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Versatile and Sensitive Vibrating-Sample Magnetometer*

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A vibrating-sample magnetometer, which measures the magnetic moment of a sample when it is vibrated *perpendicularly* to a uniform magnetizing field, is described. With this instrument, changes as small as 10^{-5} to 10^{-6} emu have been detected, and a stability of one part in 10^4 has been attained. In addition to permitting convenient measurements in the usual laboratory electromagnet, this instrument eliminates or minimizes many sources of error found in other methods. It is simple, inexpensive, and versatile, yet permits precision magnetic moment measurements to be made in a uniform magnetizing field as a function of temperature, magnetizing field, and crystallographic orientation. The mechanical design and detailed operating characteristics are presented. Applications and limitations of the method are outlined.

I. INTRODUCTION

THE usual methods of measuring magnetic moments can be divided into three major classes: measurement of a force on a material in a nonuniform magnetic field, measurement of magnetic induction in the vicinity of the sample, and indirect measurments of phenomena which involve the magnetic properties. The force method is a sensitive technique which has been employed for many years in the laboratory. With this method it is, of course, difficult to observe the magnetization in a truly uniform magnetic field since the field gradient is essential to the production of the force. Furthermore, this method is not easily adaptable to routine measurements of magnetization *versus* applied field or crystallographic orientation. Objections to the use of force methods for magnetic measurements of highly anisotropic materials have recently been raised by Wolf.[1]

Numerous indirect techniques for measuring magnetic moments include[2] measurement of the Faraday effect, analysis of galvanomagnetic effects such as the ferromagnetic Hall effect,[3] and microwave ferromagnetic resonance measurements. The advantages and disadvantages of indirect measurements are illustrated by microwave ferromagnetic resonance experiments whereby the magnetization may be obtained from a detailed knowledge of the sample shape.[4,5] The general problem of indirect techniques is that they are limited to particular phenomena which are observable in a limited class of materials about which considerable prior knowledge is required. Despite many limitations, in particular instances these indirect techniques are capable of extremely high sensitivity.

All the magnetic induction measurements involve observation of the voltage induced in a detection coil by a flux change when the applied magnetic field, coil position, or sample position is changed. Many different experimental arrangements have been employed to suit particular investigations and some are described in reference 2. Recently, oscillatory coil or sample techniques have been used to observe the magnetization of a sample by an ac method. All of these techniques have employed an arrangement in which the detection coil is symmetrically distributed about the sample with the *axis of the detection coil parallel to the applied magnetic field.* One major disadvantage of such a method is that, unless an air-core solenoid type magnet is used, the usual laboratory magnet must be extensively modified. The most convenient arrangement has been to drive either the sample or the detection coil with a rod which passes through one of the magnet pole faces; the laboratory magnet is then specifically adapted to the magnetometer. A particularly successful *vibrating-coil* technique has been developed by D. O. Smith[6] using this procedure. The difficulties encountered here are that extremely uniform magnetic fields are required, and that, furthermore, it is difficult to correct for effects produced by small nonuniformities of the field. The latter problem is particularly bothersome when the field nonuniformities are functions of the applied field. Even in a uniform magnetic field, for samples of small magnetic moment, large corrections must be made for the magnetic effects of materials which surround the stationary sample.[7]

A particularly successful *vibrating-sample* magnetometer is described in this paper. Since its inception, many labo-

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[1] W. P. Wolf, J. Appl. Phys. **28**, 780 (1957).

[2] A comprehensive review of a number of techniques for measuring magnetization can be found in L. F. Bates, *Modern Magnetism* (Cambridge University Press, New York, 1951).

[3] S. Foner, Phys. Rev. **101**, 1648 (1956).

[4] Applications of shape effects to measurements of magnetic susceptibilities using nuclear resonance techniques have been described by Reilley, McConnell, and Meisenheimer, Phys. Rev. **98**, 264(A) (1955), and B. E. Holder and M. P. Klein, Phys. Rev. **98**, 265(A) (1955).

[5] Such measurements of M_s to 1% have been made on thin Permalloy films which exhibit narrow resonance line widths. See M. H. Seavey, Jr., and P. E. Tannenwald, J. Appl. Phys. **29**, 292 (1958).

[6] D. O. Smith, Rev. Sci. Instr. **27**, 261 (1956), and more recently Dwight, Menyuk, and Smith, J. Appl. Phys. **29**, 491 (1958).

[7] The same problems would be encountered with the *rotating-coil* technique recently described by J. Kaczer and Z. Malek, Czech. J. Phys. **7**, 481 (1957).

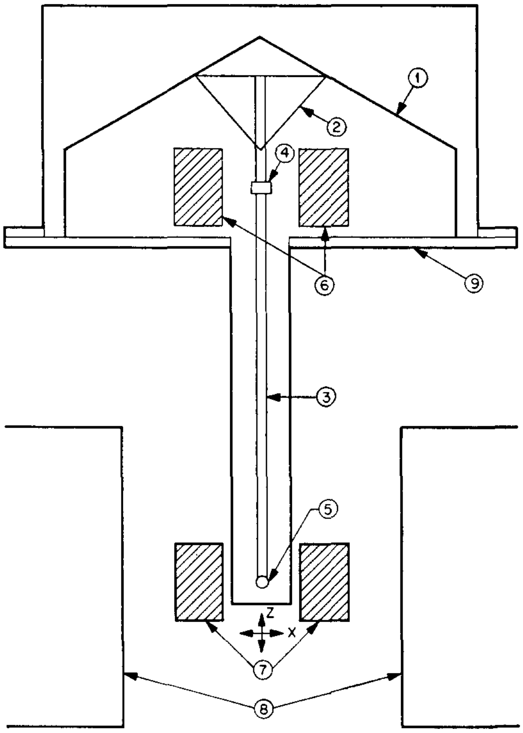

FIG. 1. Simplified form of vibrating-sample magnetometer. (1) loudspeaker transducer, (2) conical paper cup support, (3) drinking straw, (4) reference sample, (5) sample, (6) reference coils, (7) sample coils, (8) magnet poles, (9) metal container.

