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An alternative approach to vector vibrating sample magnetometer detection coil setup

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Vector vibrating sample magnetometers (VSMs) can present problems with respect to angular dependent calibration and positional dependency when they are used for measurements on thin film samples, which have dimensions comparable to or larger than the sample–coil distances. The problems are due to the fact that in conventional VSMs the sample is rotating with respect to the coils, when performing angular dependent measurements. In this article a solution is presented based on a setup of VSM detection coils, whose position is linked to that of the sample. Together with a newly designed sample holder, the above mentioned problems are prevented or reduced. The vector detection coil system shows a relatively small error in the determination of the magnetization vector (±1% in the absolute value and ±0.6° in the angle). Furthermore, it has a relatively small positional dependency (1% per mm) combined with a sufficient sensitivity (1 nA m^2 or 1 μemu at 10 s time constant) and a capability of using samples up to 10×10 mm^2. The improved sample holder for thin film measurements reduces positional problems while, at the same time, reducing the background signals of the holder (to 10 pA m^2 per kA/m or 7.958×10^{-10} emu/Oe). *© 1998 American Institute of Physics.* [S0034-6748(98)04008-8]

I. INTRODUCTION

The medium motion along the recording head during the recording process causes a continuous change in both the length and the direction of the applied field vector with respect to the medium. Therefore, a vector measurement system should be used for the analysis of recording media so that the magnetization vector is measured rather than the projection of the magnetization on the field direction. In general, for all media where the field is applied under an angle with the anisotropy direction, the magnetization vector should be measured. This presents more information than scalar measurements. An example of a vectorial measurement (on a standard γ-Fe_2O_3 audio tape at 80°) is given in Fig. 1. This figure shows the change in length and direction of the magnetization vector as a function of the changing applied field. From the maximum field, where the magnetization aligns with the field, the magnetization will rotate towards the easy axis direction (which is in plane) for decreasing fields. Close to the field where the M vector length reaches its minimum, the particles start flipping, changing their magnetization irreversibly. From that point onward, the magnetization vector will again align with the increasing negative field. This type of figure presents a lot of information on the actual reversal process that is not available from scalar measurements. To obtain this type of result, a measurement system with a sensor capable of vectorial measurements is required. An overview of several very useful applications of vector measurement systems, such as anisotropy measurements, recording simulation experiments, and intrinsic hysteresis loops, is given in Ref. 1.

One very commonly used instrument for measurements on magnetic materials is the vibrating sample magnetometer (VSM), which was introduced in 1956 by Foner,[2] who wrote a more detailed paper in 1959.[3] Other authors such as van Oosterhout,[4] Plotkin,[5] and Flanders[6] used a similar instrument but vibrated the sample in the direction of the field rather than perpendicularly as has been described by Foner and has since become the standard. In Ref. 3 and many subsequent publications various detection coil setups have been proposed for uniaxial and biaxial signal detection. One of the best setups is presented in Ref. 1, together with an overview of several other vectorial detection coil systems.

Traditionally VSMs have been used mostly for measurements on fairly small samples. However, most VSMs will show calibration problems if samples that are relatively large in one or more dimensions compared to their distance to the detection coils are used. This problem becomes visible when the sample is rotated or when the magnetization vector has an angle with the applied field. With the growing importance of thin film recording media for magnetic hard disk and videotape applications, the need arises for vectorial magnetometers capable of measuring relatively large thin film samples with low magnetic moments. Bernards[1] and Richter[7] have published systems suiting these needs. In this article, an alternative approach is presented. The detection coil system presented here does not show the need for a complicated calibration procedure as described in Refs. 7 and 8 and can be used with samples up to 1×1 cm^2 while it is at the same

