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Simultaneous Micromechanical and Electromagnetic Detection of Electron Paramagnetic Resonance

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The peculiar advantages of simultaneous observation by electromagnetic and micromechanical methods in EPR spectroscopy are discussed. The development of a novel apparatus with the capability of this simultaneous detection is described. Experiments at 23 GHz show the performance of the apparatus. The problems related to the sensitivity and to the spatial resolution are analyzed. Future prospects are presented. © 1999 Academic Press

Key Words: **EPR spectroscopy; micromechanical detection; spatial resolution spectroscopy; high fields.**

I. INTRODUCTION

Recent methodological advances in the field of magnetic resonance involved remarkable efforts to improve the sensitivity and to expand applications of this spectroscopy. In this context the possibility of using improved detection schemes plays a leading role. New techniques involving micromechanical detection of resonance signals recently joined the standard electromagnetic detection method.

Mechanical detection of magnetic resonance was first proposed by Gozzini (*1*). The procedure was based on the measurement of macroscopic torque acting on a paramagnetic sample due to the absorption, at resonance, of the angular momentum of the photons of the electromagnetic field. The value of the torque T is given by the expression

$$T = N\hbar = \frac{P}{\omega}, \qquad [1]$$

where N is the number of transitions per unit time and P is the power absorbed by the sample at the angular frequency ω. References (*2, 3*) report experiments of electron paramagnetic resonance (EPR) performed with a sample of diphenylpicrylhydrazyl (DPPH) suspended to a quartz fiber to realize a sensitive torsion pendulum.

A more refined method involving mechanically detected magnetic resonance, recently suggested by Sidles (*4*), allowed a new microscopy technique potentially capable of obtaining three-dimensional images giving the local electronic or nuclear spin density of the sample with very high spatial resolution to be developed. The basic experimental set up implemented by Rugar and co-workers (*5–8*) implied a spin-containing sample put on the free end of a cantilever, of the kind used on atomic force microscopy, and placed in a magnetic field gradient. The sample, due to the magnetization induced by the magnetic field, interacting with the inhomogeneous magnetic field, experiences a force in the direction of the field gradient. This force causes the deflection of the cantilever; variations of the magnetization change this deflection. Magnetic resonance phenomena can be so detected by observing the deflection of the cantilever. In general a modulation of the process at the mechanical resonance frequency of the cantilever will produce a sensitivity enhancement.

The main feature of this method of detection is the ability to achieve information from a small and localized amount of spins due to the use of the magnetic field gradient as an essential element of the technique. Contributions to the signal from the different zones of the sample, each in the presence of the field value locally resonant with the irradiating frequency, are drawn out by sweeping the static magnetic field value or by varying the sample position. Experiments performed up to now showed that by means of this technique the sensitivity and the spatial resolution of conventional magnetic resonance imaging can be improved (*8*).

Recently a different configuration was carried out, based on an inverted scheme, where the cantilever, equipped with a magnetic tip, supplies the field gradient and acts, at the same time, as the local sensor of the magnetic force scanning the sample along the three axes (*9*).

The ideas drawn from Gozzini's and Sidles' procedures recently merged in a scheme where a mechanical oscillator formed by a microcantilever was used in EPR experiments to detect magnetic resonance by the torque induced by angular momentum of absorbed photons (*10*). Different experimental conditions were tested and sensitivity was compared with respect to that of the force detection method.

The present work concerns EPR experiments based on the

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development of a spectrometer, working at about 23 GHz, equipped for the detection of the resonance signal simultaneously by means of both standard electromagnetic and micromechanical methods. The main features of the apparatus are as follows:

an almost standard spectrometer configuration with capabilities for the detection of resonance signals by the usual electromagnetic technique with the addition of only a special probe-head for micromechanical detection;

the use of a novel probe-head for this application, based on a dielectric resonator working in a whispering gallery resonance mode (WGM) configuration; the "open" structure of this devices allowed a cantilever with the samples to be placed very close to the resonator in order to sense the electromagnetic (em) field of the resonator and to use a modified optical apparatus based on the optical lever method for detection of the cantilever movements inside an electromagnet;

experiments at high frequencies (≈23 GHz) in order to exploit the increased sensitivity expected for higher magnetic fields.

Section II reports the main features of WGM dielectric resonators showing that the em field distribution is useful for the insertion of the samples supported by the cantilever; the same section reports the experimental measurements of these distributions in the presence of the cantilever supporting the sample. Special emphasis is given to the procedures for coupling the resonator to the microwave circuitry.

Section III contains an analysis of the overall spectrometer devised to obtain magnetic resonance signals by both electromagnetic and micromechanical ways simultaneously.

Section IV reports the experimental observations of electron paramagnetic resonance signals of samples obtained with the different techniques; measurements obtained with both torque and force micromechanical methods are reported.

Section V is especially devoted to the analysis of the sensitivity achieved with the different detection schemes and possible developments and perspectives are discussed.

II. STUDY OF WHISPERING GALLERY RESONATORS

The study of a suitable microwave resonator is a focal point when an apparatus with the micromechanical detection of EPR signals must be implemented. The cantilever, supporting the paramagnetic sample, must be placed in a location with a very intense electromagnetic field connected to the resonator itself without any significant modification of the field distribution; in addition the device must be useful for the optical detection of the movements of the cantilever related to the magnetic resonance processes. As the standard metallic cavity resonators cannot be easily used for the above purposes, Rugar and co-workers realized a microstrip resonator working at a frequency of 12 GHz (*11*).

Whispering gallery dielectric resonators have been used in the past few years for EPR applications (*12–14*). These resonators have several appealing characteristics that can be summarized here.