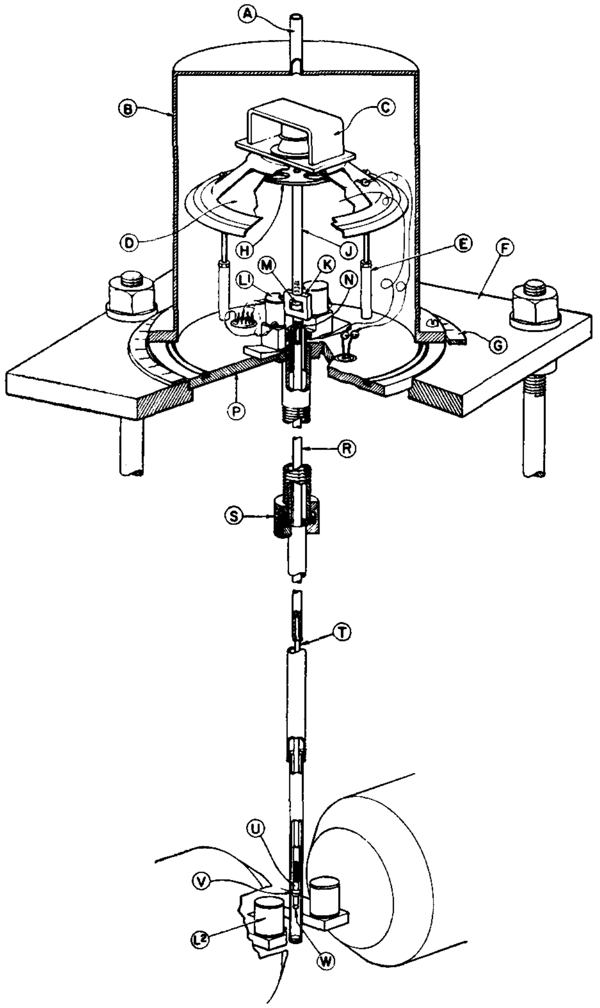

FIG. 2. Detailed mechanical construction of vibrating-sample magnetometer. The various parts are described in the text.

ratories have constructed similar instruments. The novel features of this magnetometer are: first, sample motion *perpendicular* to the applied field; and second, detection coil configurations, *with effective area-turns nonsymmetrically distributed about the axis of vibrations*, which permit this oscillating dipole field to be observed. The basic instrument, briefly described earlier[8,9] is shown in Fig. 1. The sample (5) is vibrated perpendicularly to the applied field by the loudspeaker assembly (1), (2), and (4). The oscillating magnetic field of the vibrating sample induces a voltage in the stationary detection coils, (7), and from measurements of this voltage the magnetic properties of the sample are deduced. A second voltage is induced in a similar stationary set of reference coils (6) by a reference sample (4) which may be a small permanent magnet or an electromagnet. Since the sample and reference are driven synchronously by a common member, the phase and amplitude of the resulting voltages are directly related. The known portion of the voltage from (6), phased to balance the voltage from (7), is then proportional to the magnetic moment of the sample. By this procedure the measurements can be made insensitive to changes of vibration amplitude, vibration frequency, small magnetic field instabilities, magnetic field nonuniformity, amplifier gain, or amplifier linearity. The associated electronic circuits serve the function of a null detector.

The measurements are made insensitive to the exact sample position by employing suitable pairs of coils. To do this for the coils (7) in Fig. 1 [a 3-dimensional view of these coils, (L2), is shown in Fig. 2], the sample is first centrally positioned between the coils by visual inspection. The sample coils are then rotated about the z axis for maximum signal output. (The output is then insensitive to small rotations of the coils about this axis). They are then translated in the X direction for a minimum output, along the Y direction (perpendicular to the paper) for a maximum output, and along the Z direction for a maximum output (if the coils are short). The sample is now located at a "saddle point"; the output signal is independent of small displacements of the sample in any direction.

In addition to the above features, this vibrating-sample magnetometer eliminates or minimizes many of the usual sources of error found in other methods. It is simple, inexpensive, and versatile, yet permits precise magnetic

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[8] S. Foner, Rev. Sci. Instr. **27**, 548 (1956).

[9] S. Foner, Bull. Am. Phys. Soc. Ser. II. **2**, 128 (1957).

moment measurements to be made in a uniform magnetizing field as a function of temperature, magnetizing field, and crystallographic orientation. An average stability of the balanced signals greater than one part in 10^4 has been observed. The sensitivity can be extremely high: changes of 5×10^{-5} to 5×10^{-6} emu can be detected. The combination of high sensitivity and high stability permits measurements on weakly magnetic materials, and differential measurements of very small changes of magnetic moment. Such sensitivities are useable because the vibrating-sample technique does not detect any stationary nonuniformities of the magnetic field due to the magnet or the medium surrounding the sample.[10] This emphasizes an important distinction between the moving sample and moving coil techniques—they are not equivalent unless the *detection coil plus the magnet producing the field, and the medium surrounding the sample* are moved as a unit when the sample is stationary. This procedure is usually impractical, and generally not employed in moving coil methods.

II. MECHANICAL DESIGN OF MAGNETOMETER

The simplified diagram of the magnetometer, Fig. 1, includes all the basic elements of the instrument. Detailed mechanical features of a vibrating sample magnetometer are shown in Fig. 2. The magnetometer vacuum container includes: an evacuation tube A soldered to a removable brass hat B; an O-ring vacuum seal at base plate P; and a threaded extension of P which joins a removable, segmented, brass extension-tube S which also employs an O-ring seal. The vacuum container is free to rotate about the vertical axis and rests on a counter-sunk support plate F having 3 leveling screws. The azimuth angle is given by the indicator scale G attached to F. The vibration system is composed of: a loudspeaker C glued to a plastic element H which provides rigid support for, and centers, a threaded plastic rod J; a reference sample holder K in which the reference sample M is held; a threaded plastic connector N which is glued to a thin-walled stainless extension tube R to which a solid plastic rod T is attached; a threaded sample holder U which permits convenient interchange of samples W; and finally, a Teflon centering washer V press fitted to U, which permits only a few thousandths of an inch motion perpendicular to the axis of the driving assembly, but does not add appreciable friction to the vibrating system.

