

Vector vibrating-sample magnetometer with permanent magnet flux source

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A compact magnetometer, which uses two nested 1 T Halbach cylinders to generate a variable magnetic field of up to 2 T in any direction in the xy plane, has two sets of quadrupole pickup coils to detect the components m_x and m_y of the magnetic moment of a sample vibrating along Oz . Sensitivity is 1.10^{-7} A m² with integration time of 100 ms. The instrument is used to measure longitudinal and transverse hysteresis loops and torque curves, under automatic software control. Three examples illustrating its versatility and sensitivity are provided: CrO₂ magnetic recording tape, a cobalt nanowire array in a porous membrane, and a FePt thin film. © 2006 American Institute of Physics. [DOI: 10.1063/1.2170595]

INTRODUCTION

Magnetometry is essential for any research program on magnetic materials, as well as for quality control in a production environment. The hysteresis loop $\sigma(H')$ is commonly measured in open circuit, using the vibrating sample magnetometer (VSM) pioneered by Foner in the late 1950s.¹ Here σ is the magnetic moment per unit sample mass, and H' is the applied magnetic field. A small sample (≈ 10 mg) is magnetized in a variable field and vibrated with small amplitude (≈ 1 mm) at low frequency (≈ 100 Hz) at the center of a set of quadrupole coils.² The change in flux in the coils induces an electromotive force (emf) E which is amplified and measured using lock-in detection. The sensitivity of a VSM is typically 10^{-6} – 10^{-9} A m²,³ so the instrument can be used for diamagnetic, paramagnetic, or ferromagnetic bulk material, and for ferromagnetic thin films. The VSM is robust and simple to use. The coils can be designed for operation in axial or transverse applied fields, produced by superconducting solenoids, electromagnets, or permanent magnet variable flux sources based on nested rotatable Halbach cylinders.⁴ Permanent magnet flux sources have the advantages that they are much more compact than electromagnets, and they require no high-current power supply. A compact 1.2 T permanent-magnet VSM was described 10 years ago.⁵

Another magnetometer, which is nowadays much less common than the VSM, is the torque magnetometer. It is used to measure magnetic anisotropy. The sample is suspended from a torsion fiber, and the torque is measured as the magnetic field is rotated about the axis of the fiber, usually by moving the electromagnet. The instrument is both delicate and cumbersome which accounts for its decline in popularity, although it provides data on both rotational hysteresis and the anisotropy constants of an oriented sample. The torque on a sample of volume V is given by

$$\Gamma = -V\partial E_a(\theta)/\partial\theta. \quad (1)$$

Here θ is the angle between the sample easy axis and the direction of the magnetization \mathbf{M} in the xy plane, and E_a is

the magnetic anisotropy energy density, which for the case of uniaxial symmetry can be expressed as

$$E_a = K_0 + K_1 \sin^2 \theta + K_2 \sin^4 \theta + \dots, \quad (2)$$

with K_0 , K_1 , etc., being constants independent of the angle θ . An alternative of the torque magnetometer is the vector magnetometer. If the two components of magnetic moment m_x and m_y in the xy plane can be measured, and if the field is applied at an angle θ with Ox , then

$$\Gamma = \mu_0 \mathbf{M} \times \mathbf{H} = (\Gamma_z, 0, 0) \Rightarrow \Gamma_z = \mu_0 H (m_x \sin \theta - m_y \cos \theta). \quad (3)$$

Torque is not measured directly, but it is inferred from the magnetic moment vector. This is the basis of the vector vibrating-sample magnetometer (VVSM). The field is rotated in the plane (provided the sample orientation can be controlled this is often sufficient), and the components of the moment are measured using two (ideally three) orthogonal sets of pickup coils. Examples of applications of VVSMs are readily found in the literature.^{6–8}

