A scanning Hall probe microscope for high resolution magnetic imaging down to 300 mK

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We present the design, construction, and performance of a low-temperature scanning Hall probe microscope with submicron lateral resolution and a large scanning range. The detachable microscope head is mounted on the cold flange of a commercial ^{3}He-refrigerator (Oxford Instruments, Heliox VT-50) and operates between room temperature and 300 mK. It is fitted with a three-axis slip-stick nanopositioner that enables precise *in situ* adjustment of the probe location within a $6 \times 6 \times 7$ mm^3 space. The local magnetic induction at the sample surface is mapped with an easily changeable microfabricated Hall probe [typically GsAs/AlGaAs or AlGaAs/InGaAs/GaAs Hall sensors with integrated scanning tunnel microscopy (STM) tunneling tips] and can achieve minimum detectable fields $\geqslant 10$ mG/Hz$^{1/2}$. The Hall probe is brought into very close proximity to the sample surface by sensing and controlling tunnel currents at the integrated STM tip. The instrument is capable of simultaneous tunneling and Hall signal acquisition in surface-tracking mode. We illustrate the potential of the system with images of superconducting vortices at the surface of a Nb thin film down to 372 mK, and also of labyrinth magnetic-domain patterns of an yttrium iron garnet film captured at room temperature. © *2008 American Institute of Physics.*
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I. INTRODUCTION

Some of the most interesting contemporary problems in condensed matter physics are in the area of nanoscale quantum mechanics and relate to the properties of superconducting (SC) and ferromagnetic materials at ultralow temperatures, e.g., in the study of macroscopic quantum tunneling and novel forms of quantum order. A key aspect of this work is the ability to perform high spatial resolution magnetic imaging down to ultralow temperatures and/or at high magnetic fields. Due to the major technical challenges posed in these regimes, almost no magnetic imaging system currently exists which is capable of nanoscale imaging. Over the past 10–15 years we have developed a number of scanning Hall probe microscopes (SHPMs),[1] which span the temperature range 4.2–300 K. These Hall probe systems represent a very flexible approach to magnetic imaging since Hall sensors can readily be fabricated with $\sim$100 nm spatial resolution and excellent minimum detectable fields and using high probe current densities, can be operated in magnetic fields of several teslas. All our existing SHPM systems use ^{4}He exchange gas cooling and hence, can only be operated above 4.2 K. In order to address a range of exciting contemporary problems in magnetic materials at much lower temperatures, we introduce the design of a new SHPM head that can be directly attached to the 300 mK cold flange of a commercial ^{3}He-refrigerator insert. This instrument is capable of operation in magnetic fields up to 10 T with submicron spatial resolution ($\geqslant$100 nm) and minimum detectable field $\geqslant$10 mG/Hz$^{0.5}$.

II. INSTRUMENT DESIGN

A. Cryostat

The SHPM head is housed in the inner vacuum chamber (IVC) of an Oxford Instruments Heliox VT-50 ^{3}He-refrigerator, being firmly bolted connected to its cold flange at the bottom of the ^{3}He-pot [Fig. 1(a)]. The Heliox system is designed to fit into an Oxford Instruments variable temperature insert (VTI), which forms part of a SC magnet system along with an Oxford Instruments helium cryostat. The Heliox VT-50 refrigerator is capable of reaching a 280 mK base temperature and maintaining 350 mK under 50 μW heat load for more than 8 h. The temperature of the VTI can be regulated by a needle valve which controls the flow from the helium reservoir, while the Heliox possesses its own temperature controller (ITC503). The main helium Dewar rests on pneumatic antivibration mounts to eliminate ground-born vibrations. Its large mass (more then 200 kg including the 11 T SC solenoid) contributes to the suppression of both mechanical vibrations and acoustic noise. The only pumping line, which is necessary for pumping the VTI, is flexible and passed through a massive concrete block for the effective reduction of vibrations from a rotary pump.

