LA-UR-99- 2728

96213

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Submitted to: DOE Office of Scientific and Technical Information (OSTI)

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Magnetic Resonance Force Microscope Development

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Abstract

Our objectives were to develop the Magnetic Resonance Force Microscope (MRFM) into an instrument capable of scientific studies of buried structures in technologically and scientifically important electronic materials such as magnetic multilayer materials. This work resulted in the successful demonstration of MRFM-detected ferromagnetic resonance (FMR) as a microscopic characterization tool for thin magnetic films. Strong FMR spectra obtained from microscopic Co thin films (500 and 1000 angstroms thick and 40 x 200 microns in lateral extent) allowed us to observe variations in sample inhomogeneity and magnetic anisotropy field. We demonstrated lateral imaging in microscopic FMR for the first time using a novel approach employing a spatially selective local field generated by a small magnetically polarized spherical crystallite of yttrium iron garnet. These successful applications of the MRFM in materials studies provided the basis for our successful proposal to DOE/BES to employ the MRFM in studies of buried interfaces in magnetic materials.

Background and Research Objectives

Two of the most important advances in imaging have been Atomic Force Microscopy (AFM) and Magnetic Resonance Imaging (MRI). AFM has enabled atomic scale resolution imaging of surfaces and material on surfaces. This technology has had a very significant impact on a broad range of areas ranging from materials to molecular systems to biological studies. The primary limitation of AFM and other high resolution scanning probe microscopies (such as scanning tunneling microscopy and magnetic force microscopy) is their restriction to surface studies. Sub-surface information is gained indirectly by inference from information obtained at the surface. In contrast MRI is a powerful, fully three-dimensional, non-invasive imaging technology. MRI employs a spatial variation in the applied magnetic field (applied magnetic field gradient ∇B) to distinguish magnetic resonance signals arising from different spatial locations. The magnitude of the field

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gradient and the intrinsic linewidth ΔH of the magnetic resonance signal determine spatial resolution: $\Delta z = \Delta H/\nabla B$. Although it is possible to generate very large field gradients, spatial resolution in traditional, inductively detected MRI is limited by sensitivity. Increasing the field gradient decreases the size of the volume element under study to the point where the resultant signal is insufficient to be detected with adequate signal to noise ratio. This limits the spatial resolution to tens or hundreds of microns and thus reduces the effectiveness of MRI in studies of many condensed matter and biological systems. Recently, a novel proposal [1] was made to detect magnetic resonance signals mechanically by sensitively measuring the force $F = (m \bullet \nabla)B$ between a permanent magnet and the spin magnetization. This is done by measuring changes in the oscillation amplitude of low spring constant micro-mechanical resonators (cantilevers or bridges) such as are used presently in AFM. The magnetic resonance force microscope (MRFM) exploits this approach to produce a microscopic imaging instrument, which combines the strengths of MRI and AFM.

This microscope will allow imaging and characterization of materials to depths varying from tenths of microns to several hundred microns depending on the resolution desired. The micro-mechanical resonator with a small permanent magnet attached will be scanned across the surface of the material under study. Through the coupling $F = (m \bullet \nabla)B$ between the spin magnetization m and a magnetic field gradient ∇B of the permanent magnet, the spin magnetization of the sample exerts a force on the microcantilever. As in MRI the magnetic resonance frequency, f, of the spin moment is proportional to the applied field B: $f = \gamma B$. The spatial variation of the applied field produces a correspondence between location in the sample and f. The rf field is used to eliminate the polarization of *only* those spins with the matching resonance frequency f. Periodically suppressing the spin magnetization at the characteristic frequency of the cantilever drives it into oscillation.