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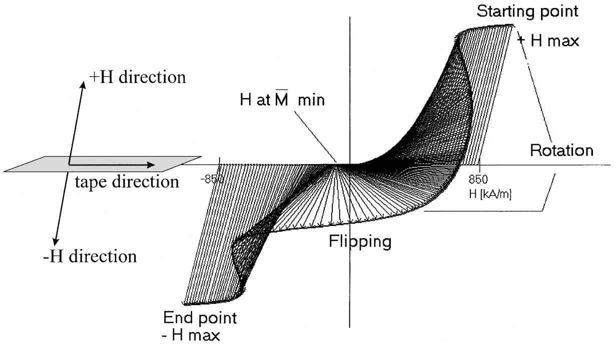

FIG. 1. Reversal process in a γ-Fe_2O_3 audio tape at 80°. The arrows indicate the magnetization vector. The downward half of a hysteresis loop is shown. The horizontal direction represents the sample plane, the vertical direction the normal to the tape.

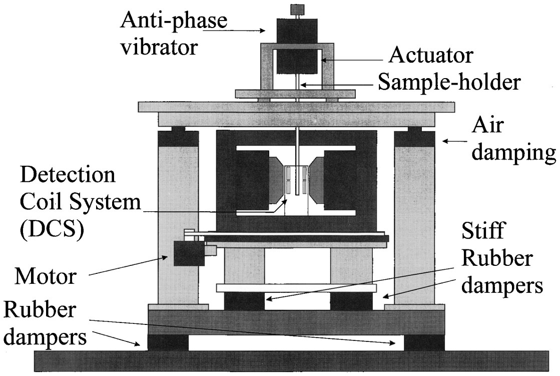

FIG. 2. Schematic VSM setup including anti-phase vibrator and vibration damping.

time somewhat more sensitive than both of these systems.

In addition, an improved sample holder will be described for thin film samples, which improves measurement reproducibility and drastically decreases the background signals caused by the sample holder and the sample substrate.

II. SENSITIVITY ISSUES

Before focusing on the factors influencing the sensitivity of the VSM, it is important to make some general remarks. The literature presents several different numbers for the sensitivity of various measurement systems without always considering the differences in application of these systems or specifying how the sensitivity number was obtained. The sensitivity of a system should be presented as either the peak to peak or the root-mean-square (rms) noise floor (in units of magnetic moment) at a certain specified effective bandwidth of the used electronics. However, the sensitivity is only relevant if the actual useable maximal sample dimensions are known, so that real magnetic signal to noise ratios for a particular magnetization can be compared. A system can be presented with a very high sensitivity, but if it is only suitable for measuring very small samples (with very small signals), the measurement results obtained on this instrument are not necessarily better than those obtained with a less sensitive system on a much larger sample. If a system demands the use of samples limited to, for example, 1 mm^2 in size, it must be 100 times more sensitive than a system in which samples of 1 cm^2 can be used, in order to produce similar quality graphs. This is of course especially the case when the third dimension is limited such as in thin films.

If the optimal sample dimensions are known for a given configuration with a known performance, the dimensions of the detection coil system can be scaled up or down proportionally to fit other sample sizes, because almost all aspects of a VSM system are scalable without changing the signal-to-noise ratio (SNR).[7]

A review of several sensitivity issues has been given in Ref. 7. The field noise problems as described by Richter have not been observed in the system that is treated here. This might be due to a more stable electromagnet power supply. If proper cabling, impedance matching, and amplification is used, the system noise can be brought back close to (within 2–3×) the theoretical limit presented by the electrical (Johnson) noise. The Johnson noise is given by $\sqrt{4kTR\Delta f}$, where R is the sensor impedance and Δf the effective noise bandwidth of the signal amplifier. Apart from the electrical (random) noise, the vibrational noise, which is generally a form of correlated noise, is a severe limitation to the sensitivity of a VSM system.