B. Microscope head

The requirement that the microscope head should fit into the limited sample space of the IVC of the Heliox VT-50 ^{3}He-refrigerator poses the first major challenge. This comprises a cylindrical space 36 mm in diameter and 135 mm high. Fortunately this is sufficient to use a stack of three

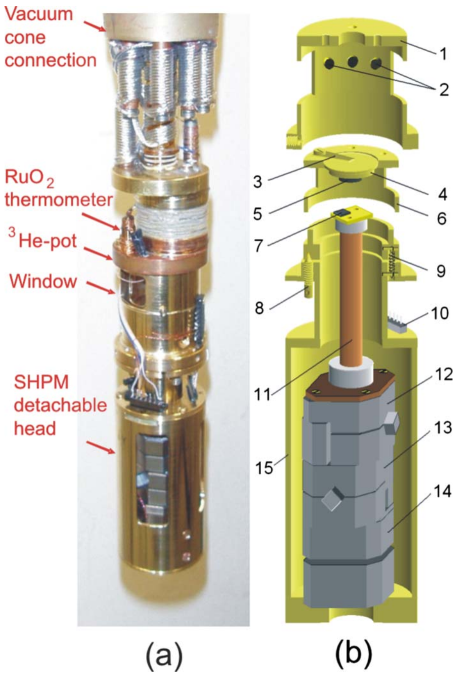

FIG. 1. (Color online) (a) A photograph of the low-temperature stage of the SHPM. (b) A sectional drawing of the SHPM detachable head: (1) mounting tube, (2) LED array, (3) flat bronze spring, (4) sample-holder disk, (5) sample, (6) sample-holder cup, (7) Hall probe, (8) alignment screw, (9) extension spring, (10) electrical connector, (11) piezoscanner tube, [(12) and (13)] ANP*x*100LT positioners, (14) ANP*z*100LT positioner, and (15) brass microscope body.

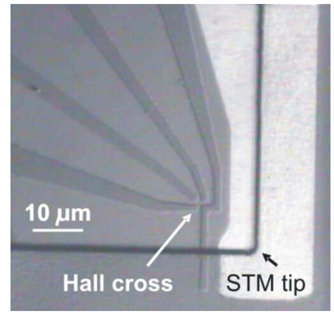

FIG. 2. (Color online) A submicron Hall probe with an integrated STM tip (see labels) that has been patterned by electron beam lithography.

commercially available slip-stick nanopositioners from Attocube Systems AG (Ref. 2) for coarse three-axis positioning along with a 2 in. piezoscanner tube as is shown at Fig. 1(b). These positioners (two ANP*x*100LT for horizontal movement and one ANP*z*100LT for vertical motion) are guaranteed to produce controlled and reasonably reproducible steps from 10 to 500 nm at helium temperatures in a vacuum environment. The given geometry makes it possible to exploit the entire translation limit ($6\times6\times7$ mm^3) of the positioners for initial adjustments of the Hall probe location to accommodate a wide variety of sample shapes and sizes. The scan range of the piezoscanner can reach $170\times170\times4.7$ μm^3 at room temperature when the full swing (±400 V) of drive voltage is applied. It decreases with temperature, but nevertheless exceeds $22\times22\times0.6$ μm^3 at 4.2 K. The Hall probe [typically a GaAs/AlGaAs or AlGaAs/InGaAs/GaAs two-dimensional electron gas (2DEG) sensor][1,3] is bonded onto a thin dielectric substrate and glued to a miniature printed circuit board with pads for Au wire bonding and two screw holes for rapid mounting. This assembly is screwed directly onto the end of the piezoscanner, which carries an array of gold-plated spring contacts to provide reliable electrical connections to the miniboard pads.

Only nonmagnetic materials were used in the construction so that the microscope is capable of operation in high magnetic fields (≤10 T). Special care was taken to achieve a highly axially symmetrical design of the head in order to prevent temperature-induced bending of the microscope, which can potentially cause unacceptably large lateral shifts of the Hall probe while cooling, leading to its damage. The microscope body is composed of a brass shell, with all external surfaces highly polished to minimize radiation into the head. It comprises the stack of positioners with the attached tube scanner, electrical connectors and an easily detachable sample-holder cup. The latter carries a 12 mm brass sample-holder disk clamped with a flat bronze spring and has side windows for visual checking and adjustment of the tilt angle between the sample surface and the Hall probe chip. This angle usually does not exceed $0.5°-1.0°$ and can be aligned by means of three fine-thread screws [Fig. 1(b)], while viewing with a binocular microscope. The correct adjustment of this angle is crucial in order to ensure that the integrated tunneling tip is the closest point to the sample surface while keeping the sample-Hall sensor spacing as low as possible. Samples are glued to the sample-holder disks with a thin ceramic or sapphire washer and can be replaced by simply sliding them through the side window without disconnecting the head from the refrigerator. The inverted head geometry largely eliminates contamination of the sample surface by dust particles during mounting.

