Ultramicroscopy 111 (2011) 1360–1365

Contents lists available at ScienceDirect

Ultramicroscopy

journal homepage: www.elsevier.com/locate/ultramic

Quantitative magnetic force microscopy on permalloy dots using an iron filled carbon nanotube probe

F. Wolny [a], Y. Obukhov [b], T. Mühl [a], U. Weißker [a], S. Philippi [a], A. Leonhardt [a], P. Banerjee [b], A. Reed [b], G. Xiang [b], R. Adur [b], I. Lee [b], A.J. Hauser [b], F.Y. Yang [b], D.V. Pelekhov [b], B. Büchner [a], P.C. Hammel [b,*]

[a] Leibniz Institute for Solid State and Materials Research (IFW), Dresden, Helmholtzstr. 20, 01069 Dresden, Germany
[b] Department of Physics, The Ohio State University, Columbus, OH 43210, USA

ARTICLE INFO

Article history:
Received 9 January 2011
Received in revised form
13 April 2011
Accepted 2 May 2011
Available online 1 June 2011

Keywords:
Magnetic force microscopy
Tip-induced magnetization
Iron filled carbon nanotube

ABSTRACT

An iron filled carbon nanotube (FeCNT), a 10–40 nm ferromagnetic nanowire enclosed in a protective carbon tube, is an attractive candidate for a magnetic force microscopy (MFM) probe as it provides a mechanically and chemically robust, nanoscale probe. We demonstrate the probe's capabilities with images of the magnetic field gradients close to the surface of a Py dot in both the multi-domain and vortex states. We show the FeCNT probe is accurately described by a single magnetic monopole located at its tip. Its effective magnetic charge is determined by the diameter of the iron wire and its saturation magnetization $4\pi M_s \approx 2.2\times10^4$ G. A magnetic monopole probe is advantageous as it enables *quantitative measurements* of the magnetic field gradient close to the sample surface. The lateral resolution is defined by the diameter of the iron wire and the probe-sample separation.

Magnetic force microscopy (MFM) is an essential technique for studying magnetic micro- and nanostructures [1–5]. It provides excellent lateral resolution ($\sim$10 nm) and is very sensitive to stray fields resulting from inhomogeneous sample magnetization (such as domains, magnetic vortices or variations in sample properties) or from boundaries of magnetic structures. MFM can provide quantitative information on magnetic structures and their magnetization reversal [6–8], but this requires accurate probe characterization [9–11].

An iron filled carbon nanotube (FeCNT) consists of an iron nanowire tens of nanometers in diameter and microns in length surrounded by a carbon shell [12]. FeCNTs are promising candidates as high resolution magnetic probes. A probe consisting of a single crystalline, single magnetic domain iron wire, with its high shape anisotropy and well known magnetization simplifies the interpretation of MFM data. Furthermore, the tough carbon shell protects the wire from mechanical damage and oxidation, making it a robust and long-lived probe. The first MFM images with FeCNT probes obtained on magnetic recording media demonstrated a high lateral magnetic resolution with magnetic feature sizes $\sim$20–30 nm [13–15].

Here we demonstrate the probe's capabilities and show that the FeCNT can be modeled as a magnetic monopole with its magnetic charge determined by the iron wire diameter and its saturation magnetization $4\pi M_s^{\mathrm{Fe}} \approx 2.2\times10^4$ G, typical for single crystal iron. We find this description to be valid for a wide range of probe-sample separations, allowing us to use our monopole model to determine the carbon shell thickness at the end of the nanotube. The applicability of this simple and reliable monopole model makes the FeCNT probe an excellent tool for quantitative MFM measurements.

The FeCNTs were prepared by chemical vapor deposition on catalyst coated silicon substrates with ferrocene as a precursor [16]. This method yields aligned multiwalled FeCNTs with a moderate distribution of lengths, diameters and filling degrees. The average FeCNT is $\approx$15 μm long and has a diameter of $\approx$100 nm. It contains one long or several short iron wires with diameters ranging from 10 to 40 nm.

