanning between 300 mK and 425 K.[6] Despite availability of many reports on SHPM for 2 K-300 K temperature ranges in the literature, only a single report from Professor Bending's group from Bath University is seen below 1 K.[7] They reported a scanning Hall probe microscope designed for ^{3}He system and operated at 372 mK. Our group also designed a SHPM for a ^{3}He system (Oxford Instruments, Heliox VT−50) and was able to measure at 322 mK.[8] So far there has been no SHPM reported below 300 mK.

In this work, we describe the design of a SHPM for a dilution refrigerator (Oxford Instruments, Kelvinox MX−400) and operation of the microscope below 40 mK for the first time. This extremely low temperature would be fascinating in the magnetic imaging techniques since there are many interesting problems below 1 K like heavy Fermion superconductivity[9] and spin-glass structures. Therefore, an ultra low temperature SHPM system would be very useful for understanding and exploring concepts of these scientific topics.

II. INSTRUMENT DESIGN

A. Cryostat

An Oxford Instruments cryostat with a 9T superconducting magnet which is capable of accepting either a dilution refrigerator insert (Kelvinox MX−400) as shown in Fig. 1(a) or a variable temperature insert (VTI) was used for the test and the experiments. The microscope head was designed such that it can be used in both of the inserts. The dilution fridge (DR) has 400 μW cooling power at 100 mK and it has the minimum base temperature level of 8 mK at mixing chamber base plate without any thermal load. The fridge has a detachable experimental insert onto which the sample holder and related wiring for the SHPM are mounted. The cryostat is mounted on a 5 cm thick triangular aluminum plate with a hole to accept the cryostat, which was floated by three air stabilizer legs[10] with 1.5 Hz nominal cut-off frequency and auto weight-balance mechanism. The floor where the vibration isolation stage sits was isolated from remaining laboratory floor where all the pumps and controller units were located. The vacuum tubes,

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Helium circulation pipes and the electrical leads attached to the fridge, and the SHPM are connected through a 60 kg concrete block in order to minimize vibration coupling to the microscope.

B. Microscope design

The microscope head was integrated on the fridge by means of a docking station as seen in Fig. 1(b). A detachable docking station was designed and mounted at the end of the experimental insert. The docking station comprises low temperature high density miniature connectors and has a large physical contact area with the mixing chamber to ensure good thermal conductivity. The microscope head, which has 25.4 mm OD and 200 mm length, is attached to the other side of the docking. The length of the microscope was designed so that the sample/sensor couples sit at the center of the magnetic field. Both the microscope and docking were made of oxygen-free high thermal conductivity copper (OFHC) for enhancing the thermal conductivity from the mixing chamber through the sample. The whole body of the SHPM and docking station are electroplated with gold. The sample holder was thermally anchored using a copper loom through the microscope body and the temperature is directly monitored by a RuO_2 thermometer placed on the microscope body.

The microscope head is composed of two concentric piezo tubes: the inner piezo tube is used for scanning and the outer one is used for the sample positioner. The length of the scan piezo is 3″ and has ∼18 μm xy scan range and ∼1.4 μm z range at 4 K. They are mounted into a metal holder concentrically to minimize thermal drift. The scan piezo is composed of quadrant electrodes. The length of the sample slider piezo is 1.5″, which has also quadrant electrodes, and a glass tube mounted at the end. A sample slider puck is loaded on this glass tube using a leaf spring and two screws. A stick-slip sample approach mechanism is used for rough positioning of the sample. The sample slider piezo can move the sample in 10 mm Z-direction and in Ø3 mm XY directions with adjustable step size from 50 nm to 800 nm.

The wiring of the microscope from ambient to the mixing chamber was optimized in order to minimize the heat transfer. A single soft stainless steel coaxial cable for carrying the tunneling signal and 24 constantan loom pairs for two piezo motors, Hall sensor, and spare connections were used. The cables were wrapped around 45 mm length copper rods for thermal anchoring at the inner vacuum can (IVC) roof, 1 K pot, still, cold plate and mixing chamber stages from top the bottom on the experimental insert, respectively.