The WG resonance in a dielectric cylinder with radius r and finite length (i.e., a disc) has an intrinsically traveling wave character where the resonance condition is roughly given by the relation

$$2\pi r \approx n\lambda, \qquad [2]$$

n being an integer number and λ the wavelength of the em radiation. The energy flows circularly near the rim of the disc; the different oscillation modes are characterized by three modal indexes $\{l\ m\ n\}$, where l is the axial, m the radial, and n the azimuthal mode index (*15*). The energy is confined in the resonator by the phenomenon of the total reflection at the surfaces of the disc; the fields outside the resonator in the proximity of the surface have an evanescent trend. Two families of modes can be excited, the quasi TE (transverse electrical field) WGE modes and the quasi TM (transverse magnetic field) WGH modes;

WG dielectric resonators present a large multiplicity of resonances (corresponding at different values of n) and can operate in very broad spectral ranges (from microwaves to the optical range). Probe-heads based on WG dielectric resonators have been already built and used at hundreds of gigahertz in high-field EPR experiments (*16*);

The coupling of the em radiation can be easily performed by partially superimposing the evanescent field of the resonator to that of a feeding dielectric circular waveguide; a proper choice of the size and materials allows a controlled coupling of the radiation to the resonator. The undercoupling, critical coupling, and overcoupling conditions can all be obtained simply by changing the distance between the rim of the disc and the cylindrical waveguide (*15*);

The quality factor Q is roughly related only to the intrinsic losses of the material forming the resonator and very high Q values can be obtained in very broad frequency ranges.

By summarizing, all of the above characteristics and the open structure suggest that the WG dielectric resonators are good candidates for applications of paramagnetic resonance spectroscopy with micromechanical detection.

The resonator used in the apparatus described here was a cylinder of alumina, 25.3 mm in diameter and 4 mm in thickness. The coupling configuration was the reaction (also called absorption) configuration and involved a fused quartz dielectric circular waveguide with a diameter of 8 mm reduced to about 2.5 mm in the zone near the disc resonator in order to achieve a good coupling condition (*15*).

A mapping of the field spatial distribution was carried out in order to locate the position where the sample could be favorably placed and to test the effects produced by the presence of the cantilever. Data were collected by using as a pick-up

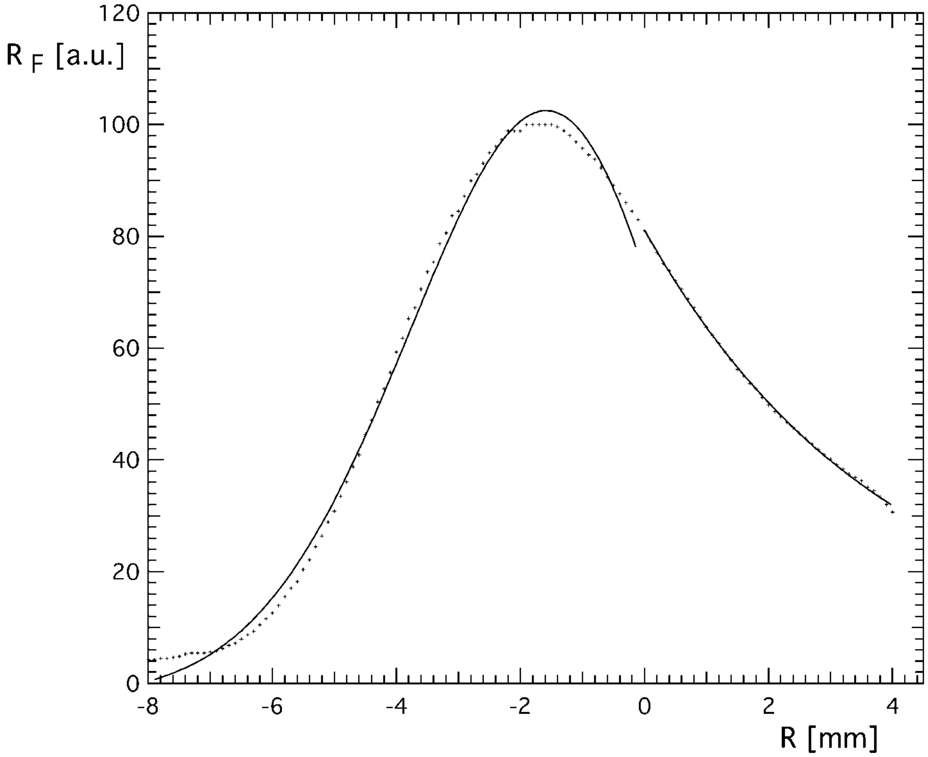

FIG. 1. Values (dots) of the field intensity distribution R_F measured in the radial direction at the WG mode dielectric resonator; the zero of the radial coordinate x was fixed at the rim of the resonator. The continuous line represents the fit of the experimental data with the expressions for the fields inside and outside the resonator.

antenna a dielectric rod properly tapered in order to obtain a satisfactory directivity (*15*) and to scan the coordinate of interest on the surface of resonator.

Figure 1 shows the measured values (dots) for the radial trend of the field intensity R_f of a WGH mode of the resonator; the zero of the x axis was fixed at the rim of the resonator; consequently the negative values of the coordinate refer to the surface of the resonator and positive values correspond to the free space outside. During these measurements the cantilever was fixed in close proximity to the circular surface of the disc. The continuous line represents the fit of the experimental data with the expression for the fields given in (*15*); in particular the exponential decrease of the evanescent field outside the rim of the resonator can be noted. Figure 2 reports the field distribution in the axial direction obtained by sensing the intensity of the radiation at the rim of the resonator. The zero of the z axis refers to the central plane of the resonator, therefore the upper and lower plane surfaces are labeled by the $+/-2$-mm coordinates. The continuous line represents once more the fit of measured data with the function $[\cos(hz)]^2$, where h is a propagation constant and z the axial coordinate, for values of z in the range $+/-2$ mm and with an exponentially decreasing function for z values exceeding the above range (*15*).

The agreement between the measured and calculated values shows that the presence of the device required for micromechanical detection does not affect the field distribution. Thus the sample supported by the cantilever can be placed at the rim of the resonator in the zone of maximum intensity of the microwave field corresponding to the mean plane perpendicular to the axial direction.

The measurements of the quality factor Q of the resonator gave values of about 5000 for a WGH mode and values of about 9000 for a WGE mode. The use of a cantilever made with silicon nitride prevented additional possible effects related to losses of the material.