The MFM probes were prepared by attaching a single FeCNT onto a conventional tapping mode AFM cantilever with the help of a micromanipulator in a scanning electron microscope (SEM) [13]. Fig. 1 shows SEM images of the FeCNT probes used in this experiment. A $\sim$3 μm long piece of FeCNT was attached to probe J2 (Fig. 1a). As shown in the inset, the iron wire at its end is $\sim$500 nm long, and $\sim$10–20 nm in diameter. This image was obtained by superimposing an SEM in-lens detector image with the inverted backscattered-electron-detector image of the same area. The iron filled parts are visible as dark regions. However, due to limited resolution the diameter of the iron filling is hard to evaluate precisely from SEM measurements alone. The FeCNT on probe K2 (Fig. 1b) is approximately 8 μm long, and is completely filled with an iron wire with a constant diameter of $\sim$20 nm (the

* Corresponding author.
E-mail address: hammel@mps.ohio-state.edu (P.C. Hammel).

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doi:10.1016/j.ultramic.2011.05.002

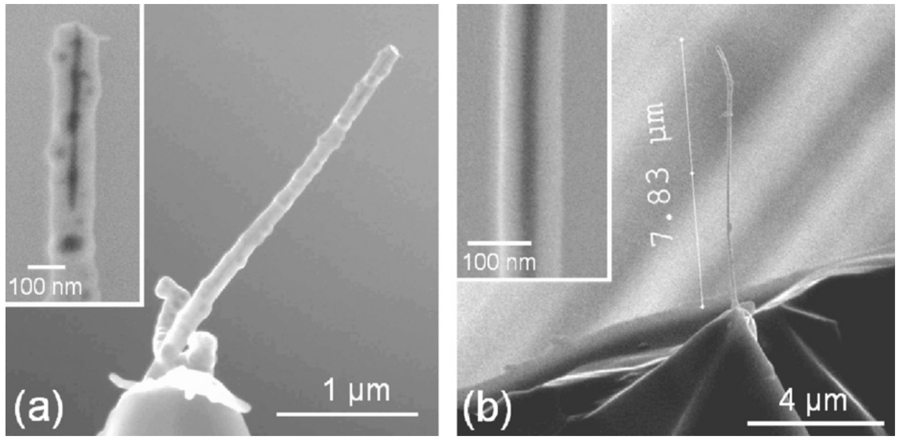

Fig. 1. SEM images of the two FeCNT MFM probes (a) J2 and (b) K2. The FeCNT is attached to the tip of a silicon cantilever. The insets show close-ups of the FeCNTs; the iron filling is darker than the carbon shells.

inset shows a region in the middle of the FeCNT). This FeCNT was cut to the displayed length with a focused ion beam (FIB) to remove unwanted iron particles from its end. Both silicon cantilevers have a resonance frequency $f_0 \sim 13$ kHz and a spring constant $k \sim 130$ dyn/cm (0.13 N/m) measured using a hydrodynamic model [17].

The FeCNT probes were used to perform MFM on permalloy (Py) disks in a high magnetic field of ~ 2 T perpendicular to the Py film plane. The Py disk array was fabricated by photolithographic lift-off process. The Py was deposited onto the silicon substrate with titanium adhesion and capping layers. The Py thickness is 40 nm, the disk diameter is 2.2 μm and the center-to-center spacing of the disks is 6 μm. The saturation magnetization of Py was measured by conventional ferromagnetic resonance (FMR) to be $4\pi M_s^{Py} = 9530$ G. The MFM experiments were performed in high vacuum and at a temperature of 5 K. The cantilever deflection is detected by fiber-optic interferometry, and its frequency is monitored by a digital signal processor [18].