The reference sample M is a thin disk permanently magnetized in the plane of the disk. The magnetic material used here, uniaxial single domains of $BaO\cdot6Fe_2O_3$ oriented and compressed in a magnetic field,[11] has a large coercive field so that the magnetization is not affected appreciably by external fields. Reference sample M may be replaced by a small coil carrying a controlled dc current when precise automatic recording of magnetic properties is desired.

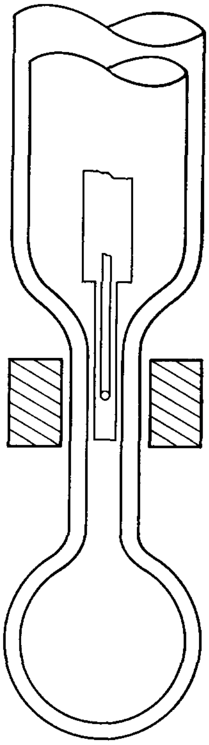

FIG. 3. Cross-sectional view of single glass Dewar used for low-temperature measurements. The two detection coils are shown outside the Dewar. Part of the sample mount and surrounding container are indicated.

The vibrating assembly from M to W is 28 in. long to permit placing the magnetometer in any electromagnet available in our laboratory. The threaded connections are convenient for disassembly when the magnetometer is to be stored in a small space, or when an element must be replaced.

The reference coils $L1$ and the sample signal coils $L2$ are adjusted as described earlier—$L1$ is glued into place whereas $L2$ is supported independently on a vertical screw arrangement for convenience in adjustment. (The supports are indicated in the figure.) Vacuum seals for electrical connections from $L1$ and to the loud speaker are also indicated.

The brass extension tube S is thin-walled (0.015 in. or less) to minimize heat losses. A thermocouple is attached to the smaller telescoping tube (0.25 in. o.d.) in order conveniently to monitor the temperature at the sample. Thermal contact between sample and outer tube is obtained by introducing helium gas into the vacuum chamber. The threaded section joining S is extended to support, and allow vertical adjustment of, a Dewar cap assembly.

A cross section of a convenient glass Dewar flask is shown in Fig. 3. The volume of the upper section is about 400 cm^3. The central section is minimized in diameter in order to obtain both maximum applied magnetic field and signal sensitivity. The small (80 cm^3) reservoir at the

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[10] If the sample is not saturated, only the field nonuniformity over the sample volume may be detected (see also Sec. VI B).

[11] Such materials are now available under the trade name of Magnadur in Europe, and under the trade name of Ferroxdure or Indox V in the United States.

bottom of the Dewar is useful for producing a slow temperature change during measurements. For further control of sample temperature, a heater may be placed in the reservoir. With such a Dewar, a warm-up from liquid nitrogen (77°K) to room temperature can be extended to several hours. Losses are sufficiently small at 77°K to allow measurements for 3 to 4 hr at this fixed temperature. The same Dewar has been used for measurements at liquid helium temperatures without a surrounding liquid nitrogen Dewar. About 20 to 30 min running time is available at 4.2°K (sufficient for several *M vs H* measurements) with a fairly rapid warmup (about 30 to 60 min warmup time from 4.2°K to 77°K).

A metal Dewar is useful for measurements over a prolonged period at 4.2°K, or for measurements below 4.2°K. Such a Dewar (with a small tail section 0.56-in. o.d. and a 500-cm^3 helium reservoir) has operated for 3 hr at 1.6°K.

For high-temperature work, a separate extension tube similar to *S*, but with a small furnace attached to the region near the sample, can be used. Of course, the materials for the sample holder and associated parts must be made of heat resistant materials. Weakly magnetic ceramics or metals may be used.

From the above description, it should be apparent that the mechanical features are sufficiently simple and flexible to permit numerous modifications to be made for particular applications.

III. COIL DESIGNS

A. General Analysis

The advantages of sample vibration perpendicularly to the applied field can be realized only if suitable detection coil arrangements can be devised. In practice many satisfactory coil configurations can be found. This is easily seen if we consider the time varying part of the vibrating dipole field. The scalar potential of a fixed dipole M at the origin and pointed along the X direction is $\phi = Mx/r^3$. If M is vibrated in the Z direction with sufficiently small amplitude a, the time varying potential in the surrounding space will be $\phi_1 e^{i\omega t}$ where $\phi_1 = -a(\partial\phi/\partial Z) = aMxZ/r^5$. The flux pattern of the time varying part of the field is given by $-\text{grad}\ \phi_1$. Its configuration in the XZ plane is shown qualitatively in Fig. 4.

Appropriate placement of pick up coils to sense this flux may be visualized easily and interesting new configurations suggested. For example, coils at 45°, or coils with horizontal axes which would fit in a narrow pole gap could be used. The latter case may require 4 coils if the instrument occupies part of the volume in the Z direction. A general feature of all the useful coil configurations is that each effective area-turn is nonsymmetrically distributed about the axis of vibration. Pairs of coils are employed in order to minimize effects of sample position or external field variations. In a similar way, the effect of slight nonuniformities of the magnetizing field, which causes M to vary during vibration, may be examined.

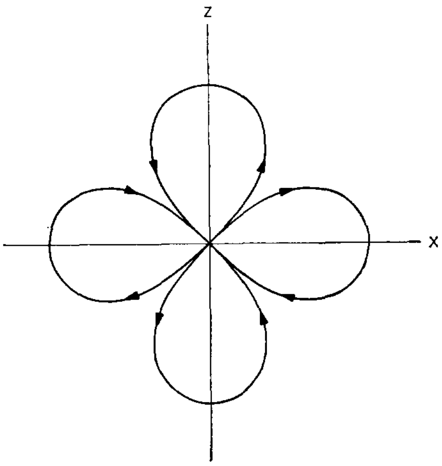

FIG. 4. Time varying part of dipole field in X-Z plane for vibration parallel to Z and dipole moment parallel to X.

