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Magnetic force microscopy

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Contents

Abstract. Principles of the operation of the magnetic force micro scope are considered. The main areas of application of magnetic force microscopy are characterised. The prospects are shown of using this method for solving problems of materials science, including the most topical problem, *viz*., **the creation of magnetic recording media with ultrahigh recording density. Unique potentials for investigation of nanostructures using a combination of magnetic force microscopy with electron spin resonance and nuclear magnetic resonance are pointed out. Primary attention is focused on the description of experimental techniques. The bibliography includes 27 references**.

I. Introduction

The interest in magnetic force microscopy is due to its unique potential, which makes it possible to solve the problems of both basic and applied investigation, *e.g*., the development of modern nanotechnologies. In chemistry, magnetic force microscopy is indispensable in the studies of the morphology, structure and properties of nanocomposites with magnetic inclusions. Magnetic force microscopy is of particular significance in the studies of magnetic properties of nanostructures, in particular, in the search for size and quantum effects. Magnetic force microscopy combines a modern technique of magnetic measurements with the unique potentials of probe microscopy.

The first probe microscopes, *viz*., scanning tunnelling microscope, made it possible to investigate conducting surfaces and to perform their targeted modification on an atomic and molecular level.[1] This type of microscope has become an ancestor of a family of probe microscopes, among which the atomic force microscope [2] and its modifications [3] are most often used.

In a scanning probe microscope there is a microprobe (a tip) with the help of which the precision mechanical system scans the surface. Simultaneously, the control unit of the microscope detects specified characteristics of the interaction between the probe and the surface under study. This can be the tunnelling current between the metal tip and the surface of a conductor in the case of the tunnelling microscope and the force of the interaction between the sharpened tip made of a hard material and the surface of the sample for an atomic force microscope. The atomic force microscope is in essence a sensitive profilometer, *i.e*., an instrument for measuring the roughness and topography of the surface, and in a sense it can be considered as a 'distant relative' of a phonograph with a diamond tip.

Tunnelling and atomic force microscopes have a spatial resolution better than 1 Å, *i.e*., these make it possible to observe individual atoms and molecules on the surface of various materials. The images of different surfaces obtained using scanning tunnelling and atomic force microscopes [4] are shown in Figs 1 and 2 as examples.

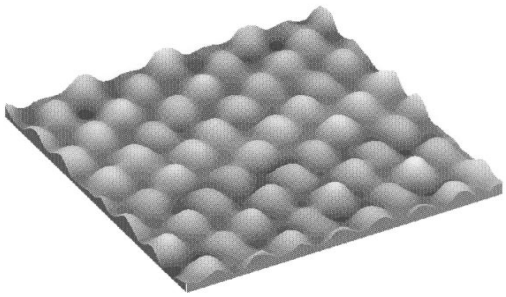

Figure 1. The surface of pyrolytic graphite.
The size of the area scanned is 2×2 nm^2; a 'Scan-8' scanning tunnelling microscope developed by the 'Advanced Technologies Centre' (Moscow, Russian Federation) was used.

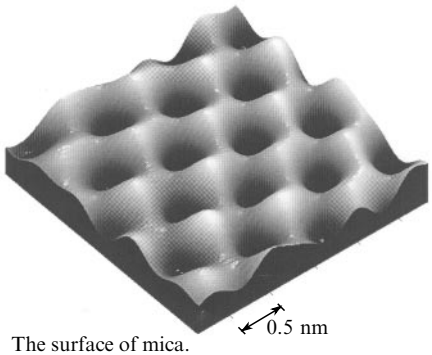

Figure 2. The surface of mica.
An NanoScope atomic force microscope (Digital Instruments, USA) was used.

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Martin and Wickramasinghe[5] have improved the atomic force microscope in order to investigate magnetic properties of the surface with submicron spatial resolution. It was proposed to use a microneedle made of ferromagnetic material as a probing tip for measuring the magnetic force experienced by this micromagnet in the vicinity of the surface of a magnetic specimen. This modification of the atomic force microscope is called the magnetic force microscope. Further stages in the development of magnetic force microscopy have been repeatedly documented (see, *e.g.*, Grütter *et al.*[6]).

