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Research and Development Report

CONSTRUCTION, CALIBRATION, AND
OPERATION OF A VIBRATING-
SAMPLE MAGNETOMETER

by

Albert E. Miller

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CONSTRUCTION, CALIBRATION, AND OPERATION OF A VIBRATING-SAMPLE MAGNETOMETER

Albert E. Miller^{*}

ABSTRACT

A vibrating-sample magnetometer of the Foner type, capable of measuring magnetic moments in the 200 emu to 10^{-4} emu range from 4.2 to 300K, has been constructed. The details of the construction are discussed and many figures are included that illustrate the electronic and mechanical systems used. Three different techniques used to calibrate the instrument, are discussed and compared. The general operating procedures employed in the use of the instrument are also presented.

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I. INTRODUCTION

The methods available for use in investigating the magnetic properties of materials are many and varied. Ideally one wants to use a method that permits rapid and accurate determination of the magnetic characteristics of materials with simple geometries. It is also advantageous to have the capability of extreme variation in both sample temperature and magnetizing field. Where single crystals are available, a built-in sample rotation feature that permits the study of the magnetization as a function of crystallographic orientation is also desirable. Several vibration magnetometers¹⁻⁷ which incorporate many of these features have been described in the literature; however, there seems to be a general lack of detail regarding construction, operation, and calibration of such instruments. The following report was written in an attempt to provide the reader with tried and proven details that are the basis of a workable magnetometer.

II. THEORY

The experimental investigations performed by Ampere²¹ and by Biot and Savart⁸ concerning the interactions of coils carrying electric currents led to a relationship from which it is possible to compute the flux density at any point in space around a circuit in which there is a current. The magnitude of the magnetic induction B (Wb/m^2) at a distance $r(\text{m})$ from a current element $ds(\text{m})$ carrying a current (amps) can be written as

$$d\vec{B} = \frac{\mu_0 i}{4\pi r^3} d\vec{s} \times \vec{r},$$

where $\mu_0 = 4\pi \times 10^{-7}$ Wb/A-m.

If we now consider a small current loop and attempt to find the field at some point P due to this loop, we find that we must integrate the above equation around the circuit. To do this we refer to Fig. 1A. and note that the change in potential $\delta\phi$ that would result if the point P were displaced by an amount $\delta\vec{a}$:

$$\begin{aligned} \delta\phi &= \vec{B} \cdot \delta\vec{a} \\ &= \frac{-\mu_0 i \delta\vec{a}}{4\pi} \cdot \oint \frac{d\vec{s} \times \vec{r}}{r^3}, \end{aligned}$$

where $\delta\vec{a}$ can be brought out of the integral because it is constant during the integration.

The same change in potential should be observed if the current element is displaced by $-\delta\vec{a}$ and the point P remains fixed. In such a displacement the current element sweeps out an area $-(\delta\vec{a} \times d\vec{s})$ and the total change in solid angle is

$$\begin{aligned} d\Omega &= \frac{-\vec{r} \cdot (\delta\vec{a} \times d\vec{s})}{r^3} \\ &= \frac{-\delta\vec{a} \cdot (d\vec{s} \times \vec{r})}{r^3}. \end{aligned}$$

Therefore, the line integral that we set out to evaluate is the total solid angle subtended at P by the area swept out by the current loop

A

B

Fig. 1. Pictorial representations of vector quantities used to derive an expression for the potential field around a magnetic dipole.