The detection coil system is extremely sensitive to flux changes in order to be able to measure the small flux changes caused by the sample motion. Therefore, vibrations of the detection coils (which change the angle between the detection coil axes and the magnetic field or move the coils in the slightly inhomogeneous applied field) will also cause flux changes in the detection coils, depending on the field strength. The use of coil pairs (connected in anti-series) will eliminate the signal produced by flux changes equally present in both coils. Vibrations with the same frequency as the sample motion are the most bothersome as other frequencies are filtered out by the lock-in amplifier. These vibrations can be caused by a mechanical coupling between the actuator (a modified loudspeaker) and the detection coil system or by other sources of vibration around the VSM. The two most common ways to reduce this problem are the use of vibration dampers between actuator and measurement system and a rigid coupling of the detection coils to the magnet. A disadvantage of commonly used vibration dampers in or directly attached to the vibrator system, is that because of the low mass of the damped system and the low frequency of the vibrations ($<$100 Hz), the damping materials must be very compliant. As a result the whole vibrator–sample holder assembly can more or less move freely, which gives rise to a poor definition of the exact sample position. In order to prevent this, the vibrator unit was mounted on a heavy table, which could be damped by much stiffer vibration dampers, so that the sample positioning remained very constant.

A third way of vibration damping is the use of passive or active anti-vibration elements. Some commercial systems like the Princeton Applied Research (PAR) and Oxford Instruments VSMs utilize passive spring mounted weights which, if properly tuned, will vibrate in anti-phase to the actuator and therewith can significantly reduce the induced vibrations in the system. Here, rather than a passive anti-phase damper, an active system is used. This incorporates a

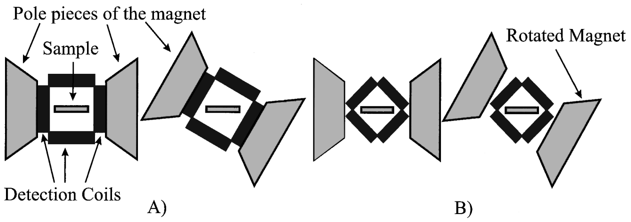

FIG. 3. Adapted Mallinson (see Ref. 10) setup compared to new coil setup.

second actuator driving a dummy mass mounted on top of the first actuator and driven at the same frequency but with amplitude and phase tuned such that the vibrations in the system can be reduced by a factor of 1000 or better, if vibration feedback is used.

Both the vibration isolation elements and the anti-phase vibration system are shown in Fig. 2.

III. VECTOR SIGNAL DETECTION

If a VSM is available with a detection coil system (DCS) suitable for the detection of the magnetization in one direction, the simplest thing to do in order to accomplish vector signal detection, is to add a similar set perpendicular to the first one. There are, however, several other approaches, which are discussed by Bernards *et al.* in Ref. 1. All standard detection coil systems are generally connected stiffly to the poles of the magnet in order to prevent vibrations of the DCS in the applied field. A change of the direction of the applied field with respect to the sample can be accomplished by rotating the magnet–DCS combination around the sample or by rotating the sample inside the magnet–DCS combination. The latter is generally easier due to weight of the magnet and its electrical wiring and cooling hoses.

A sample that is relatively small compared to its distance to the detection coils can be considered as a dipole and approached as such. However, if the sample dimensions are comparable to or larger than the distance of the sample (edges) to the detection coils, the sensitivity of the detection coil will depend on the exact position of the sample with respect to the coils. If this (large) sample is nonspherical, e.g., sheet shaped and rotated around one of the in-plane directions, then the geometric shape as seen by the sensor will change with the angle and therefore the system sensitivity will be angle dependent. If the sample holder has an angle with the sensor (in the vertical direction), rotation will cause a circular motion of the sample with respect to the sensor, making the sensitivity even more angle dependent. Therefore, when the rotation is not exactly concentric within the coils or when a relatively large sample is used, the system needs to be calibrated for each angle individually. This is a cumbersome and time consuming process that can easily lead to errors if, for example, a calibration sample is used that cannot be saturated in all directions (due to the demagnetizing field). A correct calibration procedure has been described by Richter in Ref. 7 and Bolhuis in Ref. 8.

As the sensitivity of a DCS depends on the position of the sample, the calibration for different angles will become sensitive to errors in the positioning of the sample and if a change in sample position occurs, the system needs to be recalibrated. For this reason some VSMs are equipped with a sample holder guidance.