Let us consider specific features of the operation of the atomic force and magnetic force microscopes in detail. The highest (atomic or molecular) spatial resolution is achieved if the atomic force microscope operates in the contact mode where the tip contacts the surface of the specimen under study. The resolution achieved in different 'tapping' modes is, as a rule, somewhat worse than in the preceding case. However, stable atomic resolution can also be achieved in the 'tapping' mode, *viz.*, in ultrahigh vacuum.[7] In the 'tapping' mode, a springy microconsole (a cantilever) on which the tip is mounted oscillates at a resonant frequency in the vicinity of the surface of the specimen. A cantilever is a mechanical resonator similar to a miniature tuning-fork. As the cantilever approaches the surface, the force action of the surface can cause additional damping of the oscillations of the cantilever. Under conventional laboratory conditions, the viscosity of air and the presence of adsorbed water film on the surface negatively affect the accuracy of measurements. Measurements *in vacuo* are free of these perturbing factors, which makes it possible to maintain the force of the interaction between the probe and the sample at a minimum level and thus to improve the spatial resolution up to the atomic level.[8]

In the contact-free mode, the forces acting on the tip of the atomic force microscope are due to the van der Waals interaction.

In the magnetic force microscope, the tip experiences an additional action of magnetic forces. As the tip is raised above the surface to a height of 10 to 50 nm, the universal van der Waals attraction almost completely vanishes and the tip is mainly affected by magnetic forces. In this case, it is the magnetic interaction that causes the deviation of the tip from rectilinear motion (Fig. 3). Because of its small size, the tip of the magnetic force microscope can be approximated by a magnetic dipole. The force F acting on the tip is determined by the following relationship

$$F = m_0 \mathrm{grad} H\,, \tag{1}$$

where m_0 is the magnetic moment of the tip and H is the magnetic field strength.

A magnetic microinclusion in the specimen will produce a magnetic field, the strength of which at a distance R is

$$H(R) = \frac{3r(\mathbf{r}m) - m}{R^3}\,, \tag{2}$$

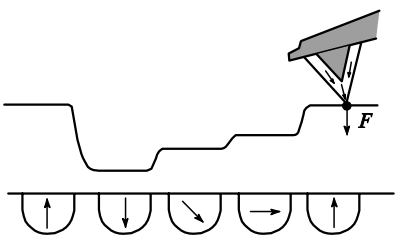

Figure 3. A diagram illustrating the action of a magnetic force on the microtip of the magnetic force microscope. The orientation of magnetic domains on the surface of the sample is shown by arrows in the bottom part.

where $\mathbf{r}$ is the unit radius-vector along the specified direction and m is the magnetic moment of the microinclusion.

Taking into account Eqns (1) and (2), the force of the interaction between the microscope tip and the magnetic microinclusion is

$$F = \mathrm{grad}\left[\frac{3(\mathbf{r}m)^2 - mm_0}{R^3}\right]. \tag{3}$$

Relation (3) can be used for approximate calculations. Let us assume that both dipole moments have the same vertical orientation along the direction of the z axis. Then the force of the interaction between them is

$$F_z = -\frac{6m_0 m}{z^4}\,,$$

and the force field gradient is

$$\frac{\mathrm{d}F_z}{\mathrm{d}z} = \frac{24m_0 m}{z^5}\,.$$

For instance, for two iron microparticles with diameters 10 nm ($m_0 = m \simeq 9\times10^{-29}$ A m^2) located at a distance of 10 nm, the magnetic force of the interaction is $\sim 4.9\times10^{-11}$ N and the force gradient is $\sim 1.9\times10^{-2}$ N m^{-1}. It is this order of magnitude of the values that are detected by the magnetic force microscope.