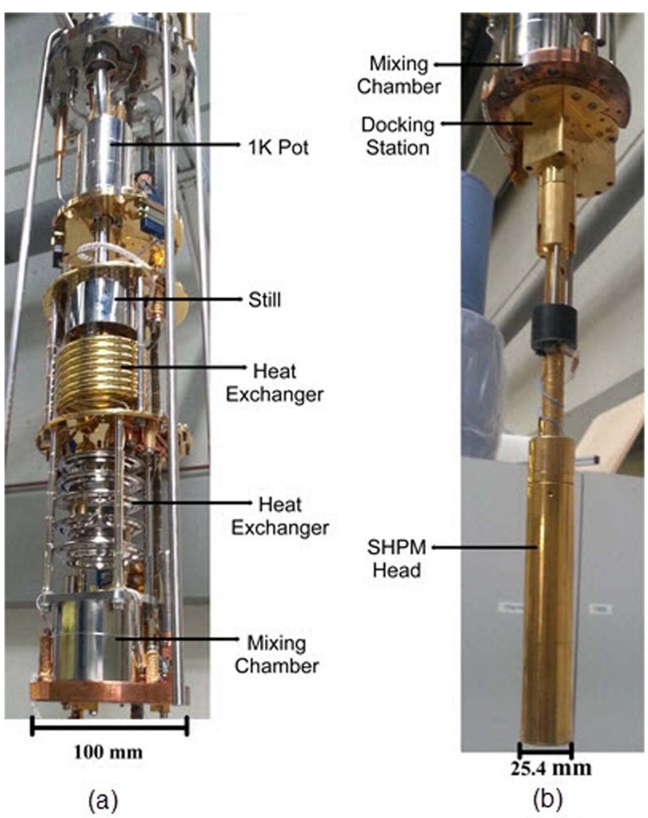

FIG. 1. (a) Dilution refrigerator and (b) integrated SHPM head at the end of the mixing chamber base plate.

C. Hall probe

An optical microscope image of a typical Hall probe (HP) used in the experiments is shown in Fig. 2. HPs were microfabricated from high mobility GaAs/AlGaAs heterostructure 2DEG materials. The Hall sensor is micro fabricated close to a gold-coated corner of a deep etch mesa, which serves as STM tip for feedback. The cross size of the Hall probe was defined as 1 μm. The Hall probe chip is mounted on a specially designed removable printed circuit board (PCB) and wire bonded with Ø12 μm gold wires from a recess etched plane, to eliminate the protruding wires.

Alignment of the Hall sensor is one of the crucial steps of the SHPM experiments. The sample is tilted ∼1° with respect to Hall probe on the sample puck ensuring that the corner of the mesa is the highest point. To achieve this, first the sample and the Hall probe were made parallel under the optical microscope guidance by means of three spring loaded M1.6 screws. Finally, one of the screws which sit on the opposite diagonal of the HP corner is loosened, giving the desired tilt angle accurately.

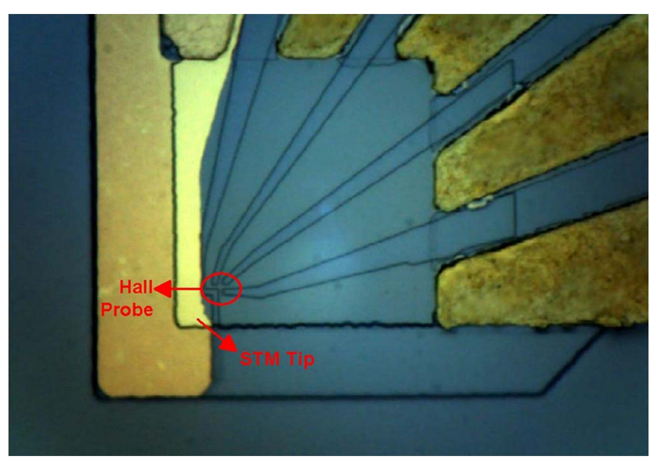

FIG. 2. Optical microscope image of 1 μm size Hall sensor. STM tip and Hall cross are shown on the picture.