DESIGN

The design of our VVSM is shown in Fig. 1. It is based on the earlier permanent-magnet VSM,⁵ except that the field is produced by a 2 T “Multimag,” where the field can be varied in both magnitude and direction in the xy plane by rotation of two nested 1 T Halbach cylinders. Each of the two cylinders can be independently rotated around the common z axis. The superposition of the homogeneous (in the central region, around the sample volume) fields created by the two magnet assemblies, leads to a controllable both in magnitude and direction in the xy plane resulting magnetic field. Each of the two quadrupole pickup coils⁹ is made of four coils of 2000 turns of 35 μm enameled copper wire. An alternative second-order gradiometer coil assembly, measuring the m_z component of the magnetic moment, was also designed following Ref. 10. The pickup coils are balanced by minimizing the total induced voltage (measured by means of lock-in detection) as the coils are fixed on their support frame in the working configuration, and placed in a solenoid producing uniform field. The solenoid is wound with a nu-

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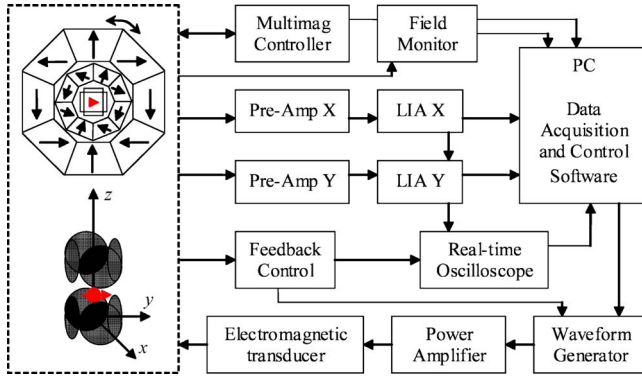


FIG. 1. (Color online) Schematic diagram of the vector vibrating sample magnetometer. Each of the two Hallbach cylinders, alone, produces homogeneous field in the central region, with a controllable direction in the plane perpendicular to Oz .

merically optimized turn-density distribution so as to ensure inhomogeneities of less than 1% over a 5 cm long working region, and is sourced with ac current from an oscillator, to which the lock-in detector measuring the total induced voltage is phase locked. By this method misbalance due to a single turn in any of the coils of the assembly can be corrected, thus maximizing the common-mode rejection ratio. The pickup assembly is fixed and stationary in the sample frame, thus avoiding the problems related to varying total flux, often occurring with samples lacking cylindrical symmetry in systems where the pickups are stationary in the magnet frame.¹¹ The sample is vibrated using a small electromagnetic transducer. (A piezoelectric stack was tried, but abandoned as the amplitude of vibration was inadequate, and a piezo bimorph was insufficiently stable.) Low-noise preamplifiers with an adjustable amplification ratio of 0 to -80 dB, connect the pickup coils to two computer-controlled lock-in amplifiers (EG&G Model 5105).

Software was written in LABVIEW 5tm to record data in two basic modes:

- (1) variable field, fixed angle, giving the magnetization curve or hysteresis loop and
- (2) fixed field, variable angle, giving the torque curve or rotational hysteresis.

Data are displayed in real time on the computer screen, and recorded to memory for subsequent analysis. The software drives the Multimag 2000-26 control system, availing it of continuous or stepped field or angle sweeps; it controls the vibrator and signal detection subsystems, and it provides real-time monitoring of the vibrator feedback, field readout, and amplified sample signals. Under software control, the amplitude and frequency of vibration are kept constant (even over sample change) to within 0.1%. The total anharmonic distortion is less than 1.5%. Sensitivity achieved for either component of the magnetization is 1.10^{-7} A m², when the vibrator is driven at 73 Hz, with an integration time of 100 ms. The useful sensitivity is limited by acoustic pickup due to the relatively high inhomogeneity of the flux source (field gradients within the volume occupied by the pickup coils can reach 2 T/m, see Table I), although measures are taken to prevent direct coupling of in-phase vibrations (the

TABLE I. Homogeneity regions of the magnetic field source used, along the z axis and on the central xy plane.