III. EXPERIMENTAL SET UP

The overall experimental apparatus is schematically shown in Fig. 3. The microwave radiation is generated at about 23 GHz by a reflex Klystron Varian Model EM1188V and controlled by a variable attenuator Waveline Model 812DR. The power, through a proper adapter, is transmitted to the cylindric circular quartz waveguide used for exciting the WG resonator in the reaction (absorption) configuration; a second identical adapter couples the power to the metallic waveguide toward the diode detector (Krytar Model 703BK). In this configuration the detected power has a minimum at the resonance frequency. An automatic frequency control (AFC) system locks the frequency of the klystron to the resonance frequency of the

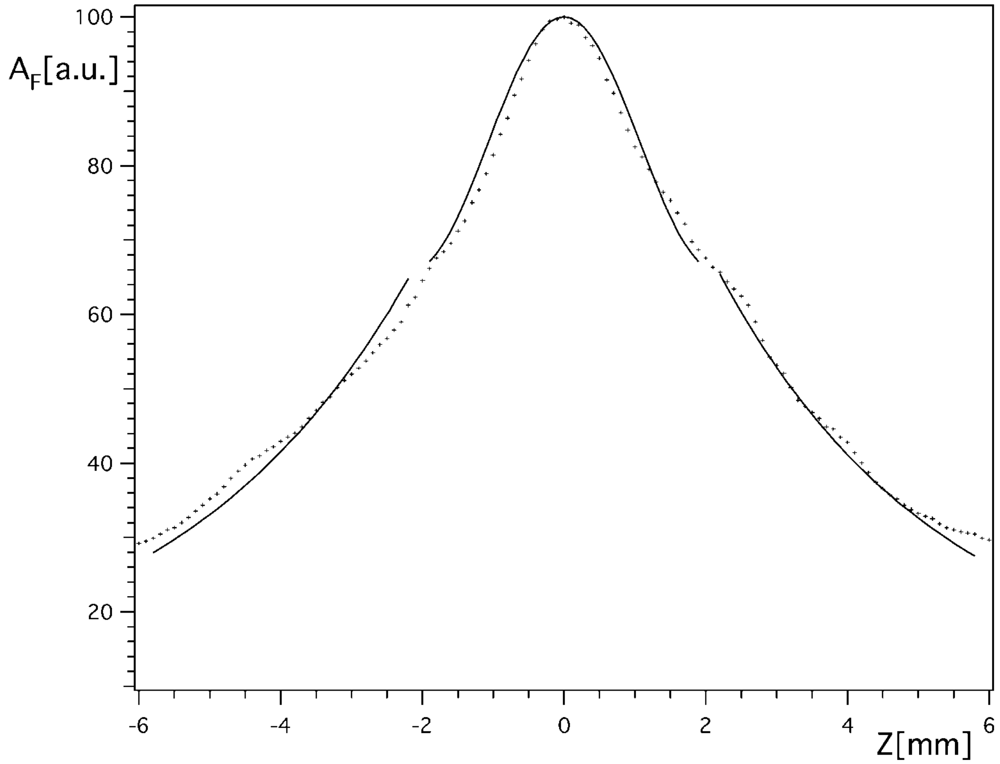

FIG. 2. Values (dots) of the field intensity distribution A_F measured in the axial direction at the rim of the WG mode dielectric resonator; the zero of the axial coordinate z refers to the central plane of the resonator. The continuous line represents the fit of the experimental data with the expressions for the fields inside and outside the resonator.

dielectric resonator. This frequency is measured by a frequency meter EIP Model 588C. The magnetic resonance signal is detected by a lock-in amplifier EG&G Model 5302 by using standard field modulation techniques.

The static magnetic field used for producing the spin polarization of the sample is supplied by an electromagnet able to giving a maximum field of about 1.8 T. Special attention must be paid to the reciprocal orientation of the static and microwave magnetic fields and to that of the long axis of cantilever integral with the sample. When a WGH mode is used, the disc resonator is placed with the axis parallel to the static field. The cantilever orientation depends on the selected micromechanical detection technique; when the torque method is used the plane of movement of the cantilever is perpendicular to the static magnetic field (*10*). On the contrary the force detection requires that the motion plane of the cantilever is parallel to the static magnetic field.

The detection of the cantilever movement was performed by means of an optical apparatus based on the optical lever method. This method uses a laser source and a four-quadrant differential photocell. The main feature of the optical detection apparatus in the application reported here was the ability to work with the laser source and the differential photocells remotely located with respect to the cantilever. In this way the problems arising when the optical and mechanical components are placed into the electromagnet could be avoided. The block scheme is shown in Fig. 4: a laser beam, 800 μm in diameter, generated by a He–Ne laser (Melles Griot, Model 05LHP151), was forwarded through a 8× beam expander and then focused, with a diameter of about 20 μm, on the bottom side of the cantilever by means of a convergent lens with 25-cm focal length. The beam reflected by the cantilever was then refocused on the photocells with a convergent lens with 10 cm focal length. In the case of the torque detection (left-hand side of Fig. 4) the laser beam directly reaches the arm of the cantilever; in the case of force detection (right-hand side of Fig. 4), since the cantilever is rotated by $\pi/2$, a mirror tilted by 45° with respect to the light propagation direction is used to focus the beam on the arm of the cantilever.

The experimental set up for force detection includes a magnetic field gradient generator. This device is carryed out by an iron cylinder with a diameter of 1 mm moved in the immediate vicinity of the sample by an accurate three-axis micropositioning mechanism.