The important point is that the rf field can *only* modify the orientation of the spin magnetization *within this selected volume* and so only those spin moments are resonantly coupled to the cantilever. This amplifies sensitivity to these spins by the quality factor Q of the resonator (several thousand to over a million). The magnetization of neighboring volumes produces a time-independent force that is easily filtered out. The oscillation amplitude of the micro-cantilever is proportional to the magnitude of the periodic force on the cantilever, and so to the spin magnetization within the selected volume. As the microscope is scanned, the variation of oscillation amplitude with position will map out magnetization density. Furthermore, once a microscopic volume of spins is selected, the well developed array of magnetic resonance tools that have been developed over 50 years of magnetic resonance research in materials can be applied.

Importance to LANL's Science and Technology Base and National R&D Needs

MRFM is unique among high-resolution probes in that one may select a well-defined *sub-surface* volume for study. As in MRI, the measurement is sensitive only to a particular volume of sample, which is selected by the application of a magnetic field gradient (arising in this case from the inhomogeneous field of the small permanent magnet). The advantage of mechanical detection of magnetic resonance is the much higher sensitivity it promises. The resolution of conventional magnetic resonance imaging is limited by the sensitivity of the detection: the volume defined for study must contain a sufficient number of spins to provide adequate signal to noise ratios. Current conventional (inductive) detection limits are on the order of $\sim 10^{15}$ nuclear spins, whereas the theoretical limit for mechanical detection is a single nucleon. Sensitivity an order of magnitude better than this was obtained in the *first* demonstration of mechanically detected nuclear magnetic resonance [2]. This increased sensitivity is the key to obtaining high spatial resolution. The ability to select a *sub-surface* volume for study with such high resolution is unique and likely to have unprecedented impact on a variety of scientific and technological areas including materials characterization and processing, biological structure, and pharmaceuticals development to name a few. Our focus, however, was on the application of MRFM to studies of layered electronic materials.

Rapid improvements in the ability to fabricate multi-layer materials with accurate control over layer dimensions and interface quality is at the foundation of the development of a range of important modern micro-electronic and magneto-electronic devices. We focused our efforts on the application of the emerging MRFM technology to the study of buried interfaces and layers in multilayer giant magnetoresistance (GMR) devices. The technological importance of these materials is clear if one recognizes that the magnetic recording industry compares to the semiconductor industry in size. As magnetic storage densities increase, inductive read heads will be entirely replaced by GMR read heads. The performance and characteristics these devices are very sensitive to microscopic details of the interfaces and to microstructure within the layers. Yet, there exist no high-resolution real-space probes of sub-surface structure. The development of characterization capabilities and instruments for these devices will have substantial impact on the magnetic recording industry, speeding research and development and possibly enabling fabrication line characterization of devices for purposes of process monitoring.

Our demonstration (described below) that MRFM/FMR can determine microscopic magnetic properties of these materials provides the basis for a new approach to understanding and optimizing these important materials.

Scientific Approach and Accomplishments

1. Ferromagnetic Resonance in a Microscopic Thin Film Magnet (presented in publication 2). We observed a ferromagnetic resonance signal arising from a microscopic (~20μm X 40μm) particle of thin (3μm) yttrium iron garnet film using MRFM (Figure 1). The large signal intensity in the resonance spectra indicates the potential for MRFM as a powerful microscopic ferromagnetic resonance technique with a micron or sub-micron resolution. We also observed a very strong non-resonance signal that occurs in the field regime where the sample magnetization readily reorients in response to the modulation of the magnetic field. This signal will be the main noise source in applications where a magnet is mounted on the cantilever.

2. Experimental Determination of Sensitivity and Spatial Resolution in Electron Spin Resonance-MRFM (presented in publication 3). The signal intensity in MRFM experiments on small samples is determined by four parameters, the rf field H_1, the modulation level of the bias field, the spin relaxation time and the "magnetic size" (dB/dz)R of the sample. Calculations of the MRFM spectra were performed for various conditions. The results were compared with experimental data on a DPPH particle and excellent agreement was found. The systematic variation of the signal intensity as a function of the rf and modulation fields provides a powerful tool to characterize the experimental setup.