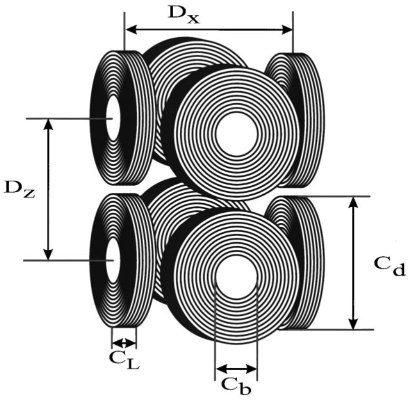

FIG. 4. Detection coil arrangement, $Cb=4$, $Cd=14$, $CL=3$, $Dx=Dy=21.5$ $Dz=19$ (all in mm).

Alternatively, one could choose to either increase the dimensions of the coil system (in so far as this is possible within the limitations presented by the electromagnet pole gap) or decrease the size of the sample, which are functionally equivalent. The disadvantage of doing this is that sensitivity is sacrificed for obtaining a lower positional dependency. The sensitivity drops with approximately the third power of the distance to the coil. Furthermore the amount of signal produced by a sample is directly proportional to the magnetic volume of the sample. This creates the dilemma of making the system either very sensitive and angular dependent or making the system insensitive to sample shape, position, and orientation, and therewith less sensitive to the signal as such.

Bernards[6] has proposed a solution to this dilemma by using a 12 coil arrangement where the coils largely compensate each others positional dependency. This has resulted in a system that is more or less insensitive to the exact position or the angular orientation of the sample for samples up to 6 mm wide while at the same time preserving a reasonable sensitivity.

IV. A DIFFERENT DETECTION COIL SETUP APPROACH

In order to prevent rather than minimize the above-described problems, a different approach has been followed here. The described problems all arise due to the relative rotation of the sample with respect to the detection coils. Therefore, in the new setup, the position of the detection coils and sample are locked to each other and the field is rotated around both the sample and the sensor. Using simple trigonometry one can easily calculate the components of the magnetization vector in the direction of and perpendicular to the applied field from the measured magnetization vector.

A similar arrangement was used in Ref. 9 to study image effects, but to our knowledge, it has never been used in a permanent setup as a magnetic thin film measuring arrangement.

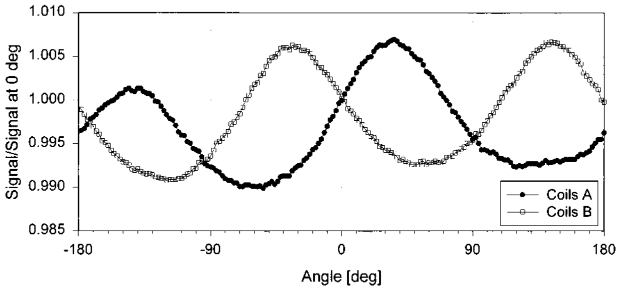

FIG. 5. Measured image effect in the experimental VSM. A and B are the two orthogonal coil sets. When the magnet is at 45° ± 180° the A coils show a maximum image and the B coils a minimum, the B coils will show a maximum for magnet angles of $-45° \pm 180°$.

If the magnet is to be rotated around the detection coil system, only a limited space for the detection coil system is available and the most obvious detection coil arrangement is an orthogonal system. In order to create an equal sensitivity for both sets of coils and in order to decrease the positional dependency of the sensitivity, the detection coils are placed under a 45° angle with respect to the sample (see Fig. 3). The signals in the sample plane and normal direction follow from simple trigonometry.

A. Advantages and disadvantages of this coil setup

The advantages of the new setup follow from the previous section describing the problems in angle dependent measurements:

(i) There is only one calibration number necessary for each set of coils;
(ii) mispositioning of the sample only leads to the change of the calibration factor, it has no influence on the relative sensitivity for different field angles.

One of the disadvantages of the system is its higher sensitivity to vibrational noise since the coils are not connected directly to the magnet. This problem has been solved by constructional improvements as described before using passive vibration damping and active anti-phase vibration cancellation.