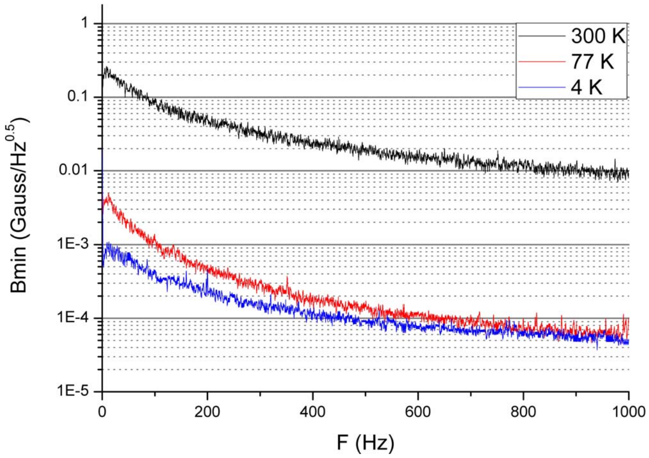

FIG. 3. Measured spectral noise density of 1 μm size GaAs/AlGaAs Hall sensor at 300 K, 77 K, and 4 K.

The serial resistances and Hall coefficient of the Hall sensor were 50 kΩ and 0.32 Ω/G at 300 K, respectively. They become 1.3 kΩ and 0.33 Ω/G at 4 K. A Rohde-Schwarz spectrum analyzer was used for the magnetic field noise measurements. The minimum detectable magnetic field, $\boldsymbol{B_{min}}$, is calculated according to the equation:

$$B_{min} = \frac{V_{Hall}}{I_{Hall}\, R_{Hall}\, G}, \tag{1}$$

where V_{Hall} is the measured voltage across the Hall sensor, I_{Hall} is the drive Hall current through the Hall sensor, R_{Hall} is the Hall coefficient, and G is the Hall voltage pre-amplifier gain factor. The Hall current, Hall coefficient, and gain factor are 50 μA, 0.33 Ω/G, and 1001, respectively, for the 4 K noise calculation. The noise level was compared with the Johnson noise of a Hall sensor at 4 K according to the equation:

$$V_{johnson} = \sqrt{4k_B T R_s \Delta f}, \tag{2}$$

where k_B, T, R_s, Δf are Boltzmann constant, temperature, resistance of the Hall sensor, and bandwidth, respectively. The Johnson noise is calculated to be 7.7×10^{-10} V/$\sqrt{\text{Hz}}$ which corresponds 0.1 mG/$\sqrt{\text{Hz}}$ at 4 K. The minimum detectable magnetic fields of the HPs were measured to be 1 mG/$\sqrt{\text{Hz}}$ at 4 K as shown in Fig. 3.

D. SHPM controller

Dedicated controller electronics and software[11] were used for operating the scanning Hall probe microscope. The controller contains very low noise power supply units for related modular cards. It has four channels of low noise high voltage amplifiers to drive scan piezo to ±200 V swings. This card is driven by 3 channels of XYZ 24Bit digital to analog converters. A DC Coupled Hall probe amplifier with x1,001 gain is used to detect the Hall voltage with 4 nV/$\sqrt{\text{Hz}}$ noise floor. The DC Hall currents between 10 nA to 1 mA can be generated digitally in the Hall Probe Card and the Hall voltage can be amplified with software controlled gain of x1, 10, 100, 1000. An I-V converter with −1 V/pA gain and 4 fA/$\sqrt{\text{Hz}}$ noise floor is used for tunnel current amplifier. The tunnel current is digitized with 16 Bits ADC at 250 kHz. A Digital PID loop is operated at 250 kHz for STM feedback, which gives analog 24 Bits signals to drive the Z position and 32 Bits digital output for the software. A sample slider card produces exponential pulses up to 380 V for the stick-slip coarse approach mechanism of the sample slider as well as XY sample positioning with adjustable step size.

