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Magnetic Measurement Techniques for Materials Characterization

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Preface

Magnetic Materials pervade every aspect of our daily lives, being used in a wide variety of important commercial and technological applications. These range from mobility applications, as an integral part of the power train of personal mobility devices (PMD), to anti-counterfeiting measures implemented in bank notes, passing through the storage of information in multiple formats. The sustainability of our energy-hungry way of life depends on the discovery and use of efficient and environmentally friendly energy conversion devices. Many of these energy conversion processes rely on magnetic materials and their improvement would have a significant influence on society. From a materials perspective, permanent magnetic materials are used in electrical motors, hybrid vehicles, etc. Soft magnetic materials are used for high frequency power electronics, power conditioning, and grid integration systems. Magnetic thin films and multilayers are used in high density recording media. Magneto-caloric effect materials have application in magnetic refrigeration systems. Nanomagnetic materials have applications in spintronics, medical diagnostics, and targeted drug delivery.

The discovery of new materials for any specific application goes through different stages that involve formulation, synthesis, and characterization. And while characterization techniques in quality control laboratories can be performed in a routine way, the use of the same characterization methods in the materials discovery process requires a thorough understanding of the experimental techniques used for their characterization. When the materials properties are not previously known, it is necessary to evaluate if the newly found features are intrinsic to the material, if they are related to the specific characteristics of the sample under study, or if they are the consequence of some peculiarities of the experimental technique being employed. The use of advanced and costly equipment as a “black box,” rather extended nowadays among those researchers who start on a new topic, can lead to blunt artifacts in the measurements or erroneous interpretation of the data. While the scientific literature has valuable research articles on the different techniques, it is rare in the literature to find a single source where the most relevant techniques are presented and compared, showing their advantages and limitations.

This book aims to fill this gap and will discuss the most commonly used techniques for characterizing magnetic material properties and will present examples of their application to several relevant magnetic materials.

The target audience of the book is rather broad, including graduate students starting their research on magnetic materials, more senior researchers who want to know more details about the fundamentals of the techniques that they are using or they plan to use, and those who are changing their focus of research into other types of materials and would like to know which are the appropriate techniques for the characterization of the properties that they are interested in.

Magnetism is probably the only field of research in which two systems of units unofficially coexist in the scientific literature. The reluctance to change is not only due to tradition, but also because even the equations of magnetism change in the different systems of units. The recent redefinition of the International System of Units (SI) in 2019 had some fundamental effects on the magnetic constants. Therefore, Part I of the book starts with a chapter on the units for magnetic quantities. We hope that the coexistence of different systems of units in magnetism will soon fade out, with all of us adhering to SI. There are some research subtopics in magnetism where the centimeter-gram-second (CGS) system of units still prevails and, therefore, you will still find such legacy units in some of the chapters of this book.

The rest of the book is structured into five parts. The first four, consisting of 15 chapters, focus on the description of the measurement techniques, showing the underlying principles that make them operate in the way they do and possible limitations for their use. We have grouped the techniques by the principle of measurement. Magnetization is most frequently measured by using inductive- or force-based methods, which are described in Part II. These methods provide average information of the magnetic response of a material. In current technology, it is necessary to have more local information and the ability to visualize the different domains in which magnetic moments arrange; this is dealt by imaging techniques that use visible light, X-ray photons or electrons. These techniques are presented in Part III. There are additional techniques that are for more specific applications, or so general that they do not fit in the previous more restricted approaches. These are grouped in Part IV. Field sensing is necessary in any magnetic technique, but it is also possible to use it to infer the magnetization of a sample by detecting the field produced by it. In this part, we have also included two chapters on neutron scattering targeted to different audiences: the first is a description of the possibilities of neutron techniques oriented to those who are new to the technique, and the second is a thorough description of the fundamentals accompanied by practical examples. While most of the previous techniques are quasistatic or involve relatively low frequencies, technology is continuously evolving towards higher frequencies in the MHz to GHz range. Characterization of materials in these higher frequency ranges yield fundamental information about magnetization dynamics and are described in Part V.

Part VI of the book, consisting of 10 chapters, presents a set of current examples in which the magnetic properties of the materials are relevant, the peculiarities associated with measuring that kind of material or property are discussed, and illustrative results are presented. First-order reversal curves (FORC) has become a go-to technique for the characterization of hysteresis of any kind. In magnetism, it is used in fields as different as geomagnetism and nanomagnetism. Efficient energy conversion using magnetic materials is related to soft magnetic materials, permanent magnets, and magneto-caloric materials. In this latter case, it is shown that magneto-caloric characterization is, by itself, a tool to study phase transitions even for materials that will never be used in a magnetic refrigerator. The study of thermomagnetic hysteresis by FORC is also included in this chapter. Magnetic actuation or sensing by magnetostrictive materials, new trends in information storage technology using heat-assisted magnetic recording (HAMR), and the biological and medical applications of magnetic nanoparticles close the spectrum of emerging applications.

The idea for this book was developed by the Editors in October 2018 while Victorino Franco was a visiting scientist at Lake Shore Cryotronics. In December of 2018, Springer Nature authorized the Editors to develop the book. The Editors then proposed a table of contents, identified and invited authors to contribute chapter content. The broad distribution of scientific topics of the chapters in the book is also matched by a geographical distribution of the sources. Authors of the different chapters are all specialists in their respective fields and are from 9 countries: the United States, Canada, Austria, Germany, Romania, Spain, Switzerland, the United Kingdom, and Japan. Magnetics research nowadays is focused both on fundamental science and technological applications, thus authors from Academia, Industry and National Laboratories have contributed (each sector contributing 16, 6 and 4 chapters, respectively). This distribution is also evident in the editors of the book, with one being from Academia and the other from Industry.

Every project is always subject to contingencies and, in the case of this book, it was given the name of the novel Coronavirus COVID-19. This severely impacted the schedules of the contributing authors in 2020. All authors had to implement remote (virtual) learning curriculum, or adapt to remote working conditions with limited access to labs, which caused delays in research programs, etc. Despite the COVID-19 situation, authors never waivered in their enthusiasm for the book, and in their commitment to contribute high-quality content.

We would like to thank Springer Nature for providing us with the opportunity to develop this book. We are also deeply indebted to all authors for their excellent contributions, and their support of this project. Each of them had their public or private funding sources, listed at the end of each chapter. The whole collection is too long to be mentioned here. But we want to explicitly acknowledge all those funding agencies, foundations, and companies that financially support advancements in science that ultimately improve our society. At the same time, we also acknowledge the enthusiastic support of Lake Shore Cryotronics and the University of Seville

that enabled the successful completion of this endeavor. We hope this book proves to be a valuable resource for researchers, engineers, and students working in the field of magnetism and, equally importantly, to those who are new to this rich field of research.