3. Novel Coil Design (presented in publication 4). We devised a novel radio-frequency coil design based on the Alderman-Grant coil and applied it in the MRFM. The rf field of the so-called Modified Alderman-Grant Coil (MAGC) has a magnitude comparable to that of a solenoidal coil of similar size (for the same input power) but the coil has a much smaller inductance. This is advantageous in electron spin resonance MRFM experiments that would benefit from rf frequencies in excess of 1000 MHz. The open design of the MAGC is also advantageous because it provides superior access to the sample mounted on a mechanical cantilever by the optical fiber and permanent magnet and so allows the sample to be placed at the center of the coil.

4. Magnet on Detector. The FMR experiment reported in Item 1 above was the first published MRFM experiment performed with a magnet on the detector. This demonstrated that large spurious forces arising from forces between the large permanent moment of the magnet and the applied time-dependent forces produce a cantilever response that mimics a genuine MRFM signal. Unfortunately these signals can be orders of magnitude larger than the desired MRFM signal. An example of this (labeled "NR mode") is evident in Figure 2. A detailed study of the consequences of placing a magnet on the sensitive force detector is reported in Publications 5 and 6. We analyzed the spurious detector response arising from interactions between miniature, magnetically polarized $Nd_2Fe_{14}B$ particles and various external applied fields. The large magneto-crystalline anisotropy of $Nd_2Fe_{14}B$ prevents its magnetization from rotating easily to align with the applied magnetic field. As a consequence the interaction between the applied field and the moment of the polarized $Nd_2Fe_{14}B$ particle attempts to rotate the entire particle and exerts a torque on the force detector. This modifies the apparent restoring force of the mechanical resonator shifting its resonant frequency. Because this torque depends on the magnitude of the applied field, the resonant frequency of the detector becomes field dependent. In a high Q system (Q~10,000-80,000 for the present detectors), a very small frequency shift results in a very substantial shift in the sensitivity of the system at a given excitation frequency. This drift in the system sensitivity as field is swept is undesirable and must be eliminated.

Two solutions to this problem were presented in these of publications. The polarized $Nd_2Fe_{14}B$ particles show promise as magnetic probe tips if the particles can be glued to the detector in the apparatus and in the presence of the magnetic field used for making measurements. This will ensure that the moment of the magnetic probe is accurately parallel to the applied fields eliminating the torque responsible for the field-dependent frequency shift of the mechanical resonator. An alternative approach is to use soft magnetic materials with small crystalline anisotropy shaped into spheres. In this case the moment can easily reorient relative to the crystalline axes to align with the applied field, thus minimizing the field-dependent torque. In particular, spherical YIG particles are readily available and are promising candidates for magnetic probe tips.

5. First Demonstration of MRFM Detection of FMR in Co Thin Films (publication 8). The primary objective of this LDRD project was the development of the MRFM into an instrument useful for studies of scientifically and technologically important materials. The accomplishment of this objective was demonstrated in this publication, where we reported high sensitivity mechanical detection of ferromagnetic resonance signals from microscopic Co single-layer thin films using a MRFM.

Ferromagnetic resonance (FMR) is an important tool for characterizing magnetic materials [4,5]. It has played a particularly important role in studies of the magnetic multilayer systems that are becoming widely used in the recording industry as recording read heads and/or media. For example, FMR has been used to measure the dependence of a key parameter, the interlayer exchange coupling, on the thickness of the spacer layer separating the ferromagnetic layers. The ability to perform *microscopic* FMR would be extremely valuable, enabling characterization on a microscopic scale of distributions of magnetic anisotropy and exchange energies in magnetic devices.

Microscopic FMR cannot be performed using conventional techniques for two reasons. First, the sensitivity of conventional FMR is inadequate; for most magnetic thin films, such as Co and Fe, sample areas on the order of $(mm)^2$ are needed in order to obtain sufficient signal at X-band. Second, conventional FMR is performed in a uniform magnetic field so there is no means to identify the spatial origin of a particular contribution to the FMR signal.

