



Review

Magnetic Force Microscopy in Physics and Biomedical Applications

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Abstract: Magnetic force microscopy (MFM) enables to characterize magnetic properties with submicron (nanoscale) resolution and without much demand on sample surface preparation. MFM can operate in a wide range of temperatures and environmental conditions, that is, vacuum, liquid, or air, therefore this technique has already become the most common tool used to characterize variety of magnetic materials ranging from ferromagnetic thin films and 2D materials to biomedical and/or biological materials. The purpose of this review is to provide a summary of MFM basic fundamentals in the frame of other related methods and, correspondingly, a brief overview of physics and chiefly biomedical as well as biological applications of MFM.

Keywords: magnetic force microscopy; 2D materials; biomedical materials; biological materials; nanoparticles; magnetic materials; thin films



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1. Introduction

There have been various methods developed to observe magnetic structures. Basically, two major classes of visualization methods of magnetic structures can be distinguished: (i) Magnetic measurement methods related to a beam (electron beam or light beam), that is, for example, Lorentz force microscopy [1], magneto-optical microscopy employing either Kerr [2] or Faraday effect [3,4], scanning electron microscopy with polarized analysis [5,6] and X-ray magnetic dichroism spectromicroscopy [7–9]; and (ii) magnetic measurement methods related to scanning probe-based techniques realized by means of magnetic force microscopy (MFM), magnetic resonance force microscopy (MRFM) [10–12], magnetic exchange force microscopy [13], magnetoelectric force microscopy [14], scanning Hall probe microscopy [15,16], and magnetometry with nitrogen-vacancy defects in diamond [17,18]. Note that some well-known methods such as the Bitter method [19] do not fall into either of the above two classes.

MFM [20–24], originally developed with the goal to visualize magnetic fields arising from the surface of a flat ferromagnetic sample, was derived from the scanning probe microscopy (SPM) [25]. In general, SPM includes the scanning tunneling microscopy (STM) [26,27] and the atomic force microscopy (AFM) [28]. Both the mentioned methods enable surface imaging and spectroscopy with an atomic resolution. In general, SPM imaging consists of collecting various local surface properties during scanning of the surface with an SPM probe in small lateral steps within an area of interest on the sample surface (raster imaging). According to Ref. [27], illustratively put, the scanning tunneling microscope is “a mechanically positioned, electrically sensitive kind of nanofinger for sensing, addressing, and handling individually selected atoms, molecules, and other tiny objects”. The principle of STM is based on the concept of quantum tunneling between a metal sharp tip and a conductive specimen separated from the tip by an insulating layer (usually vacuum). In the most common imaging mode, the image is formed by contours of constant tunneling current. Tunneling spectroscopy is then a technique that locally separates the individual contributions of the various electronic states to the tunneling current.

The AFM probe working as a force sensor also comprises a sharp tip (placed at the end of a tiny cantilever) that is used to scan the surface area of a sample with no need for conductivity. As soon as the tip is brought close to the sample surface with the separation gap in the nanometer range, both the attractive and repulsive forces between the tip and the sample result in cantilever deflection. The cantilever's deflection is measured in various ways, e.g., by the optical interferometric system or using a sectional photodiode combined with a reflected laser beam. In general, AFM measurement can be separated in the static (contact mode) and the dynamic mode. The contact mode is the simplest mode of AFM, where the surface topography is obtained from the feedback signal which keeps track of the cantilever deflection as a result of its changing force due to surface height variations. Dynamic mode measurement includes (i) the amplitude modulation (AM) AFM (also known as tapping mode [29]), and (ii) frequency modulation (FM) AFM (also called the non-contact AFM) [30]. The introduction of dynamic AFM modes is related to the existence of several parameters sensitive to the tip-sample interactions: amplitude, frequency, phase shift, and cantilever deflection. Although AM AFM is usually performed, FM AFM also has some advantages [31,32]. The modulation is usually realized through a cantilever driver in the form of a piezoelectric element. However, some other driving principles such as electrostatic, magnetic, thermo-optic, and acoustic coupling drivers have also been proposed [33]. Note that in the pioneering design of Binnig et al. [28], which is now obsolete, AFM ran in four different modes, the amplitude of an oscillating cantilever with a conductive surface was sensed via tunneling current using an STM probe stacked between the AFM probe and the AFM head. The modulation frequency was near the fundamental resonance frequency of the cantilever. While scanning the surface, a user-set value of the amplitude was kept constant by controlling the distance between the cantilever and the sample.