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Contents

About the Editors

Victorino Franco is a professor in the Condensed Matter Physics Department of the University of Seville, Spain. His main research interests cover magnetic materials for energy applications, including soft magnetic and magnetocaloric materials. He has published more than 200 peer-reviewed technical articles on these topics. He received the Young Scientist Award from the Royal Physical Society of Spain in 2000 and was named IEEE Magnetic Society Distinguished Lecturer in 2019. He has served as chair of the Spain Chapter of the IEEE Magnetics Society, chair of the Magnetic Materials Committee of the Minerals, Metals & Materials Society (TMS), and member of the Steering Committee of the European School of Magnetism. He has been Editor and Publications Chair of several Magnetism and Magnetic Materials (MMM) conferences and the General Chair of the 2022 Joint MMM-INTERMAG Conference.

Brad Dodrill graduated from the Ohio State University in 1982 with a BSc degree in Physics. He completed 2 years of graduate studies in Physics and Electrical Engineering and took a position with Lake Shore Cryotronics in 1984 as a Research Scientist. He was promoted to Systems Applications Engineer (1989–1996) and served a key role in the development of a commercial AC susceptometer/DC magnetometer and a vibrating sample magnetometer. He later served as an Applications Scientist and Product Manager (1996–2002), and in 2002 he was appointed VP of Sales. In 2018 he transitioned to his current position as VP of Applications and Senior Scientist. He has 49 paper publications to his credit and holds 3 U.S. patents. He has lectured at numerous universities and given invited and contributed talks at technical conferences in the U.S., Europe, Asia, and Mexico on vibrating sample magnetometry, AC susceptometry, alternating gradient magnetometry, and on the use of the first-order reversal curve (FORC) measurement and analysis technique for the characterization of magnetic materials.

Part I
Units in Magnetism

Units for Magnetic Quantities

Ronald B. Goldfarb

Abstract The centimeter-gram-second (CGS) system of units was adopted by the pioneers of electromagnetism in the nineteenth century. By the early twentieth century, two limitations of the CGS system became apparent: its inability to gracefully incorporate the electrical units common in engineering and inconvenient factors of 4π in electromagnetic equations. Giovanni Giorgi was most responsible for the development of the rationalized meter-kilogram-second-ampere system, which evolved into the International System of Units (SI). In 2019, the SI was redefined in terms of seven defining constants of nature, which set the value of the elementary charge. A direct consequence is that the value of the magnetic constant, the permeability of vacuum, is no longer fixed in the SI. Some conversions from CGS electromagnetic units to SI units in an updated conversion table thus involve the redefined permeability of vacuum, whereas other conversions require only powers of 10 and factors of 4π. The effect on magnetism and magnetic measurements is more philosophical than practical.

Keywords Magnetism · Magnetism history · Magnetic units · Electromagnetic units · International System of Units · Giorgi system · Permeability of vacuum · Magnetic constant · Conversion table · Units of measure · Magnetic quantities · International Bureau of Weights and Measures

1 The Centimeter-Gram-Second System of Units

In 1873, the same year that James Clerk Maxwell published the first edition of *A Treatise on Electricity and Magnetism,* the Committee for the Selection and Nomenclature of Dynamical and Electrical Units, under the leadership of William Thomson (later known as Lord Kelvin), presented its first report at the 43rd

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meeting of the British Association for the Advancement of Science. It formally recommended the adoption of the centimeter-gram-second (CGS) system of units [1].

The following year, noting that “students usually find peculiar difficulty in questions relating to units,” the Committee commissioned a book to explain the new CGS system and give examples of its application to physical measurements [2]. The book, authored by the Committee’s secretary, Joseph David Everett, contained an appendix that reproduced the Committee’s first report to the British Association [3].

However, the appendix omitted the dissent for the record by Committee member George Johnstone Stoney, who objected that “the centimetre was recommended as the unit of length against my earnest remonstrance,” stating that “it is far too small.” Stoney predicted that “the metre must in the end be accepted as the standard unit of length” [1] (the British spelling “metre” is used in the original). Indeed, the Committee’s recommendation reversed the decision of its predecessor, the British Association’s Committee for Standards of Electrical Resistance, which had adopted the meter-gram-second (MGS) system [4, 5]. But by 1873, the CGS system was preferred over the MGS system because it had the advantage “of making the value of the density of water practically equal to unity” [1].

The CGS system is an “absolute” system, that is, one based on the fundamental mechanical units of length L, mass M, and time T. Thus, the quantities in the electrostatic (ESU) and electromagnetic (EMU) subsystems of CGS all resolve to whole or fractional powers of centimeters, grams, and seconds. For example, the dimensions for magnetic moment in EMU are $L^{5/2}\ M^{1/2}\ T^{-1}$, with units $\mathrm{cm}^{5/2}\cdot\mathrm{g}^{1/2}\cdot\mathrm{s}^{-1}$. Although magnetic moment has no named unit in EMU (recourse is often made to writing “emu” as a pseudo-unit), the units for magnetic moment correspond to those for the ratio of ergs per gauss: $\mathrm{cm}^2\cdot\mathrm{g}\cdot\mathrm{s}^{-2}/\mathrm{cm}^{-1/2}\cdot\mathrm{g}^{1/2}\cdot\mathrm{s}^{-1}$. (The name “erg” was recommended as the unit for work and energy by the British Association in 1873. The name “gauss” was assigned, initially, to magnetic field strength by the International Electrical Congress in 1900 and, later, to magnetic flux density by the International Electrotechnical Commission in 1930.)

It was the intent of the British Association’s Committee for the Selection and Nomenclature of Dynamical and Electrical Units that “one definite selection of three fundamental units be made once for all” so “that there will be no subsequent necessity for amending it” [1].

It was not to be.

2 The Rationalized Meter-Kilogram-Second-Ampere System

One of Oliver Heaviside’s many accomplishments was the reformulation of Maxwell’s cartesian equations in compact vector calculus notation. He believed that the factor of 4π in electromagnetic equations was simply an illogical convention, and he made a strong case for rationalization of the CGS system, that is, removal of the irrational number 4π in most equations, including those of Maxwell [6].

Giovanni Giorgi viewed rationalization as an optional but convenient adjunct to a four-dimensional, meter-kilogram-second (MKS) system, in which the fourth, electromagnetic unit was initially not specified [7]. Giorgi respectfully submitted preprints of his papers to Heaviside, who was 21 years his senior and quite famous. Heaviside was skeptical, as evidenced by his notations on Giorgi's correspondence, currently in the archives of the International Electrotechnical Commission [8]. In Giorgi's typewritten letter of 11 March 1902 to Heaviside, he outlined the differences between their two systems: "My object was in fact not only to get rid of the 4π, but to bring the practical electrical units into agreement with a set of mechanical units of reasonable size, and then to have a system which is absolute and practical at the same time." Years later, Giorgi extended the classical definition of an absolute system of units by noting the equivalence of mechanical and electrical energy and thus applied the coveted "absolute" adjective to his four-dimensional MKS system [9].