Homogeneity region size (mm)	2% error limit	5% error limit	Nominal field (T)
Along Oz (by magnitude)	15 26	45 45	0.12 2.00
Along Oz (by angle)	11 35	23 40	0.12 2.00
On the central xy plane	2×2 10×10	6×6 15×15	0.12 2.00

transducer support is pneumatically suspended and driven at a frequency far from the mechanical resonances of the structure). The field resolution is better than 1 mT and the angular resolution is better than 0.5° . The VVSM can accommodate samples with volume up to 100 mm³ or area up to 25 mm² in the 8 mm free bore, which is at ambient temperature.

EXAMPLES

In order to illustrate the capability of the VVSM, we discuss data on three samples:

- (1) a CrO₂ commercial magnetic recording tape;
- (2) cobalt nanowires grown by electrodeposition in an alumina membrane; and
- (3) a CoPt thin film.

In the first case, the hysteresis of a tape sample 4 mm² in area is measured both with the field in the direction of orientation of the acicular CrO₂ particles, and in the perpendicular direction [Fig. 2(a)]. The torque curve is shown in Fig. 2(b). The magnetization of CrO₂ is 79 A m² kg⁻¹, so the sample mass is inferred to be 68 μ g. The coercivity $\mu_0 H_c$ is 40 mT. The anisotropy field deduced graphically from the perpendicular magnetization curve is $\mu_0 H_a = 200$ mT. Fitting the torque curve to $E_a = K_1 \sin^2 \theta$ ($\Gamma = K_1 \sin 2\theta$) gives K_1

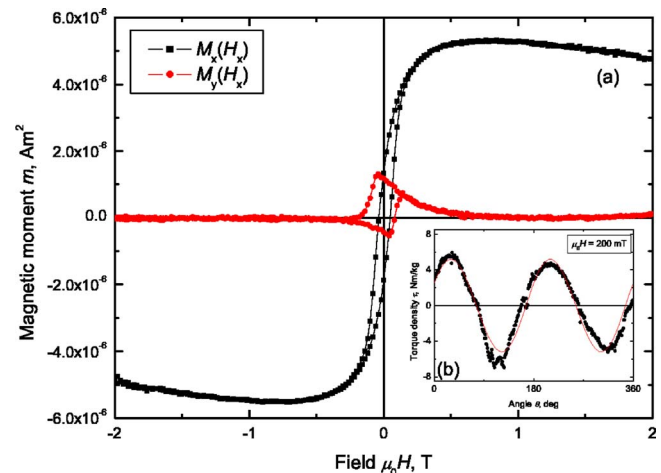


FIG. 2. (Color online) Magnetization curve of CrO₂ magnetic recording tape with the field applied perpendicular to the plane (a), 1 s per point. Torque curve for the same sample recorded at 200 mT, 100 ms per data point.

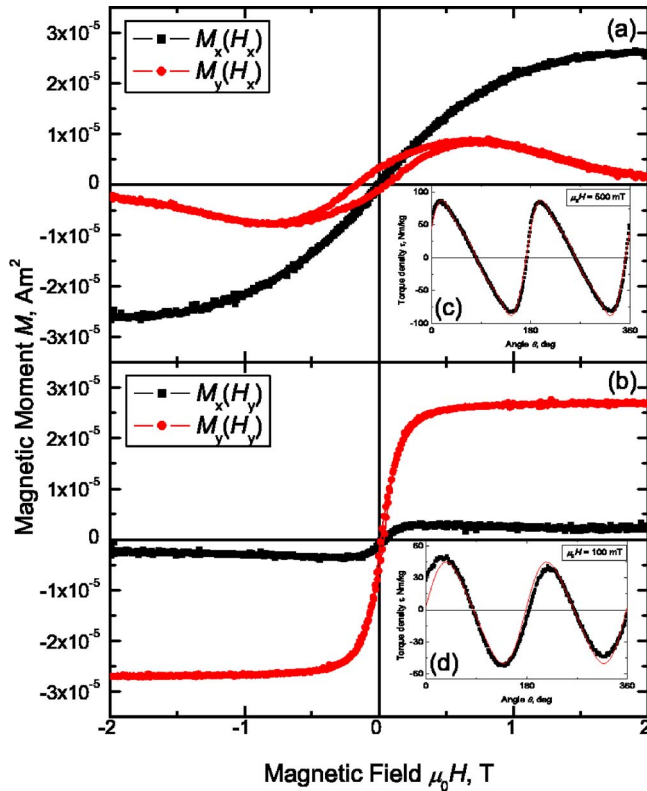


FIG. 3. (Color online) Magnetization curve for an array of partially oriented Co nanowires (100 nm diameter, 2 μm long) recorded with the field applied parallel to the wires (a) and perpendicular to the wires (b). Torque curves recorded at 500 mT (c) and 100 mT (d) for the same sample.