Depending on the tip, the sample surface and the distance between the tip and the surface, various forces can be sensed such as van der Waals forces, capillary forces, magnetostatic and electrostatic forces, etc. The output of AFM in its simplest setup is the topography of the surface obtained usually using the tapping mode AFM and sensing van der Waals forces. For sensing magnetostatic forces, it is necessary to lift the probe away from the sample to the height, where van der Waals forces are negligible and in the best case, the probe experiences only magnetostatic forces. However, the possible presence of electrostatic interactions which are also the long-range interactions can cause artefacts during magnetic force scanning. Distinguishing magnetostatic and electrostatic interactions can be accomplished by, for example, a combination of Kelvin probe force microscopy and MFM [34].

The initial development of the AFM inspired Martin and Wickremasinghe who described, in 1987 [35] for the first time, the MFM method for mapping the magnetic field distributions on a microscopic scale. The first MFM cantilever was an iron L-shaped wire with a diameter of 25 μm . The shape of the cantilever's tip was manufactured by tapering the wire down to a 2 μm diameter and electro-etching in order to achieve a 100 nm diameter tip apex. An electric coil placed around the cantilever wire gives the possibility that magnetization of the tip could be modulated by pulsing the exciter current. Today, the majority of magnetic force microscope (MFM) probes consist of a cantilever with a ferromagnetic tip. Back to the first MFM measurement, as of the examined sample, an IBM 3380 thin-film magnetic recording head was used. Hence, a local magnetic field on the sample could be modulated at a certain frequency by modulating the recording head current. Two modes of the MFM were run, that is, either with modulated magnetic field on the sample or modulated current of the exciter coil current. In both the cases, the force probe was kept at constant height over the sample surface while the cantilever vibrations (the amplitude signal) were detected using a laser heterodyne probe. The amplitude change was proportional to the perpendicular component of the local magnetic field gradient. The maximum spatial resolution reached at that time was 100 nm. The typical forces detected were on the order of 10^{-10} N. In the same year, Saenz et al. [36] presented an observation

of magnetic forces using AFM. The resolution was also about 100 nm. Today, the spatial resolution has been improved to typically ~50 nm [24]. In some MFM devices, resolution reached less than 50 nm [13,37]. However, the reached lateral resolution also depends on the type of studied materials and the prevalent orientation of magnetization in a sample, that is, either perpendicular or in-plane magnetization. A higher lateral resolution is usually reached with magnetically hard materials (e.g., FeNdB, SmCo) than magnetically soft materials (e.g., Permalloys). Sensitivity of the force sensor as a part of MFM might today reach values of order 10 pN [38]. It is worth noting that the force sensitivity (the magnitude of the smallest detectable force) is proportional to the quality factor [39]. In order to improve the force sensitivity, AFM or MFM often operate in a vacuum.

Standard two-pass MFM is the most common MFM mode. In the first pass, the probe scans the surface usually in tapping (semi-contact) mode, sensing van der Waals forces and mapping the topography of the sample by keeping constant amplitude of the oscillating cantilever with its resonant frequency; in the second pass, the probe lifts a distance away from the sample and senses the long-range interactions via, for example, the frequency change of the vibrating cantilever while keeping the probe-sample distance constant. Sensing the long-range interactions is due to the magnetostatic interactions between the ferromagnetic tip and the sample surface. MFM tips are most commonly magnetized along their axis, although lateral magnetizing fields can also be used [40,41].

MFM, which may be equipped with (i) a thermal or vacuum chamber, (ii) a source of an external constant or variable magnetic field, or (iii) other auxiliary devices, plays an important role in studying the formation of magnetic domains in magnetic materials [42,43], magnetic nanoparticles in a nonmagnetic matrix [44,45], vortex dynamics in superconductors [46], vortices in a nanodot array made of thin permalloy film [47], magnetic skyrmions, small swirling topological defects in the magnetization/texture [48] with a prospect of new magnetic recording, in SrIrO₃/SrRuO₃ bilayers [49] or magnetic domains in diluted magnetic semiconductors [50], antiphase boundaries in bulk Ni-Mn-Ga Heusler alloy [51], etc., to name some cases of MFM usage. MFM imaging is rather qualitative imaging, generally. In some special cases, such as perpendicularly magnetized thin samples, it is theoretically possible to use MFM's force distribution within the scanned area to obtain the corresponding magnetization distribution [52,53].

The faster development of MFM is limited by the fact that, in principle, atomic resolution cannot be achieved in MFM due to the long-range magnetic forces between the tip and the sample. However, new SPM methods that have evolved from MFM (both directly and indirectly) [54], such as scanning superconducting quantum interference device microscopy, nitrogen vacancy center scanning probe microscopy, magnetic resonance force microscopy, magnetic exchange force microscopy, and scanning Hall probe microscopy, provide some new magnetic field sensing capabilities. Some of the abovementioned SPMs do have atomic resolution. Nevertheless, a description of the evolved methods is beyond the scope of the present review.