Comparative analysis of different methods used to study magnetic properties of the surface shows that magnetic force microscopy is highly sensitive to the magnetic flux (at a level of 10^{-4} T) and makes it possible to achieve a unique spatial resolution as compared to that obtained by other methods of magnetic measurements.[9] The sensitivity of the magnetic force microscope to the magnetic flux is only slightly lower than that of the SQUID-based scanning microscope.[10]

II. The principle of operation of magnetic force microscope

A block diagram of the mechanical part of a magnetic force microscope is shown in Fig. 4*a*. The specimen is mounted on a piezoscanner, which provides spatial movement of the specimen along the three coordinate axes. Usually, the piezoscanner is a thin-walled tube made of piezoceramics coated with a system of metal electrodes. The electric voltage applied to the electrodes changes the geometric size of the tube because of the inverse piezoelectric effect. The piezoscanner is characterised by a high

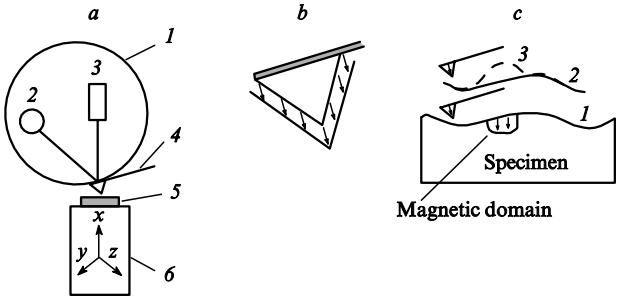

Figure 4. A block diagram of the mechanical system of a magnetic force microscope (*a*), the cantilever tip coated with a ferromagnetic material (*b*) and the trajectory of the motion of the microtip (*c*);
(*a*): (*1*) small displacement; (*2*) photodetector; (*3*) laser; (*4*) cantilever; (*5*) specimen; and (*6*) piezoscanner; (*c*): (*1*) trajectory in the course of recording the surface profile; (*2*) trajectory in the course of recording the magnetic profile; and (*3*) deviations from the selected trajectory caused by the interaction between the microtip and a magnetic domain in the specimen.

mechanical rigidity and, hence, it is insensitive to seismic and acoustic interferences, simple to control and has a long lifetime. It is these advantages that have determined wide use of this design of the piezoscanner in actual microscopes. Errors of movements, which are due to, *e.g.*, nonlinearity of the properties of the material of the piezoscanners are the drawbacks of this type of piezoscanner. However, the errors of the movement of the object under study can be reduced by software control, *viz.*, by applying a controlling voltage to the electrodes of the piezoscanner in accordance with special correcting algorithms. In metrological systems, additional capacitive or optical transducers are used; they provide the linear movement of the specimen in the range from 1 to 250 μm in the plane of the specimen (along the x and y coordinates) and from 1 to 15 μm normal to this plane (along the z coordinate) for different piezoscanners.

In a magnetic force microscope, the cantilever is above the specimen. The magnetic force F acting on the tip causes a bending of the cantilever and a vertical movement of the tip (Fig. 4 *b*). According to Hooke's law, this movement is determined by the mechanical rigidity of the cantilever (by the spring constant) lying typically in the range from 0.1 to 10 N m^{-1}.

Bending of the cantilever is detected using a small displacement transducer. Among different transducers (capacitive, inductive, tunnelling, *etc.*), optical sensors have found the widest practical use. They detect angular deflection of the light beam reflected from the surface of the cantilever. A laser beam is focused on the reflecting surface of the free (unfastened) end of the cantilever and the changed position of the reflected beam, indicating a bending of the cantilever, is determined by a split photodiode.

When scanning the specimens under actual experimental conditions (the relative displacement of the probe and the surface under study), the probe successively scans each surface area and the electronics detects the total force of the interaction in the probe–sample system. The results of measurements, displayed after processing of the data, are three-dimensional (3D) images of the surface. If the bending of the cantilever is maintained constant during the scanning process, the images obtained correspond to the surfaces of constant force. This mode is called a'constant force' mode. In the 'deflection mode' the cantilever moves in the horizontal plane above the specimen and the bending of the cantilever is detected, which is proportional to the force acting on the tip.