Typical MFM images measured with the FeCNT probe J2 at different probe-sample separations d_s, and at a temperature of 5 K are presented in Fig. 2a. The MFM signal is the cantilever frequency shift monitored during the scan at constant d_s without SPM feedback. Force-distance curves (DC force on the cantilever as a function of probe height) were used to determine the probe touch point ($d_s = 0$) with an accuracy of 10–15 nm. The microscope does not have vibration isolation inside the vacuum can, so the accuracy of the d_s measurement is limited by motion of the probe in response to mechanical vibrations induced by boiling liquid helium. The DC deflection of the cantilever due to DC MFM forces during a scan was estimated to be less then 1 nm. The peak-to-peak cantilever oscillation was kept much smaller than d_s and was usually set to 10 nm; for $d_s < 80$ nm it was reduced to 5 nm. The cantilever frequency shift δf due to a force gradient is given by

$$\delta f(x,y) = \frac{f_0}{2k}\frac{\partial F}{\partial z}(x,y)$$

where f_0 is the cantilever resonance frequency, k its spring constant and $\partial F/\partial z$ is the force gradient in the direction of the cantilever oscillation $\hat{z}$.

We calculate the MFM force gradient $\partial F/\partial z$ under two assumptions (see Fig. 3). First, we consider the iron wire in the CNT to be uniformly magnetized along its long axis. In this case its magnetization can be described by two monopoles Q and $-Q$ positioned at the ends of the wire. The monopole charge $Q = \pi d^2 M_s^{Fe}/4$ is determined by the diameter d of the iron wire and its saturation magnetization M_s^{Fe}. Our experiments were performed in a magnetic field of 20 kG, close to $4\pi M_s^{Fe}$. If the iron wire is not parallel to the external field its magnetization will tilt slightly away from the wire axis reducing the monopole at the wire end. However, for moderate FeCNT tilt angles ($\leq 20-30^\circ$) the monopole description is still reasonable. We also assume the Py film to be saturated in the direction of the external field, perpendicular to the film plane. Consequently the magnetization of the Py film can be represented by two effective monopole layers with an effective charge per unit area $q = M_s^{Py}$ defined by the saturation magnetization of Py (see Fig. 3).

Since the stray field of the film falls off rapidly compared to the iron wire length, the influence of Q associated with the remote end of the wire (see Fig. 3) can be neglected. We can express the MFM force gradient induced by the sample's upper monopole layer as

$$\frac{\partial F}{\partial z}(x,y) = \int \left.\frac{\partial H_z}{\partial z}\right|_z (x-x',y-y')q(x',y')\,dx'\,dy' \equiv \left.\frac{\partial H_z}{\partial z}\right|_z * q$$

where $\mathbf{H} = -Q\mathbf{r}/r^3$ is the magnetic field created by the tip monopole $-Q$, $\mathbf{r}$ is the radius vector, and $*$ indicates a convolution. The total MFM force gradient created by both Py monopole layers can be written as

$$\frac{\partial F}{\partial z} = \left.\frac{\partial H_z}{\partial z}\right|_z * q - \left.\frac{\partial H_z}{\partial z}\right|_{z+t} * q$$

where t is the Py film thickness. This predicts the MFM image will be cylindrically symmetric about the center of the Py disk but we observe (see Fig. 2a) considerable asymmetry in the $\hat{x}$ (horizontal) direction. This can be explained by the tilt of the cantilever and its oscillation relative to the z-axis (Fig. 3). In our setup this tilt is in the xz plane and the tilt angle is $\theta = 15-20^\circ$. In this case the MFM force gradient is given by

$$\frac{\partial \mathbf{F}}{\partial \mathbf{l}} = \left.\frac{\partial \mathbf{H}}{\partial \mathbf{l}}\right|_z * q - \left.\frac{\partial \mathbf{H}}{\partial \mathbf{l}}\right|_{z+t} * q \tag{1}$$

where $\mathbf{l}$ is a vector parallel to the direction of cantilever oscillation. A calculation of $\partial \mathbf{H}/\partial \mathbf{l}$ in our geometry gives

$$\frac{\partial \mathbf{H}}{\partial \mathbf{l}} = \frac{\partial H_x}{\partial x}\sin^2\theta + \left(\frac{\partial H_x}{\partial z} + \frac{\partial H_z}{\partial x}\right)\sin\theta\cos\theta + \frac{\partial H_z}{\partial z}\cos^2\theta \tag{2}$$

$$\frac{\partial H_x}{\partial x} = -Q\frac{r^2-3x^2}{r^5}$$