The aim of this paper is to provide researchers and engineers with a brief overview on engineering and biomedical applications of MFM. As such, this review is organized as follows. In Section 2, we outline the fundamental principles of MFM. Engineering classical applications of MFM that include magnetostriction in thin films, IT circuits, and nanoparticle reinforced structures are given in Section 3. Section 4 presents the biomedical application of MFM.

2. Principle of Magnetic Force Microscopy

For obtaining magnetization distribution from MFM data, various models can be used [55,56]. The models usually take the tip shape into account and assume a regular stripe domain structure with anisotropy perpendicular to the sample surface. In the case of the analytical approach and a simple geometry shape of the probe tip, the magnetostatic interaction energy and the resulting force can be adopted from, for instance, Ref. [57]. In the simplest case, the unknown magnetization vector field near the probe apex is modeled

by a homogeneously magnetized prolate spheroid [58]. It is worth noting that a theoretical spatial resolution calculated as a function of the geometric parameters of a magnetic tip has also attracted attention [55,56].

Free vibration of the MFM cantilever can be described by a well-known Euler–Bernoulli beam equation [59]:

$$EI \frac{\partial^4 w(x, t)}{\partial x^4} + \mu \frac{\partial^2 w(x, t)}{\partial t^2} = 0, \quad (1)$$

where $w(x, t)$ is the displacement of the cantilever beam in z direction (see Figure 1 for orientation of x, y, z axes in respect to the cantilever), E, I , and μ are the Young's modulus, the second moment of area of the beam's cross section, and mass per unit of length, x is the spatial coordinate along the cantilever beam, and t is time. In the case of the forced vibration, a force term would be added to the right side of Equation (1). It is useful to describe the motion of the cantilever tip as the motion of a forced harmonic oscillator with damping [60]:

$$m \frac{d^2 z}{dt^2} + \frac{m\omega_0}{Q} \frac{dz}{dt} + kz = F_{ts} + F_0 \cos(\omega t). \quad (2)$$

where m and k are the mass and cantilever stiffness, respectively; F_0, F_{ts} , and ω are the driving force amplitude, the tip–surface interaction force, and angular frequency of the driving force, respectively; Q and ω_0 are the quality factor and angular resonance frequency, respectively.

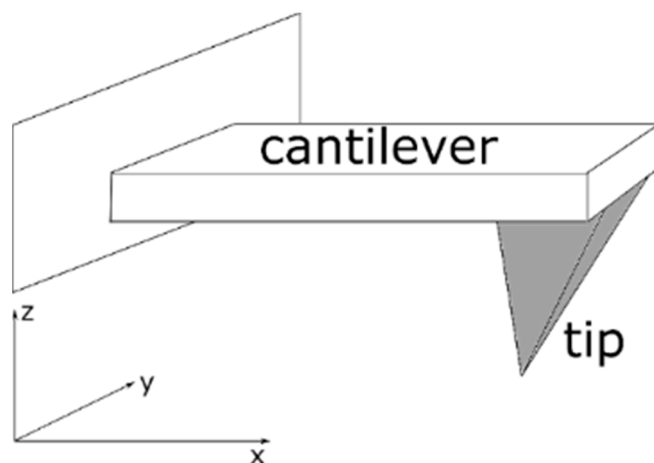


Figure 1. Schematics of the standard MFM microcantilever with tip and the Cartesian coordinate system.

As described in the previous paragraphs, in the MFM second pass, the probe lifts a distance away from the sample and senses the magnetostatic interactions between the ferromagnetic tip and the magnetic areas at the sample's surface via the amplitude, frequency, or phase change of the vibrating cantilever while keeping the probe–sample distance constant. Most commonly, magnetic contrast in the second pass is represented by the phase change of MFM cantilever vibrating at resonance frequency $\omega_0/(2\pi)$. To understand MFM principles, it is desirable to express the phase change analytically:

$$\Delta\varphi(\omega_0) = \arctan\left(\frac{k}{Q \frac{dF_{ts}}{dz}}\right) \approx \frac{Q}{k} \frac{dF_{ts}}{dz}. \quad (3)$$

It is evident from Equation (3) that MFM sensitivity can be notably improved by increasing Q and/or decreasing k . Equation (3) is also useful in the case of phase detection in the second pass. In the first pass, van der Waals forces are sensed and MFM works as an AFM microscope, e.g., in the non-contact mode (FM) as described in [61]. Note that in the FM detection method, the cantilever for force sensing is vibrated at the mechanical

resonant frequency and its frequency shift due to the force interaction is detected as the force gradient acting on the tip.