III. EXPERIMENTAL RESULTS

$LiHoF_4$ has been well studied as a transverse-field Ising model ferromagnet, which orders below $T_C = 1.53$ K and undergoes a quantum phase transition in a transverse field of $H_C = 49.3$ kOe.[12] One aspect of $LiHoF_4$ which makes it such an interesting material is remarkably well defined and relatively simple microscopic Hamiltonian.[13,14] A recent theme of investigation is the effect of transverse magnetic field on the hardness of this ferromagnet, observed via bulk magnetization measurements taken at 100 mK.[15] A logical complement would be therefore to directly image how the domain structure is modified during this process. The $LiHoF_4$ single crystal was grown from a melt using the Czochralski method. A 12 × 12 × 2 mm^2 piece of this crystal was cut, polished, and gold sputtered with a 50 nm thick film of gold for utilizing STM tracking. This was then glued onto the sample holder using silver paste to ensure electrical contact between the sample surface and the puck where the sample bias voltage was given. The crystal was aligned so that the imaging plane was perpendicular to the Ising axis (crystallographic c-axis).

For SHPM imaging, 20 μA Hall current was driven through the Hall sensor and Hall voltage was measured across the scan area. The STM feedback was utilized with −100 mV sample bias and 0.5 nA set tunneling current set point. Therefore, topography and magnetic domains of the sample could be imaged simultaneously in Figs. 4(a) and 4(b). As in $LiHoF_4$ the Ho^{3+} moments are Ising and have their maximum expectation value, the real domain structure will essentially consist of a pattern of positive and negative domains with equal magnetizations (per domain volume). Taking the second derivative of the measured hall signal and assigning positive numbers correspond to positive domains and vice-versa reveals the underlying structure shown in Fig. 4(c). A pattern of "textured bubbles" is observed in contrast to the theoretically predicted parallel stripe configuration.[16] The textures seen in the bubbles are most likely due to a surface branching effect.[17]

Furthermore, by looking at the statistical distribution of the values of Hall signal measured, it becomes possible to determine the behavior of the bulk magnetization of the sample (as the zero in the signal is arbitrarily defined). This is done by making a histogram of the Hall signal measured as shown in Fig. 4(d). The distribution shows two peaks, one corresponding to positive domains and the other to negative domains. Fitting both peaks with Gaussian line shapes and comparing the distance between their centers gives the relative magnetization. By using this method we have measured several points on the M(T) order parameter curve, which agree well with neutron diffraction measurements[18] as shown in Fig. 4(e).