In general, the pickup coil dimensions are not small compared to their distance from the sample. Furthermore, the coil geometries often do not follow the symmetries of the time varying field. For these reasons, the voltage induced in most useful pick up coils can not be calculated in closed form.

One of the most convenient detection-coil arrangements is the double-coil shown in Fig. 5(a). The spatial variations of relative output signal for two typical double-coil assemblies are plotted in Figs. 6 and 7. These results were obtained experimentally by observing the output signal due to the magnetic moment of a small magnetically saturated nickel sphere as it was positioned at various points in space. For this measurement, the entire magnetometer was positioned at various points in the XY plane by a suitable micrometer arrangement, and the detection coils were moved in only the Z direction. Data were obtained to at least 0.5% accuracy with the null detection procedure described in Sec. IV. The experimental points were obtained at intervals of 0.010 to 0.020 in., and deviate from the curves by less than 0.25%.

An additional feature of this double-coil system is that the two coils are connected series-opposing in order to obtain a net output signal. This arrangement to a large extent eliminates the effects of the background noise due to magnetic field instability or mechanical vibrations of the magnet and coil systems. These effects are also independently minimized by each coil since a minimum of area-turns is presented to the applied field.

The coil configuration shown in Fig. 5(a) has been employed extensively for almost all our magnetic measurements. This arrangement has proved both easy to assemble and most convenient in operation. Oval-shaped coils, shown in Fig. 5(c), have also been used extensively. Their characteristics have the general features of Figs. 6 and 7.

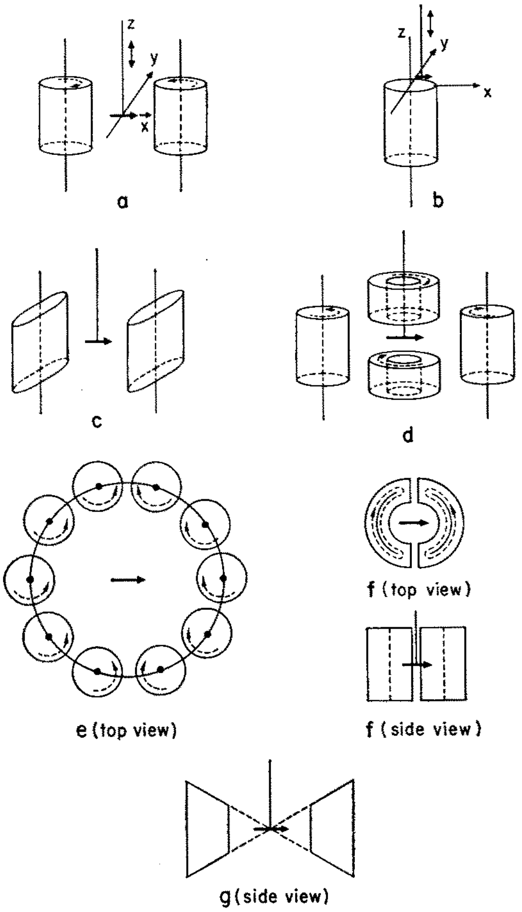

Fig. 5. Examples of useful detection coil arrangements described in the text. The sample, indicated by the heavy arrow, is vibrated along the Z direction.

An arrangement of a single coil, useful when very high fields are required, is shown in Fig. 5(b). The output voltage in such a case can be maximized for position in the X or Y direction so that the output signal is then insensitive to small sample displacements in these directions. This arrangement suffers, however, from a strong Z dependence of the output signal. Some characteristic results for a single-coil are shown in Fig. 8 to illustrate the dependence observed. Measurements have been made using such an arrangement, with almost as high an accuracy as the double-coil arrangement in Fig. 5(a), when an additional normalizing point was obtained with the Fig. 5(a) coil arrangement. Annoying spurious voltages, introduced by external vibrations, can be minimized by placing a second compensating coil of comparable area-turns in the magnetic field. The magnet air gap can be reduced by about a factor of three with an appropriate field increase, when the single-coil arrangement is employed. As shown at the top right of Fig. 8, a gain of almost 10 in output signal can be realized when the sample is positioned close to the top of the coil.

Additional examples of useful coil arrangements (all with axes parallel to Z) are shown in Fig. 5. In particular, Fig. 5(a) may be modified by the addition of a pair of coils coaxial with the Z axis [see Fig. 5(d)]. With such an arrangement, the magnitude and direction of the magnetic moment vector in space can be determined—the Z component is detected by the coaxial pair, and the component in the XY plane is determined by rotating the double coil. Figure 5(e) shows a multiple-coil arrangement which attempts to intercept a maximum of the sample dipole field, but at the expense of additional thermal noise of the coils. Four coils of this multi-coil array have been used for high-field configurations. An efficient modification of Fig. 5(e) is shown in Fig. 5(f); this coil geometry, however, is not easily fabricated. Finally, the cross section of a coil geometry which reflects most of the dipole field symmetry properties is shown in Fig. 5(g). It is directly derived from Fig. 5(f), and leads to rather simple computations of output voltage *versus* geometric parameters.

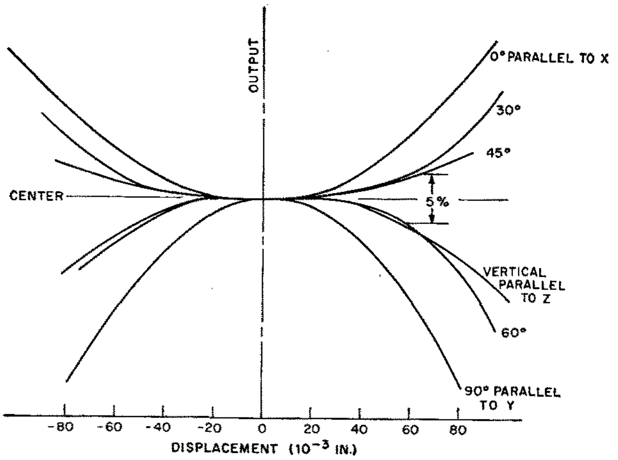

Fig. 6. Relative output signal from a double-coil system *vs* sample position. The contours of the "saddle point" are illustrated by measurements along various directions in the XY plane. Circular detection coils, each $\frac{5}{8}$ in. diam and $\frac{1}{2}$ in. long, with $1\frac{1}{8}$ in. between centers, were used.