3. Aspects of Magnetic Force Microscopy for Various Engineering Fields

3.1. MFM Design Ideas Applied in Magnetostrictive Thin Films

Giant magnetostriction has been observed in numerous rare-earth/iron alloys and a number of applications which include the surface acoustic wave devices, actuators, magnetic sensors [62], etc., have been anticipated. Although various device applications are envisaged for bulk elements, ribbons, and thin films, the latter are particularly advantageous for the production of miniature sensors with a low power consumption.

Giant magnetostrictive thin films have predominantly been used as various micro-actuators for applications in microsystem technology. Different geometries and boundary conditions are common, of which the most important are related to cantilevers, membranes, and plates [63]. Upon acting of an external magnetic field, the magnetostriction in a magnetostrictive film causes the substrate with the deposited magnetostrictive film to bend, similar to the bending of a bimetallic transducer. An example is a micro-electro-mechanical system (MEMS)-based magnetostrictive magnetometer with a sensitivity near $1\ \mu\text{T}$. The example is not directly related to MFM; however, it shows some design ideas adopted from MFM. In the given example, the active transduction element of the magnetometer was a commercial (001) silicon microcantilever coated with an amorphous thin magnetostrictive film which offers a number of advantages for device applications with small size, high sensitivity, potential integration on a silicon chip, and with a vector capability [64–66]. The schematic of this operating principle is given in Figure 2.

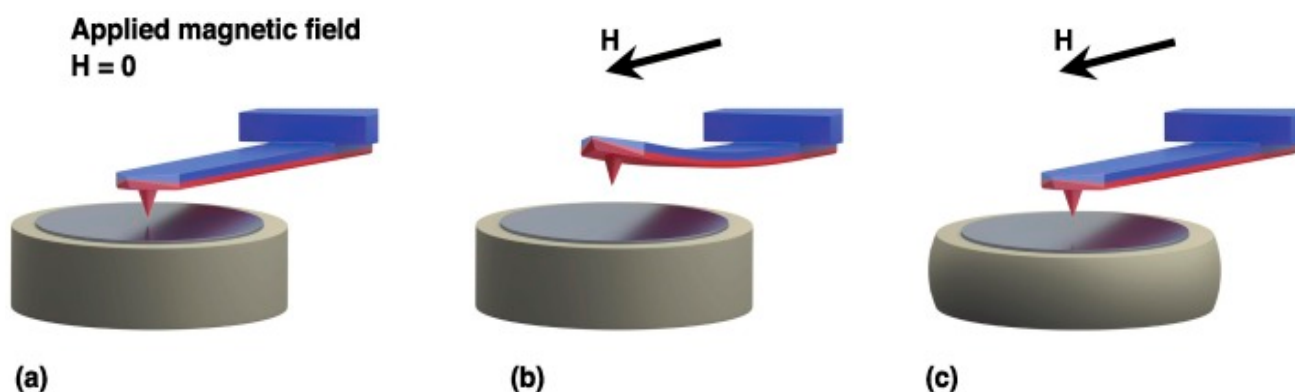


Figure 2. Working scheme of the setup. (a) At $H = 0$, the cantilever (blue) coated with the magnetostrictive film (red) is straight; the tip is engaged to the sample surface (the light blue disk), and the AFM piezoelectric scanner (gray) is in its default position. (b) At $H > 0$, the bimorph bends upward. (c) The AFM feedback causes the piezoelectric scanner to retract, until the cantilever is straight again, keeping the deflection point constant. The representation is not to scale and the increased tip–sample distance is exaggerated. For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article [64] reprinted with permission from Elsevier.

In another example [67], MFM is used to characterize a local evolution of the magnetic domain structures of Terfenol-D ($\text{Tb}_{0.27}\text{Dy}_{0.73}\text{Fe}_2$) under a variable magnetic field. An asymmetry in magnetostrictive strain is observed in Terfenol-D under the opposite magnetic field. This technique is useful in characterizing the evolution of magnetic domains for a wide range of ferromagnetic materials under an external field. Figure 3 displays the magnetostrictive strain of Terfenol-D surface in the direction of parallel and perpendicular to the magnetic field.