C. Hall probe

An optical micrograph of a typical Hall probe is shown in Fig. 2. The Hall sensor used in most of the studies described here was fabricated from a high mobility GaAs/AlGaAs heterostructure 2DEG [$n_{2d}(4.2\text{ K})\sim3.3\times10^{11}$ cm^{-2}, $\mu(4.2\text{ K})>10^6$ cm^2/V s]. Au/Ge Ohmic contacts were first deposited by optical lithography, thermal evaporation and lift-off. These were alloyed at 425 °C for 15 s to realize low resistance contacts. Coarse Hall probe features were then patterned using optical lithography and wet etching in $H_2SO_4:H_2O_2(30\%):H_2O$ 1:8:80. Optical lithography and lift-off was then used to deposit a thin Ti/Au (10nm/50nm) scanning tunnel microscopy (STM) tip metallization on the corner of a deep mesa etch. Finally the Hall probe was patterned <10 μm from the STM tip using polymethyl methacrylate and electron beam lithography followed by wet chemical etching in $H_2SO_4:H_2O_2(30\%):H_2O$ 1:8:1000. The geometrical width of the wires comprising the Hall "cross" was 1 μm, yielding an electronic width of <800 nm due to edge wall depletion.

D. Thermal anchoring, wiring, and thermometry

The only part of the head which is permanently mechanically coupled to the cold flange of the ^{3}He-pot is the mounting tube. It plays the role of thermally anchoring the microscope body to the refrigerator. This tube also has side windows which match the ones on the sample-holder cup, and it carries a light emitting diode (LED)-array placed on its wall [see Fig. 1(b)], which allows one to briefly illuminate the Hall probe *in situ*. Exposing the probe to optical photons at low temperatures persistently excites trapped carriers and usually leads to improvements in the probe sensitivity and a reduction in the noise level. The LEDs can also be used as an additional heater while evacuating exchange ^{4}He gas from the IVC and for heating the sample in vacuum.

The SHPM scanner, coarse positioners, and sensors require many cables to be connected to room temperature control electronics. Radio-frequency (rf) pickup and heat conduction of the cables can significantly contribute to the heat load of the refrigerator and limit the base temperature. We used small-diameter stainless-steel coaxial cables (UT 20-SS-SS from Microcoax) for the sensors and the scanner, and a combination of Cu wire joined to Nb–Ti SC wires for the positioners. In order to reduce heat sources we firmly anchored all the electrical leads first at the upper flange of the IVC and then at the 1 K plate of the Heliox VT-50 insert. In addition, winding cables onto the thermal anchor posts forms highly inductive paths which partially suppress parasitic rf signals.

We embedded a Lake Shore Cryogenics, Inc. Cernox CX-1010-SD sensor into the copper sample-holder disk in addition to the main RuO_2 thermometer, which is placed on the ^{3}He-pot. The sample holder can be attached directly to the ^{3}He-pot by means of a flexible copper braid to ensure excellent thermal anchoring. The Cernox sensor is separated from the sample by only a 0.3 mm thick sapphire substrate and hence accurately monitors its actual temperature. Low-temperature runs of the microscope with this sample holder have demonstrated the establishment of almost perfect temperature equilibrium between the thermometers with a time constant of about 1 s.

E. Electronics

The NanoMagnetics Instruments Ltd. system that we use for controlling the microscope provides scanning, automatic surface approach, and data acquisition. It also features user-friendly software for image processing. We constructed several modules to meet the demands of our hardware developments. First a multichannel controller for the Attocube positioners was created. It is triggered by the same high-voltage (HV) pulses the scanning probe microscope (SPM)-controller utilizes for the slider motor of the commercial Nanomagnetics SHPM and converts them into the standard Attocube positioner waveforms with simultaneous adjustment of the step size in accordance with software set values. The custom-designed HV piezoscanner controller with output swing ± 400 V for each channel was built around Apex PA94 low-noise HV operational amplifiers in order to increase the scanning range by two to three times. For the tunnel current and the Hall probe voltage measurements we designed a current-to-voltage converter (10^8 V/A) and a low-noise differential preamplifier with gain $G=1000$ respectively, operating at room temperature. They are incorporated in a screened case, which is mounted at the top of the Heliox VT-50 insert, minimizing the input cable capacitance and preventing unwanted pickup along the long (1.5 m) cables to the main controller rack.

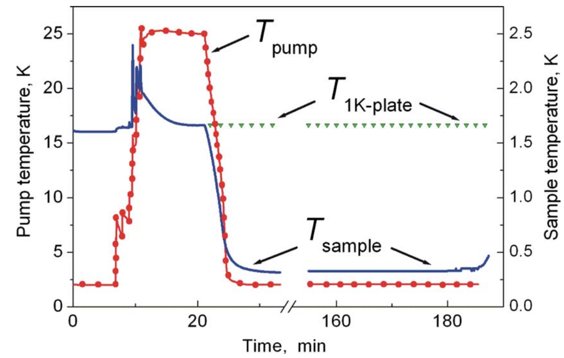

FIG. 3. (Color online) A typical thermal cycle of full microscope operation during which the base temperature $T=320$ mK was achieved (the sorption pump temperature—line+dots, the sample temperature—line, and the 1 K plate temperature—triangles).