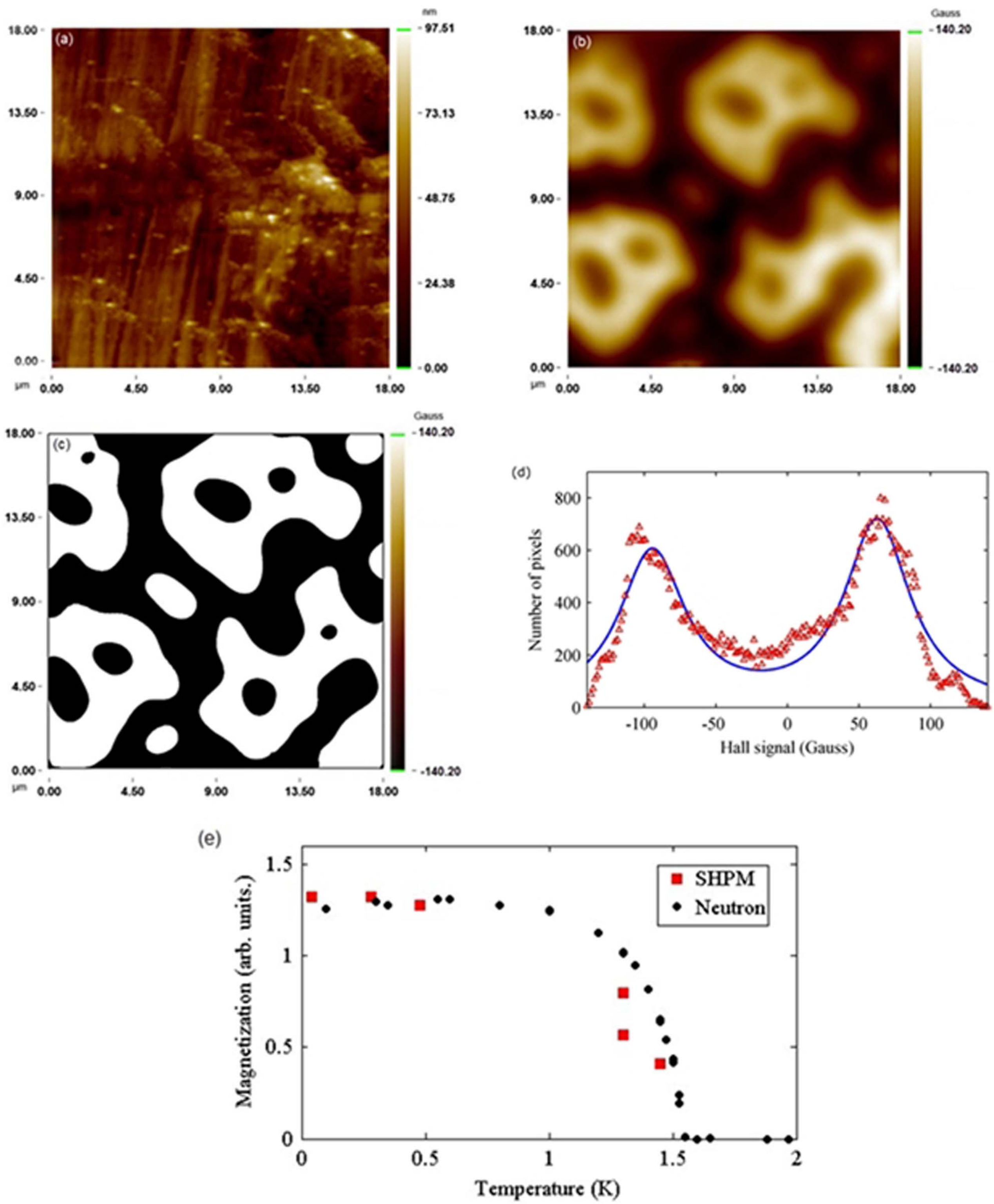

FIG. 4. (a) Topography and (b) SHPM image of the $LiHoF_4$ below 40 mK. (c) Proposed underlying domain structure extracted from topography image. (d) Histogram of Hall signal in SHPM image fit to two Gaussian line shapes. By comparing the distance between their centers the (e) M(T) order parameter curve has been generated.

During the measurement procedure, the temperature stability has been measured using the thermometer on the microscope body as shown in Fig. 5. The image shows the temperature variations during several approaches to the surface, two scans and retracting from the surface for removal. The vertical dashed lines indicate the starting and stopping times when the sample has been moved using stick-slip motion. This reveals that the coarse motion of the sample does produce a very large amount of heat on the sample due to the friction loaded sample puck. The microscope body heats up by over 100 mK, depending on the step rate during stick-slip motion, and takes up to 15 min to thermalize back to base temperature. SHPM

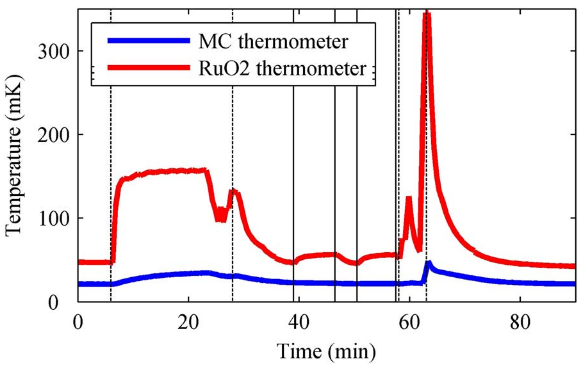

FIG. 5. Image showing the temperature stability of the microscope during stick-slip motion (vertical dashed lines) and scans (vertical solid lines).

scans are started and stopped at the positions marked by solid lines and show gradual heating of up to 10 mK throughout the scan. This indicates that while coarse repositioning sample cannot be done while staying cold, the actual measurements do not significantly heat the sample.

IV. CONCLUSIONS

We have designed and constructed a scanning Hall probe microscope for a dilution refrigerator and operated below 40 mK for the first time, successfully. The magnetic domain structure of $LiHoF_4$ single crystal has also been studied for the first time at these ultra low temperatures. The microscope is also a good platform for standalone STM, AFM/MFM, or any other scanning probe microscopy applications at this ultra low temperature.

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