MAGNETIC SENSITIVE SCANNING PROBE MICROSCOPY

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INTRODUCTION

Magnetic imaging has become increasingly more important and challenging for scientist and engineers, mainly driven by the advances in nanotechnology, and the phenomenal capacity increases in the magnetic data storage industry. This increase is accomplished by giant magnetoresistance (GMR) and tunneling magnetoresistance (TMR) effects. Traditional magnetic imaging methods like magneto optic Kerr effect (MOKE), Bitter decoration (Bezryadin and Pannetier, 1996), and scanning electron microscope with polarization analysis (SEMPA) (Cameron and Judy, 1988) can achieve 1 μm, 30 nm, and 10 nm resolution, respectively as shown in Figure 1. However, these methods would not be very practical for routine magnetic analysis because they do not have the resolution for MOKE, damage the

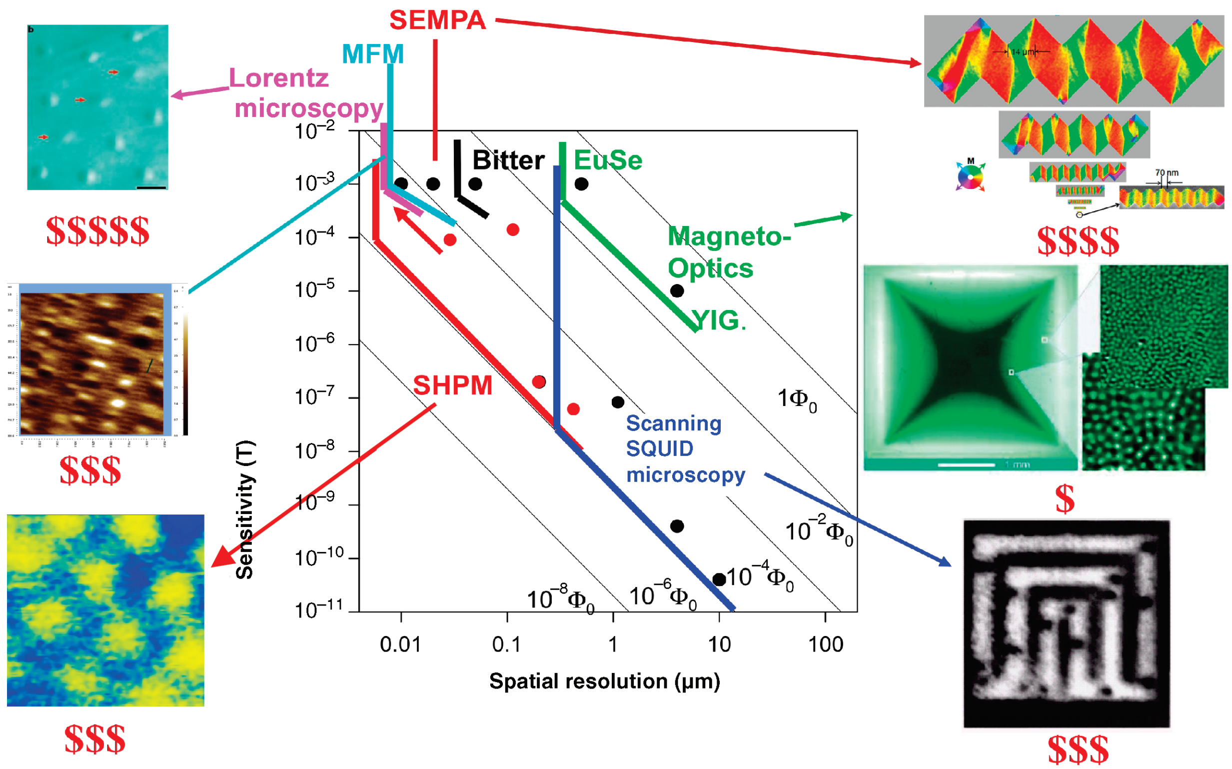

Figure 1. Comparison of magnetic imaging methods: magnetic resolution versus spatial resolution.

specimen for the Bitter decoration method, and are very complicated and expensive for SEMPA. Invention of magnetic force microscope (MFM), first demonstrated by Martin and Wickramasinghe (1987), was a natural development after the invention of atomic force microscope (AFM) in 1986 (Binnig et al., 1986). In MFM, a magnetically coated AFM tip is used to sense the weak magnetic forces between the specimens and tip to visualize the magnetic domains. MFM could achieve 100 nm resolution in the early days. The method has been improved over the years; magnetic materials can now be imaged down to 10 nm resolution with MFM (Karci et al., 2011). Since a magnetic tip is used to image the specimen, the field emanating from the tip can change the magnetization state of the region of interest for a magnetically soft sample. The tips' magnetization state may also be altered by the very specimen it is trying to image. Furthermore, application of external magnetic field may also alter or switch the magnetization state of the tip, resulting in a completely different contrast in variable field experiments. Quantification of the MFM images obtained by MFM is usually not straightforward and may require lengthy procedures. Furthermore, these procedures may not be conclusive as the tip magnetization state usually changes during the experiment.