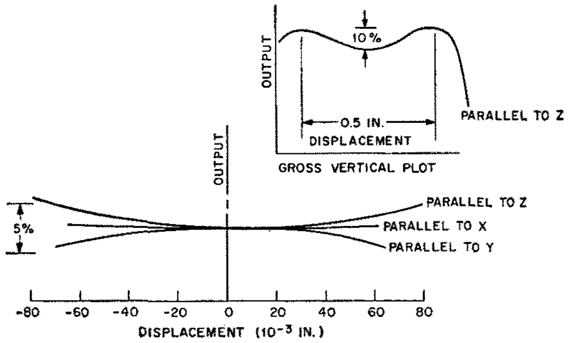

Fig. 7. Relative output signal from a double-coil system *vs* sample position. Bottom curves show relative output *vs* displacement along the three orthogonal directions in neighborhood of "saddle point." Upper right insert shows relative output for large sample displacement along the Z direction. Circular detection coils, each $\frac{5}{8}$ in. diam and 1 in. long, with 1 in. between centers, were used.

IV. ELECTRONIC CIRCUITS

The functions of the associated electronic circuits are: (1) to permit accurate calibration of the signal output obtained from the detection coils, (2) to produce a convenient dc output signal which is directly related to the input and which can be recorded, (3) to provide sufficient amplification for high sensitivity operation. These functions can be performed by a variety of circuits.

A functional block diagram of the circuit used for the magnetometer is shown in Fig. 9. The loudspeaker transducer assembly is driven at about 90 cps by a low-power amplifier fed by an audio oscillator which is peaked at the tuned amplifier pass-band. The combination of phase shifter, divider, and mixer performs the function of a calibrated ac bridge; the tuned amplifier and following circuits then indicate any unbalance of the ac bridge, but also may incorporate a feedback circuit to permit continuous balancing. An oscilloscope is used as a convenient visual null indicator of the ac signal prior to detection—the rectified, filtered, dc output is presented on an X-Y recorder.

The low-frequency and narrow band of operation permit standard circuits to be employed. For highest sensitivity the input circuit must, however, be carefully constructed in order to avoid extraneous voltages of millimicrovolt level. The reference permanent magnet signal furnishes the standard comparison voltage across the reference coils. Although the amplitude of this signal may vary with vibration amplitude, a proportional signal change is also produced at the sample coils, so that to high approximation this effect can be eliminated. A calibrated, constant-impedance, decade resistance-divider, incorporating a linear (3600°, helical wire-wound) potentiometer, is employed in the reference signal circuit for precise voltage division. The resultant voltage is then compared with the sample signal. In order accurately to balance the reference signal against the sample signal, the two signals must be carefully adjusted to be 180° out of phase. The difference between sample and reference signals is fed to the primary of a well-shielded audio transformer. At balance, there is no current flow produced in this transformer primary circuit; and hence the reference divider circuit serves as an accurate voltage divider of an ac potentiometer. The magnetic moment of the sample is then proportional to the reference voltage divider setting, and independent of vibration amplitude, frequency, or the following electronic circuits which then only serve as a null-detector amplifier. The main sources of error of null measurements involve the calibration of the voltage divider network and the standard comparison sample.

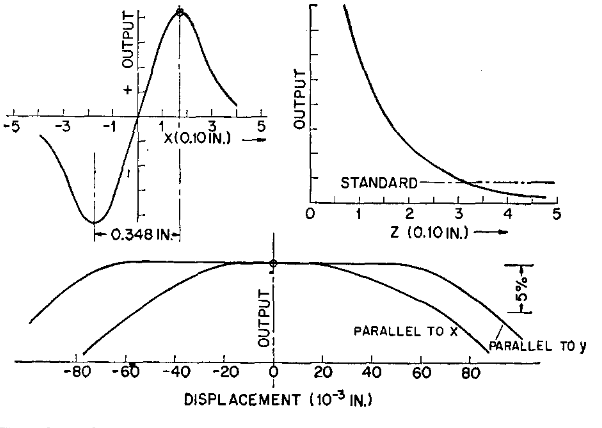

FIG. 8. Relative output signal from a single coil [Fig. 4(b)]. Upper right shows output *vs* Z, the distance between the center of the sample and the top of the coil. Output level obtained with the standard double-coil is indicated by the broken line. Upper left shows output as sample is displaced along the X direction while the Z position is maintained—no output is obtained at the coil axis ($X=Y=0$). Bottom shows sensitivity to sample displacement in the XY plane in region of maximum output. Dimensions of single-coil correspond to one coil of Fig. 6.

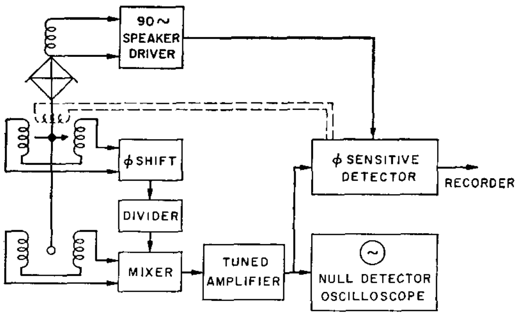

FIG. 9. Block diagram of electronic system for magnetometer. Relevant components of the magnetometer are indicated pictorially.