The amplitude of the cantilever motion, and thus the sensitivity, was increased by modulating the intensity of the microwave field at the mechanical resonance frequency of the cantilever and using a phase detection technique for the signal. This was obtained by forcing the klystron with a square wave with large amplitude; in this way during a half period of this

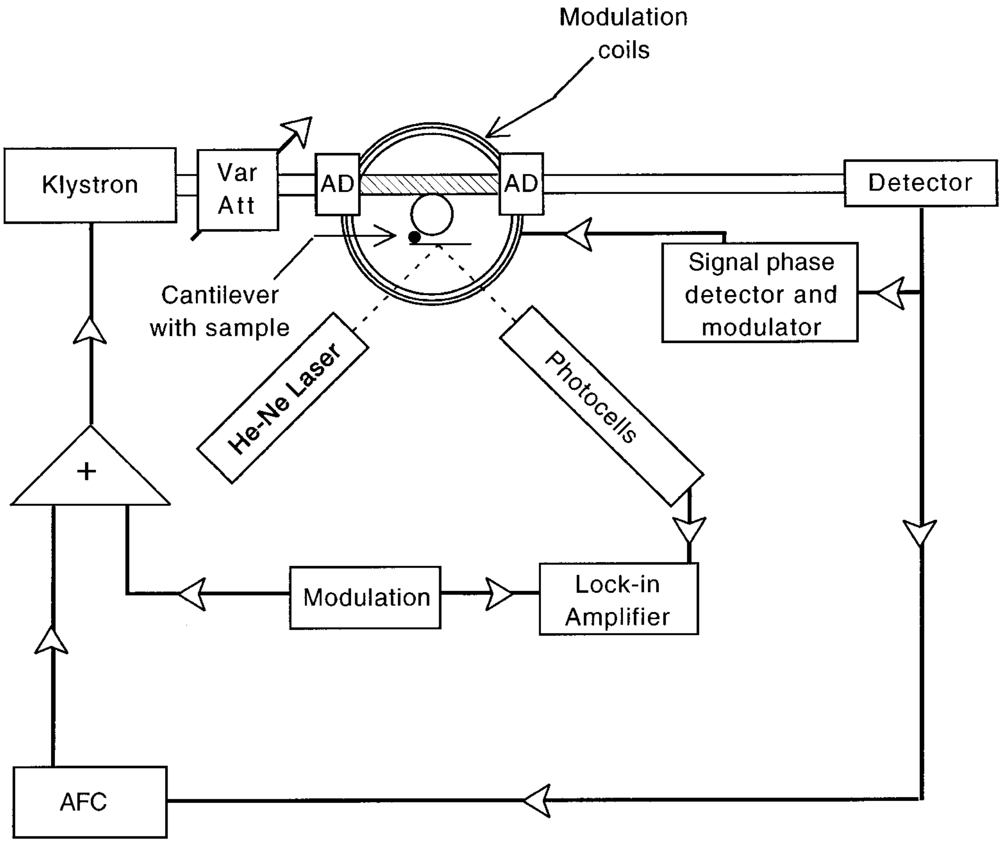

FIG. 3. Block scheme of the overall experimental apparatus.

square wave the klystron is locked to the resonance frequency of the resonator, and during the other half period it is pushed out of the response curve of the resonator, and the sample is subjected to a 100% modulation of the power producing magnetic resonance. Special care must be taken so that AFC operation is not affected by the above modulation procedure; this is obtained by regulating properly the AFC time constant in comparison with the modulation frequency. The same amplitude modulation can also be obtained by using a PIN diode switch inserted in the main arm of the microwave path.

The mechanical resonance frequency of the cantilever charged with the sample was measured accurately by studying

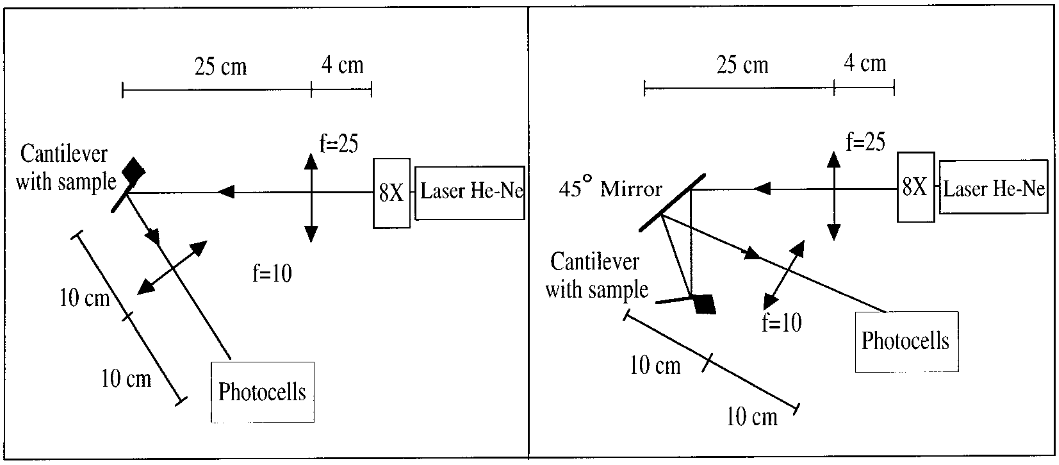

FIG. 4. Block schemes of the optical apparatus used for the detection of oscillations of the cantilever in the case of torque detection (left) and of force detection (right).