Alternative methods have also been developed to overcome the shortcomings of the MFM. Scanning Hall probe microscopy (SHPM) (Chang et. al., 1992) is a quantitative and noninvasive technique for magnetic imaging, which uses a nano-Hall sensor to form the magnetic image with high spatial and magnetic field resolution of ~50 nm and 3×10^{-8} T/$\sqrt{\text{Hz}}$ (Oral et al. 1996, 2002; Oral, 2007), over a wide range of temperatures, 30 mK–300 K. The scanning SQUID microscopy (SSM) is similar to the SHPM, where the Hall sensor is replaced by a low T_c or high T_c SQUID (Kirtley et al., 1995). The SSMs can operate down to 200 nm spatial and 10^{-7}T/$\sqrt{\text{Hz}}$ magnetic field resolution (Finkler et al., 2012), but they have to be operated at low temperatures to achieve this.

Magnetic resonance force microscopy (MRFM) is proposed by Sidles (1991) to improve the spatial and spin resolution of magnetic resonance imaging (MRI) microscopy and implemented first by Züger and Rugar (1993), quickly followed by a number of other groups. Even though the MRFM measures the magnetic forces between the interacting (electron and proton) spins at the tip and the specimen, it is actually used to image the spin density of samples, potentially leading to 3D imaging of atoms if single proton resolution is eventually achieved. MRFMs can now achieve single electron spin resolution at 25 nm length scale (Rugar et al., 2004) and $\sim 4\times10^4$ proton spins at <10 nm resolution (Degen et al., 2009) at ultra-low temperatures (300 mK) and high magnetic fields. A detailed and useful review of the MRFM method has recently been compiled by Poggio and Degen (2010).

Spin polarized scanning tunneling microscopy (SP-STM, see also article MAGNETIC SENSITIVE SCANNING TUNNELING MICROSCOPY) (Wiesendanger et al., 1990) can achieve atomic resolution using the spin dependence of the tunnel current. However, the surfaces have to be extremely clean, and the microscope has to be operated in ultrahigh vacuum (UHV) with special and complicated *in situ* tip preparation, in addition to the sample preparation.

Magnetic exchange force microscopy (MExFM) was first proposed by Wiesendanger in an SP-STM paper (Wiesendanger et al., 1990) but took quite a few years to demonstrate because of instrumental limitations. In MExFM minute, magnetic forces due to exchange interactions are measured between the atoms. Since the length scale of the exchange interaction is very small, one has to get very close to the surface. Advances in the noncontact atomic force microscopy have improved the force resolutions of the AFMs dramatically, which in turn made MExFM operation possible (Kaiser et al., 2007). One can now achieve atomic resolution with MExFM, but these microscopes have to be operated in UHV, at low temperatures (~4 K) and at high external magnetic fields, ~7 T.

Even though magnetically sensitive scanning probe microscopes, MFM, SHPM, SSM, MRFM, and MxFM, can solve many interesting academic and industrial problems, scientists in academia and engineers in the magnetic storage industry need better tools with higher spatial and magnetic resolution, perhaps down to a few nm and down to single spin to study magnetism at the nanoscale.

PRINCIPLES OF THE METHOD

Almost all of the magnetic sensitive force microscopes have the same ingredients, typical for scanning probe microscopes (SPM): a sensitive probe to measure magnetic interaction at nanoscale, a feedback mechanism to keep the probe at desired height, above the surface, a scanning mechanism in XYZ coordinates, and finally a control electronics and software to acquire the data and display the information.