When scanning a rough surface, the contribution from the topography should be separated from that of the magnetic forces. To this end, the tip scans the same surface area twice. For the first pass, it moves contacting the surface of the specimen and the trajectory of the movement of the tip, which corresponds to the profile of the surface under study, is stored in a computer. The magnetic properties of the specimen (neglecting the surface deformations, which are small, as a rule) have no effect on the observed trajectory. For the second pass, the microconsole moves along the known trajectory above the same surface area without contacting the specimen. In this case, the tip is affected by long-range forces rather than by contact forces, as in the first case. The deviation of the tip from the specified trajectory depends on the magnetic properties of the specimen (Fig. 4 *c*). In actual experiments, to achieve the maximum sensitivity, oscillations are induced of the cantilever at a natural resonant frequency and the tip also scans the specimen twice, *viz.*, first in the 'tapping' mode and then in the free oscillations mode at some distance from the surface. Recording the amplitude, phase or frequency of the oscillations provides more precise information on the magnetic inclusions (domains, clusters) in the specimen under study.

III. Areas of application of magnetic force microscopy

Magnetic force microscopy is used in the creation of materials for magnetic recording media [magnetic tapes, hard ('Winchester') disks, magneto-optical disks,[11, 12] *etc.*[13]], in the optimisation of the recording modes of magnetic heads,[14] in the studies of the structure and properties of nanoparticles, alloys,[15] nanocomposites and thin films,[16] in the development of the methods for magnetic recording with ultrahigh density,[17] in the studies of superconductors as well as in biological and geophysical studies. This makes it possible to observe single magnetic domains whose sizes vary from several nanometres to several tens of nanometres.

For instance, the formation of Ni particles on a quartz glass (silicon dioxide) resulting from autocoalescence of an 'island' Ni film upon annealing (at 800 °C) in an atmosphere of hydrogen was studied.[18] Using magnetic force microscopy, it was shown that particles with sizes from 40 to 100 nm are single domain particles (Fig. 5), which agrees well with theoretical estimates.

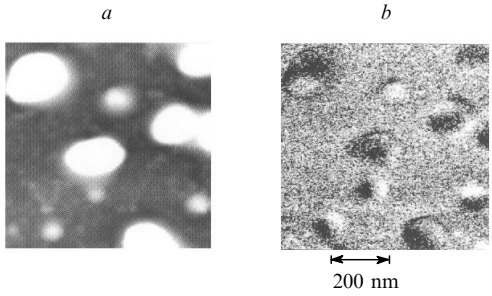

Figure 5. A microtopographical (*a*) and the corresponding magnetic (*b*) image of Ni particles.

Magnetic force microscopy has found an important application in the development of novel methods for magnetic recording with ultrahigh density: the leading manufacturers of magnetic recording media make wide use of commercial magnetic force microscopes in the steps of design and quality control of finished products.

In Fig. 6, a topographical profile of a fragment of a surface of a magneto-optical disk (the dark lines seen are rectilinear microrecesses, Fig. 6 *a*) and a magnetic image of the microstructure of magnetised domains on the surface are shown. Elongated islands of dimensions 2×1 μm^2 corresponding to recording of one data bit (Fig. 6 *b*) are fairly clearly seen. The domain structure of the magnetic film in the area of recesses (light longitudinal strips) is also visible. Magnetic force microscopy makes it possible to distinguish single grains of size 50 nm. The magnetic structure of the surface is intensively studied in research laboratories of the leading manufacturers of recording media in order to minimise the surface area corresponding to one data bit and to solve the problems of noise immunity, noise reduction during the recording and reproduction of the data. The results of these investigations are used in the development of novel principles and devices for magnetic and magneto-optical recording. We note that it is the use

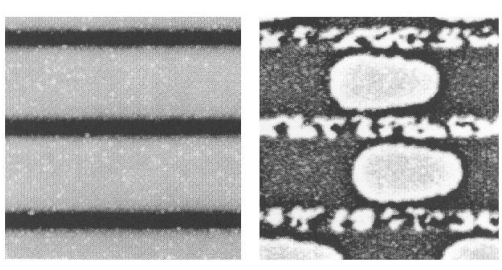

Figure 6. The surface of a disk of size 5×5 μm^2 consisting of microtracks separated by longitudinal recesses (*a*) and the magnetic structure of the same surface (*b*).

of modern methods of investigation such as magnetic force microscopy that made it possible to provide a virtually exponential increase in the magnetic recording density on magnetic and magneto-optical disks in recent years. Currently, the storage capacity of hard disks can be as high as 20 Gbytes and even more.