In standard detection coil systems where the coils are placed close to the magnet pole faces, the image effect enhances the sensitivity and is therefore not bothersome (as long as the pole faces do not saturate completely). In the system described here, the coils had to be placed close to the sample and relatively far from the pole faces in order to reduce the image effect sufficiently (to approximately ±1%). This is necessary since in this case, the rotating magnet would otherwise cause an image effect that would change with the angle.

B. Realization of the coil setup and its performance

Using the formulas given in Ref. 1 a DCS has been developed based on a compromise between the following desired specifications:

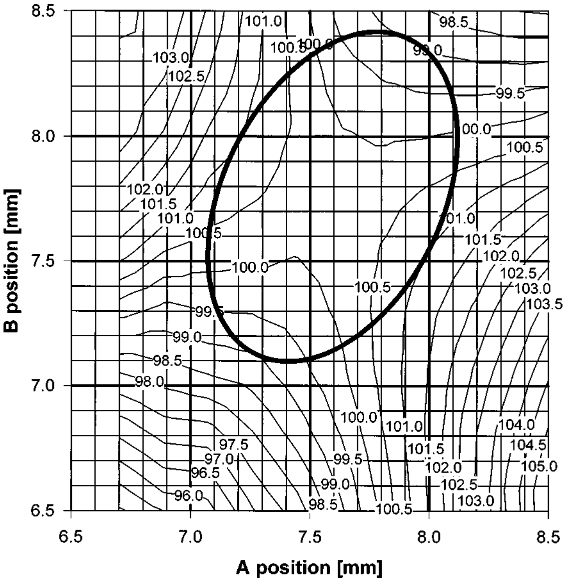

FIG. 6. Normalized measured positional sensitivity, obtained with a 1 cm^2 sample. The A and B direction are the directions of the coil axes of the two orthogonal coil sets. The elipsoid subtends the region within 1% error.

(i) low image effect (<2%);
(ii) reasonable sensitivity;
(iii) low positional dependency.

The sensitivity increases with decreasing distance between the sample and the detection coil system, as does the positional dependency. The image effect drops for decreasing sample–detection coil distance (the sensitivity towards the image is approximately $1/r_i^{5/2}$, with r_i the effective coil–image distance). As only a limited, fixed pole shoe gap (50 mm) was available, and the image effect was to be kept low, the compromise led to the system shown in Fig. 4. The coils were wound using 0.15 mm diam copper wire coated with a thermo-setting epoxy.

The effect of the extra image signal can be monitored by rotating the magnet–pole shoes (at zero field, the magnet current compensating for the pole–shoe remanence) around a sample in remanence. As the remanence of the sample doesn't change, the change in measured signal can be attributed to the image effect. The signal measured as a function of the magnet angle for a (square) 1 cm^2 sample in remanence is given in Fig. 5. The signal is normalized against the

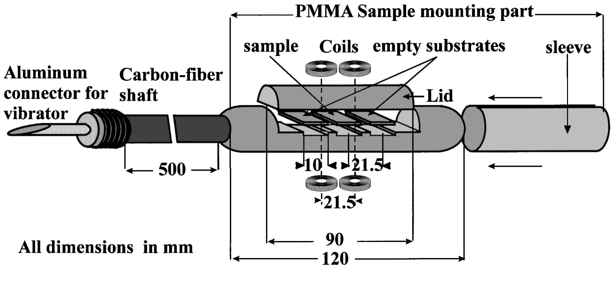

FIG. 7. Sketch of the sample holder. The wedge shaped top of the sample holder connector guarantees a reproducible placement of the sample holder. The samples and substrates are placed in three slits guaranteeing a reproducible and stable positioning. If the sample holder itself has very shallow slits and deeper slits are created in the ''lid'', than the same sample holder can be used for different sample thicknesses simply by using different lids.