A servo loop coupled to the reference voltage divider may be employed to obtain null measurements of magnetic moment. This complication can be avoided by balancing reference and sample signals at one point and recording the amplified, rectified, unbalanced signal which is closely proportional to the change of sample magnetic moment. The output trace is then calibrated to compensate for any nonlinear circuit characteristics by comparison with several appropriate settings of the reference divider. Effects of unbalanced currents in the input circuits can be eliminated by feeding the reference and the sample signals to high-impedance inputs of two balanced cathode-follower circuits.

If the reference sample is replaced by a small coil (indicated by the dashed lines in Fig. 9) energized by an adjustable dc current, automatic recording of magnetic properties can be made with the inherent advantages of null measurements. The direct current is regulated by a servo-loop monitoring the null output signal, and a voltage proportional to the balancing direct current is recorded. This system is essential for measurements of weakly magnetic materials because the long integrating time makes point-by-point balancing difficult. Such a system is, of course, ideal for magnetic measurements in general. In most cases we have found that the permanent magnet reference is less complicated for measurements over extreme ranges of magnetic moment, and is therefore more convenient for routine measurements.

V. OPERATING CHARACTERISTICS OF MAGNETOMETER

A. Calibration

The reference voltage divider reading at null is directly proportional to the sample magnetic moment and to the coil conversion factor. As mentioned in Sec. III, the induced voltage has not been calculated in closed form for many of the useful coil arrangements. In principle this can be done so that an absolute measure of magnetic moment may be calculated. It is doubtful, however, that the accuracy of such a calculation will approach that of a comparison method. An expedient method of calibration involves standardizing the reference voltage divider by replacing the sample with a material of known magnetic properties and mass. In this way, one can easily calibrate the magnetometer for any useful sample-coil configuration. The saturation moment of a small sphere of pure nickel (about 8 mg) has been used as a secondary calibration standard for most measurements. The two major reasons for choosing nickel are that it can be obtained in high purity, and that magnetic saturation of nickel is easily attained at low magnetic fields, so that the calibration is not dependent on the applied magnetic field.

Calibration for very weakly magnetic materials is somewhat more difficult because the sample may occupy a comparatively large volume of irregular shape whereas small samples act as simple dipoles and size effects are negligible. Calibration for such large samples can be accomplished by measurement of the sample susceptibility *in vacuo* and then in pure oxygen gas at standard temperature and pressure. The difference of the two measurements determines the susceptibility of the vibrating hole in the O_2 gas (occupied by the sample) which is just the negative of the well-known susceptibility of O_2. By this substitution method an accurate correction for higher order shape effects of large samples is made without recourse to tedious, less accurate computations. One expects such shape effects to become important when the sample dimensions approach those of the coil spacings. As an example, tests with paramagnets of irregular shapes with linear dimensions up to about 3 mm were made. The magnet moments agreed within one percent for these cases, indicating the shape effects were still small.

Absolute calibration of a particular coil configuration can be made by vibrating a sufficiently small direct current loop of known area-turns in place of, or surrounding, the sample.

If the sample is weakly magnetic, but of uniform cross section (e.g., a cylinder) one can wind a coil on the sample to produce a current shell of equivalent magnetic moment. This method can be used effectively to cancel the induced dipole moment in the sample when the axis of the cylinder is parallel to the applied field.[12] The reference voltage circuit is thus replaced by a dc current measurement for the equivalent canceling current shell. Of course, all the advantages of null measurements are still obtained. Absolute measurements of magnetic properties as a function of crystallographic orientation can be obtained by using several cylinders suitably oriented. The current shell method eliminates the need for the reference coil system; the sample coils then function as part of the null detection circuit by indicating the unbalance between the current shell and magnetic moment. Unfortunately, the practical limiting currents, sample shapes, and complexity, limit this method to special problems with weakly magnetic materials.

The *absolute* accuracy of the instrument depends on the knowledge of the magnetic properties of the calibration standard and reproducibility of sample position. When the substitution method of calibration is used, the major error, ±1%, is introduced by the estimate of the nickel-standard magnetic moment. The *relative* accuracy of this instrument depends on accurate calibration of the precision resistor divider network. The total error here can be kept to less than 0.5%.

Repeated tests showed that when the sample coils were repositioned as described earlier, the output signal was reproduced to 0.25%. Recalibrations of the coils, maintained with a fixed spacing, showed that calibration could be maintained within ±0.5%.

B. Sensitivity Limits

The limits of sensitivity are determined by signal to noise ratio at the input circuit where noise is defined as any signal not due to the sample magnetic moment. The major sources of noise arise from the Johnson noise of the wire used for the pickup coils, and from the magnetic properties of the sample holder which, of course, superimposes an in-phase signal on the wanted signal. Use of a minimal mass of weakly diamagnetic material for a sample holder, carefully checked to contain no ferromagnetic impurities, is essential to minimize this coherent noise contribution. Corrections for the small magnetic contribution of the sample holder can then be made by measurements with the sample removed. Obviously, this correction is much less than the equivalent case with a moving coil system.

Tests with the double-coil system [Fig. 5(a)] showed that the detectable signal was within a factor of two of the theoretical thermal noise of the coil winding at room temperature. Further improvements of signal to noise can be

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[12] A. Arrott and J. E. Goldman, Rev. Sci. Instr. 28, 99 (1957) employed such a technique with a moving sample. Here again, the sample motion was *parallel to the applied field direction.*

made in several ways. The thermal noise of the detection coils can be reduced substantially by immersing them in a low-temperature bath, at a sacrifice of magnetizing field and convenience in adjustment. Some gain can be obtained by optimizing coil geometry. As an example of an extreme case, a gain of about 10 in signal and a decrease in resistance may be attained by using the less convenient arrangement of a single coil [see Fig. 5(b) and Fig. (8)]. Finally, some additional gain can be obtained by increasing the vibration amplitude of the sample. Peak amplitudes of 1 mm can be obtained without damaging the loudspeaker, whereas this amplitude usually is kept near 0.1 mm. In principle, no gain is expected by increasing the number of area-turns per unit volume of coil if we assume impedance matching to the input circuit and a constant space-factor are maintained.