In Fig. 7, a magnetic 'image' is shown of lithographically patterned Co metal particles.[19] The procedure of patterning of metal 'islands' is used in the technology of production of magnetic media with digital type of data recording. Using a magnetic force microscope, it is possible to 'see' the domain structure of individual clusters (Fig. 7 *a*). Smaller magnetic clusters (Fig. 7 *b*) look like single magnetic dipoles.

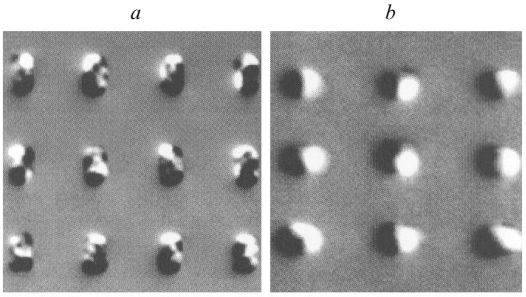

Figure 7. Image of metallic Co 'islets' obtained using a magnetic force microscope.
The 'islet' area: (*a*) 200×400 nm^2; (*b*) 1.7×1.7 μm^2.

The magnetic force microscope can be used not only for the observation (mapping) of the magnetic field, but also for magnetic recording with ultrahigh density. In Fig. 8, the projections correspond to the magnetic domains formed on the surface of a TbFeCo film using the microscope tip. These domains are formed at an instant when a certain threshold value of the strength of the magnetic field produced by an external magnet and the tip is achieved. The threshold magnetic field strength is determined by the coercivity of the magnetic film. The magnetised domains decrease in size and gradually disappear as the net magnetic field decreases successively. In this case the recording density is 10 Gbit cm^{-2}, which is substantially higher than that achieved for hard ('Winchester') disks and magneto-optical disks used in modern computers.

In Figs 9 *a* and 9 *b*, a magnetic image of a hard disk and the result of averaging of the magnetic image in the central surface area of the disk (between the dashed lines in Fig. 9 *a*) over a large number of cross-sections are shown, respectively. Figure 9 *c* represents a signal in the reading magnetic head that had scanned the same surface area of the disk. The curves shown in Figs 9 *b* and 9 *c* are identical even in minor details, which indicates that the signals detected by the magnetic force microscope and conventional magnetic head coincide. The magnetic force microscope provides a much higher spatial resolution, which makes it possible to use this instrument in the detailed analysis and optimisation of read – write modes of standard magnetic heads.

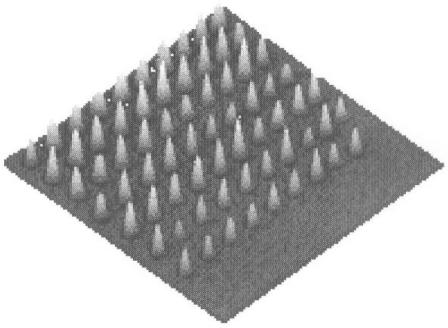

Figure 8. Single magnetic domains (a bit array) formed on the surface of TbFeCo carrier with the aid of the tip of a magnetic force microscope in the presence of external magnetic field. The same tip was used to obtain the magnetic image. The surface area scanned is 12×12 μm^2.

