TUNNELING STABILIZED MAGNETIC FORCE MICROSCOPY:
PROSPECTS FOR LOW TEMPERATURE APPLICATIONS TO SUPERCONDUCTORS†

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Abstract

We have recently demonstrated an imaging technique referred to as tunneling stabilized magnetic force microscopy or TSMFM. TSMFM is performed using a scanning tunneling microscope (STM) with a flexible, magnetic, tunneling tip in place of the usual rigid tunneling tip. TSMFM images are therefore combinations of topography and the magnetic forces between the tip and the sample. Room temperature TSMFM images of magnetic bit tracks on a hard disk have 100 nm resolution and are comparable to Bitter patterns made using a ferrofluid. We have built a low temperature STM with the hope of getting TSMFM images of the flux lattice in superconductors. Preliminary TSMFM images of a $\text{YBa}_2\text{Cu}_3\text{O}_x$ (YBCO) film ($T_c = 88$ K) in a 50 mT field show that relatively large magnetic forces are acting on the flexible tip while scanning at 48 K.

Introduction

Tunneling stabilized magnetic force microscopy¹ (TSMFM) is a recent addition to a growing number of proximal probes² based on scanning tunneling microscopy (STM). In contrast to magnetic force microscopies having atomic force detection schemes,³ TSMFM is a rudimentary variation of STM. The single difference between STM and TSMFM is that the rigid tunneling tip of the former is replaced by a magnetic flexible tunneling tip of the later. This means that TSMFM images are combinations of sample surface topography and surface magnetic forces on the flexible tunneling tip.

TSMFM tip geometry and composition are shown in Fig. 1. Tips consist of a triangular magnetic film a few micrometers thick that is fastened to a support wire with silver paint. Free standing magnetic films can be made by evaporating magnetic materials onto a glass microscope slide. If the films are thick enough they spontaneously separate from the glass slide upon cooling to room temperature. They are then cut with scissors into an isosceles triangle pattern with a base and height of approximately 1 mm. The flexible thin film tips are mounted on the piezoelectric tube scanner of our STM. We estimate from standard beam deflection formulas that the spring constant for the triangles described above is about 0.1 N/m with a corresponding minimum resonant frequency of about 1 MHz.⁴ Choosing the appropriate spring constant and resonant frequency is critical. The compliance has to be high enough that the magnetic force contribution to the TSMFM image is distinguishable from sample surface topography. The resonant frequency has to be high enough so that the apparatus is unaffected by laboratory vibrations and the resonant modes of the tube scanner (greater than 10 KHz).

TSMFM has an advantage over other MFM's in cryogenic applications because of its relative ease of implementation. We have built a STM stage that fits into a variable temperature bathysphere cryostat⁵ for measurements at temperatures from 4 K to 300 K. The bathysphere has the potential of being inserted into the bore of a high field magnet. In this paper, we discuss the construction of our cryogenic STM and its operation at low temperatures. We present results showing images when a Au coated Ni flexible tip is used for TSMFM of the surface of a computer hard disk and TSMFM of the

Figure 1. Flexible magnetic film tunneling tip for tunneling stabilized magnetic force microscopy (TSMFM).

surface of a $\text{YBa}_2\text{Cu}_3\text{O}_x$ (YBCO) thin film at low temperatures in a magnetic field.

Apparatus

A cross sectional view of our cryogenic STM is shown in Fig. 2. The essential feature is an inverted stainless steel double-wall Dewar that insulates a vapor space from a surrounding cryogenic fluid. The STM head, thermometers, and heater are located in the vapor space as shown in the figure. The STM tube scanner is surrounded by a copper can to facilitate thermal stability. A fiberglass-epoxy end plug fits snugly into the bottom of the Dewar. The end plug prevents convection and vapor oscillation that may cause thermal instabilities in the vapor space, yet allows exchange of gas between the vapor space and the surrounding cryogen (liquid helium or liquid nitrogen).

The tunneling current is measured using a remote preamplifier mounted at the top of the cryostat at room temperature. A coaxial cable with a driven shield helps to isolate the tunneling current lead from capacitive pick-up. The driven shield improves STM image quality compared to images obtained with a grounded shield. Alternatively, low temperature preamplifiers could be used locally near the tunneling contact.

The high voltage leads for driving the piezoelectric tube scanner consist of twisted pairs. Short (2 cm), fine wire (38 gauge) jumpers connect the tunneling current coax to the tunneling tip collet. The driven shield is connected to a pair of guard rings on either side of the tunneling tip. These precautions minimize pickup between the tunneling current lead and the high voltage leads.

The tube scanner has a continuous inner electrode and four equally spaced outer electrodes for controlling X, Y, and Z motion of the tunneling tip relative to the sample. The tunneling tip is mounted at the center

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Figure 2. Sectional view of the low temperature STM head mounted in a cryogenic bathysphere cryostat.