Several methods were used to estimate the ultimate sensitivity of the vibrating sample magnetometer with the double-coil configuration. Because it would be difficult to insert a sample of a very small, but known, susceptibility without also introducing background effects of the sample support and other impurities, differential methods were used. The simplest measurement used the standard nickel sample. A change in susceptibility,[13] $\Delta\chi$, of 5×10^{-9} could be detected at the oscilloscope, and $\Delta\chi\simeq5\times10^{-10}$ could be observed after synchronous phase detection (band width $\simeq2\times10^{-2}$ cps). The other tests used a small current carrying coil mounted in place of the sample. Either a direct current was passed through this coil as it was vibrated at 90 cps, or an alternating current at 90 cps was passed through the coil which remained stationary. In both cases, $\Delta\chi\simeq2\times10^{-10}$ was detectable with optimum conditions and synchronous detection. Probably a factor of 10 improvement can be expected with minor modifications. These results compare favorably with some of the most sensitive force methods. A particularly high sensitivity force method, developed by Stevens and Crawford[14] permits $\chi\simeq2\times10^{-10}$ to be measured.

C. Stability Tests-Differential Measurements

With only the tuned amplifier (band width ≈1 cps) and the oscilloscope as a null detector, it was found that the 8 mg Ni sample signal could be balanced reproducibly to one part in 8000. Such reproducibility indicated that the long time drifts caused by the combined effects of vibration amplitude changes, frequency changes, varying average sample position, and any other effects not considered here were indeed negligible. When a synchronous phase detector was added (band width $\simeq2\times10^{-2}$ cps) differential changes about one-tenth this size could be recorded reproducibly. These results demonstrate that the average stability of the instrument is exceptional.

D. Vibration Amplitude

The peak-to-peak vibration amplitude has been varied from less than 0.1 mm up to 1.0 mm in order to examine errors caused by amplitude changes. Such tests show that the measured magnetic moment varied by less than $\pm0.5\%$ over this range of amplitude, although a somewhat sharper balance is obtained at higher vibration amplitudes because of the larger signals involved. Measurements usually are made with vibration amplitudes of about 0.1 mm. Insensitivity to small amplitude variation is also demonstrated by results of Sec. VC.

E. Image Effects

When a magnetic material is placed near a highly permeable medium, a magnetic moment is induced in that medium. This effect, called the "image effect," may be eliminated by using the absolute current shell calibration technique (Sec. VA), so that no net moment is present when measurements are made, by using sufficiently large air gaps, or by using air-core solenoids. As mentioned earlier, the current shell technique is not useful for strongly magnetic materials because impractical currents are required to balance the induced moment. Estimates of the image effect can be made by observing the measured magnetic moment when the sample and coils are positioned at points far from the central plane of the magnet air gap. The largest effect should then be observed when one of the coils almost touches a pole face. Such measurements with the nickel standard sphere have been made using an air gap of about 2 in. and fields up to 8 kilogauss. An image effect was not detectable and is thus estimated to be less than 0.2%. It is expected that an image effect will be more important for smaller air gaps, in which case such measurements can be used to obtain suitable corrections.

Image effects were also examined with a small vibrating coil carrying a dc current. The image effect was no greater than $\pm1\%$ for fields up to 18 kilogauss produced in an air gap of $1\frac{3}{4}$ in. Undoubtedly, there is an image induced in the magnet poles. It appears, however, that when the sample is vibrated, the effective image vibration is reduced by eddy-current shielding.

F. Vibration Frequency

The vibration frequency is not critical. High-frequency operation is limited by the driving mechanism and capacitive shunting in the detection coils. Frequencies of 100 cps or less permit use of inexpensive components and minimize eddy-current shielding by the vacuum chamber. The meas-

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[13] For convenience whenever values of χ are considered, the units of χ are cgs units/g normalized to correspond to the change detectable for a 1-g sample in an applied field of 10 kilogauss.

[14] D. K. Stevens and J. H. Crawford, Jr., Phys. Rev. **92**, 1065 (1953). Specifically they report $\chi=10^{-10}$ cgs units/g can be measured for a 1-g Ge sample when $H=18$ kilogauss and $\partial H/\partial x\simeq3$ kilogauss/cm.

urements are completely independent of eddy-currents in surrounding parts if measurements and calibration are made at the same temperature. A correction for changes of penetration depth with temperature may be required if the conducting parts are thick. The thickness of conducting parts has been minimized, so that the temperature dependence of penetration depth is less than 1%. Finally, low frequencies may be desirable for measurements below 1°K in order to minimize introduction of phonon energy.

G. Vibration Problems

Mechanical coupling between the vibrating system and the fixed detection coils must be avoided. Although the coils are arranged for minimum sensitivity to external vibration, a noticeable background signal is obtained when the vacuum chamber contacts the detection coils. Such mechanical effects are difficult to eliminate electronically because the spurious background signal has the same frequency as the sample signal and maintains a constant phase difference with respect to the sample signal. The unwanted background signal may also be field-dependent if the coils subtend a region of nonuniform field. Usually the magnetometer and detection coils are both supported by the magnet, so that some mechanical coupling may be noticed at highest sensitivity. This effect can be eliminated by shock-mounting either the magnetometer or the detection coil system as required by the particular experiment. Rigid clamping of the detection coils to the magnet pole faces has also been successful.