when it is displaced by $\delta \vec{a}$. This in turn is equal to the change $d\Omega$ in solid angle due to the displacement of P by $\delta \vec{a}$. Now we can write the change in the potential as

$$\delta \phi = \mu_0 i \frac{d\Omega}{4\pi},$$

where Ω is the total solid angle that the circuit subtends at P. Now from Fig. 1B, we find that

$$d\Omega = \frac{\vec{r} \cdot \vec{n} ds}{r^3},$$

where ds is a surface element. We now have

$$d\phi = \frac{i \mu_0 \vec{r} \cdot \vec{n} ds}{4\pi r^3}.$$

If we now define $i \vec{n} ds$ ($A \cdot m^2$) as the magnetic dipole moment $d\vec{m}$, we then have

$$d\phi = \frac{\mu_0 d\vec{m} \cdot \vec{r}}{4\pi r^3},$$

and the potential of an individual dipole $\vec{m}$ is

$$\phi = \frac{\vec{m} \cdot \vec{r} \mu_0}{4\pi r^3}.$$

If we consider a fixed magnetic dipole, $\vec{m}$, at the origin of a Cartesian coordinate system and if the dipole is assumed to be oriented along the X axis, the scalar potential can be written as

$$\phi = \frac{\mu_0 m \lambda}{4\pi r^3}.$$

If the dipole is now given a sinusoidal motion in the Z direction by

$$Z = Z_0 + \delta \sin \omega t,$$

then the scalar potential in the surrounding space will vary with time in the manner

$$\Phi(Z, t) = \Phi(Z) \exp(i\omega t)$$

where

$$\begin{aligned} \Phi(Z) &= -\delta \left[\frac{\partial \phi}{\partial Z} \right] \\ &= -\delta \left[\frac{\partial \left(\frac{\mu_0}{4\pi} \frac{mX}{r^3} \right)}{\partial Z} \right]. \end{aligned}$$

Substituting $r^2 = X^2 + Y^2 + Z^2$ into the above equation and performing the indicated differentiation we get

$$\phi(Z) = \frac{\delta M X Z}{r^5}.$$

The -gradient $\phi(Z)$ establishes the flux pattern of the time varying part of the field.⁹ The configuration of the flux pattern in the X-Z plane is shown in Fig. 2. The problem now becomes one of choosing the most advantageous coil arrangement to sense the dipole field. A discussion of several possible choices of coil configurations is given by Foner¹ and an analysis of a particularly efficient coil arrangement is given by Mallinson.¹⁰

Fig. 2. Time varying part of dipole field in X-Z plane for oscillation parallel to Z and dipole moment parallel to X.

III. DESCRIPTION OF A VIBRATING-SAMPLE MAGNETOMETER

A. General Description

The vibrating-sample magnetometer (VSM) to be described in detail below is of the Foner type in that the sample motion is perpendicular to the magnetizing field. The principles involved in determining the magnetic characteristics of materials using this technique are best discussed with reference to the schematic drawing of the electronic system shown in Fig. 3. When the magnetic dipole, produced by applying a static magnetic field to the sample under study, is vibrated perpendicular to the applied field, the dipole field that exists in the surroundings becomes time varying. This time varying part of the dipole field induces an ac signal into suitably constructed and oriented multiturn coils located in the immediate vicinity of the oscillating dipole. This sensed signal is then added to a reference signal of exactly the same magnitude but 180° out of phase with it. The net result of this is, of course, zero. If the magnitude of the mixed reference signal has been previously calibrated with materials with a known magnetic behavior, then the magnetic characteristics of other magnetic dipoles are readily determined.

A schematic of the physical arrangement of the apparatus is shown in Fig. 4; and Fig. 5 is a photograph of the entire system.

In designing and constructing a vibration-magnetometer there are several rather general points that must be kept in mind. Probably the most important item that must be considered in this system, as in

Fig. 3. Schematic diagram of electronic system for magnetometer.

Fig. 4. Physical arrangement of transducer, pick-up coils and dewar.

Fig. 5. Photograph of entire system for magnetic property studies.

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all ac bridge networks, is the signal-to-noise ratio. The signal-to-noise ratio can be improved in vibration-magnetometers by increasing the frequency or the amplitude or both. However, such increases place severe limitations on the mechanical design and a compromised situation must be adopted. After trying several possible combinations it was decided that a system operating at 88.2 cps for maximum hum rejection of the line frequency and a vibration amplitude of ± 1 mm provided the most usable situation.