A single crystal yttrium iron garnet (YIG) thin film was used in our previous demonstration of microscopic FMR using the MRFM (publication 1). YIG was chosen because it has very strong FMR intensity and narrow resonance linewidth (<1 Gauss). However, typical magnetic devices are composed of metallic ferromagnets such as Co and Fe. These have much larger FMR linewidths (of order 100 Gauss) and hence weaker signals, making the signals much harder to detect. It is also essential that the resonant field be substantially larger than the linewidth, hence that the irradiation frequency exceed ~2-3 GHz.

We successfully detected the FMR signal from a microscopic, single-layer Co thin film by means of an MRFM instrument. Irradiation at high frequency (~ 8 GHz in this case) and implementation of a novel MRFM geometry (the perpendicular geometry described in detail in publication 8) were essential features of the experiment. We further demonstrated the ability to determine materials properties from these microscopic FMR measurements. Variations in the magnetic anisotropy field and the inhomogeneity of the films were clearly observed in the FMR spectra of microscopic Co thin films 500 and 1000 angstroms thick and ~40 x 200 μm in lateral extent. This demonstrated the important potential that MRFM detection of FMR holds for microscopic characterization of spatial distribution of magnetic properties in magnetic layered materials and devices.

6. First Demonstration of MRFM/FMR Imaging (publication 7). Lateral one-dimensional imaging of cobalt (Co) films by means of microscopic FMR detected using the MRFM was demonstrated. Magnetic resonance imaging employs a magnetic field gradient to identify the spatial origin of a resonance signal. Through the magnetic resonance condition $f=\gamma B$ for a noninteracting spin (γ is the gyromagnetic ratio, and B is the applied field), an applied field gradient allows the spatial origin of the signal to be inferred from the resonance frequency. This relies on having the resonance frequency ω_0 as a *local* function, g,of applied field B; that is, $f(r)=g[B(r)]$. Because of strong dipole couplings to neighboring spins in a ferromagnet, the resonant frequency at a particular spatial location is non-local; that is, it is determined by the magnetization of neighboring regions in addition to the value of the field applied at that point. Thus, imaging by means of an applied field gradient is not as straightforward as in the case of non-interacting spins, such as occurs in NMR.

In fact, in earlier work we (publication 1) and Wago et al. [3], found that the linewidth of the FMR signal was unaffected by the applied field gradient indicating that the entire crystal was resonating a single frequency regardless of the variation of the field across the sample. Thus we chose to employ a novel approach in which we scanned a small, magnetically polarized spherical crystallite of ferromagnetic yttrium iron garnet. This produces a spatially localized field that shifts the resonant frequency of the material in the immediate vicinity of the magnetic probe.

Figure 3 shows FMR spectra of two microscopic patches of Co film and their dependence on the spatial position of the localized magnetic probe. Two patches of Co film were deposited through masks to define their location and size. The magnitude of the frequency shift depends on the location of the magnetic probe relative to the patches of Co, and the intensity of the signal indicates the volume of the sample brought into resonance at this higher value of local field. This is shown in Figure 4. We were able to resolve the ~20 micron lateral separation between the two Co films. We further showed that in the large field gradient produced by the small YIG sphere, the FMR resonance frequency could indeed be made to vary across the contiguous Co film. This demonstrates that magnetic resonance imaging will be possible using FMR if a sufficiently large applied field gradient can be applied.