III. OPERATION AND PERFORMANCE

Although the 300–400 mK base temperature hold time with the SHPM head attached can be as long as 24 h, the operation of the microscope in scanning mode with all the electrical leads connected reduces this time to about 3 h. After that a new ^{3}He-condensation cycle is needed. It takes about 20 min to restore the base temperature again. A typical time diagram for the condensation cycle and stabilization of the 320 mK sample temperature for more than 2 h is shown at Fig. 3. During almost the whole cycle of operation the temperature of the 1 K plate, which is used for ^{3}He condensation, remains at a constant value of 1.55 K which is governed by the pumping rate of the VTI.

For calibration of the scanner and investigation of its stability as well as sensitivity to vibrations we operated the microscope in topographic STM mode with the integrated Hall sensor replaced by a high quality STM tip prepared from a piece of $Pt_{80}Ir_{20}$ wire, which allows one to obtain much higher quality STM images and hence get a more reliable impression of the true performance and mechanical stability of the whole system. Two STM images of a 1 μm period Pt-coated calibration grid from Veeco are shown in

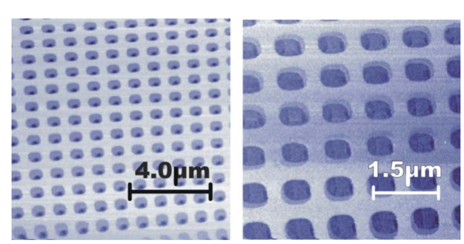

FIG. 4. (Color online) Two topographic STM images of a 1 μm period platinum-coated square calibration grid at 300 K ($I_t=0.2$ nA).

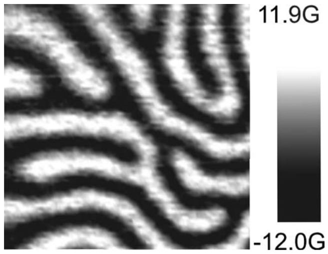

FIG. 5. Unfiltered $170\times 170\ \mu\text{m}^2$ room temperature image of labyrinth magnetic-domain patterns in a YIG film captured at a scan rate of $300\ \mu\text{m/s}$.

Fig. 4. Further observations of the tunnel current behavior revealed that the boiling of cryogenic liquids inside the cryostat did not have a noticeable influence on the STM tracking and imaging modes. The latter test demonstrated the high rigidity of the microscope frame and the stability of the system under working conditions.

An example of instrument operation in SHPM mode at room temperature is given in Fig. 5, where a submicron Hall probe with integrated STM tip, similar in design to that shown in Fig. 2, was employed to image the magnetic structure at the surface of an yttrium iron garnet (YIG) film. Prior to the experiment a thin layer of gold (50 nm) was deposited on the insulating YIG surface in order to enable tunnel tracking. Scanning at speeds as fast as $300\ \mu\text{m/s}$ was possible with the integrated STM tip lifted just out of tunnel contact. During these scans the full HV swing was applied to the piezoscanner to achieve the maximum scan range of $170\times 170\ \mu\text{m}^2$. The unfiltered image of labyrinth magnetic domains in the YIG film captured at high speed is presented in this figure.

Low-temperature SHPM mode testing was carried out on a 700 nm thick sputtered SC Nb film. We have imaged SC vortices at various temperatures down to 372 mK [see Figs. 6(a) and 6(b)]. Our experimental data allowed us to