Probably the best method of improving the signal-to-noise ratio is to combine a narrow band amplifier with a detector system. This produces a system with a band width of about 0.05 cps and markedly improves the signal-to-noise ratio.

When setting up the electronics care must be exercised in establishing the proper grounding. In order to prevent ground loops it was necessary to isolate the equipment from the cabinet, shield all interconnections and ground only the amplifier-detector chassis. With this arrangement extraneous noise problems were eliminated.

B. Electronic System

The electronic circuits shown in Figs. 6, 7, 8 were designed by W. Rhinehart and constructed by the Ames Laboratory electronics shop personnel to perform the functions outlined in the schematic, Fig. 3. A transistorized version of the above amplifier-detector system has been developed by Rhinehart and Mournalam.¹¹

A 50-w, push-pull type, audio oscillator is used to drive the

Fig. 8. Power supply for vibrating-sample magnetometer.

transducer system at 88.2 cps. The oscillator is generally operated in the 12-w range which results in a vibration amplitude of about 2 mm, peak to peak. Once the oscillator has been set for the desired amplitude, which is determined by the voltage drop across the driver coil, the amplitude and frequency stability is better than 0.1% for 8 h.

The reference signal is generated by moving a circular coil in a radial magnetic field. This signal is fed into a precision attenuation selector and then into a two-stage electronic phase shifter. The phase shifter (Fig. 7a) has the disadvantage that each stage attenuates the signal by as much as 5%. However, amplitude independence of the phase shifting network can be achieved by fixing R_1 and then monitoring the output voltage of the phase shifter as R_2 is varied over its range. An attenuation curve such as shown in Fig. 9 can then be obtained for each stage. It is observed that a considerable range of adjustment of R_2 exists, corresponding to a phase shift of about 20° , over which the amplitude change is less than 0.1%. By setting R_2 in the middle of this range, adjusting R_1 until the reference signal is 180° out of phase with a sample (nickel) signal, and then permanently locking R_1 , amplitude insensitive phase adjustment is possible by using only the fine adjustment, R_3 . The fine adjustment has a maximum phase shift capability of about 3° ; however, phase adjustment has seldom been observed to be larger than a few minutes except in the case of very low resistivity materials, e.g., gold-rare-earth alloys, where phase adjustments from the previously established 180° phase difference point of about 50° are required. For such materials the attenuation

Fig. 9. Attenuation characteristics of phase shifting network.

characteristics of the phase shifter must be taken into account.

The reference signal enters the mixer section through an input transformer that serves to isolate the reference section from the amplifier. Direct coupling of the reference signal to the mixing stage results in instability in the amplifier. A portion of the reference signal is then added to the signal induced in the pick-up coils by the oscillating sample dipole. The addition or mixing is done with a high-resolution, 1 part in 10^5 , potentiometer decapot which serves as a voltage divider in the reference signal circuit.

The output of the mixer section is the lower level signal point in the system. The minimum signal that can be detected by the amplifier at this point is significantly lowered by conditioning the signal with a well-shielded, precision input-transformer which has a turns ratio calculated according to relations given by Rhinehart and Mourlam.¹² This gives the low-level signal an essentially noiseless gain of about 7, greatly improves the resolution of low signal levels, and also extends the range of sample moments that can be studied.

The conditioned signal from the mixer is then amplified in a two-stage, narrow band (1 cps), variable gain amplifier. The amplifier has a maximum gain of about 10^6 , an amplitude stability of 0.05%, and a noise level referred to the input of less than 0.5μ volts.

The amplified signal is then put on the Y axis of an oscilloscope synced with the oscillator. When the magnitudes of the two signals

are not equal or when the signals are not 180° out of phase (or when both of these conditions exist simultaneously) the oscilloscope trace is sinusoidal. By watching for an amplitude minimum in the trace as the phase of the reference signal is adjusted, it is quite easy to get the two signals 180° out of phase. A balanced condition is then obtained by adjusting the precision potentiometer. For large sample signals it is quite easy to obtain balances to within 0.1% by using the oscilloscope and rather high (32db) amplifier attenuations.