Publications

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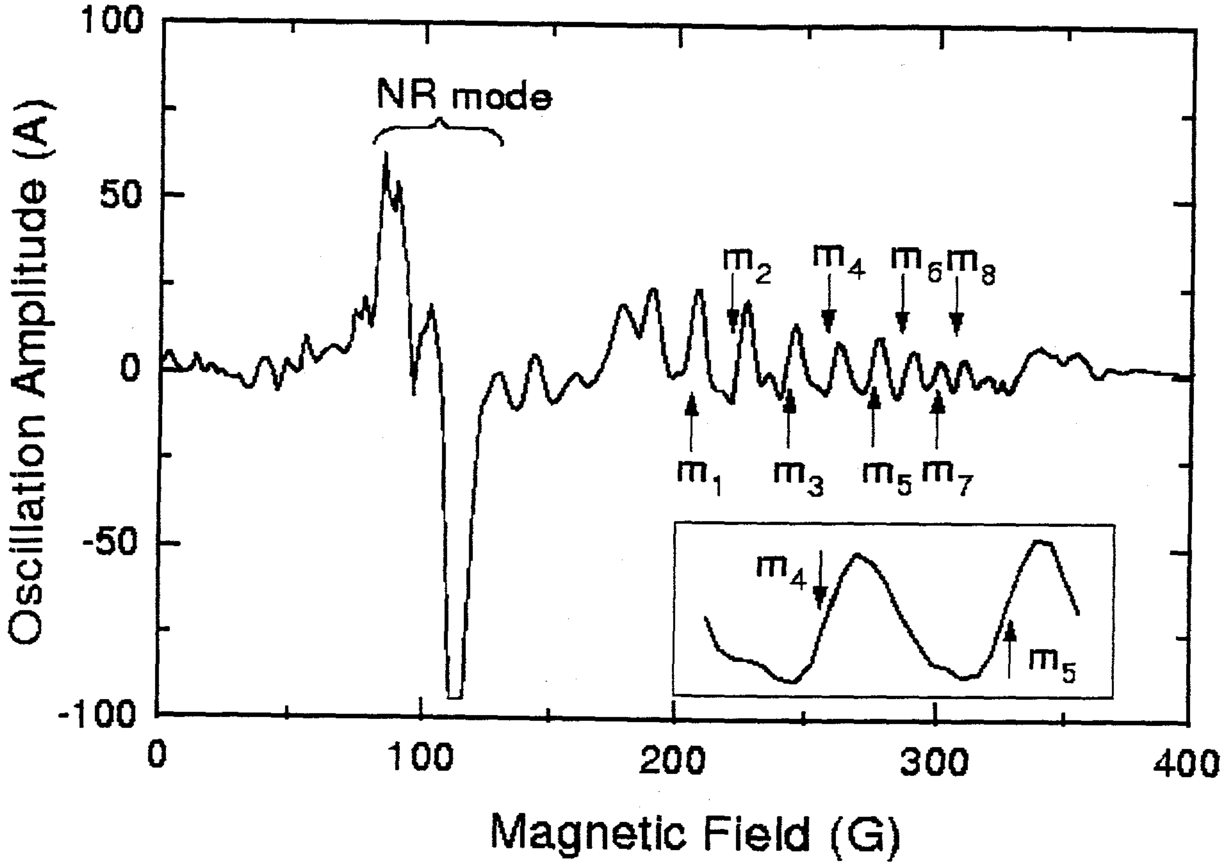

Figure 1: We show an experimental MRFM spectrum (cantilever oscillation amplitude as a function of applied field in Gauss) from a single crystal YIG film. A family of magneto-static modes (labeled as m_1, m_2, ...) is evident alongside the non-resonance (NR) mode. The locations of the magneto-static modes are indicated by the arrows in the inset. The resonance is excited by means of an oscillating magnetic field with an amplitude of 2 Gauss and a frequency of 825 MHz; this rf magnetic field is 100% amplitude modulated at 41.27 kHz. The bias field is ramped at 1.5 Gauss/s and modulated at a frequency of 36.01 kHz and with a modulation amplitude of 4 Gauss.