The first MFM utilized an etched and bent nickel tip from a wire, whose magnetization state was modulated using a small coil wrapped around to its base. An optical interferometer operating in free space was employed to measure the cantilever deflection in the first MFM. As the AFM technology was developed, the optical beam deflection method (Meyer and Amer, 1988) has become the mainstream deflection measurement method and microfabricated silicon and silicon nitride cantilevers replaced the handmade ones and became the off-the-shelf force sensors. MFMs also benefited from this mass production and magnetically (Co, Ni, Fe, NiFe, or CoPt) coated MFM cantilevers became the main workhorse. Since the magnetic forces are small, MFMs usually use soft cantilevers with ~3 N/m stiffness to increase the force resolution of the microscope. The typical MFM force sensor uses soft tapping mode, 3 N/m and ~70 kHz, cantilevers with hard magnetic (high coercivity) or soft magnetic (low coercivity) coating. Remnant magnetic moment and coercivity of these coatings are typically 150 emu/cm^3 and 250 Oe for hard magnetic tips and 225 emu/cm^3 and 0.75 Oe for soft magnetic tips. Some researchers are also using stiffer cantilevers up to 20–40 N/m, mainly for noncontact mode MFM to avoid snap to contact to the specimen. Even though optical beam deflection method is very easy to operate in ambient conditions, it is not very suitable for low temperature or UHV operation. Fiber interferometers, piezoresistive, and piezoelectric quartz crystal tuning fork force sensors are used for MFMs, mainly at low temperatures and UHV conditions, where the space is limited or the alignment procedure is difficult.

Cryogenic MFMs were developed by a number of research groups, Roseman and Grutter (2000), Hug et al. (1993), and Karci et al. (2011), to study and image magnetic materials and superconductors as a function of temperature and magnetic field. Abrikosov vortices in superconductors were imaged by MFMs using fiber optic interferometers (Hug et al., 1993) and piezoresistive cantilevers (Volodin et al., 2000). Fiber interferometers can achieve extremely low noise levels compared to piezoresistive and piezoelectric displacement detection and can be used with wide range of cantilevers. However, the design of cryogenic fiber optic interferometer-based MFM is quite challenging since the space is limited and optical re-alignment is usually necessary due to thermal contractions from 300 K to 0.3 K.

In the MFM, the cantilever senses the total forces acting in the z-direction. The magnetic forces are typically much lower than the chemical and the van der Walls forces, which are responsible for image formation in AFMs. Therefore, separation of magnetic forces is necessary from the other forces to obtain a magnetic image. Most commonly used method is the lift-mode MFM, which was patented by Digital Instruments (now Bruker). In the lift-mode MFM, topography of the specimen is obtained in the forward scan line. On the backward scan line, the feedback loop is suspended, and the cantilever is lifted from the surface by a predetermined amount, typically 20–100 nm as shown in Figure 2. As the tip is moved along the backward scan line, it is moved up and down to follow the topography of the specimen and sustain the same lift-off height. This is achieved by the MFM controller, using the measured topography from the forward scan line. In the lift mode, the long-range magnetic forces will modulate the cantilever, and the magnetic image will be collected either from the DC

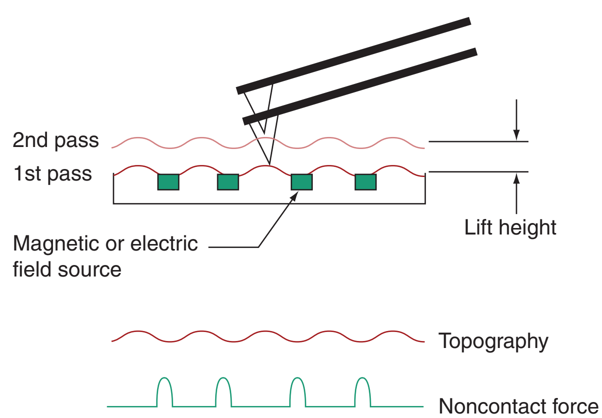

Figure 2. Lift-mode operation of MFM (Digital Instruments).

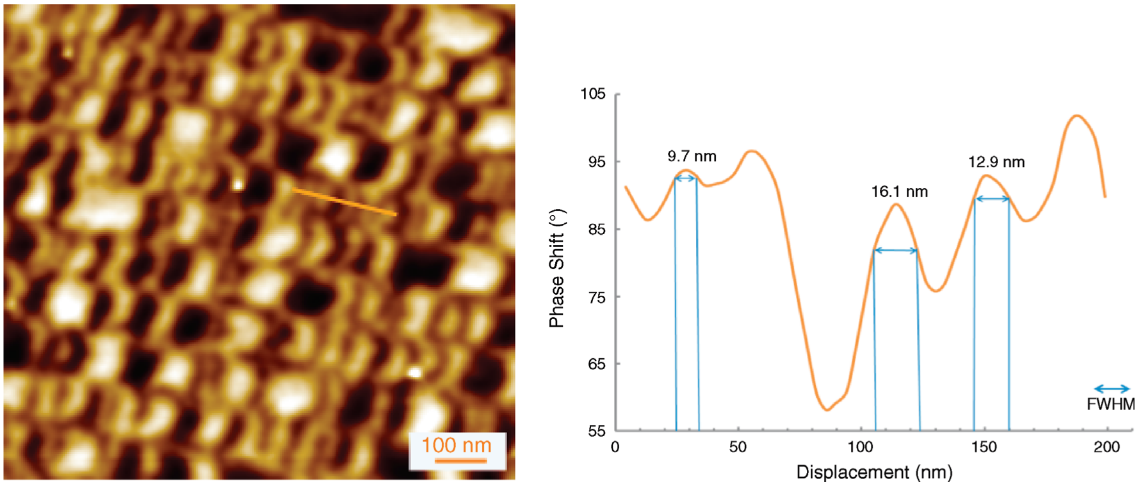

Figure 3. Low temperature-MFM image of Seagate 394 Gbpsi hard disk at 77 K, $k = 8$ N/m (Karci et al., 2011).