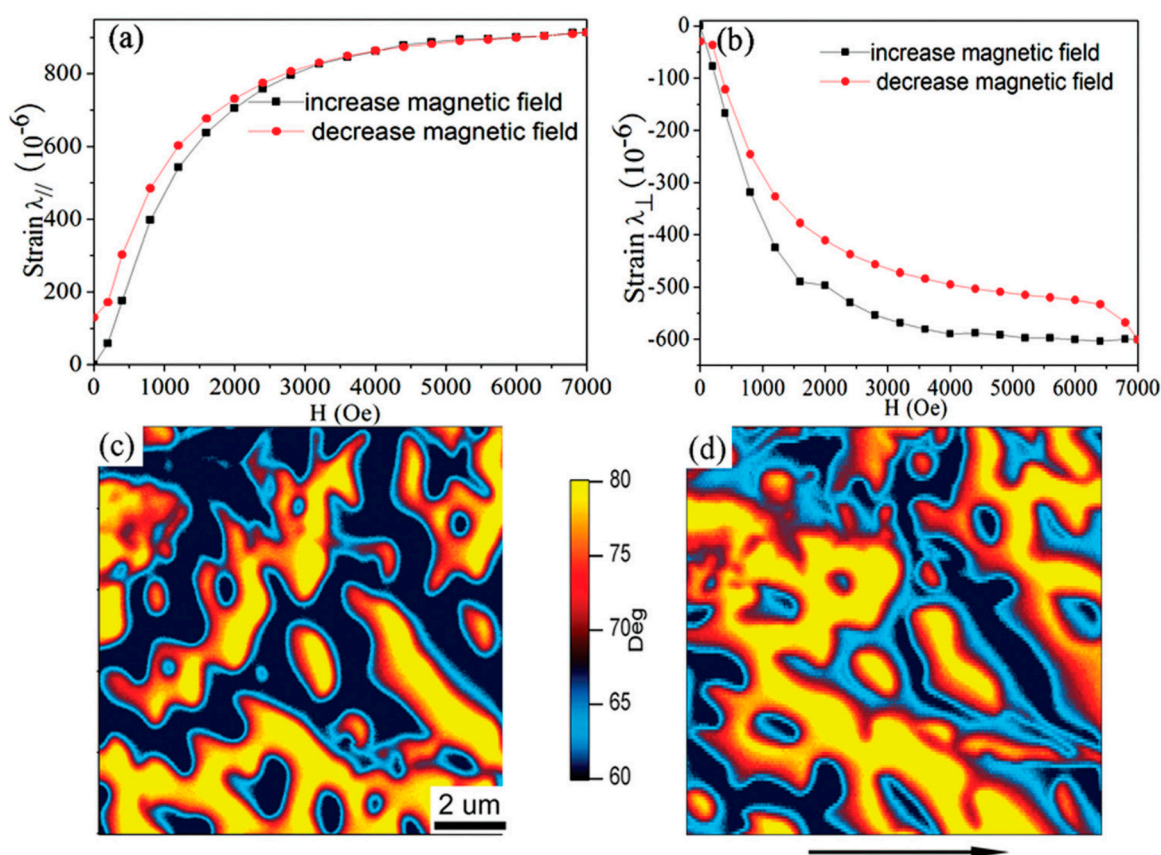


Figure 3. The magnetostrictive strain and MFM phase images of the Terfenol-D disk at saturated magnetization (a) parallel, (b) perpendicular to the applied magnetic field; (c) MFM phase image before, (d) after magnetization [67].

3.2. MFM for IC Current Used in Conductors and Superconductors

In integrated circuits (IC) failure analysis, current is often used to indicate the presence of a defective device. The increasing complexity of IC requires an internal contactless test technique for their function and failure analysis. By imaging the magnetic field produced by a current flowing in an IC, the defective devices can be located using MFM. MFM has been applied to image currents in the internal IC conductors with the ability to analyze the current direction and magnitude with a sensitivity of approximately ~ 1 mA ($1 \mu\text{A}$) dc (ac). MFM methods are also used to determine magnetic vortex core observation in circular dots of permalloy. A ferromagnetic dot with a curling magnetic structure is provided by MFM imaging of circular dots of permalloy ($\text{Ni}_{80}\text{Fe}_{20}$) 0.3 to $1 \mu\text{m}$ in diameter and 50 nm thick [68–70]. MFM methods are also designed on the high- T_c superconductors $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ and conventional type-1 superconductor Sn, with low temperature phase. The low-temperature MFM may be used to investigate the distribution of the superconductive phase on the surface of the superconductors. All MFM images of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ at $T < T_c$ show some perturbations and smooth regions, which may be treated as the regions with the disturbance of the superconductivity of the sample. The possible physical reason for the existence of such regions could be imperfections in the sample structure or more complex phenomena of the magnetic field penetration into the high- T_c superconductor, which is caused by the tip [71–73].