VI. APPLICATIONS

A. Magnetic Measurements

1. *Low-Conductivity Materials*

The vibrating-sample magnetometer has been in use for routine magnetic measurements as a function of temperature and field of ferromagnetic, ferrimagnetic, antiferromagnetic, paramagnetic, and diamagnetic materials for over three years.[15] The samples are first weighed, then glued to a standard sample mount. The magnetic moment per gram is determined, and from this result conversion to other appropriate units can be made with additional information. Samples can be inserted in the instrument and saturation moment measurements completed in about a minute. Spherical or approximately ellipsoidal sample shapes are used for magnetic moment *versus* field measurements of ferri- or ferromagnetic materials, so that accurate corrections for demagnetizing fields can be made. Measurements on single crystals are generally made with spherical samples of constant demagnetizing factor, although thin disks are also satisfactory. Crystals with cubic structure are mounted with the (110) plane perpendicular to the vibration direction so that measurements as a function of H along the three principal crystallographic directions may then be made.[15]

2. *High Conductivity Materials*

If the material is strongly magnetic, usually negligible corrections need be made for the diamagnetic effect of currents induced when the sample vibrates in a nonuniform field. However, many high-conductivity metals are weakly magnetic and these induced currents will then add large contributions to the magnetic moment. Such effects can be minimized by vibrating the sample in a uniform region of magnetic field, and, as a last resort, by laminating the sample. Even in a uniform field, a voltage is developed in a moving conductor. The effect is analogous to the Hall effect where we replace the primary current by the moving conductor. The resulting currents, perpendicular to both the vibration direction and the applied field, would produce a magnetic field configuration which to first order would be completely canceled in each of the pickup coils for the standard configuration. Furthermore, these currents are small and can be reduced further by suitable lamination of the sample if necessary. Susceptibility measurements on a solid cylinder of high purity (99.999%), oxygen-free, copper from 77 to 300°K demonstrated that these effects were indeed negligible. If these currents were appreciable, their magnetic effects could be observed with an appropriate coil arrangement and the Hall effect deduced.

B. Measurement of Magnetic Field

The magnetometer may be used as a field measuring device by using a paramagnetic sample. If the susceptibility is not too large and is field independent, the proportionality constant, accounting for the particular coil geometry and the susceptibility, is obtained by calibration with the nickel standard above magnetic saturation, or by an independent field measurement. The field measured is an average value over the sample volume (which can be made small). A relative accuracy of about 1% is obtained with the double-coils; the absolute accuracy depends on the calibration method.

Extremely small field volumes can be examined by vibrating a small thin flat disk such as a thin film of Permalloy in this region. In this case, the low anisotropy, high permeability, accurately known demagnetizing factor (4π), and high saturation magnetization permit measurements of fields up to 10 kilogauss when the field is perpendicular to the film. The magnetic moment is then proportional to the applied field. Small fields may be measured when the film is placed parallel to the applied field. A small additional dc solenoid around the sample can then be used to produce a variable field which almost balances the un-

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[15] See, for instance, S. Foner and J. O. Artman, J. Appl. Phys. 29, 443 (1958).

known field. Field gradients can be measured accurately when the sample is replaced by a small coil.

C. General Purpose Instrument

Many of the refinements presented here for this vibrating sample magnetometer were directed toward the development of a high sensitivity, versatile, laboratory instrument. A production line instrument, useful for quality control measurements of ferri- or ferromagnetic materials at room temperature, can be made with the minimum elements of Fig. 1. Since the magnetic moment will be relatively large, the simplest of detection systems can be used; 60 cps can be used for a driving frequency, and tolerances are not critical. The magnetizing field can be supplied by a small permanent magnet. A production line instrument in this form can be quite inexpensive and yet of quite high accuracy.

This inexpensive version can also be useful for teaching purposes in general physics. The magnetic properties of materials such as demagnetizing factors, anisotropy, magnetization processes, and magnetic saturation, can be conveniently demonstrated. Examination of various useful coil configurations for this instrument is also very instructive.

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Low-Temperature Adiabatic Calorimeter with Automatic Shield Control*

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The design and operation of an adiabatic low-temperature calorimeter employing a somewhat different design of automatic shield control is described. Thermocouple difference is sensed by a dc μv amplifier, which is followed in the circuit by a recorder-controller and a Leeds & Northrup Series 60 (current adjusting type) control unit. The output of the latter is used to control the voltage across the shield heaters through a Westinghouse "Magamp" saturable reactor. This design has been used successfully with a conventional low-temperature adiabatic calorimeter (50-ml sample size). In most cases the recorder showed no variation from zero (±0.002°) during heating and equilibrating periods, while momentary disturbance at "on" and "off" was less than 0.05°.

I. INTRODUCTION

To carry out our program of obtaining thermodynamic properties of various materials of interest, a calorimeter capable of medium accuracy was needed for those materials not sufficiently pure or stable to be appropriate for the laboratories engaged in high-accuracy work. For this purpose it was decided that a conventional adiabatic calorimeter with automatic shield control would best fill our needs.

The particular design of shield control was decided upon after consideration of the following requirements: (1) the components should be commercially available wherever possible, and (2) the circuitry should be highly stable and require a minimum of maintenance.

The general aspects of automatic shield control have been thoroughly discussed in recent years by a number of authors[1–4] and will not be repeated here.

II. CALORIMETER

The calorimeter is of the conventional adiabatic vacuum type, similar to that described by Southard and Brickwedde.[5] The cylindrical, 50-ml sample container is constructed of thin-walled copper and contains eight radial vanes for heat distribution. A false bottom of much thinner copper acts as a radiation shield for the calorimeter heater and resistance thermometer. The capsule type platinum resistance thermometer and Manganin heater are held in a central well by eutectic solder, which provides good thermal contact. The sample container is tinned internally and gold plated externally and is suspended by nylon thread in the adiabatic shield. Both sample container and shield are surrounded by a brass vacuum jacket. A cryostat capable of maintaining temperatures from 50° to 370°K, is placed around the calorimeter.

III. MEASURING CIRCUITS

The circuit for measuring the electrical energy supplied to the calorimeter and for measuring its temperature are

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[*] Taken from paper presented at Twelfth Annual Calorimetry Conference, Portsmouth, New Hampshire (September, 1957).

[1] T. M. Dauphinee and S. B. Woods, Rev. Sci. Instr. **26**, 693 (1955).

[2] Worthington, Marx, and Dole, Rev. Sci. Instr. **26**, 698 (1955).

[3] Zabetakis, Craig, and Sterrett, Rev. Sci. Instr. **28**, 497 (1957).

[4] E. D. West and D. C. Ginnings, Rev. Sci. Instr. **28**, 1070 (1957).

[5] J. C. Southard and F. G. Brickwedde, J. Am. Chem. Soc. **55**, 4378 (1933).