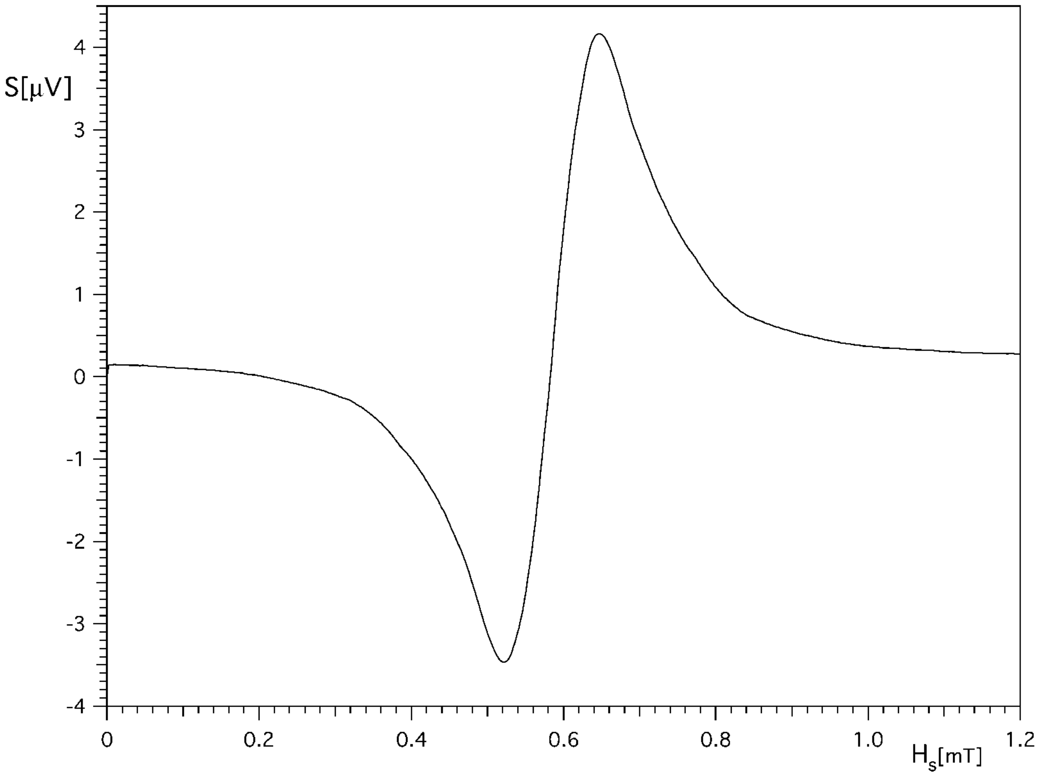

FIG. 5. Magnetic resonance signal S of a microsample of DPPH detected in the electromagnetic mode. The sample was positioned on the cantilever.

the frequency dependence of the noise by means of a FFT spectrum analyzer Stanford Research Model SR760.

All data relevant to the experiments are collected by a computer system that, through a standard interface system, controls the essential parameters.

Measurements have been performed on a paramagnetic sample of DPPH glued with epoxy resin to the tip of a silicon nitride AFM cantilever 320 μm in length, 0.6 μm in thickness, supplied by PARK Scientific Instrument. Typical samples of DPPH were spheroids with a diameter of about 100 μm. The cantilever was placed very close to the curved surface of the WG resonator.

It must be noted finally that, owing to the field distribution on the surface of the resonator, two nearly equal perpendicular components of the microwave magnetic field are present in the sample site and therefore the sample is submitted to an essentially circularly polarized field; the resonance signal accordingly changes strongly when the field or the microwave propagation directions is inverted.

IV. OBSERVATIONS OF ELECTRON PARAMAGNETIC RESONANCE

A. Electromagnetic Detection

Experiments have been performed at 23 GHz. The signal, in the electromagnetic mode, was phase detected by modulating the static field by means of a pair of coils in Helmholtz position.

The magnetic field sweep was accurately calibrated by varying in a controlled way the resonance frequency of the WG resonator by means of a conducting ring displaced axially near one of the plane surfaces of the resonator (*17*).

The cantilever with the sample was kept in a fixed position in air and at room temperature.

Figure 5 shows the resonance signal S of a sample of DPPH detected with the apparatus in the electromagnetic mode. The static field sweep (H_s) of 1.2 mT through the resonance was carried out in 65 s, the time constant of the lock-in amplifier being 500 ms, and the spectrum was averaged two times. The observed half height linewidth of the resonance curve is about 0.2 mT.

B. Torque Detection

Figure 6 shows the EPR signal T_s of a sample of DPPH in the torque mode. The sample had a mass of a fraction of a microgram; the cantilever charged with the sample had a mechanical resonance frequency of 1.695 kHz with a quality factor of the order of 30. The magnetic field sweep (H_s) of 1.6 mT was carried out in 1000 s, the detection time constant being 10 s, and the spectrum was not averaged. The half height linewidth gets out, as in the case of Fig. 5, about 0.2 mT, so confirming the condition far from saturation of the sample.

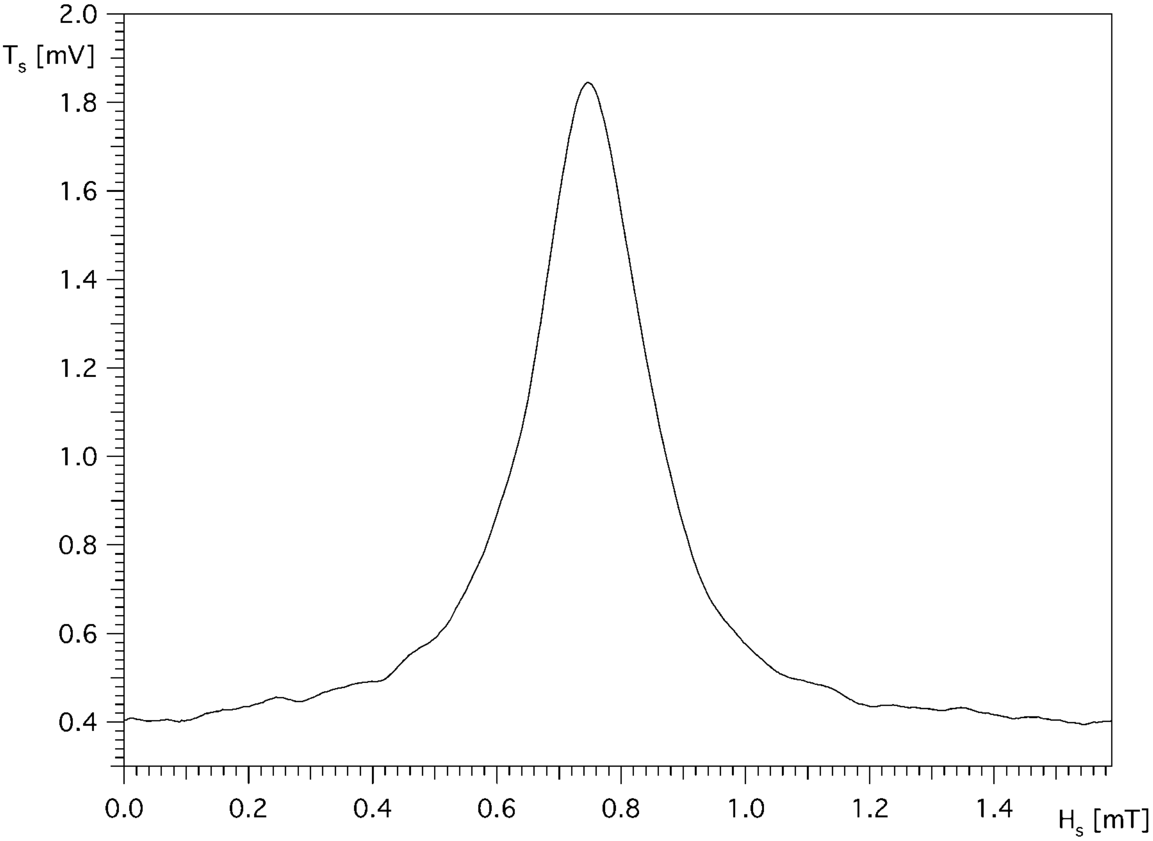

FIG. 6. Magnetic resonance signal T_s of a microsample of DPPH detected in the torque mode.