force, the phase of cantilever oscillation in tapping mode or frequency shift (Δf) in noncontact mode AFM. The feedback is turned on at the end of the scan line, and the lift off is reduced to zero. The scan continues like this to form the complete magnetic and topographic images simultaneously. Figure 3 shows a typical MFM image of hard-disk specimen (Karci et al., 2011). The magnetic resolution in MFM mainly depends on the shape and magnetic moment of the MFM tip as well as the lift-off height. One can try to improve the resolution by reducing the lift-off height, but the topography will start to appear in the magnetic image through the van der Waals and chemical forces. In the other extreme cases, topography will be affected by the magnetic force between the specimens with extremely high magnetic moments and the MFM cantilever, for example, NdFeB, SmCo permanent magnets. The magnetic image will start to appear in topography in these specimens, and operating the MFM will be very difficult.

In the SHPM, a nano-Hall sensor as shown in Figure 4 is replaced with the MFM cantilever. The Hall sensor is fabricated near a mesa corner, which is coated with a thin layer of gold to act as an STM tip as shown in Figure 5. STM feedback is usually used to track the surface in SHPM. Compared to the MFM topography is completely separated from the magnetic signal and simultaneous magnetic image, albeit shifted a few micrometers as shown in Figure 6. The main advantage of SHPM is its ability to give quantitative map of vertical component of magnetic field without disturbing the specimen. It is especially useful for soft magnetic materials or Abrikosov and Josephson vortices, which can be easily magnetized or moved by the tip of the MFM cantilever. Furthermore, the magnetic signal is linear from milligauss to tens of kilogauss and does not saturate or switch its state compared to MFM cantilever. The state-of-the-art SHPMs have extremely low noise floors, sufficient to image Abrikosov vortices in room-temperature superconductors if someone discovers them in the future.

The magnetic field **B(x,y,z)** is a vector. Almost all of the magnetic sensitive scanning probe microscopes are either sensitive to one component of this vector or the signal is a complex and usually unknown interaction of this field, **B(x,y,z)** with the tip or sensor. Furthermore, this interaction may change during the imaging due to tip switching and wear in the case of MFM. Several attempts can be found in the literature (Fedor et al., 2003) to measure all the components of the magnetic field vector **B(x,y,z)**, but most of them suffer from lack of resolution, 30–40 μm. Recently, Dede (2009) have demonstrated an innovative 3D-SHPM, where three components of the **B(x,y,z)** can be measured simultaneously on a magnetic surface at 700 nm

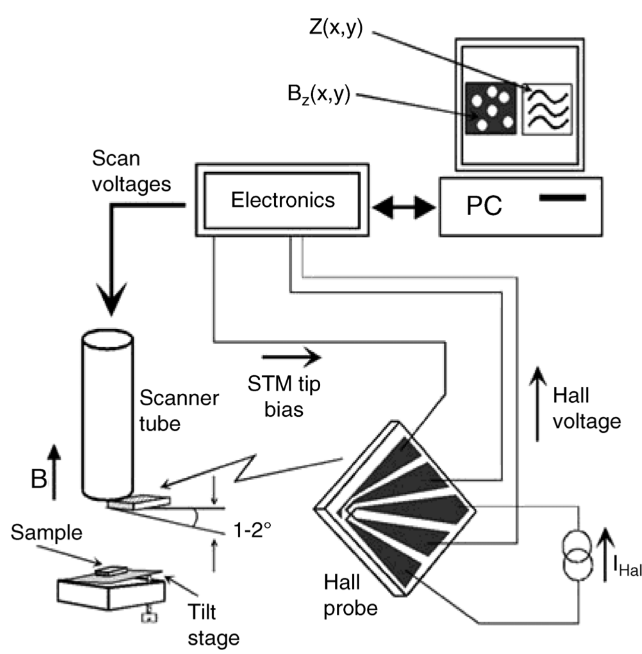

Figure 4. Principle of scanning Hall probe microscope (SHPM).