The meaning of the permeability of vacuum μ_0 was central to Giorgi's system [10]. He noted, "In my system, [μ_0] is not a numeric, nor do I assume any special value for it; it is a physical quantity, having dimensions, and to be measured by experiment" [11]. Thus, he regarded both μ_0 and the permittivity of vacuum ε_0 as subject to experimental refinement, with $\mu_0 \approx 1.256 \times 10^{-6}$ henries per meter and $\varepsilon_0 \approx 8.842 \times 10^{-12}$ farads per meter, and both subject to the condition that $(\mu_0\ \varepsilon_0)^{-1/2}$ is equal to the speed of light $c \approx 3 \times 10^8$ m/s. He noted that his four-dimensional system "is neither electrostatic nor electromagnetic, because neither the electric nor the magnetic constant of free ether is assumed as a fundamental unit" [12].

Opposition to the full adoption of Giorgi's system was led by Richard Glazebrook, a former student and intellectual heir of Maxwell, who served as the chair of the Symbols, Units, and Nomenclature (SUN) Commission of the International Union of Pure and Applied Physics. The SUN Commission accepted the three-dimensional MKS as a parallel system but with μ_0 as just a fixed scaling factor with respect to the CGS system [10].

3 The International System of Units

Eventually, in 1954, the 10th General Conference on Weights and Measures (CGPM) approved the ampere as the fourth base unit, thereby formalizing the "MKSA" practical system of units. In 1960, the 11th CGPM adopted the name *Système International d'Unitès,* with the abbreviation "SI," for the practical system of units. In the SI, the "definition of the ampere was based on the force between two current carrying conductors and had the effect of fixing the value of the vacuum magnetic permeability μ_0 (also known as the magnetic constant) to be exactly $4\pi \times 10^{-7}$ H·m^{-1} = $4\pi \times 10^{-7}$ N·A^{-2}" [13].

On 16 November 2018, in Versailles, France, the 26th CGPM adopted the most significant change in units of measure since 1954. It went into effect on

20 May 2019, World Metrology Day. The revised SI fixed the values of formerly measurable constants: the Planck constant, h; the elementary charge, e; the Boltzmann constant, k; and the Avogadro constant, N_A, thereby, individually or in combination, redefining the units kilogram, ampere, kelvin, and mole. The cesium 133 hyperfine transition frequency, $\Delta\nu_{Cs}$; the luminous efficacy of radiation of frequency 540×10^{12} Hz, K_{cd}; and the speed of light in vacuum, c, had already been fixed by the CGPM in 1967, 1979, and 1983, respectively, which defined the units second, candela, and meter [13].

The motivation for the use of defining constants is explained carefully in the 9th edition of the *SI Brochure* [[13], pp. 125–126]:

> Historically, SI units have been presented in terms of a set of—most recently seven—base units. All other units, described as derived units, are constructed as products of powers of the base units.
>
> Different types of definitions for the base units have been used: specific properties of artefacts such as the mass of the international prototype for the unit kilogram; a specific physical state such as the triple point of water for the unit kelvin; idealized experimental prescriptions as in the case of the ampere and the candela; or constants of nature such as the speed of light for the definition of the unit metre.
>
> To be of any practical use, these units not only have to be defined, but they also have to be realized physically for dissemination. In the case of an artefact, the definition and the realization are equivalent—a path that was pursued by advanced ancient civilizations. Although this is simple and clear, artefacts involve the risk of loss, damage or change. The other types of unit definitions are increasingly abstract or idealized. Here, the realizations are separated conceptually from the definitions so that the units can, as a matter of principle, be realized independently at any place and at any time. In addition, new and superior realizations may be introduced as science and technologies develop, without the need to redefine the unit. These advantages—most obviously seen with the history of the definition of the metre from artefacts through an atomic reference transition to the fixed numerical value of the speed of light—led to the decision to define all units by using defining constants.
>
> The choice of the base units was never unique, but grew historically and became familiar to users of the SI. This description in terms of base and derived units is maintained in the present definition of the SI, but has been reformulated as a consequence of adoption of the defining constants.

Instead of the definition of the ampere fixing the value of μ_0, the 2019 revision of the SI defines the ampere in terms of the fixed value of e. As a result, the value of μ_0 must be determined experimentally. Similarly, the permittivity of vacuum $\varepsilon_0 = 1/(\mu_0 c^2)$ must be determined experimentally (as it was before c was fixed in 1983). The product $\mu_0\varepsilon_0 = 1/c^2$ remains exact. The experimental value of μ_0 is now based on that of the dimensionless fine-structure constant α, the coupling constant of the electromagnetic force: $\mu_0 = 2h\alpha/ce^2$, where h is the newly fixed Planck constant, c is the fixed speed of light in vacuum, and e is the newly fixed elementary charge (equal to the absolute value of the electron charge). The relative standard uncertainties in μ_0, ε_0, and α are identical [14].

It was reasonable to fix the value of e instead of μ_0 because, by the 1990s, the realization of the ampere was by Ohm's law, the Josephson effect for voltage, and the quantum Hall effect for resistance (both in terms of the 1990 recommended values of e and h [15]), not by the force on currents in parallel wires. A definition of the ampere and the kilogram in terms of fixed values of e and h, respectively, brought the practical quantum electrical standards into exact agreement with the SI [13].

4 Conversion Factors

Conversion tables are helpful for magnetics researchers who want to compare data appearing in published articles. The need will diminish with time as the SI becomes universal for instruction in electromagnetism. Magnetics researchers who currently measure in SI units and analyze using SI equations do not have to worry about conversion factors, but even they occasionally need to refer to published data in EMU.

Units of measure have been examined and reexamined vigorously. The monograph by Silsbee is noteworthy for its completeness [16]. The appendixes in the textbooks by Jackson [17] and Coey [18] are good resources. Few articles deal specifically with units for magnetic properties. Bennett et al. published a conversion guide especially for magnetics in which they pointed out, to the surprise of many, that “emu” is not actually a unit [19]. During an evening panel discussion on magnetic units at the 1994 Joint Magnetism and Magnetic Materials—International Magnetics Conference, different perspectives were advanced by seven practitioners [20], some of whom recapitulated their recent articles or prefaced their future articles on the subject [21, 22, 23].