The peculiar behavior of the torque signal when experiments involving the inversion of the magnetic field are carried out, as in the case reported in Ref. (*10*), must be pointed out; owing to the circular polarization of the microwave field mentioned above, the inversion of the magnetic field forces the inversion of the direction of microwave propagation. This implies the

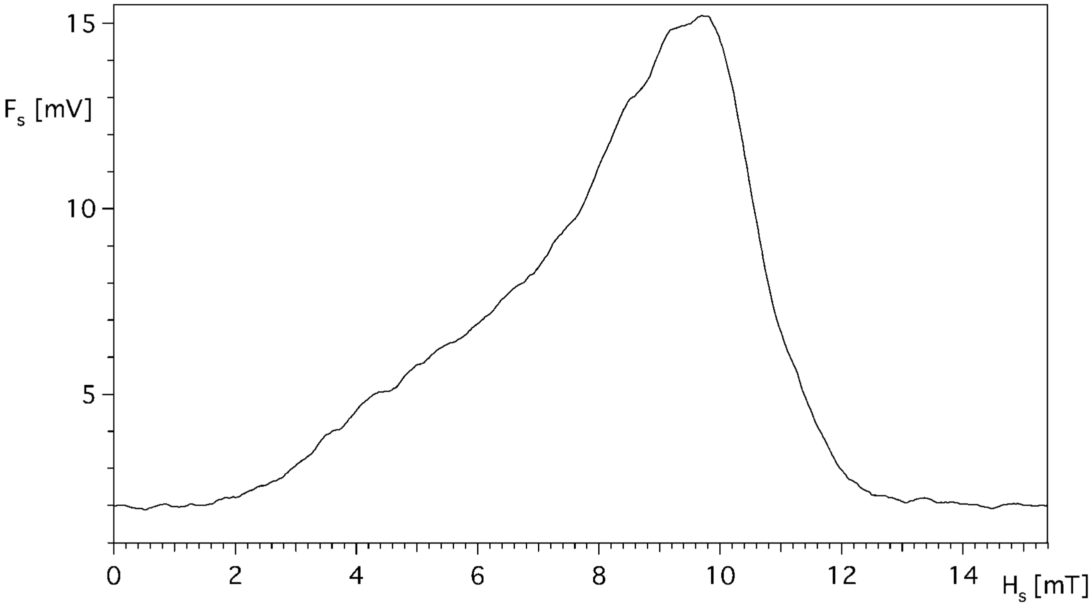

FIG. 7. Magnetic resonance signal F_s of a microsample of DPPH detected in the force mode. The calculated field gradient value was about 100 T/m.

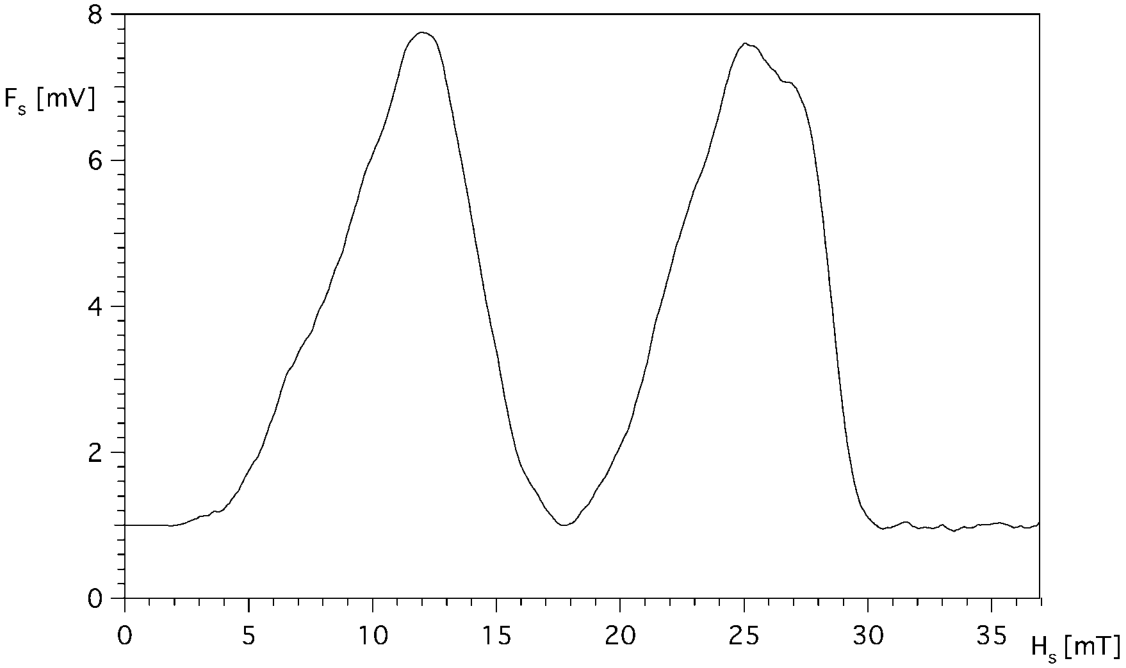

FIG. 8. Magnetic resonance signal F_s of a microsample of DPPH detected in the force mode. The sample is distributed in two spatially separated spheroids. The field gradient, parallel to the axis connecting the centers of the two spheroids, had a calculated value of 180 T/m.

absorption of photons with opposite angular momentum with respect to the previous case. Consequently the motion of the cantilever presents a π phase change and the detected signal is inverted.