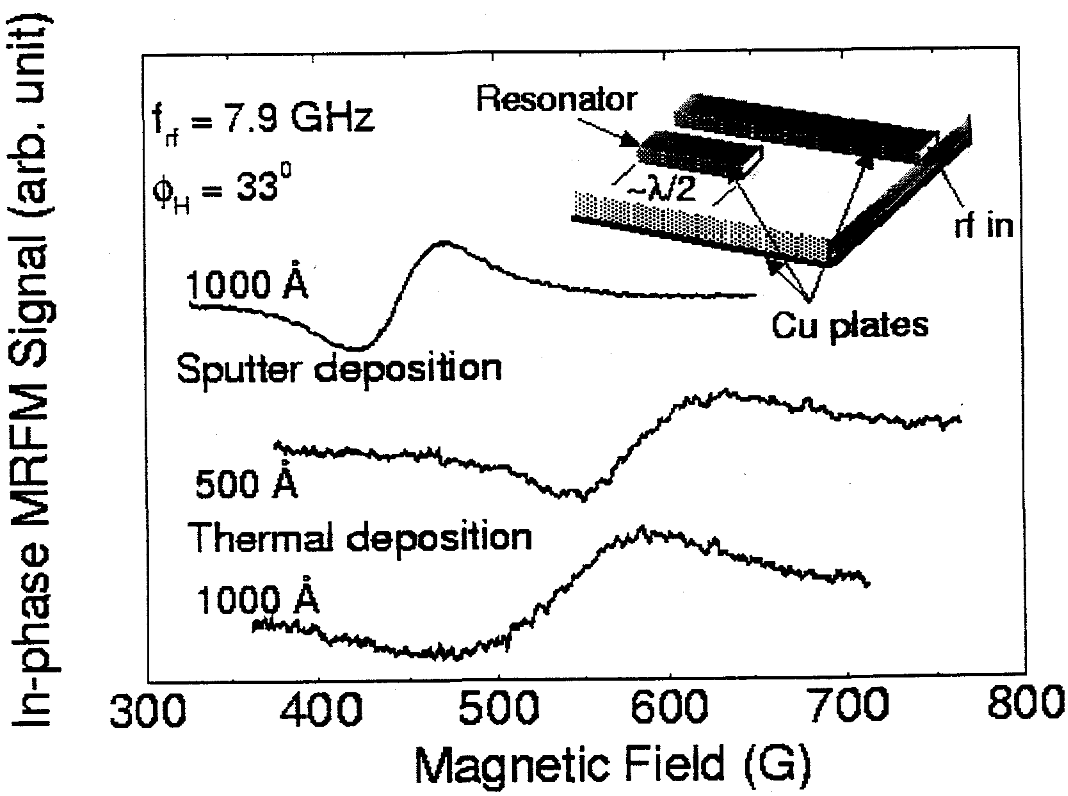

Figure 2: MRFM spectra of three single-layer Co thin films using transverse geometry MRFM. The experiment was performed in air and at room temperature. The angle between the external field and the film plane is ϕ~33. The inset shows the design of the novel microstrip resonator that provides the 8 GHz rf magnetic field used to excite the magnetic resonance.