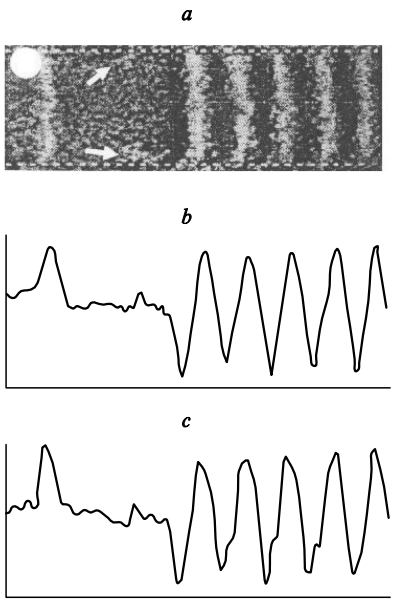

Figure 9. Image of a track of a magnetic disk obtained using a magnetic force microscope (*a*), averaged cross-section of the magnetic profile in the area between the dashed lines (*b*) and the signal in the reading magnetic head detected in the same area of the track (*c*).

The use of magnetic force microscopy in the studies of magnetic properties of biological objects is also promising. Micromagnets play a significant role not only in artificial systems, but also in nature. For instance, bacteria *Aquaspirillum magnetotacticum* can move along the magnetic field lines. These bacteria contain specific organelles, *viz*, magnetosomes. The magnetosomes are chains of 10 to 25 permanent magnets, *viz*., iron oxide crystals of size ~ 50 nm. Single crystals are clearly seen in nonstained cells using transmission electron microscopy. Magnets inside the cells determine the specified direction of motion of the bacteria in sea-water: to the north in the Northern hemisphere and to the south in the South hemisphere. It has been possible to measure the magnetic moment of a single bacterium *Aquaspirillum magnetotacticum* using a magnetic force microscope.[20] The length of the bacterium was 2 μm and the measured magnetic moment was negligibly small, *viz*., 10^{-16} A m^2.

Currently, 'Digital Instruments' and 'Park Scientific Instruments' companies (both from USA) produce magnetic force microscopes for use both in industry and scientific research. In the 'Centre of advanced technologies' (Moscow, Russian Federation), a promising model of scanning probe microscope, 'Femto-Scan' has been developed, which permits measurements of magnetic properties of the surface.

Cantilevers for both magnetic force microscopy and atomic force microscopy are fabricated by microlithography using a silicon technology, which means that one or several cantilevers at a time are formed at the edges of a silicon plate. Then the surface of the tip is coated with a thin layer of a ferromagnetic material. A scheme of the components for the probe of a magnetic force microscope made by Park Scientific Instruments (USA) is shown in Fig. 10.

A problem of calibrating the magnetic force microscope appears in relation to metrological measurements of magnetic forces. This problem has successfully been solved by Kong and

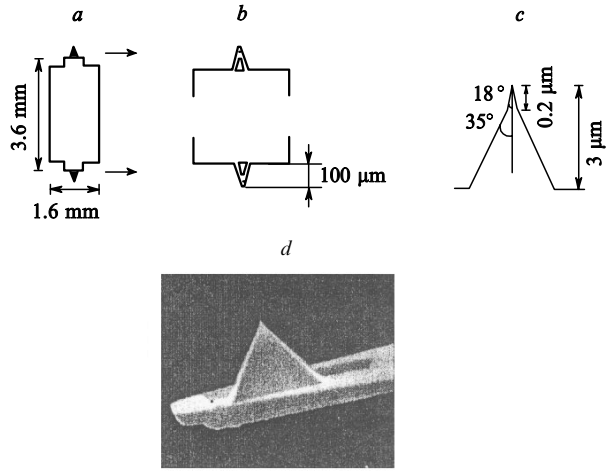

Figure 10. Scheme of manufacture of a cantilever with a tip ($a-c$) and the image of the tip mounted on a microconsole obtained using an electron microscope (d); (a) silicon plate holder with two cantilevers (b); (c) tip.