In the MKSA system and the SI of 1960, μ_0 served both as a conversion factor and as a means for rationalization with respect to EMU. Thus, the 2019 revision of the SI, which made μ_0 an experimental constant, has consequences for magnetics. A conversion guide for magnetic quantities from EMU to SI may now distinguish between conversions based on an experimental determination of μ_0 and conversions based on rationalization of EMU. As first noted by Davis, conversion factors to CGS systems, such as EMU, which made use of the exact relation $\{\mu_0/4\pi\} \equiv 10^{-7}$, are no longer exactly correct after the SI revision of 2019 [24] (The curly brackets mean that one removes the units associated with the quantity within.)

Table 1 is a conversion guide from EMU to SI that reflects the redefinition of the SI. Conversion factors formerly based on the fixed permeability of vacuum $\{\mu_0\} \equiv 4\pi \times 10^{-7}$ are here replaced explicitly by the symbol $\{\mu_0\}$. However, factors based only on the conversion of centimeters to meters, grams to kilograms, and rationalization of EMU retain the factor of 4π; for example, the sum of the three axial demagnetizing factors of an ellipsoid is 4π in EMU and unity in the SI.

Magnetism in the SI is concordant with the Sommerfeld constitutive relation $B = \mu_0(H + M)$ for magnetic flux density B, magnetic field strength H, and magnetization M. However, magnetic polarization J and magnetic dipole moment j, derived from the Kennelly convention, $B = \mu_0 H + J$, are also recognized. In both conventions, B and H have units different from each other.

Table 1 Conversion of units for magnetic quantities. In the right column, $\{\mu_0\}$ refers to the numerical value of μ_0, the recommended value of which may change slightly over time. Factors of 4π originate from the conversion of unrationalized EMU to rationalized SI units. In the absence of units, a dimensionless quantity is labeled with its associated system of units (EMU or SI). The arrows (→) indicate correspondence, not equality. From [10], after [25]

SI Symbol	SI Quantity	Conversion from EMU and Gaussian Units to SI Units [(a)]
Φ	Magnetic flux	1 Mx = 1 G·cm^2 → 10^{-8} Wb = 10^{-8} V·s
B	Magnetic flux density, magnetic induction	1 G → 10^{-4} T = 10^{-4} Wb/m^2
μ	Permeability [(b)]	1 (EMU) → $\{\mu_0\}$ H/m = $\{\mu_0\}$ N/A^2 = $\{\mu_0\}$ Wb/(A·m)
H	Magnetic field strength, magnetizing force	1 Oe → $10^{-4}/\{\mu_0\}$ A/m
m	Magnetic moment	1 erg/G = 1 emu → 10^{-3} A·m^2 = 10^{-3} J/T
j	Magnetic dipole moment	1 erg/G = 1 emu → $10^{-3}\{\mu_0\}$ Wb·m
M	Magnetization, volume magnetization	1 erg/(G·cm^3) = 1 emu/cm^3 → 10^3 A/m
		1 G → $10^{-4}/\{\mu_0\}$ A/m
J, I	Magnetic polarization, intensity of magnetization	1 G → 10^{-4} T = 10^{-4} Wb/m^2
σ	Specific magnetization, mass magnetization	1 erg/(G·g) = 1 emu/g → 1 A·m^2/kg
χ	Susceptibility, volume susceptibility	1 (EMU) → 4π (SI)
χ_ρ, χ_m	Specific susceptibility, mass susceptibility	1 cm^3/g → 4π × 10^{-3} m^3/kg
w, W	Energy product, volume energy density [(c)]	1 erg/cm^3 → 10^{-1} J/m^3
N, D	Demagnetizing factor	1 (EMU) → $(4\pi)^{-1}$ (SI)

(a) EMU are the same as Gaussian units for magnetostatics: Mx = maxwell, G = gauss, Oe = oersted. SI: Wb = weber, T = tesla, H = henry, N = newton, J = joule.
(b) In the SI, relative permeability $\mu_\mathrm{r} = \mu/\mu_0 = 1 + \chi$. In EMU, permeability $\mu = 1 + 4\pi\chi$. Relative permeability μ_r in the SI corresponds to permeability μ in EMU.
(c) In the SI, w [J/m^3] = B [T] · H [A/m] = μ_0 [Wb/(A·m)] · M [A/m] · H [A/m]. In EMU, w [erg/cm^3] = $(4\pi)^{-1}$ B [G] · H [Oe] = M [erg/(G·cm^3)] · H [Oe].

In EMU, $B = H + 4\pi M$, where B and H have the same units with different names, gauss (G) and oersted (Oe). As has been noted, "the magnetization, when written as $4\pi M$, is also in gausses and may be thought of as a field arising from the magnetic moment. When magnetization is expressed simply as M (the magnetic moment m per unit volume), its units are erg·G^{-1}·cm^{-3}. In terms of base units, erg = cm^2·g·s^{-2} and G = cm$^{-1/2}$·g$^{1/2}$·s^{-1}; therefore, erg·G^{-1}·cm^{-3}, the units for M, are dimensionally but not numerically equivalent to G" [21].

In the table, dimensionless quantities are labeled with their associated system of units (EMU or SI) to distinguish them. In magnetic materials with permeability μ, $B = \mu H$, where μ is dimensionless in EMU. The conversion of dimensionless volume susceptibility χ from EMU to SI is based on the correspondence between $\mu = 1 + 4\pi\chi$ in EMU and relative permeability $\mu_\mathrm{r} = \mu/\mu_0 = 1 + \chi$ in SI; that is, $4\pi\chi$ (EMU) corresponds to χ (SI); $\{\mu_0\}$ is not involved. This also follows from the definition $\chi = M/H$, in both EMU and SI, and $4\pi\chi$ (EMU) having units of gausses per oersted (dimensionless). The conversion of specific (mass) susceptibility follows from that of volume susceptibility.

The SI redefinition of the ampere implies that the EMU abampere (the prefix "ab" means "absolute") does not convert exactly to 10 amperes, as was similarly footnoted by Quincey and Brown in relation to the abcoulomb and coulomb [26]. This affects the conversion of magnetic field strength H from oersteds (the named unit for gilberts per centimeter, which corresponds to $(4\pi)^{-1}$ abamperes per centimeter) to amperes per meter by requiring the use of $\{\mu_0\}$. Alternatively, the conversion factor of $10^{-4}/\{\mu_0\}$ in the table may be considered to arise from the equivalence of oersted and gauss in EMU, the conversion of gauss to tesla, and the relationship $B = \mu_0 H$ in vacuum. The same factor is used in the table for the conversion of magnetization *M,* when formulated as $4\pi M$ in gausses, to amperes per meter.

Conversions based on transformations from gausses to teslas and ergs to joules do not involve $\{\mu_0\}$. For example, magnetization in gausses converts to magnetic polarization in teslas without involvement of $\{\mu_0\}$. However, magnetic moment, when expressed in EMU as ergs per gauss (or "emu"), converts to magnetic dipole moment in weber meters with a required factor of $\{\mu_0\}$.