C. Force Detection and Spatial Resolution

Experiments in the force mode imply in general a broadened lineshape, owing to the use of the field gradient generator as an essential part for the detection of spectra. Figure 7 shows the spectrum of a small sample of DPPH detected in the force mode. The observed linewidth is about 10 mT. It must be pointed out that the possibility of measuring simultaneously the characteristics of the unperturbed EPR signals of the sample in the operation position in the electromagnetic mode is essential in order to use, in the deconvolution procedures employed for determining the spatial spin density distribution, the right lineshape. This is particularly important when spectra which are not already well known are studied and/or when the saturated conditions must be controlled.

In the case of Fig. 7, since the observed linewidth is exceedingly large in comparison with the unperturbed linewidth due to the use of a relatively intense field gradient, the line profile resembles the sample shape in the direction of the field gradient. The accurate knowledge of the field sweep and of the sample size allowed us to calculate the field gradient value that in the case of Fig. 7 was about 100 T/m. The spatial resolution obtained in this condition was about 2 μm.

Figure 8 shows the EPR signal in the force mode of a microsample of DPPH with a particular spatial distribution. The sample is distributed in two spheroids placed in opposite sides of the cantilever arm with an additional spacer made of a pyrex slide with a thickness of about 20 μm. A microphotograph of the sample and the cantilever is shown in Fig. 9. The field gradient was along the axis connecting the centers of the two spheroids. Also in this case the observed lineshape resembles the spatial sample distribution. The calculated field gradient value is 180 T/m and the spatial resolution is about 1.2 μm.

The value of the field gradient and consequently of the spatial resolution can be further increased by putting the iron cylinder generating the gradient closer to the sample.

V. SENSITIVITY CONSIDERATIONS AND PERSPECTIVES

One of the aims of the magnetic resonance force microscopy (MRFM) technique, here implemented in the force detection mode, is the increase of sensitivity in terms of the minimum number of spins detectable. It is evident that the concept of sensitivity in the electromagnetic mode discussed here is not directly comparable with the one considered in the case of standard EPR spectrometers. In fact, the samples of interest for MRFM have in general microscopic sizes and they are inserted in resonant structures only in order to have high field intensity at the sample site. In this way the filling factor, which plays a dominant role in obtaining high sensitivities in the standard

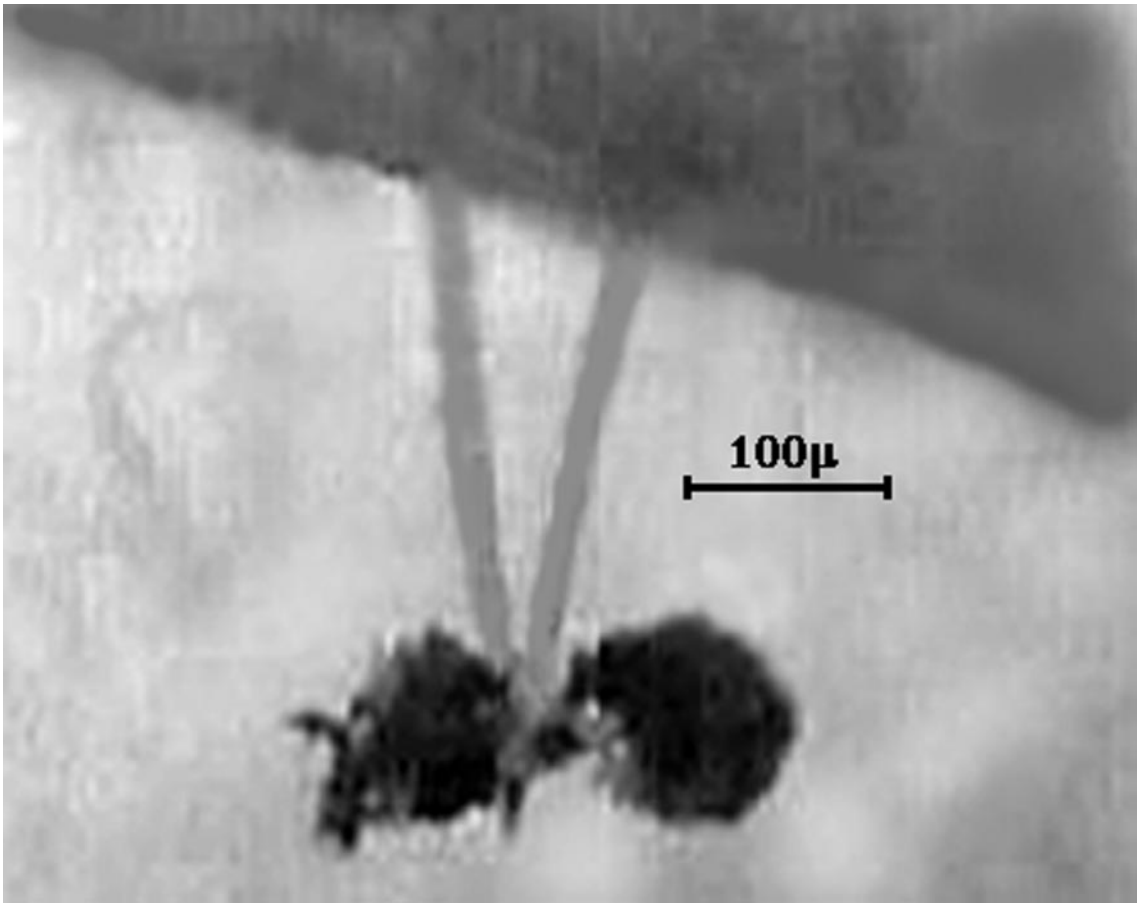

FIG. 9. Microphotograph of the sample and the cantilever used for observing the signal reported in Fig. 8.