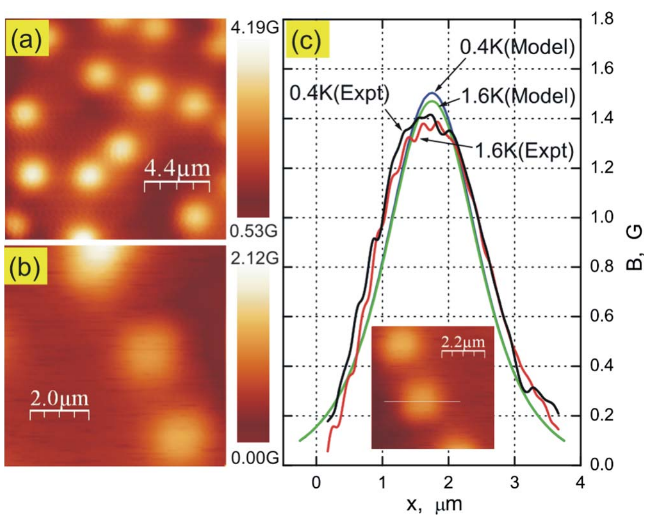

FIG. 6. (Color online) SHPM images of SC vortices in a 700 nm thick Nb film at various temperatures: (a) $T=1.575$ K; (b) $T=372$ mK; (c) line scans across the same single vortex (inset) at $T=400$ mK and $T=1.6$ K along with model results for the vortex profile at these two temperatures (see text).

investigate the temperature dependence of the profile across a single vortex, and compare this with the results of a model.

The theoretical modeling of the emergent field of vortex structures was performed within the London approximation, applicable for extreme type-II superconductors. For an isotropic superconductor, this problem was first solved by Pearl,[4] for the case of extremely thin films. In a more general case, the field outside the SC film can be found as the free space solution of the London equation matching the appropriate boundary conditions at the SC surface (i.e., at $z=\pm d/2$, where d is the thickness of the film).[5] Let $r=\sqrt{x^2+y^2}$ be the *radial* distance measured from the center of the vortex, while a denotes the vertical distance from the top surface of the SC film ($a=z-d/2$). The radial and transverse components of the magnetic field created by a single vortex above the SC film are as follows:

$$h_{vr}(r,a)=\frac{\phi_0}{4\pi\lambda^2}\int_0^\infty \frac{qdq}{Q}J_1\left(\frac{qr}{\lambda}\right)e^{-(qa/\lambda)-(1/2)(k^2/\kappa^2)},$$

$$h_{vz}(r,a)=\frac{\phi_0}{4\pi\lambda^2}\int_0^\infty \frac{qdq}{Q}J_0\left(\frac{qr}{\lambda}\right)e^{-(qa/\lambda)-(1/2)(k^2/\kappa^2)}. \qquad (1)$$

Here, $\phi_0=h/2e$ is the magnetic flux quantum, and λ and ξ are the penetration depth and the coherence length of the superconductor, respectively, and their ratio $\kappa=\lambda/\xi$ is usually known as the Ginzburg–Landau parameter. In Eq. (1), $k=\sqrt{1+q^2}$, $Q=k[k+q\coth(kd/2\lambda)]$, and $J_\nu(x)$ denotes a Bessel function of order ν. The term $e^{-(1/2)(k^2/\kappa^2)}$ is a cutoff introduced in order to account for the effects of the finite vortex core size, which are not included in the London theory.[6]

The best fit with experimental data has been obtained assuming the temperature dependence of the penetration depth scales as $\lambda(T)=\lambda(0)/[1-(T/T_c)^4]^{1/2}$ with $\lambda(0)=80$ nm, $\xi(0)=40$ nm, and $T_c=9.2$ K. In simulations we use the known thickness of the Nb film, and take the scanning height, a, as a free parameter. For all three vortices shown in Fig. 6(b), we were able to obtain excellent fit, with $a=0.7$, 0.85, and 0.9 μm, for the bottom to top vortex, respectively. This fitting procedure also accounted for the finite size of the Hall probe, and the vortex field at each point was calculated as a spatial average of the z-component of the field [Eq. (1)] over the active area of the Hall cross ($\sim 0.8\times 0.8\ \mu\text{m}^2$). The resulting fit is shown in Fig. 6(c), as a horizontal line scan across the central vortex [see inset image in Fig. 6(c)].

This procedure was repeated at two temperatures, namely, $T=400$ mK and 1.6 K. As shown in Fig. 6(c), the amplitude of the vortex field only increases by 33 mG as the temperature is lowered to 400 mK. Nevertheless, even such a small field increase remains detectable by our experimental setup.

IV. CONCLUSION

We have described the design, construction and performance of a novel SHPM for milliKelvin magnetic imaging. It has been demonstrated to be an effective tool for the direct, quantitative and noninvasive measurements of local magnetic fields at submicron length scales. This instrument can be considered, moreover, as a versatile low-temperature platform for a much wider range of experiments. Its modular design enables easy and low-cost minor modifications, which would convert the instrument into a low-temperature AFM, MFM, or a purpose-built STM. Special measures were taken to provide good access to the sensors and the sample holder for their safe and rapid replacement. The mechanically robust and rigid body of the detachable low-temperature microscope head makes it possible to exploit it over a wide range of temperatures, even in stand-alone room temperature applications.

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