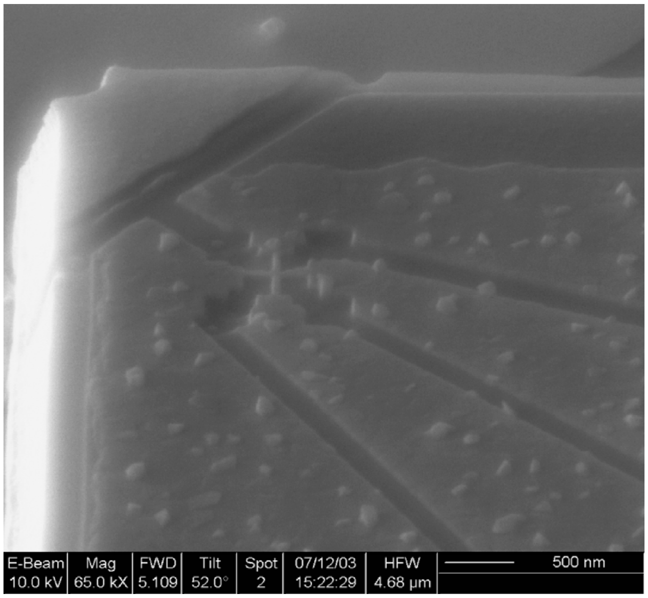

Figure 5. Bismuth thin film nano-Hall sensor integrated with an STM tip.

resolution, using a single Hall sensor. Figure 7 shows simultaneous measurement of B_x, B_y, and B_z images on the surface of a magnetic hard disk specimen. The resolution can be improved further as it is limited by the size of the Hall sensor used in the experiment, 700 nm.

One of the most interesting features of the SHPM is its ability to perform local magnetization measurements. Figure 8 shows *B–H* curves measured on 2 μm diameter iron disks at different locations on the sample. This method is actually better than the SQUID magnetometers as the filling factor is almost one.

In scanning SQUID microscopes (SSM), a micro or nano-sized SQUID is scanned across the specimen to image the local magnetic flux density (Kirtley et al., 1995). In the original microscopes, usually step or DC motor-driven mechanical stages were used for scanners as shown in Figure 9. In the recent systems, S-bender-type large area piezo scanners are utilized for ease of use. The separation between the SQUID pickup loop and the size of the loop determines the spatial resolution of the SSM, similar to SHPMs. DC, RF, high T_c, and low T_c SQUIDs were used by different research groups.

MRFM usually operate at low temperatures as low as 100 mK and high vacuum. These microscopes use extremely soft, ~1 μN/m and ~5 kHz, AFM cantilevers

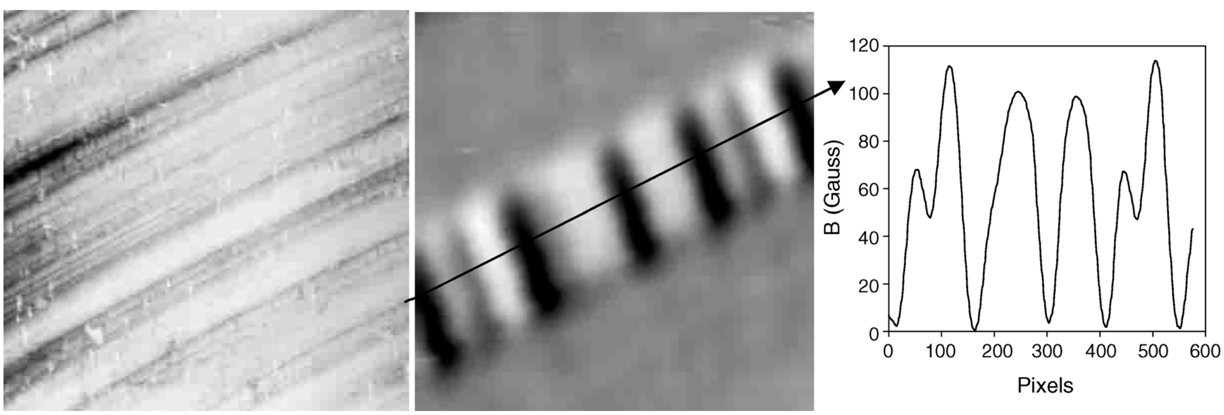

Figure 6. Topography, B_z image, and cross section of magnetic hard disk obtained by an SHPM image (Howells et al., 1999).