3.3. MFM Measurement for Magnetic and Non-Magnetic Particles Reinforced Matrix

Embedding superparamagnetic iron oxide nanoparticles (SPIONs) in functional thin films is of high interest for nanocomposites as well as for biomedical applications which are discussed in Section 4. However, there is still a need for the high-resolution mapping of the hidden nanoparticles. Magnetic force microscopy (MFM) has proven to be a suitable

technique for imaging SPIONs at ambient conditions and providing information about spatial distribution, size, and magnetic behavior in a single pass. MFM enables high-resolution mapping of magnetite nanoparticles of various sizes embedded in polymer films of various thicknesses. A quantification of the magnetic signals is used to determine the size of the magnetic core of the embedded nanoparticles as also shown in Figure 4 [36,74,75]. MFM is also used to detect probe-sample interactions from superparamagnetic nanoparticles *in vitro* in ambient atmospheric conditions. By using both magnetic and nonmagnetic probes in dynamic lift-mode imaging and by controlling the direction and magnitude of the external magnetic field applied to the samples, it is possible to detect and identify the presence of superparamagnetic nanoparticles (see Figure 5) [76–79]. Magnetic dichroism in the high-energy spectroscopies has opened new opportunities for the spatially resolved investigation of the magnetic microstructures. Involving characteristic electronic levels of the solid, soft X-ray magneto-dichroic effects combine the magnetic sensitivity with elemental selectivity and, correspondingly, permit element-specific analyses of magnetic phenomena. Of particular interest is their use as a contrast mechanism in magnetic domain imaging [7]. Magnetic hysteresis measurements on heteromagnetic materials are of interest. Dramatically different Fe and Co hysteresis curves of Fe/Cu/Co trilayers were obtained by recording the magnetic circular dichroism at their respective L3 white lines as a function of applied magnetic field [80].

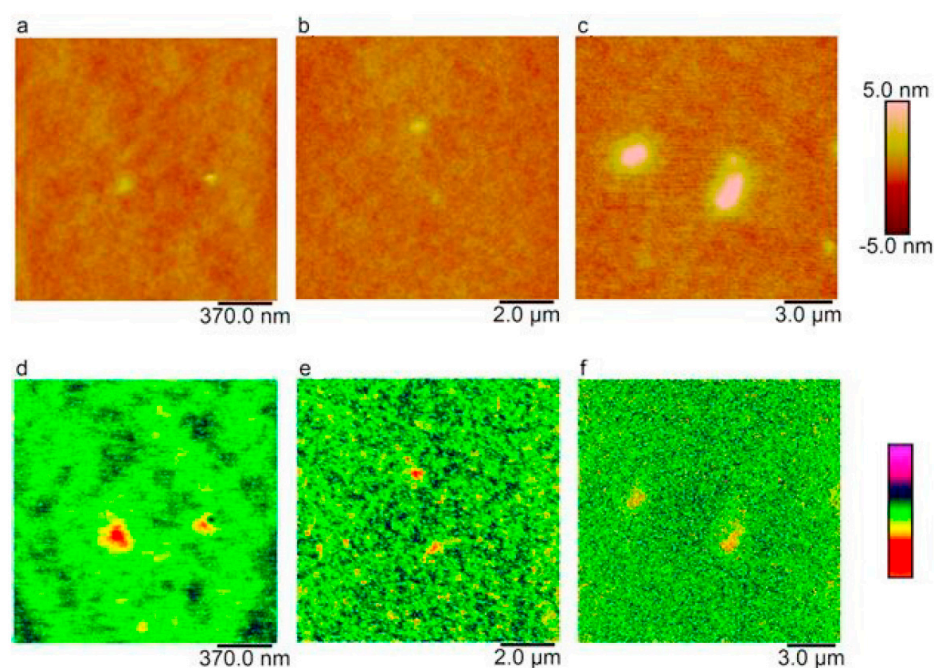


Figure 4. Atomic force and magnetic force microscopy measurements of iron oxide nanoparticles embedded in a resin layer. Topography of 10 nm nanoparticles in a 25 nm layer (a), 20 nm nanoparticles in a 150 nm layer (b), and 80 nm nanoparticles in a 350 nm layer (c). Negative phase shifts due to the attractive magnetic force are observed in corresponding phase images (d–f). The color scale is -1 to 1° in (d), -0.25 to 0.25° in (e), and -0.1 to 0.1° in (f). All phase images were taken in 15 nm lift height. These data were reprinted from Ref. [74] with permission from Wiley Online Library.