For small sample signals for which low amplifier attenuations (2db) are used, a phase sensitive detector is used to obtain the null. Here, a signal representative of the amplifier output is chopped at the operating frequency of the system which is determined by the frequency of the amplifier. The oscillator, which is tuned to the amplifier, also provides the reference frequency to operate the detector. For the detector to operate properly the reference voltage must be in phase with the input voltage to the detector, a condition that yields the maximum dc output. For this reason an adjustable phase shifter is included in the detector reference-voltage circuit so that the in-phase requirement can be insured.

The output of the detector is heavily filtered and suitable for display on a recorder. This feature, however, has not been used to any great extent to date due to the type of materials being investigated.

The balancing process can be completely automated by incorporating a servo amplifier and a digital read-out system into the output of the detector. These units are found in digital temperature

sensing devices and the modular components can be obtained from manufacturers of these devices.

C. Mechanical Construction

The heart of the VSM system is the transducer-reference unit shown in Fig. 10 and fully detailed in Figs. A1-A4 of the Appendix. The driver magnet and driver coil were obtained from a 30-w, dual-coil speaker manufactured by University Speakers Inc. The driver coil is attached to an aluminum rod which passes through the center of the driver magnet. The rod is connected to phosphor bronze cantilever springs above and below the driver magnet platform. A plexiglas disc is used to center the drive rod and drive coil in the aluminum housing after which the driver magnet is easily positioned with respect to the driver coil and permanently clamped to the platform.

The cantilever springs are constructed of 20 mil phosphor bronze, Fig. A4, with the long dimension of the springs parallel to the "grain." The springs shown were designed to produce a system with a resonant frequency in the neighborhood of 90 Hz. The resulting transducer system is extremely stable and responds well to the 88.2 cps used in this system in addition to being rugged enough to withstand considerable abuse. Although the cantilever spring system shown should produce some lateral motion such motion has not been observed up to 4 mm peak-to-peak amplitudes.

The reference signal system consists of a small 4 Ω speaker coil

Fig. 10. Transducer-reference unit and positioning mechanisms.

that is vibrated in the radial field of a high quality, 5-w speaker magnet. Some care must be exercised in selecting a speaker constructed from a magnetic material with a low temperature coefficient and a large coercive force. This, however, presents no real problem since most high quality speaker materials satisfy both conditions. Once the reference coil has been firmly attached to the shaft, the reference magnet is easily positioned with respect to the coil by "feel." After the centered position is determined, the magnet is firmly fixed to its platform.

The signal that results from the reference system is about 0.5 V peak-to-peak and is very stable. However, it is somewhat sensitive to the mean position of the reference coil in the magnet. Close examination of the signal shows that the mean position of the coil has about a 6-mm range over which the amplitude change is less than 0.1%. On both sides of this region, not only does the amplitude change but the wave form is also considerably distorted. Since the vibration amplitude normally used is of the order of ± 1 mm, centering the coil in the above-mentioned range results in an extremely stable reference voltage.

The transducer-reference system is mounted on a circular plate which can be rotated, Figs. 10 and A5, and is in turn mounted on a table which has a continuously adjustable vertical position. The electrical leads for the reference and driver are brought through the circular plate via insulated vacuum feed throughs soldered into the plate. The leads then go to phosphor bronze strips, Figs. A6, A7 and

A8, which are attached to movable cars that are spring loaded by the phosphor bronze strips to brass tracks. The tracks are pressed into plexiglas mounts that are fastened to the elevator table. Electrical leads from the slip rings are brought through the elevator table and terminated at Amphenol-type connectors attached to the underside of the elevator table. Small pins in the bottom of the rotating table engage the cars. The two sets of tracks are separated by a curtain of Co-Netic AA foil to prevent magnetic pickup. This slip ring system allows the sample to be rotated without entangling the electrical leads.

