Stable magnetization of iron filled carbon nanotube MFM probes in external magnetic fields

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Abstract. We present results on the application of an iron filled carbon nanotube (Fe-CNT) as a probe for magnetic force microscopy (MFM) in an external magnetic field. If an external field is applied parallel to the sample surface, conventional ferromagnetically coated MFM probes often have the disadvantage that the magnetization of the coating turns towards the direction of the applied field. Then it is difficult to distinguish the effect of the external field on the sample from those on the MFM probe. The Fe-CNT MFM probe has a large shape anisotropy due to the high aspect ratio of the enclosed iron nanowire. Thanks to this the direction of the magnetization stays mainly oriented along the long nanotube axis in in-plane fields up to our experimental limit of 250 mT. Thus, the quality of the MFM images remains unchanged. Apart from this, it is shown that Fe-CNT MFM probe yields a very good magnetic resolution of about 25 nm due to the small diameter of the iron filling.

1. Introduction
Magnetic force microscopy (MFM) is a powerful tool to image the magnetic stray field on the surface of micro- to nanoscale magnetic structures. In the last years many different approaches were developed to produce MFM probes with better magnetic resolution. Some of these are for example FIB-milled probes [1], magnetically coated carbon nanotubes [2], magnetic nanowires [3] and spherical magnetic particles [4].

Iron filled carbon nanotubes (Fe-CNTs) consist of a cylindrical single domain iron nanowire which is surrounded by several carbon shells. They possess various properties that make them ideal for the application as MFM probes. The extraordinary mechanical stability of carbon nanotubes [5] ensures a very long probe lifetime. In contrast to using magnetically coated carbon nanotubes as MFM probes, the iron nanowire is protected from oxidation by the surrounding carbon shells. Concerning the magnetic properties, the elongated iron nanowire enclosed in the nanotube can be regarded as an extended magnetic dipole of which only the monopole close to the sample surface interacts with the magnetic stray field of the sample [6]. This could simplify the MFM data evaluation considerably. Furthermore, the small diameter of the iron nanowire provides a very good magnetic resolution.

MFM measurements can be carried out in an external magnetic field, e.g., to determine the switching fields of small magnetic structures and particles or to investigate the magnetization reversal mechanism of a sample. When the magnetic field is applied parallel to the sample surface, the probe magnetization can be affected. In the case of a conventional magnetically
coated MFM probe with a pyramidal tip it can be necessary to use special calibration samples in order to determine the magnetization state of the probe tip in the external field for a clear evaluation of the MFM data [7]. In some cases the reorientation of the probe’s magnetization along the external field might be an advantage, e.g., to selectively image specific components of the sample stray field [8]. However, when only the perpendicular sample stray field component is to be imaged depending on the external field, this is an unwanted effect. We use an Fe-CNT MFM probe in an external in-plane magnetic field to compare its behavior to that of a conventional probe. The iron nanowire of an Fe-CNT has a very high aspect ratio (often larger than 1:100) and thus a large shape anisotropy. This should lead to a stable magnetization along the long nanotube axis even in a moderate external field perpendicular to this axis.

2. Probe Preparation
The multi-walled Fe-CNTs used for the MFM probe fabrication are grown by chemical vapor deposition (CVD) on catalyst-coated silicon wafers with ferrocene as a precursor [9]. Subsequently, a single filled nanotube is attached to a conventional atomic force microscopy (AFM) cantilever in a scanning electron microscope (SEM) with the help of a micromanipulator [10]. In figure 1, an MFM cantilever prepared with this method is shown. The SEM image in figure 1(a) shows that the attached nanotube has a length of about 15 µm. The upper 5 µm of the nanotube are filled with iron as can be seen in figure 1(b). The diameter of the filling is approximately 20 nm, the nanotube diameter is 90 nm.

![Figure 1. Fe-CNT attached to an AFM cantilever. (a) SEM micrograph. (b) Backscattered electron contrast (the bright region of the nanotube is iron filled).](image1)

![Figure 2. MFM image of a magnetic hard disc taken with the Fe-CNT probe shown in figure 1. Scan size 3×3 µm. The inset shows a section along the white line with a lateral magnetic resolution of 20-30 nm.](image2)

3. Results and Discussion
3.1. Magnetic resolution
To find out what lateral magnetic resolution can be obtained with an Fe-CNT MFM probe, an MFM scan of a high density magnetic hard disc was performed. The measurement was performed in a Nanoscan high-resolution magnetic force microscope (hr-MFM) in high vacuum. Figure 2 shows a 3×3 µm scan of a 320 GB Western Digital hard disc. The magnetic bits are magnetized parallel or antiparallel to the probe magnetization corresponding to darker or lighter grey in the MFM image. To determine the resolution in this case, a line section through a row
of bits was extracted (inset in figure 2, an average over 4 scan lines is displayed). Peaks with a width of 25-35 nm can be found and even some smaller features with widths below 20 nm appear as shoulders on bigger peaks. Thus the magnetic resolution of the Fe-CNT MFM probe already comes close to the so far maximum reported resolution of 10 nm [1, 2]. A further improvement of the resolution should be possible with the use of Fe-CNTs with smaller diameters.