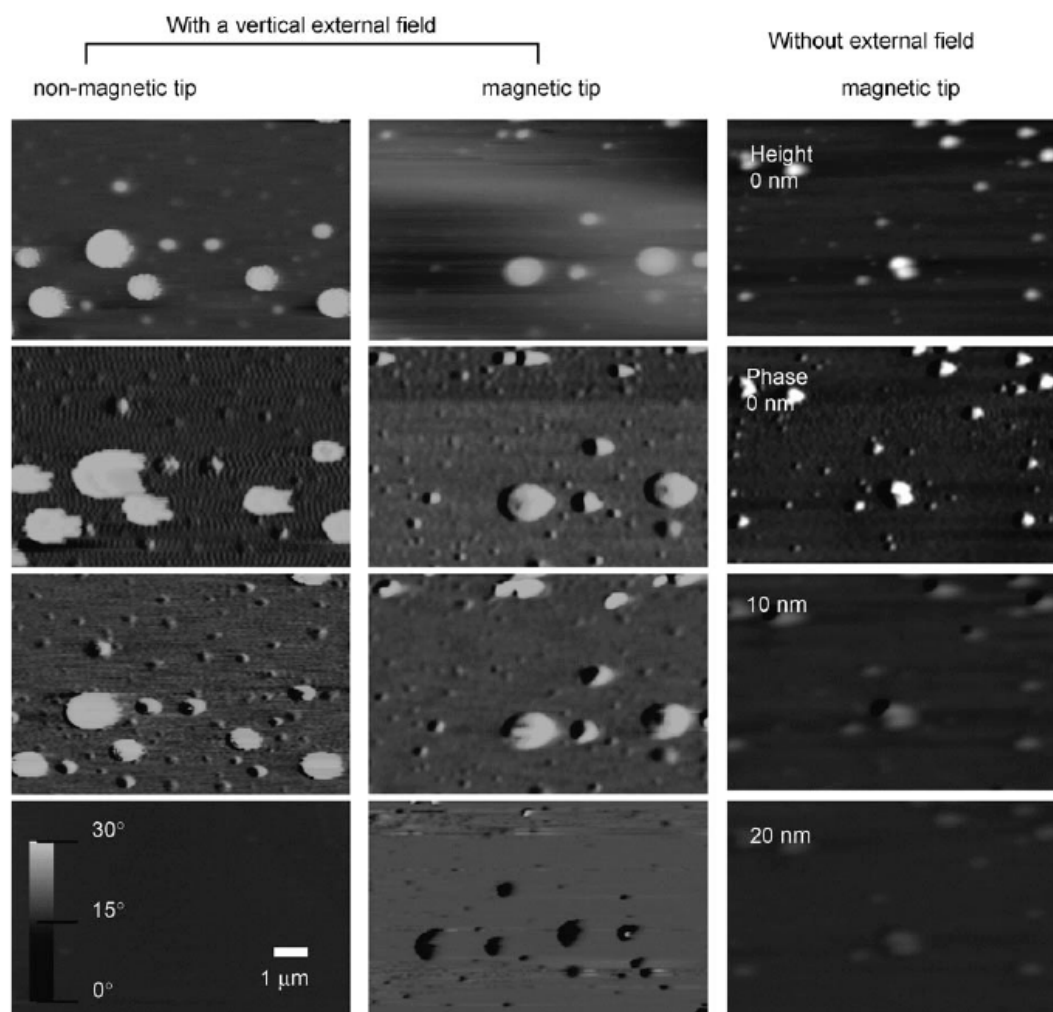


Figure 5. Tapping-mode height (row 1) and phase images (rows 2 to 4) of SPNs obtained in lift-mode using a nonmagnetic or magnetic AFM probe with or without the presence of an externally applied vertical magnetic field, as indicated. In each series of images, the amplitude of cantilever oscillations was kept constant for various lift heights. Lift height for each row of images is indicated on the top left of the images in Column 3. Grayscale for phase values and scale bar for image size are shown as insets in the bottom-left image and apply to all panels. Figure was reprinted from Ref. [77] with permission from Wiley Online Library.