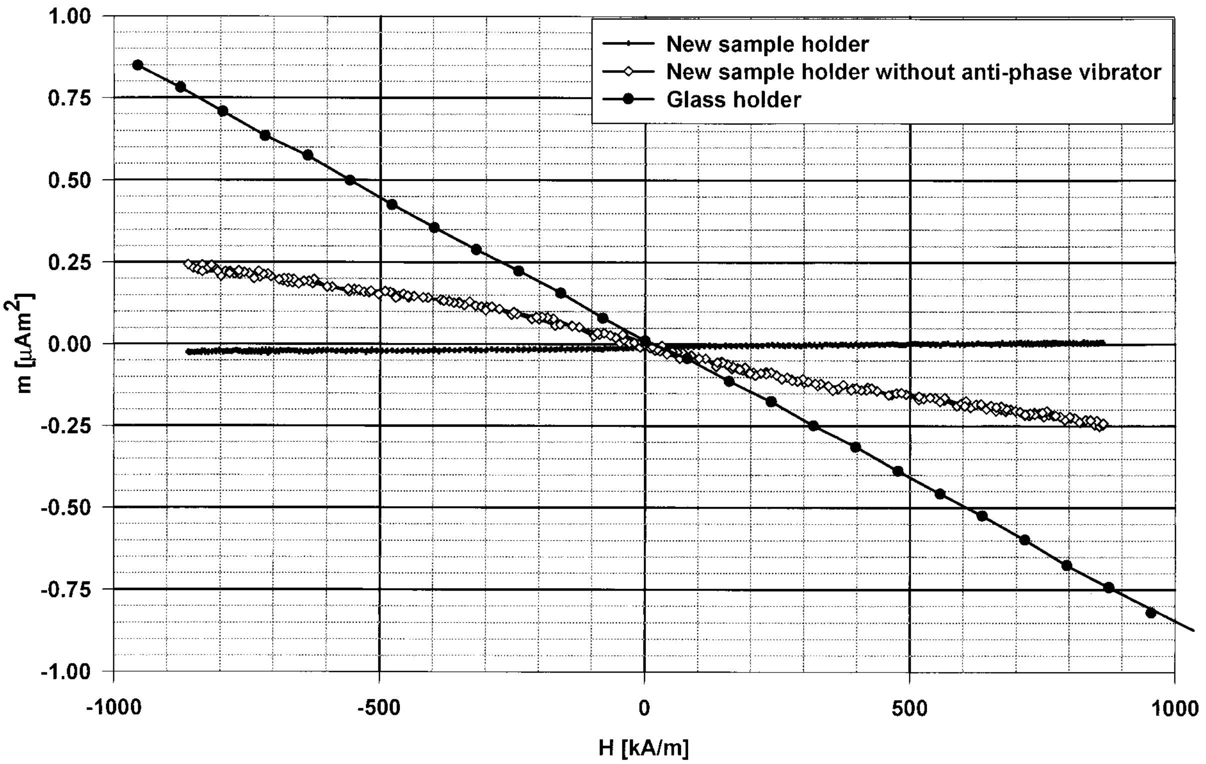

FIG. 8. Background curves for the new sample holder, the new sample holder when the anti-phase vibrator is switched off, and a standard commercial (DMS) glass sample holder.

signal at 0°. Due to the 45° rotation of the system, the extrema in the graph are reached at $45° \pm 90°$. One sees the expected sensitivity behavior showing that the image effect is indeed limited to only ±1%. The difference between the shape of the image effect curve and a sine function can be attributed to the small differences between the coils and the not exactly centered sample.

The positional dependency of the sensitivity can be monitored by moving a remanent measurement sample in the area between the coils. The experimental results for a 1 cm^2 thin film sample are shown in Fig. 6. One can see from the gray area in the figure that a positional error of ±0.5 mm will cause a signal change of approximately 1.5%. This is acceptable in this system however since the sample position with respect to the coils does not change during the measurement in contrast to other systems where the rotation of the sample around its axis is mostly accompanied by a small motion of the rotation center. As the VSM is equipped with a micrometer precision positioning system, a positioning error of less than 0.5 mm can be accomplished.