5 Epilogue

While the accepted value of $\{\mu_0\}$ will change slightly over time with changes in the experimental fine-structure constant α, $\{\mu_0\}$ is currently equal to $1.256\,637\,0621 \times 10^{-6} \pm 0.000\,000\,0019 \times 10^{-6}$, based on the latest quadrennial adjustment to the fundamental physical constants by the International Science Council's Committee on Data [27]. That is, the value of $\{\mu_0\}$ is equal to $4\pi \times 10^{-7}$ to nine significant figures. Thus, the distinction between $\{\mu_0\}$ and $4\pi \times 10^{-7}$ is largely philosophical and hardly practical; their difference is much smaller than the total uncertainty in any magnetic measurement.

In the revised SI, it is compelling to regard *B* as the primary magnetic field vector, μ_0 as an experimental constant, and *H* as an arithmetically derived auxiliary vector [10]. For displays of measurement data, the symbol B_0 could be used for applied magnetic field in units of teslas, much as $\mu_0 H$ is sometimes used, where B_0 is distinguished from the flux density *B* in magnetic materials. Magnetic volume susceptibility χ should remain defined as M/H (dimensionless), not M/B_0, because M/H is embedded historically in EMU, the MKSA system, and the SI.

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References

1. W. Thomson, G.C. Foster, J. Clerk Maxwell, G.J. Stoney, F. Jenkin, C.W. Siemens, F.J. Bramwell, J.D. Everett, First report of the Committee for the Selection and Nomenclature of Dynamical and Electrical Units, *Report of the Forty-Third Meeting of the British Association for the Advancement of Science,* pp. 222–225 (1873). https://www.biodiversitylibrary.org/item/94452
2. W. Thomson, G.C. Foster, J. Clerk Maxwell, G.J. Stoney, F. Jenkin, C.W. Siemens, F.J. Bramwell, W.G. Adams, B. Stewart, J.D. Everett, Second report of the Committee for the

Selection and Nomenclature of Dynamical and Electrical Units," *Report of the Forty-Fourth Meeting of the British Association for the Advancement of Science,* p. 255 (1874). https://www.biodiversitylibrary.org/item/94451

3. J.D. Everett, *Illustrations of the Centimetre-Gramme-Second (C.G.S.) System of Units* (Taylor and Francis, London, 1875). https://archive.org/details/b22295641
4. A. Williamson, C. Wheatstone, W. Thomson, W.H. Miller, A. Matthiessen, F. Jenkin, C. Bright, J. Clerk Maxwell, C.W. Siemens, B. Stewart, J.P. Joule, C.F. Varley, Report of the Committee on Standards of Electrical Resistance, *Report of the Thirty-Fourth Meeting of the British Association for the Advancement of Science,* pp. 345–367 (1864). https://www.biodiversitylibrary.org/item/93072
5. A.E. Kennelly, I.E.C. adopts MKS system of units. Trans. Am. Inst. Electr. Eng. **54**, 1373–1384 (1935). https://doi.org/10.1109/T-AIEE.1935.5056934
6. O. Heaviside, *Electromagnetic Theory, Vol. 1, Ch. 2* (The Electrician, London, 1893), pp. 116–127. https://archive.org/details/electromagnetict01heav
7. G. Giorgi, Unità razionali di elettromagnetismo, Atti dell' Associazione Elettrotecnica Italiana, **5**, 402–418 (1901). Republished as: "Sul sistema di unità di misure elettromagnetiche," Il Nuovo Cimento, **4**, 11–30 (1902), https://doi.org/10.1007/BF02709460. English translation in: C. Egidi, Ed. *Giovanni Giorgi and His Contribution to Electrical Metrology,* (Politecnico di Torino, Turin, Italy, 1990), pp. 135–150.
8. IEC, *Giovanni Giorgi's Correspondence with Oliver Heaviside* (Int. Electrotech. Comm., Geneva, 1902). https://www.iec.ch/history/historical-documents
9. G. Giorgi, *Memorandum on the M.K.S. System of Practical Units,* (International Electrotechnical Commission, London, 1934). Republished: IEEE Magn. Lett., **9**, 1205106 (2018). https://doi.org/10.1109/LMAG.2018.2859658
10. R.B. Goldfarb, Electromagnetic units, the Giorgi system, and the revised International System of Units. IEEE Magn. Lett. **9**, 1205905 (2018). https://doi.org/10.1109/LMAG.2018.2868654
11. G. Giorgi, Rational electromagnetic units, in *Electrical World and Engineer*, vol. 40, (McGraw-Hill, New York, 1902), pp. 368–370; editorial comment on p. 355. https://books.google.com/books?id=e15NAAAAYAAJ
12. G. Giorgi, Proposals concerning electrical and physical units. J. Inst. Electr. Eng. **34**, 181–185 (1905). https://doi.org/10.1049/jiee-1.1905.0013
13. BIPM, *The International System of Units*, 9th edn. (Bureau Int. des Poids et Mesures, Sèvres, France, 2019). https://www.bipm.org/utils/common/pdf/si-brochure/SI-Brochure-9.pdf
14. R.B. Goldfarb, The permeability of vacuum and the revised International System of Units (editorial). IEEE Magn. Lett. **8**, 1110003 (2017). https://doi.org/10.1109/LMAG.2017.2777782
15. N. Fletcher, G. Rietveld, J. Olthoff, I. Budovsky, M. Milton, Electrical units in the new SI: Saying goodbye to the 1990 values. NCSLI Measure J. Meas. Sci. **9**, 30–35 (2014). https://doi.org/10.1080/19315775.2014.11721692
16. F.B. Silsbee, Systems of electrical units. J. Res. Natl. Bur. Stand. **66C**, 137–183 (1962). https://doi.org/10.6028/jres.066C.014. Republished: Nat. Bur. Stand. Monograph 56 (1963). https://nvlpubs.nist.gov/nistpubs/Legacy/MONO/nbsmonograph56.pdf
17. J.D. Jackson, *Classical Electrodynamics*, 3rd edn. (Wiley, New York, 1998), pp. 775–784. https://www.wiley.com/en-us/Classical+Electrodynamics%2C+3rd+Edition-p-9780471309321
18. J.M.D. Coey, *Magnetism and Magnetic Materials* (Cambridge University Press, Cambridge, 2010), pp. 590–594 and inside back cover. https://doi.org/10.1017/CBO9780511845000
19. L.H. Bennett, C.H. Page, L.J. Swartzendruber, Comments on units in magnetism. J. Res. Natl. Bur. Stand. **83**, 9–12 (1978). https://doi.org/10.6028/jres.083.002. Republished from: Proc. 1975 Magn. Magn. Mater. Conf., Philadelphia, Penn., *AIP Conf. Proc*. **29**, xix–xxi (1976). New York: Am. Inst. Phys.
20. C.D. Graham, S. Chikazumi, R. Street, A. Arrott, J.M.D. Coey, M.R.J. Gibbs, R.B. Goldfarb, Panel discussion on units in magnetism. IEEE Magn. Soc. Newsl. **31**(4), 5–8 (1994) https://www.ieeemagnetics.org/files/newsletters/IEEEMS-N-31-4-Oct-1994.pdf. Republished: *Magnetic and Electrical Separation*, **6**, 105–116 (1995)