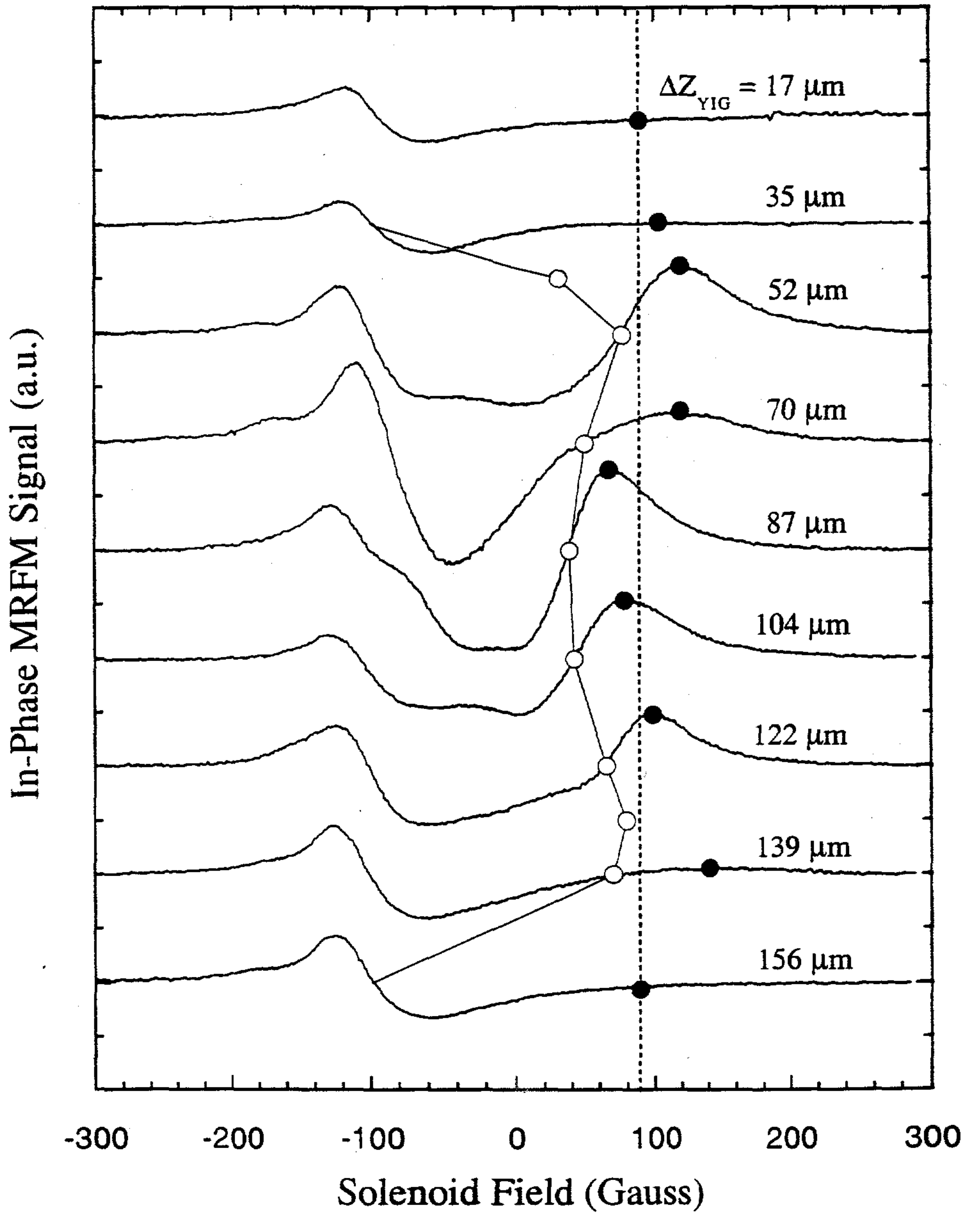

Figure 3: A series of FMR spectra from which the lateral positions of two Co films can be determined is shown. These are obtained by scanning a small sphere of YIG (whose position is indicated by Δz_{YIG}) over the films while performing FMR/MRFM. The FMR spectra (signal intensity vs. solenoid field) are shown for several values of Δz_{YIG}. The positions of the maximum signal deviation (•) and the center of the additional signal (°) are shown. The dotted line indicates the value of the solenoid field at which the signal intensity as a function of Δz_{YIG} (shown as a dotted line in Figure 4) is determined.