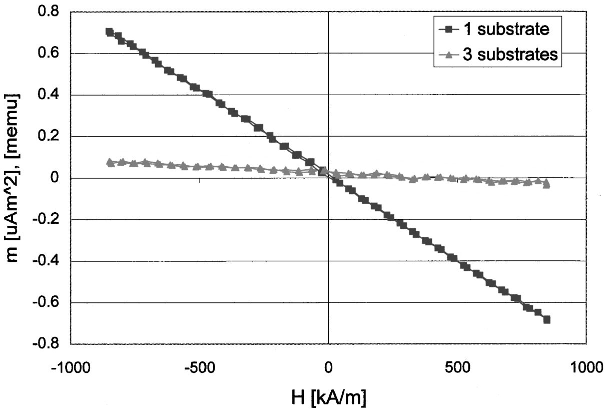

FIG. 9. Background curves when using a single (clean) substrate or three substrates that largely compensate each other due to the design of the sample holder.

The rms noise in the system is approximately 1 nA m^2 (1 μemu), using a measurement time constant of 10 s (12 mHz effective noise bandwidth). This noise is independent of the applied field, which indicates good vibration isolation.

At a maximum field (800 kA/m) the noise is virtually the same, which indicates that field noise is not a significant problem in this system. Both noise figures are independent of sample size but are accomplished in a system that allows thin film samples with a maximum size of 1 cm^2.

V. DESIGN OF AN IMPROVED SAMPLE HOLDER FOR THIN FILM MEASUREMENTS

VSM measurements on thin films will often be diluted by the contribution to the signal of the moving diamagnetic or paramagnetic sample holder as well as by the magnetic properties of the substrate on which the thin film is deposited. For very low output samples substantial corrections are needed in order to recover the signal coming from the magnetic layer under investigation. This is especially true when measurements are done on for example hard disk samples, where the (very thin) recording layer is deposited on a thick substrate of glass or aluminum. Background signals from hard disk substrates can be as high as 2 μA m^2 (2 memu) at a field of 800 kA/m (~10 000 Oe), which is in many cases larger that the signal produced by the thin film itself. Recovering the measurement loop from such a signal can lead to extra noise and problems if the measurement vector needs to be retrieved.

As a separate issue, the reproducibility and accuracy of the measurement depend to a high degree on the reproducibility of the position and orientation of the sample in the system. We describe a sample holder which precisely reproduces the position and orientation of the sample as well as reduces the contribution of both holder and substrate to the signal measured. The reproducibility of the sample position has been established by deepening the part in which the sample should reside. The sample is clamped to its fixed position by means of a lid and an elastic sleeve of the same material as the rest of the holder (Fig. 7). This way the background signal from the support rod has been minimized by keeping its cross section as uniform as possible along the z axis. The parts that are disturbing the uniformity of the cross section should be repeated symmetrically along the z axis at each side of the centers of the coils. Therefore two more slits are made, where the distance between the centers of the three slits should be equal to the distance between the centers of the coils along the z direction.

As a result there are two more positions in the sample holder for putting in a sample. Substrates of the same size, shape, and material as that on which the thin magnetic film is deposited can be mounted in these two extra slits in order to automatically compensate for the substrate background signal.

The material used for the sample holder is polymethylmethacrylate (PMMA) which is fairly easy to machine and has a smooth surface, which makes it easy to clean. For cleaning the holder we use hydrochloric acid (30%) in an ultrasonic cleaning bath for at least 1 h.

The connector to the vibration unit has been made in such a way that it exactly reproduces the angle of orientation of the holder. Because we use a loudspeaker based actuator the distance between the actuator and the center of the magnet is quite large (>70 cm). Therefore a carbon–fiber shaft which is very stiff, straight, and light, is used to extend the sample mount part towards the connector. As a result the background signal in a hysteresis curve, with the empty sample holder installed, has a slope of less then 10 pA m^2 per kA/m (7.958×10^{-10} emu/Oe). The background signal for this new sample holder is roughly 10× lower than the background signal for a commercial glass sample holder, as can be seen in Fig. 8. This figure also shows that the background signal due to vibrations is significantly reduced by means of the anti-phase vibrator. The empty substrates in the holder will reduce the substrate background signal by approximately a factor of 10, as can be seen in Fig. 9.

ACKNOWLEDGMENTS

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