21. R.B. Goldfarb, Magnetic units and material specification, in *Concise Encyclopedia of Magnetic and Superconducting Materials*, ed. by J.E. Evetts, 1st edn., (Pergamon Press, Oxford, 1992), pp. 253–258. https://www.worldcat.org/title/concise-encyclopedia-of-magnetic-superconducting-materials/oclc/924972388
22. A.S. Arrott, "Magnetism in SI and Gaussian units," Ch. 1.2, in *Ultrathin Magnetic Structures I*, ed. by J.A.C. Bland, B. Heinrich, (Springer-Verlag, Berlin & Heidelberg, 1994), pp. 7–19. https://www.springer.com/gp/book/9783540219552
23. J. Crangle, M. Gibbs, Units and unity in magnetism: A call for consistency. Phys. World **7**, 31–32 (1994). https://doi.org/10.1088/2058-7058/7/11/29
24. R.S. Davis, Determining the value of the fine-structure constant from a current balance: Getting acquainted with some upcoming changes to the SI. Am. J. Phys. **85**, 364–368 (2017). https://doi.org/10.1119/1.4976701
25. R.B. Goldfarb, F.R. Fickett, *Units for Magnetic Properties, National Bureau of Standards Special Publication 696* (U.S. Gov. Printing Office, Washington, D.C., 1985). https://archive.org/details/unitsformagnetic696gold
26. P. Quincey, R.J.C. Brown, A clearer approach for defining unit systems. Metrologia **54**, 454–460. https://doi.org/10.1088/1681-7575/aa7160
27. CODATA, Committee on Data of the International Science Council, Task Group on Fundamental Physical Constants (2018). https://physics.nist.gov/cuu/Constants/index.html, https://codata.org/initiatives/strategic-programme/fundamental-physical-constants

Part II
Inductive and Force-Based Techniques for Measuring Bulk Magnetic Properties

Vibrating Sample Magnetometry

Brad Dodrill and Jeffrey R. Lindemuth

Abstract A magnetometer is an instrument to measure the magnitude and direction of a magnetic field. The most commonly used magnetometric technique to characterize magnetic materials is vibrating sample magnetometry (VSM). VSMs can measure the magnetic properties of magnetically soft (low coercivity) and hard (high coercivity) materials in many forms: solids, powders, single crystals, thin films, or liquids. They can be used to perform measurements from low to high magnetic fields employing electromagnets, Halbach rotating permanent magnet arrays, or high-field superconducting magnets. They can be used to perform measurements from very low to very high temperatures with integrated cryostats or furnaces, respectively. And, they possess a dynamic range extending from 10^{-8} emu (10^{-11} Am^2) to above 10^3 emu (1 Am^2), enabling them to measure materials that are both weakly magnetic (ultrathin films, nanoscale structures, etc.) and strongly magnetic (permanent magnets). In this chapter, we will discuss the VSM measurement technique and its implementation in an electromagnet. We will also discuss relevant extensions of the technique that provide variable temperature capability, a vector VSM for magnetic anisotropy studies, and implementation of data acquisition algorithms for first-order reversal curve (FORC) measurements for characterizing magnetic interactions and coercivity distributions in magnetic materials. We will present typical measurement results over a range of experimental conditions for various materials to demonstrate the VSM capability for magnetic materials characterization.

Keywords Magnetometry · Hysteresis · Vibrating sample magnetometer, VSM · Vector VSM, magnetic anisotropy · Variable temperature measurements · First-order reversal curve · FORC

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1 Magnetic Measurement Techniques

Magnetometry techniques can be classified into several broad classes: inductive-based, for example, vibrating sample magnetometry (VSM) and superconducting quantum interference device (SQUID) [1] magnetometry; force-based, for example, alternating gradient magnetometry (AGM) [2] and Faraday [3] and Gouy balance [4]; and optically based, for example, nitrogen vacancy defects in diamond [5].

Some magnetic materials such as nanowires, nanoparticles, thin films, etc., typically possess weak magnetic signatures, owing to the small amount of magnetic material that is present. Thus, one of the most important considerations in determining which type of magnetometer is best suited to specific materials is its sensitivity, as this determines the smallest magnetic moment that may be measured with acceptable signal-to-noise ratio. Measurement speed, i.e., the time required to measure a hysteresis loop, is also important because it determines sample throughput, and it is particularly important for First Order Reversal Curve (FORC) measurements because a typical series of FORCs can contain thousands to tens of thousands of data points. The final consideration is the temperature and field range over which measurements are to be performed, and this is dictated largely by the magnetic materials that are being studied.

Commercial VSM systems provide measurements to field strengths of ~34 kOe (3.4 T) using conventional electromagnets [6, 7], as well as systems employing superconducting magnets to produce fields to 160 kOe (16 T) [8, 9]. In an electromagnet-based VSM, the magnetic field can be swept at up to 10 kOe/s (1 T/s), and a typical hysteresis loop measurement can take as little as a few seconds to a few minutes, and a typical series of FORCs takes minutes to hours. When used with superconducting magnets, higher field strengths are possible, which are necessary to saturate some magnetic materials such as rare-earth permanent magnets; however, the measurement speed is inherently slower due to the speed at which the magnetic field can be varied using superconducting magnets due to their large inductance. Field sweep rates are typically limited to 200 Oe/s (20 mT/s), and thus a typical hysteresis loop measurement can take tens of minutes or more, and a typical series of FORCs can take a day or longer. Magnetometers employing superconducting magnets are more costly to operate since they require liquid helium. Cryogen-free systems employing closed-cycle refrigerators, or liquefiers that recover helium in liquid helium-based systems, are available, but these represent an expensive capital equipment investment. The noise floor of commercially available VSMs is in the 10^{-7}–10^{-8} emu (10^{-10}–10^{-11} Am^2) range.