(versus side) of the end of the tube to prevent distortion of the image at low temperatures. The displacement (nm/V) for the X-Y motion of the STM is shown as a function of temperature in Fig. 3. The data are determined from STM images of a Au coated holographic grating (see Fig. 4). The X-Y scanning range of our STM decreases by a factor of three upon cooling from room temperature to 76 K; the Z motion displacement, on the other hand, decreases by about a factor of four.

Figure 3. Lateral displacement of the STM piezoelectric tube scanner as a function of temperature.

Figure 4. Full scan range STM images of a Au coated holographic grating with a $1.0\ \mu\text{m}$ spacing at (a) room temperature and (b) 76 K.

TSMFM images

All of the TSMFM images in this paper were taken with the same flexible tunneling tip. The tip was a $1\ \mu\text{m}$ thick evaporated Ni film with a 60 nm Au coating. The Ni film was cut to the geometry shown in Fig. 1 before it was coated with evaporated Au. Poor STM images indicated that stick-slip contact with the sample occurred if the Ni tips were not coated with Au. Apparently an oxide layer on the Ni film has high enough resistance to cause the tip to rub on the surface of the sample.

Figure 5. TSMFM images of a computer hard disk at (a) room temperature (scan range $50\text{ }\mu\text{m}$) and (b) 76 K (scan range $3.6\text{ }\mu\text{m}$). Magnetic bit tracks dominate the image in Fig 5a.

Figure 5a is a TSMFM image of a computer hard disk showing magnetic bit tracks. The Ni tip was mounted in our room temperature long scanning range (full scan range of $50\text{ }\mu\text{m}$) STM head. The bits in the TSMFM image are 150 nm "high" obscuring any topographic features of the disk surface. The bit contrast (magnetic sensitivity) and resolution for this Ni film are roughly ten times larger than previous TSMFM results using $5\text{ }\mu\text{m}$ thick (presumably stiffer) Fe film tips.⁶ Due to our limited scan range of $3.6\text{ }\mu\text{m}$ for our low temperature STM head, we could not see bits in the TSMFM image taken at 76 K . However, the polishing groove topography is visible as shown in Fig. 5b.

A laser ablated in situ YBCO film⁷ ($T_c = 88\text{ K}$, $0.3\text{ }\mu\text{m}$ thick) was also imaged at low temperatures. The YBCO film was coated with a 60 nm layer of Au to improve tunneling contact. The film was mounted on top of a small pellet magnet in the STM so that a 50 mT field

Figure 6. TSMFM images of a YBCO film at (a) room temperature (scan range $9.3\text{ }\mu\text{m}$) and (b) 48 K (scan range about $3\text{ }\mu\text{m}$).

penetrated the film perpendicular to its surface at the tunneling contact.

Figure 6 shows some of the TSMFM images of the film. The top image (Fig. 6a), is a TSMFM full range scan at room temperature. The bottom image (Fig. 6b) is a TSMFM full range scan at 48 K . The scale in Fig. 6b is approximate since we have not yet calibrated the instrument at temperatures below 76 K . The grain structure of the film is apparent in Fig. 6a with a 50 nm roughness scale. These surface features are similar to those visible in STM images taken with a rigid PtIr tip. However, the grain structure is obscured in Fig. 6b by giant features (greater than 900 nm) we believe to be caused by magnetic forces on the tip. Even though the features in Fig. 6a repeated after several scans we caution the reader that we have not tried different samples or tips and therefore consider this image preliminary data.

Conclusion

In conclusion, we have operated a STM in a cryogenic bathysphere cryostat. The bathysphere cryostat will afford measurements from 4 K to 300 K in the bore of a high field magnet. The STM has been operated in two modes at low temperatures; the STM mode with a rigid tunneling tip, and the TSMFM mode with a flexible magnetic tunneling tip. We have imaged magnetic bit tracks on a hard disk with submicrometer resolution in the TSMFM mode at room temperature with a Au coated Ni film flexible tip. The TSMFM contrast improved markedly with this type of tip versus thicker Fe film tips used previously. Magnetic bits were not visible in TSMFM images of the hard disk taken at 76 K. This was probably due to the limited scan range at low temperatures. Preliminary TSMFM measurements on a YBCO film show grain structure at room temperature. At 48 K the TSMFM image was dominated by very large features probably due to magnetic forces on the tip.

What is the nature of these forces? Perhaps the remnant magnetization of Ni film is repelled by the YBCO film because of the Meissner effect and the TSMFM image is showing normal and superconducting sections of the film. Or, perhaps the Ni film is interacting with flux bundles penetrating the film from the pellet magnet. In the future we hope to answer the question by doing low temperature TSMFM of YBCO films and single crystals with and without applied fields as well as TSMFM of conventional superconducting Nb and Pb alloy thin films.

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