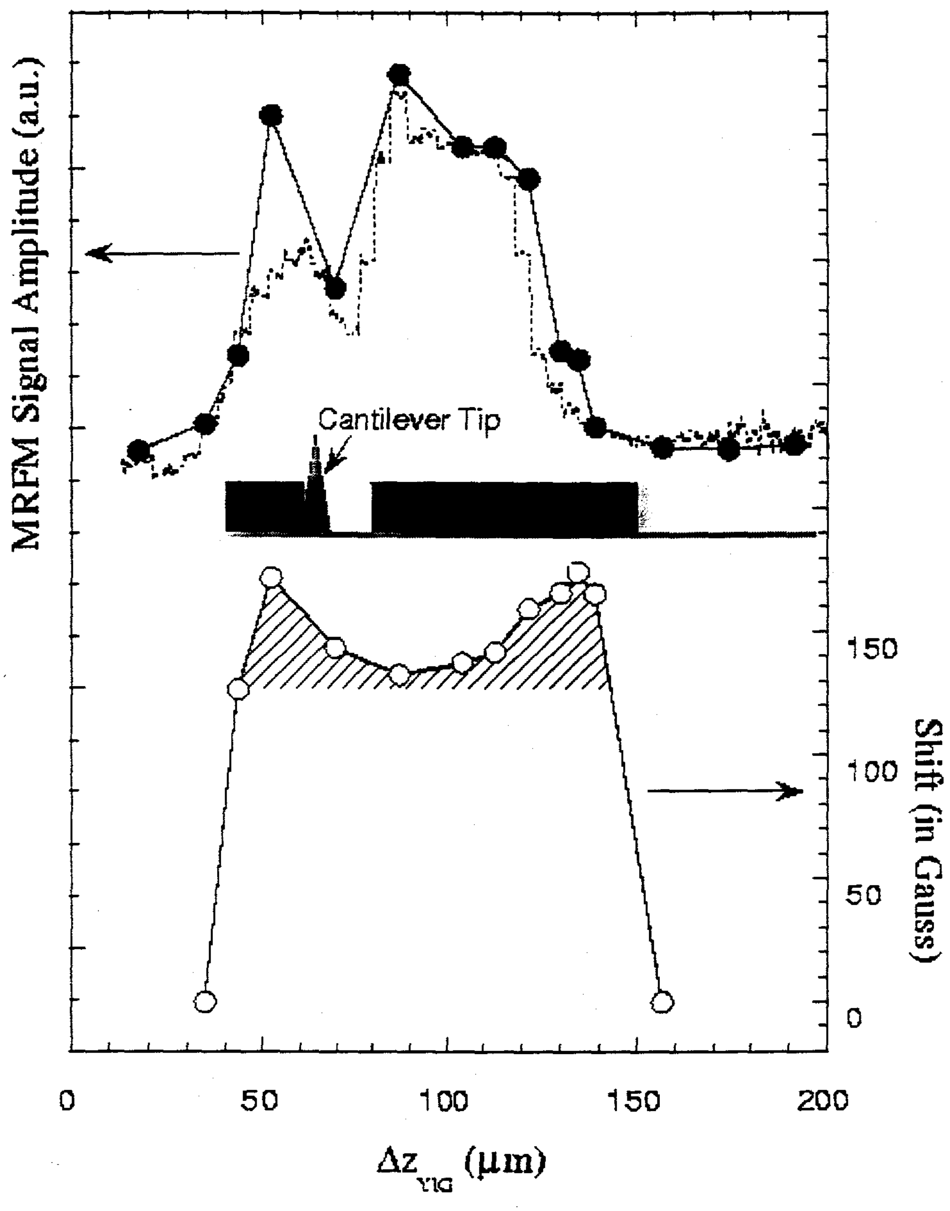

Figure 4: The dotted curve in the upper panel shows the variation of the signal amplitude as a function of the position (Δz_{YIG}) of the YIG sphere at a fixed value of solenoid field B=90 Gauss. The curve indicated by the solid circles shows the variation of the maximum deviation of the additional signal arising from the Co film in close proximity to the YIG sphere as it is scanned. The lower curve (open circles) shows the magnetic field shift of this additional signal with respect to the position of the original signal. An estimated sample profile obtained from knowledge of the shadow mask dimensions and from optical microscopic examination of the Co sample is indicated schematically in the center of the figure for comparison.