The most common measurement used to characterize a material's magnetic properties is measurement of the major hysteresis or $M(H)$ loop as illustrated in Fig. 1. The main parameters extracted from the hysteresis loop that are used to characterize the properties of magnetic materials include the saturation magnetization M_s (the magnetization at maximum applied field), the remanence M_r (the magnetization at zero applied field after applying a saturating field), and the coercivity H_c (the field required to demagnetize the sample).

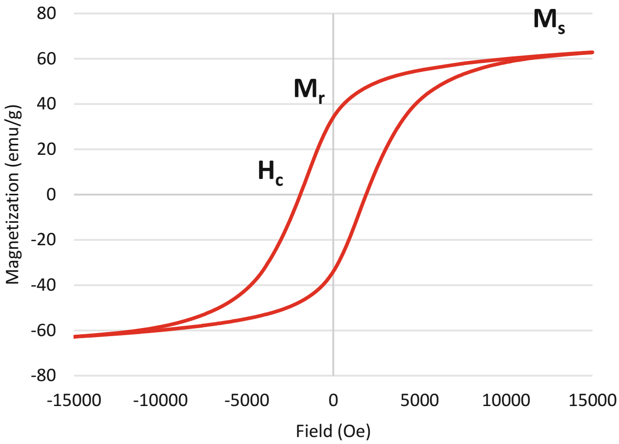

Fig. 1 A typical hysteresis loop showing the extracted parameters M_s, M_r, and H_c

More complex magnetization curves covering states with field and magnetization values located inside the major hysteresis loop, such as minor hysteresis loops and FORCs, can give additional information that can be used for characterization of magnetic interactions [10].

All VSM results presented in this chapter were recorded using a Lake Shore Cryotronics Model 8600 electromagnet-based VSM.

2 Electromagnet-Based VSM

The vibrating sample magnetometer was originally developed by Simon Foner [11] of MIT's Lincoln Laboratory. Foner patented the VSM technology [12] and sold an exclusive license to Princeton Applied Research Corporation (PARC) to develop and market the VSM. The early VSMs were called Foner magnetometers.

In an electromagnet-based VSM, a magnetic material is vibrated within a uniform magnetic field H generated by an electromagnet, inducing an electric current in suitably placed sensing coils. The resulting voltage induced in the sensing coils is proportional to the magnetic moment of the sample. Variable temperature measurements can be performed from <4.2 to 1273 K using integrated cryostats and furnaces, respectively.

Figure 2 shows a schematic representation of an electromagnet-based VSM. A variable magnetic field in the x direction is produced by an electromagnet energized by an appropriate bipolar power supply. Four-coil transverse detection or sensing coils [13] are mounted on the pole faces of the magnet, two on each face. The coils are balanced so as to produce zero signal (voltage) in the absence of a sample. A Hall probe, which is connected to a gaussmeter, is also mounted on the electromagnet

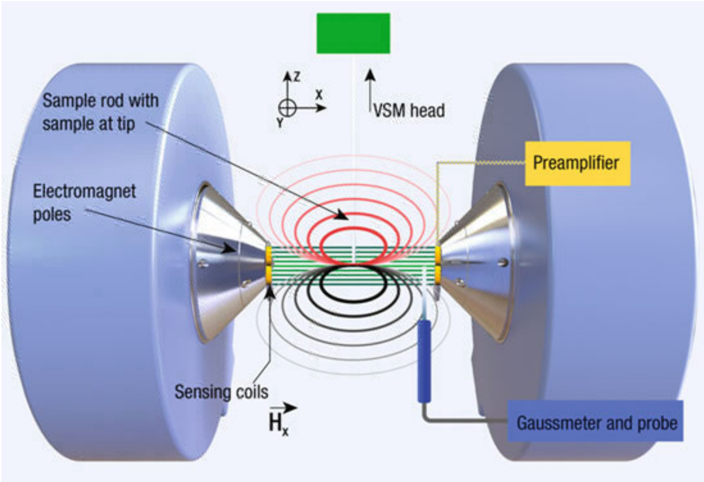

Fig. 2 Schematic representation of a VSM. The red and black contours represent the dipole magnetic field of a magnetized sample

pole face for closed-loop control of the magnetic field. A sample of any form (solid, powder, thin film, etc.) is placed in a suitable non-magnetic sample holder which is attached to the end of the VSM sample rod, which is in turn attached to the VSM head. The sample is vibrated in the z direction within the sensing coils, and the resulting induced voltage is passed through a preamplifier and then to a narrow bandwidth lock-in amplifier (LIA). The LIA reference is phased locked to the head drive vibration frequency.

The voltage induced in the VSM sensing coils is given by:

$$V_{\mathrm{emf}} = mAfS \tag{1}$$

where:

m = magnetic moment.
A = amplitude of vibration.
f = frequency of vibration.
S = sensitivity function of the VSM sensing coils.

S is determined by calibrating the VSM with a magnetic calibrant [14], i.e., a material with known magnetization at a specified applied field H.

A VSM's sensitivity depends on a number of factors:

- Electronic sensitivity.
- Noise rejection through signal conditioning.
- Amplitude and frequency of mechanical drive.
- Thermal noise of sensing coils.

- Optimized design and coupling (proximity) of sensing coils to the sample under test.
- Vibration isolation of the mechanical head assembly from the electromagnet and VSM sensing coils.
- Minimization of environmental mechanical and electrical noise sources, which can deleteriously effect VSM sensitivity.

It is clear from Eq. (1) that increasing A, f, or S will improve moment sensitivity; however, there are practical limitations to each. Frequencies of less than ~100 Hz are typically used so as to minimize eddy current generation in magnetic materials that are electrically conductive, and it is also important to avoid frequencies that are close to the line frequency and its higher-order harmonics. The vibration amplitude should be sufficiently small to ensure that the sample is not subjected to inhomogeneous magnetic fields arising from the field source. S may be increased by optimizing the design of the sensing coils (i.e., number of windings, coil geometry, etc.) and by increasing the coupling between the sense coils and the sample under test (i.e., minimize gap spacing). When the sensing coils and sample are very close together, finite sample size effects [15] can lead to errors in the measured sample magnetization. These errors can be mitigated by using a calibrant that is geometrically identical to the sample. At first glance, it would seem that all that one needs to do to increase S is to maximize the number of windings in the coils; however, this increases the resistance of the coils, which in turn increases their thermal noise which negatively impacts their signal-to-noise ratio. Finally, increasing signal averaging of the LIA also improves signal-to-noise ratio.

VSM Sensitivity Examples: Figure 3 shows typical noise measurement results at 100 ms/point (top) and 10 s/point (bottom) averaging. Note that the vertical axis is expressed in nemu = 10^{-9} emu (10^{-12} Am^2). The RMS noise values are noted in the figure caption.