$=5.4 \text{ J/kg}$ (26 kJ m^{-3}). The corresponding anisotropy field $\mu_0 H_a = 2K_1/M_s$ is 136 mT.

The cobalt nanowires have a partial crystallographic order along the wire axis. The torque curve is shown in Fig. 3(c). Assuming a torque density $\tau(\theta)$ of the form

$$\tau(\theta) = \left(K_1 + K_2 + \frac{15}{16}K_3\right)\sin 2\theta - \left(\frac{1}{2}K_2 + \frac{3}{4}K_3\right)\sin 4\theta + \frac{3}{16}\sin 6\theta. \quad (4)$$

An excellent fit is obtained with the three anisotropy constants $K_1 = 155 \text{ J kg}^{-1}$, $K_2 = -131 \text{ J kg}^{-1}$, and $K_3 = 54 \text{ J kg}^{-1}$ (1.3, 1.2, and 0.5 MJ m^{-3} , respectively).

The third example is a thin film of $\text{Co}_{80}\text{Pt}_{20}$ which has a coercivity of 0.25 T, and an easy axis perpendicular to the film plane. The anisotropy field deduced from the in-plane magnetization curve (Fig. 4) is 0.5 T. Analysis of the torque curve with a single anisotropy constant gives $K_1 = 47 \text{ J kg}^{-1}$ (0.5 MJ m^{-3}). The mass of the sample is 8.1 μg .

These three examples demonstrate the versatility and sensitivity of the instrument. The time taken to record a hysteresis curve with 250 points is 25 min, and the time for a torque curve with points every 5° is 8 min.

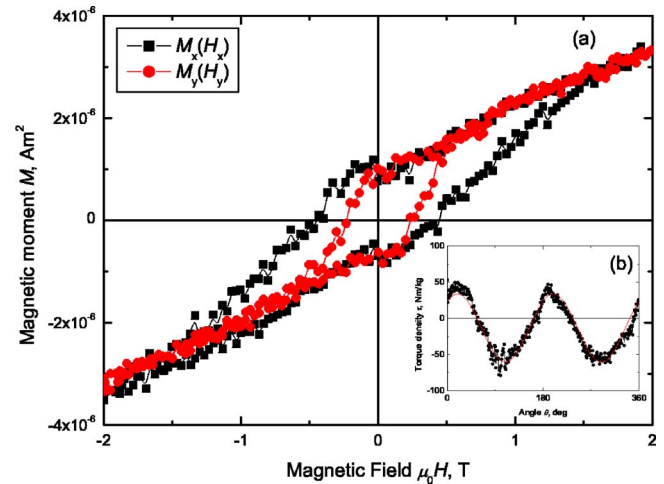


FIG. 4. (Color online) Magnetization curves for a $\text{Co}_{80}\text{Pt}_{20}$ thin film with the x direction being perpendicular to the film plane, recorded at 100 ms per data point (a). Torque curve (b) for the same sample recorded at 2 T (100 ms per data point).

CONCLUSIONS

In conclusion, the permanent magnet vector vibrating-sample magnetometer is a compact and versatile laboratory instrument, which has operated reliably for 2 years. It combines the functionality of a VSM and a torque magnetometer in a single, robust instrument with none of the delicate sample mounting or cumbersome magnet rotation associated with a standard torque magnetometer. Full three-dimensional vector capability can be achieved by adding a third set of pickup coils that measures m_z , although the field produced by the Multimag is confined to the xy plane.

ACKNOWLEDGMENTS

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