Chou,[21,22] who have used metal microrings. The calibrating rings with diameters of 1 and 5 μm (Fig. 11) were made using electron-beam lithography (metal strips on a silicon surface). The magnetic tip moved at variable distances above the rings and its magnetic moment was evaluated from the electric current induced in the rings. The axial magnetisation of the silicon tip coated with a 65-nm thick cobalt film measured using this procedure was 3.8×10^{-22} A m^2. Considerable attention is given to the procedure for fabrication of the tips, since it is the tips that largely determine the quality of the magnetic image of the surface.[23]

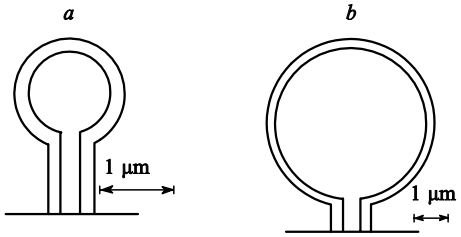

Figure 11. Microrings used for calibration of a magnetic force microscope.

IV. Magnetic-resonance force microscopy

In the year 1991, it was suggested that a magnetic force microscope can be used to obtain three-dimensional images of individual biomolecules.[24] To this end, it was proposed to combine the existing methods of nuclear magnetic resonance and magnetic force microscopy. The novel method has been called magnetic-resonance force microscopy.[25,26] Its major advantages are safety for the object under study (*i.e.*, it is a nondestructive method), a subnanometre spatial resolution and the possibility of studying individual biomolecules.

At present, magnetic-resonance force microscopic experiments are carried out as follows. A specimen under study of negligible mass ($\sim 10^{-11}$ kg) is mounted on a springy cantilever analogous to that used in atomic force microscopy (Fig. 12). A constant magnetic field in the specimen is produced by a magnet placed near the cantilever. The magnetic force from the side on which the tip is mounted, acting on the surface atoms of the specimen, is proportional to the spin of the unpaired electrons or to the magnetic moment of the nuclei. In an alternating magnetic

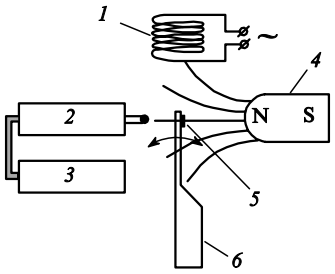

Figure 12. A block diagram of a magnetic-resonance force microscope; (*1*) magnetic coil; (*2*) fiber optic interferometer sensor; (*3*) detection electronics; (*4*) magnet; (*5*) specimen; (*6*) cantilever.

field, a quantum transition occurs between the energy sublevels of the electron or nucleus under the conditions of resonance, which causes a change in the orientation of the magnetic moment of the system. By periodically changing the frequency of the alternating magnetic field produced by an additional coil, one can modulate the magnetisation of the sample. The modulation frequency is chosen to be equal to the resonant frequency of the cantilever. The amplitude of the forced oscillations of the cantilever is measured with a small displacement transducer (*e.g.*, a fiber interferometer). For a chosen geometry of experiment, the conditions of nuclear and electron spin resonances in the system are held in the region of a specimen of subnanometre size.

The method of magnetic-resonance force microscopy has nontrivial possibilities for the structure determination of proteins and viruses. The results of the first successful experiments on the observation of electron paramagnetic resonance are documented.[27] Spin resonance in a sample of diphenylpicrylhydrazyl of mass 30 ng was detected. The force acting on the cantilever was 10^{-14} N. According to an assessment made, the observation of resonance from a single molecule requires the enhancement of sensitivity to force at least by 4 orders of magnitude. Recently, the construction of a microcantilever of length 230 μm and thickness of only 60 nm with a micromagnet mounted atop was reported.† The magnetic field of the micromagnet interacts with the magnetic moments of the atoms of a specimen. The magnetisation of the specimen is varied periodically (as in the preceding case) to achieve the conditions of electron (paramagnetic) or nuclear magnetic resonance. The force measured was 6×10^{-18} N.