Figure 4 shows typical low moment measurement results for a CoPt bit-patterned (bit size <100 nm) magnetic media (thin film) sample with saturation moment m_{sat} < 20 μemu (20 × 10^{-9} Am^2). The hysteresis loop was recorded to ±5 kOe (0.5 T) in 25 Oe (2.5 mT) steps at 100 ms/point averaging. The total loop measurement time was 1 min 25 s. Figure 5 shows results for a synthetic antiferromagnetic thin film with m_{sat} < 2 μemu (2 × 10^{-9} Am^2). The hysteresis loop was recorded to ±500 Oe (50 mT) in 2.5 Oe (0.25 mT) steps at 2 s/point averaging. The total loop measurement time was 28 min.

3 VSM Components and Extensions

Aside from the electromagnet and associated bipolar power supply, the principal VSM components include the sensing coils, vibration head, control and measurement electronics, and data acquisition software. In this section, we will discuss the coils, head, and electronics.

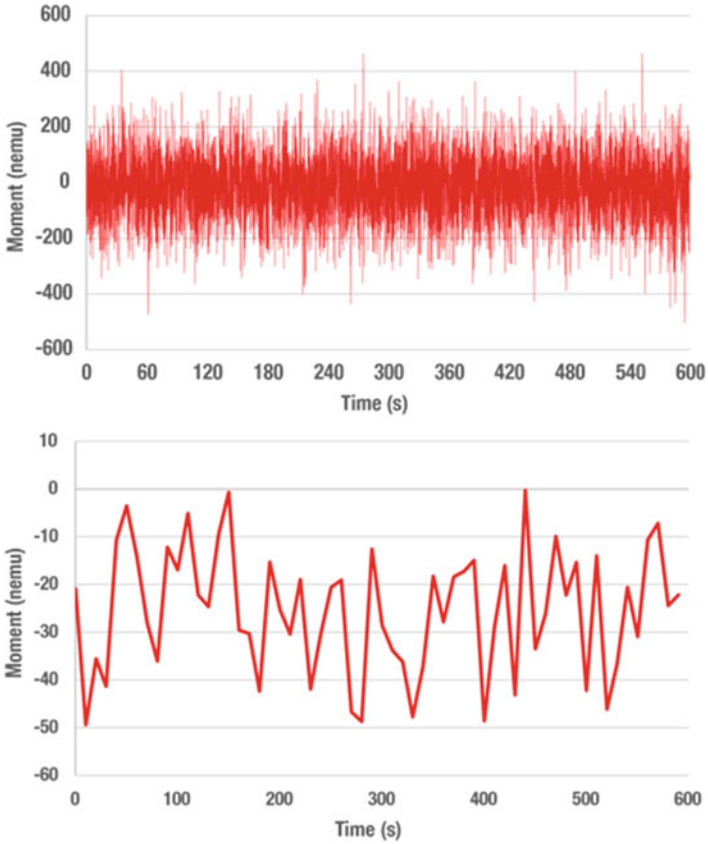

Fig. 3 (**a**, **b**) Noise at 100 ms/point and 10 s/point (**a**, top) and 10 sec/point (**b**, bottom) averaging. The observed noise is 119.5 nemu and 13 nemu RMS, respectively

Sensing Coils: Various transverse detection or sensing coil configurations have been proposed, but the most commonly employed in an electromagnet VSM is the four-coil configuration proposed by Mallinson [16] shown in Fig. 6. Practical detection coil arrangements utilize an even number of coils to minimize sensitivity to sample position. The coils are typically balanced (i.e., geometrically identical with precisely the same number of windings). A perfectly balanced coil set produces zero signal in the absence of a sample. A nonzero signal is a consequence of not having a perfectly balanced coil set, but such offsets are usually small and can be removed with appropriate nulling electronics. Finding the appropriate balance between the number of windings and the resistance of the coils is important to maximize their sensitivity without creating thermal noise. The size (or diameter in the case of circular coils) of the coils should be larger than the sample under test and at the same time sufficiently small to ensure they are in a homogeneous magnetic field. In the configuration shown in Fig. 6, the axes of the coils are parallel

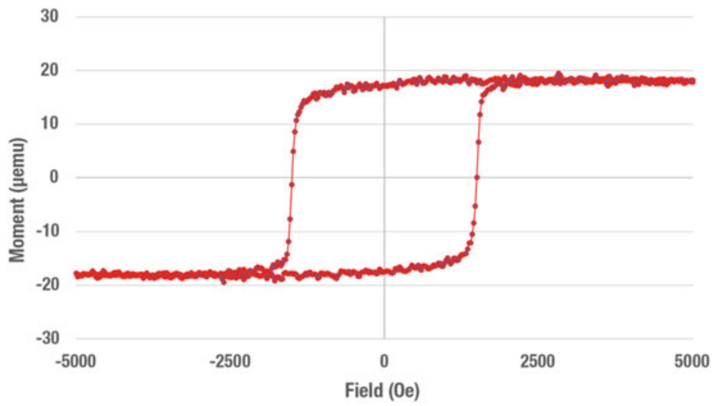

Fig. 4 Hysteresis loop for a 20 μemu CoPt nanomagnet array

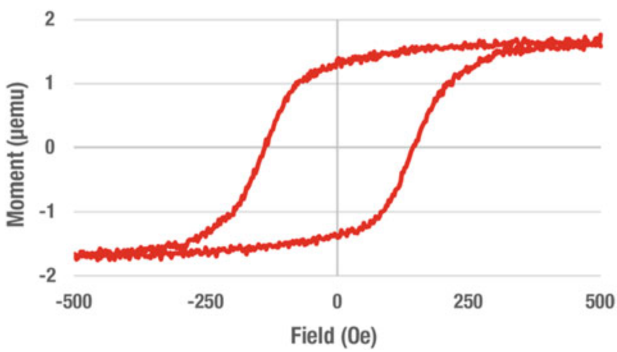

Fig. 5 Hysteresis loop for a < 2 μemu synthetic antiferromagnetic thin film

to the applied field (x) and transverse to the sample vibration (z). A magnetic sample is properly positioned within the sensing coils by adjusting the xyz position of the sample via micrometers attached to the vibrating head to maximize the signals in the z and y directions and minimize the signal in the x direction.

Vibrating Head: The vibrating head must provide a vibration of constant frequency and amplitude as a function of time. If either drifts, then the voltage induced in the sensing coils drifts, which produces an apparent drift in a samples magnetization. Frequencies should not be close to the line frequency or its higher-order harmonics and should be less than 100 Hz to minimize eddy currents in electrically conductive materials. The drive amplitude should be sufficiently small to ensure that a sample is not subjected to an inhomogeneous magnetic field, and it should be less than the sensing coil diameter. The head should provide a stable reference signal for lock-in detection of the signal induced in the sensing coils. And, finally, the head should be either passively or dynamically decoupled from the