3.2. MFM measurements in an external in-plane magnetic field

To see the influence of an external in-plane magnetic field on the magnetization of a conventional and an Fe-CNT MFM probe, we wanted a test sample whose magnetization isn’t influenced much by this external field. We chose a sample of Fe-CNTs grown perpendicular to the surface on a silicon wafer and afterwards embedded in an SiO$_2$ matrix. The sample was then polished so that the ends of the magnetic nanowires are located directly at the sample surface. Each of these iron nanowires has a magnetic stray field similar to a small bar magnet. In the MFM, an in-plane magnetic field that can be varied from -250 to 250 mT was set up. Before the measurement, the MFM probes were magnetized perpendicular to the sample surface. Thus, the external magnetic field was applied parallel to the sample plane and perpendicular to the original probe magnetization.

![MFM images](image_url)

**Figure 3.** MFM images of a sample of iron nanowires (oriented perpendicular to the sample plane) taken at different values of the external magnetic field (applied parallel to the sample plane from the left to the right) with (a) a CoCrTa coated MFM probe, from left to right: 0 mT, 75 mT, 100 mT, 0 mT and (b) an Fe-CNT MFM probe: 0 mT, 100 mT, 250 mT, -250 mT. The circles mark equal structures in the different MFM scans to facilitate the comparison. Scan size: 7×5 μm.

Figure 3(a) shows four MFM scans of the embedded nanotubes taken at different values of the external magnetic field. A conventional magnetically coated MFM probe was used, in this case a silicon cantilever with a sharp pyramidal tip at its end coated with 50 nm of a CoCrTa alloy. The magnetic moment of this probe was originally parallel to the sides of the coated pyramid resulting in a net magnetization along the pyramid axis. From the left to the right image, the magnetic field was increased stepwise from 0 to 100 mT and then back to 0 mT. When the MFM probe is magnetized along the z direction (perpendicular to the sample surface), it only images the z component of the magnetic stray field gradient. Thus, in the zero field MFM scan, one can only see the magnetic stray field emanating from the ends of the iron nanowires as black or white dots corresponding to a nanowire magnetization parallel or antiparallel to the MFM probe magnetization. Starting at external field values between 75 and 100 mT, all black and
white dots are transformed into neighboring black-white and white-black structures. From this one can conclude that the MFM probe magnetization is starting to tilt out of the z-axis into the direction of the applied field. This way, not the sample stray field along the z direction is imaged but the stray field along the direction of the effective tip magnetization. In the rightmost image of Figure 3(a) it can be seen that the probe moment stays tilted even when the external field is switched off again. In order to restore the probe magnetization perpendicular to the sample surface, it needs to be remagnetized. Especially for MFM measurements conducted in vacuum this represents a bothersome additional effort.

In the MFM measurements shown in figure 3(b) the same procedure as described above was repeated with an Fe-CNT MFM probe. One can clearly see that with this probe, even in a maximum applied in-plane field of 250 mT, the black and white dots still remain the same as in the zero field measurement. The probe magnetization remains stable along the probe Fe-CNT axis and images the z component of the magnetic sample stray field gradient. The last image in figure 3(b) shows an MFM scan performed at the maximum applied magnetic field in the opposite direction. One can see that the probe magnetization direction switched, but still no in-plane components can be seen. It has been shown by other groups that if the direction of the external magnetic field deviates from the long nanowire axis up to a perpendicular orientation, the magnetic moments inside the nanowire start to deviate from the wire axis [11]. When the applied field becomes stronger, the deviation of the moments from the nanowire axis also becomes larger. Yet our MFM measurements at applied fields even up to the nanowire switching field do not show a magnetic component parallel to the sample surface. This could mean that the deviation of the magnetization from the wire axis is still small and the stray field of the magnetic monopole at the nanowire end outweighs the existing in-plane components.

4. Conclusion

It has been shown that Fe-CNT MFM probes yield a very good lateral magnetic resolution in the range of 20-30 nm. There is also still great potential to improve the resolution by the fabrication of MFM probes with Fe-CNTs of smaller diameter. Furthermore, our probes exhibit a stable magnetization in in-plane magnetic fields of up to 250 mT. This is a great advantage since the change in sample magnetization can be measured without a disturbing change in the MFM probe magnetization. This property could still be enhanced by fabricating MFM probes with the Fe-CNT’s axis aligned with the designated external field direction. Then one could apply large magnetic fields that would only lead to a further stabilization of the wire magnetization while retaining the probes z-sensitivity.

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