EPR instrumentation, is always near zero. The part of the apparatus used for the electromagnetic mode was implemented for the purposes discussed above and it was not completely optimized with respect to sensitivity. The sensitivity in the electromagnetic mode measured in the presence of the device generating the field gradient was about 2×10^{13} spins/0.1 mT. As far as the sensitivity in the force mode is concerned, the value measured from the spectrum reported in Fig. 8 was about 10^{11} spins/0.1 mT. It must be noted that in the case of the force mode the signal increases as the field gradient is increased; consequently, the sensitivity in terms of the signal-to-noise ratio must increase. If, however, the field gradient becomes so large as to produce a broadening of the spectral linewidth, the trend of the maximum of the signal S_{max} flattens. On the contrary the sensitivity $S^{\mathrm{i}}_{\mathrm{max}}/n$, where the signal is $S^{\mathrm{i}}_{\mathrm{max}}$ integrated on the broadened linewidth continues its growth. This trend is shown in Fig. 10, where the sensitivity in the force mode is measured for a sample of DPPH as a function of the field gradient value changed by varying the distance d between the magnetic cylinder and the sample. The crosses represent the values of the signal-to-noise ratio S_{max}/n as observed; the circles represent the quantity $S^{\mathrm{i}}_{\mathrm{max}}/n$ corresponding to the signal integrated on the broadened linewidth.

Improvements to the performance of the apparatus can derive from the operation of the cantilever with the sample under vacuum and at low temperature. These improvements are in progress also by exploiting the good characteristics of the dielectric materials forming the microwave resonator and the coupling waveguide. In addition, a crucial point is the possibility of performing MRFM spectroscopy at high field/frequency; this allows the enhancement of the sensitivity by the increase of the sample magnetization and by the possibility of using higher field gradient values (*18*). The experimental scheme discussed in the present paper is particularly useful for this purpose since dielectric resonators and related circuitry identical to the one described above have already shown excellent characteristics when used as sample resonators for high field–high frequency EPR applications up to about 400 GHz (*16*).

Finally it can be noted that the method and apparatus presented here could be particularly useful for the introduction of a novel capability of standard EPR instrumentation; it could be sufficient to introduce the microwave network formed by the switch and the probe-head including the micromechanical oscillator in the path of commercial spectrometers in place of standard cavities, thereby implementing an apparatus where

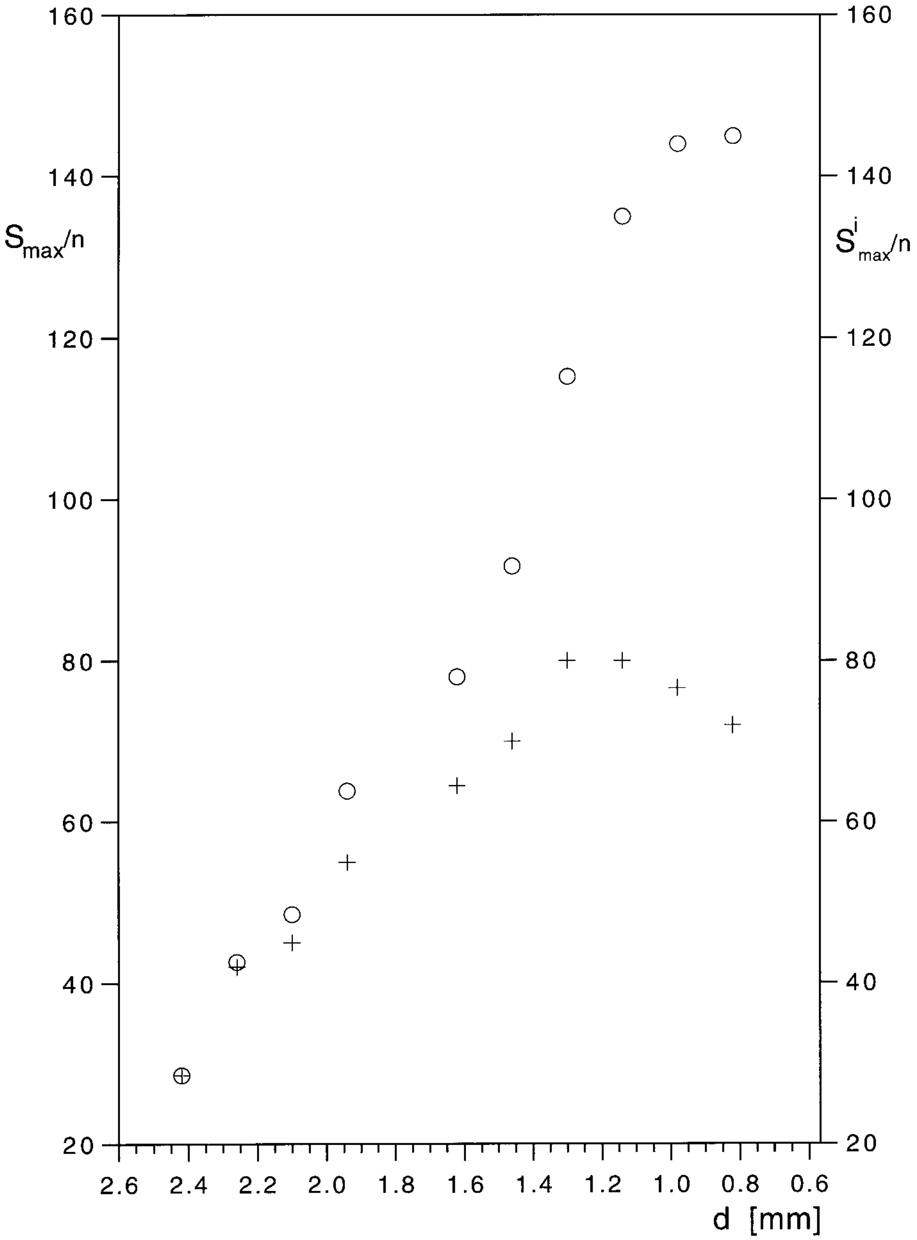

FIG. 10. Trend of the sensitivity in the force mode measured for a microsample of DPPH versus the field gradient changed by varying the distance d between the magnetic cylinder and the sample. The crosses represent the values of the signal-to-noise ratio S_{max}/n as observed; the circles represent the signal-to-noise ratio S^{i}_{max}/n calculated in terms of the signal integrated on the broadened observed linewidth.

micromechanical detection and spatial resolution become an “option.”

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