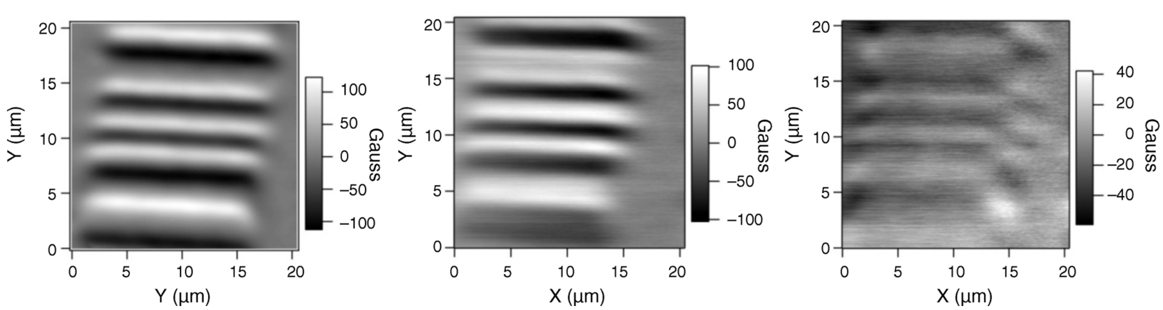

Figure 7. B_x, B_y, and B_z images of a hard-disk sample surface obtained with 3D-SHPM (Dede, 2009).

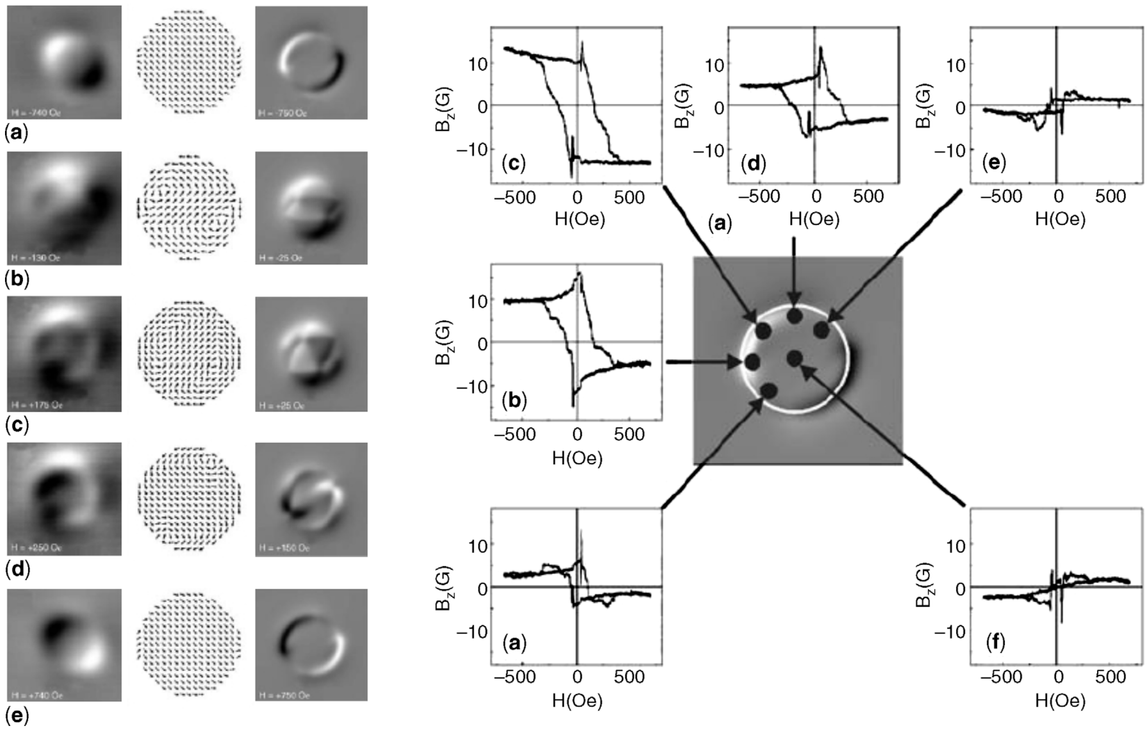

Figure 8. Local magnetization measurements performed on a 2 μm Fe disks using SHPM. Reprinted from (Neal et al., 2006), Copyright 2006, with permission from Elsevier.

to detect nuclear or electron spins in specimen as shown in Figure 10. A small strong permanent magnet (SmCo) is glued at the end of the cantilever and trimmed using the FIB method. Magnetic resonance condition is achieved in a parabolic slice as shown in Figure 10. Extremely high-quality factors can be achieved at very low temperatures despite very low resonance frequencies. Laser interferometers operating at nW power levels are usually employed not to warm up the cantilever. Single electron spin MRFM can provide single electron spin resolution at 25 nm length scale (Rugar et al., 2004).