4. Magnetic Force Microscopy for Bio-/Medical Applications

MFM was originally developed for the characterization and study of the inorganic materials such as the magnetostrictive films or IC components that were also briefly discussed in the previous section. Nevertheless, some biological [81] and organic/inorganic [82] films or even single proteins [83] can exhibit magnetic properties; therefore, MFM has recently become the suitable candidate for their characterization. For example, iron deficiency in human bodies is usually evaluated based on the ferritin protein level [84]. Nevertheless, for individuals with injury or those with inflammation, the iron level regulation is affected by an interplay between the ferritin (iron-replete) and apoferritin (iron-deficit) proteins [85]. In addition, ferritin protein has been also considered as a possible carrier for anticancer drugs [86]. It has already been demonstrated that MFM is able to distinguish between the iron-deficit “apoferritin” and iron-replete “ferritin” protein from their mixtures in vitro [83]. We emphasize here that to preserve the native structure of molecules, it is of a great importance to perform the characterization of the investigated biological system in either a native environment or at least in the aqueous solution that is usually relevant to majority

of the biological systems [87,88]. As a result, the application of MFM in the study of soft or biological materials is still undergoing extensive development [89].

Magnetic nanoparticles have extraordinary magnetic properties such as super-paramagnetic behavior and size ranges from 1 to about 100 nm [90,91]. Therefore, they are of a great importance in biomedical applications, especially for catalyst or cancer target therapy [92]. We remind the reader that the standard chemotherapeutic drugs which are injected to the blood circulatory system suffer from a low control of drug release and, correspondingly, they often cause serious damage to not only the cancer cells but also to healthy tissues [93,94]. In contrast, targeted drug delivery enables to reduce the toxicity and notably improve the drugs' effectiveness [95]. For these purposes, many different types of magnetic nanoparticles have been prepared and studied as possible magnetically guided drug nanocarriers [95–100]. Among them, iron-oxide nanoparticles, particularly Fe_2O_3 (maghemite) and Fe_3O_4 (magnetite) or their combination, are the most considered ones. This is mainly due to the fact that they have a lower toxicity and higher biocompatibility than other magnetic nanoparticles (e.g., ZnFe_2O_4) [99]. It is noteworthy that the iron-oxide nanoparticles exhibit a magnetic behavior in response to the applied external field [100]. As such, the supermagnetic iron oxide nanoparticles have been also considered as a tracer in nuclear medicine [101]. However, in order for these magnetic nanoparticles to reach the desired functionalities, that is, the correct interpretation of the data obtained by for example MRI, their magnetic properties must be evaluated [102]. MFM enables to disentangle tip forces such as van der Waals and magnetic interactions; therefore, it is widely used for the characterization of magnetic nanoparticles [103]. Note that the nanoparticles are commonly prepared by the various synthetic methods [104,105] and, as such, their non-uniform stress and shape together with lattice defects and crystal orientation may generate magnetic anisotropy [105]. Nocera et al. demonstrated magnetic anisotropy measurement on a single superparamagnetic nanoparticle with diameter of ~20 nm by means of MFM [106]. More recently, the characterization of the superparamagnetic systems of magnetite with diameter below 20 nm embedded in the silicon dioxide matrix by MFM was demonstrated in a study by Fuentes-Garcia et al. [107]. Similarly, the programmable domains aerogel-based magnets comprising of the ferromagnetic nanoparticles to achieve the magnetic anisotropy was fabricated and, correspondingly, the magnetization in the single domain of these prepared "magnetic" structures was evaluated by MFM [108]. We emphasize here that the achievable magnetic domains programmability can find an application in, for example, soft robotics for drug delivery [109]. The resolution in the nanoparticles' magnetic moment measurement of 10^{-18} A m² was demonstrated by Sievers et al. [110]. It has been shown that by increasing the tip–substrate distance above the superparamagnetic nanoparticle enables to achieve a 10 nm resolution, that is, an increase in the tip–substrate distance causes the reduction of the electrostatic force in a positive phase shift, whereas the magnetic force yields a negative phase shift [111]. Arajua et al. demonstrated the capability of a nonstandard, that is, cylindrical current sheet model, to study the magnetic properties of various nanoparticles [112]. This new method has been proven to be highly suitable particularly for the low-mass samples. MFM can also be used to study not only the various magnetic nanoparticles [113] but also the Fe_3O_4 nanowires with DNA cores [114].

5. Conclusions

With current progress in medicine and nanotechnology, the magnetic force microscopy has become a necessary tool to characterize and study various magnetic nanoparticles. In this context, there is ongoing progress in the sensitivity and resolution improvement of the magnetic force microscopy used to study the various nanoparticles. In summary, in this review article, we briefly summarize the principles and the engineering application of MFM. We particularly discuss the recent needs for characterization of the superparamagnetic nanoparticles that show promise in the target therapy. We envisage that the next generation of magnetic force microscopy microscopes will enable to characterize the various biological

samples with sizes ranging from tens of nm to μm and in even a native environment, i.e., physiological fluids—a goal that is still beyond the majority of current microscopes.

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