A description of early successful experiments on the observation of electron spin resonance using the magnetic force microscope is given in Ref. 27. The strength of the constant magnetic field is modulated, which changes the magnetisation of the specimen under conditions of spin resonance (Fig. 13). During the modulation period, the system will be at resonance twice and, in the case of sufficient strength of the alternating magnetic field, the absorption of energy reaches its maximum. This leads to the levelling of the populations of magnetic energy sublevels corresponding to the quantum numbers lying in the range from $-m$ to $+m$ and to the decrease in the magnetisation down to zero. To eliminate interference, the signal is detected at a frequency of the second-harmonic component of the modulation signal. The calculated and experimental spectra (Fig. 14) virtually coincide.

In early experiments on the observation of 3D images, a 2-μm particle of diphenylpicrylhydrazyl was mounted on a cantilever in a constant nonuniform magnetic field produced by a conical tip made of NdFeB. The magnetic field gradients were 4.3 and 0.94 G μm^{-1} normal to the specimen and in the longitudinal direction, respectively. The transverse and longitudinal spatial resolutions were 1.2 and 5.3 μm, respectively. A 3D image of the sample with a resolution of $\sim$ 3 μm was obtained in the observa-

† The report was posted in January, 1997 at the URL http://www.aip.org/physnews

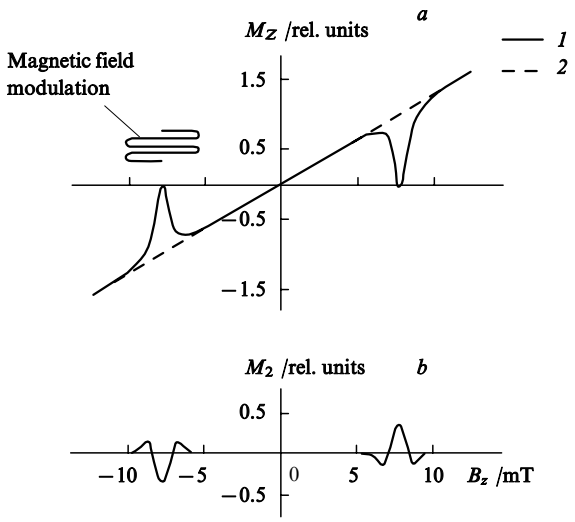

Figure 13. Dependences of the magnetisation of a specimen (*a*) and the amplitude of the magnetisation signal measured at the frequency of the second-harmonic component (*b*) as functions of the constant magnetic field strength.
In the presence (*1*) and in the absence (*2*) of high-frequency field.

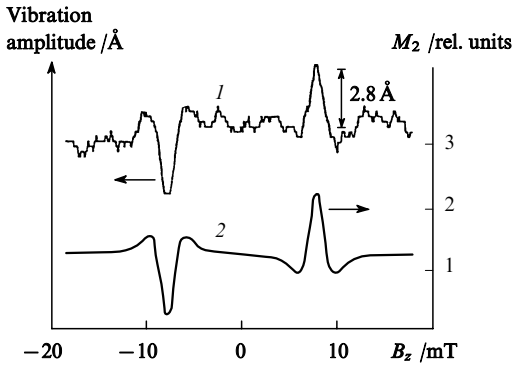

Figure 14. Experimental (*1*) and calculated (*2*) electron spin resonance spectra obtained using a magnetic-resonance force microscope.

tion of NMR in ammonium nitrate at a field gradient of 22 G μm^{-1}.

Production of a constant magnetic field with the highest uniformity in the bulk of the entire specimen is the principal condition that should be met in the classical ESR and NMR methods. On the contrary, in the case of magnetic-resonance force microscopy the higher the nonuniformity of the constant magnetic field, the smaller the spatial region in which the resonance condition hold and, hence, the higher the spatial resolution obtained when constructing 3*D* images.

V. Conclusion

The material presented in this review shows that the investigations in the field of magnetic force microscopy are being intensively developed in the last decade. This method is used in both fundamental science and industry. Of particular interest and fruitfulness are the results of application of the magnetic force microscopy in the creation of magnetic media that make it possible to perform magnetic recording with ultrahigh density.

The latest studies have shown that combination of magnetic force microscopy and traditional methods of electron paramagnetic resonance and nuclear magnetic resonance opens unique potentials for the investigation of nanostructures including biomolecules.

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