Magnetic exchange force microscopes (MExFM) can be operated in UHV and under high magnetic fields, typically ~7 T to magnetize the tip. The system usually is based on a noncontact AFM in UHV at low temperatures and high fields. Most of the groups

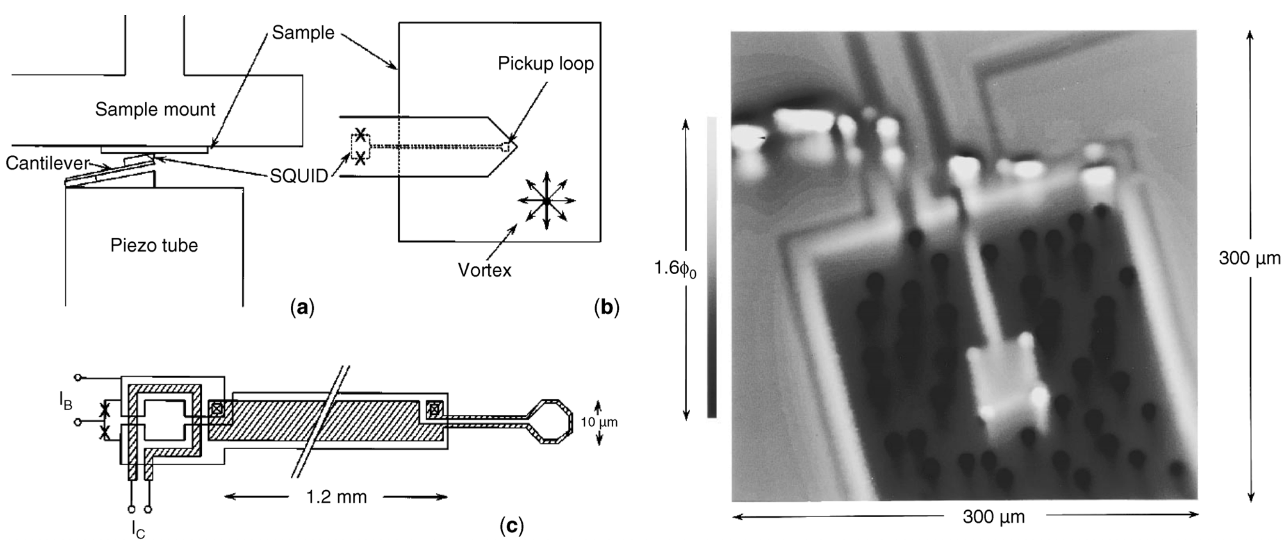

Figure 9. SSM and SSM image of a thin-film high-T_c washer SQUID, with vortices trapped in the bulk of the washer, in a scratch in the SQUID, and at the inside corners of the square SQUID hole. Reprinted with permission from (Kirtley et al., 1995). Copyright 1995, American Institute of Physics.

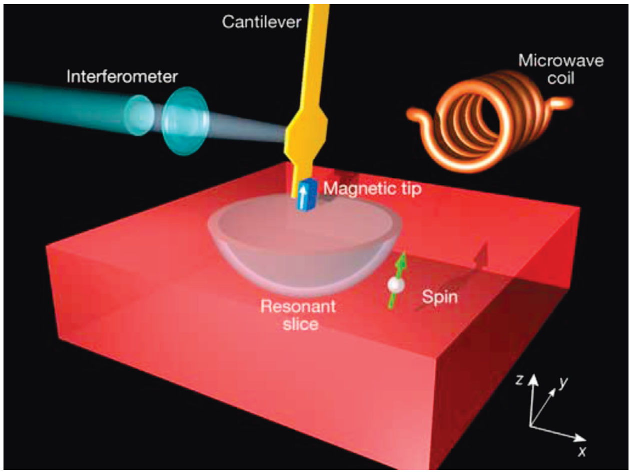

Figure 10. Principle of MRFM.

working on MExFM build their own microscopes because of extremely high sensitivity required to operate the systems. Figure 11 shows the image of antiferromagnetic ordering of atoms in NiO(1 0 0) crystal (Kaiser et al., 2007). The magnetic image contrast is extremely small in the MExFM images, typically 1 pm.

DATA ANALYSIS AND INITIAL INTERPRETATION

Quantification of the SHPM, SSM, and MxFM data is pretty straightforward. Even though the quantification of MFM data can be quite difficult, a number of groups have attempted to solve this problem. Hug et al. (1993) has developed a spectral analysis method to model the MFM tip by scanning the specimen and obtaining a model magnetic structure of the tip, field transfer function, HTF(*k*). They then deconcolve the MFM images to improve the spatial resolution and quantify the field. However, the tip structure does change due to tip–sample interaction during scanning. Garcia et al. (2001) has used micromagnetic modeling to extract the tip's strength and model and then used this to quantify the MFM images.

SAMPLE PREPARATION

MFM, SHPM, and SSM do not require any special specimen preparation for flat samples. STM guided SHPM needs conductive surfaces; therefore, insulating specimens should be coated with a thin, 10–20 nm layer of gold. However, AFM guided has recently been shown for conventional Hall sensors in SHPM (Dede, 2009), making this step unnecessary. Magnetic exchange force microscope requires extensive specimen preparation in UHV, typically cleaving, sputtering, and annealing the samples, depending on the nature of the samples, to obtain atomically clean surfaces.

SPECIMEN MODIFICATION

For most of the magnetically sensitive scanning probe microscopy methods, the specimen is not affected. The exception is MFM, unfortunately the most commonly used magnetic SPM technique. The magnetic field emanating from the MFM tip can be as high as few hundred Gauss, Thiaville et al. (1997). The specimen's magnetic structure may be modified severely if the sample is magnetically soft and hard magnetic tip is used for MFM imaging. Even if the sample's coercivity is comparable to the tips coercive field, there is a danger of modifying the magnetic structure.

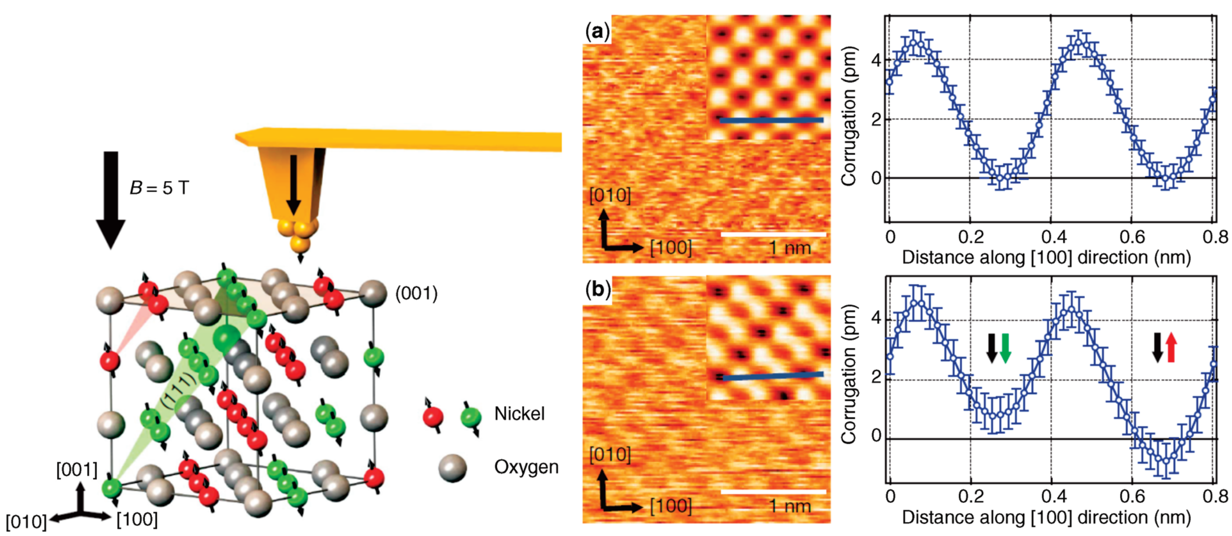

Figure 11. MExFM and MExFM image of NiO(0 0 1) surface after unit cell averaging. Reprinted by permission from Macmillan Publishers Ltd: Nature (Kaiser et al. 2007), copyright 2007.

PROBLEMS

The specimen surface should be fairly flat to obtain good images in almost all magnetically sensitive scanning probe microscope methods. The sample surface should also be grounded as the magnetic tip will also be sensitive to the electrostatic forces due to surface charges in MFM. The major problem for MFM is the calibration and quantification of the magnetic data as the tip's magnetization state is usually unknown, and it usually changes during the experiment. The MFM spatial resolution can be improved further, but this requires extremely low-noise force sensors, which have been shown to operate in the literature for a few noncontact atomic force microscopes. In principle, MFM lateral resolution could be improved to 5 nm range. SHPMs give quantitative data, but the magnetic field is still averaged across the small nano-Hall sensor. Graphene Hall sensors may be promising alternatives to increase the spatial resolution toward 5 nm range. SSM can be operated with high resolution, while the SQUID is at the same temperature with the specimen. This limits the usable range of temperatures. The specimen temperature can also be controlled independently for some SSMs where a sapphire window is used to separate the SQUID from the sample, decimating the spatial and magnetic resolution. MExFM can see magnetic state of the atoms (mainly antiferromagneticaly ordered samples), but it cannot be applied to every sample. Moreover, the system requires UHV and superconducting magnets; hence, lots of care should be given in addition to serious capital expenditure.

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