The transducer table is connected to a hollow stainless steel shaft, Figs. A5, and A9, which is in turn brought through the elevator table via a pair of opposed and preloaded roller bearings. This arrangement provides the needed rotation without introducing any slop into the system that would result in a rocking motion. A simple worm-and toothed gear arrangement, Figs. A9 and A10, pinned to the stainless steel shaft makes accurate angular positioning a simple process.

Vertical positioning of the transducer-reference system is accomplished with a multiple screw and guide system shown in Fig. 10 and detailed in Figs. A9 and A10. This arrangement provides a very easily controlled and accurate vertical movement that is free of any twisting action that would be particularly troublesome when the direction of motion is reversed.

The transducer-reference and vertical positioning unit are

mounted on a commercially available, compound vise table, Fig. 5. This table provides the elevator table and transducer-reference system with two degrees of accurate horizontal motion and complete rotational freedom.

The remainder of the mounting system, shown in Figs. A11, A12 and A13, was designed for use with a Harvey-Wells (now Ventron-Magnion) Model L-158, 15 inch electromagnet. The mounting frame fits into a gap in the housing of the magnet and performs the additional function of a dewar support, Fig. 5. The dewar, Figs. A14 and A15, is aligned and centered in the magnet by eye at first and more precisely adjusted later when the sample tube is in place. The entire operation of mounting and dewar positioning takes less than two hours to perform.

The sample chamber, Fig. 11, is made of Pyrex glass that has been hand picked for straightness. Its overall length is 93 cm, about 29 cm of which is 6 mm-o.d., 5 mm-i.d. tubing that has been prepared by carefully etching standard 7-mm o.d. tubing with HF(aq.). The remainder of the chamber is 12 mm o.d. connected to a $\frac{1}{2}$ -inch Kovar graded seal that has been selected on the basis of the concentricity of the glass and metal portions. The Kovar seal is soldered into a $\frac{1}{2}$ -inch Cenco type vacuum fitting that fits into a 3-inch long coupling the sole purpose of which is to provide an exit for the thermocouple and heater wires attached to the sample. The electrical lead coupling is fitted with a $\frac{3}{4}$ -inch Cenco-type seal which fastens onto a mating extension section that is permanently

screwed into the hollow stainless steel pipe that passes through the roller bearing arrangement. All connections utilize "O"-rings for vacuum sealing.

The sample rod is made of Refrasil fiberglass sleaving that has been impregnated with Araldite epoxy.* These rods are easily made by slipping the sleaving over a straight 1/16-in. by 3-ft. piece of drill rod that has been dressed with a good quality of floor wax. Three coats of the epoxy are then applied producing an o.d. of about 1/8 inch. The drill rod is then heated resistively by passing about 30 A through it. The epoxy-fiberglass tube is then stripped off the hot rod and hung until the now rather limp tube regains its rigidity. The resulting tube is not as rigid as a comparable size quartz rod; however, it has the advantage that it withstands rough handling without breakage. One end of the rod is sanded down slightly and a 6-inch piece of 1/8-inch stainless steel tubing is slipped over the tube and epoxied to it. Four 1/16-inch thick teflon discs with about 0.001 inch clearance with the sample chamber and tapered edges are epoxied to the tube at regular intervals to prevent lateral movement of the rod and sample.

A small tube of thin, high purity copper, Fig. 11, is glued to the other end of the tube and serves as a heater and thermocouple mount to which small high purity copper sample mounts are attached. A 50 Ω heater of No. 40 Manganin wire is noninductively wound on the copper mount. A No. 34 Au-0.5 at. % Fe wire and a No. 40 Au-2.1 at. % Co wire share a No. 36 copper wire and form two thermocouples that provide accurate temperature measurement from 1.2 to 300K. All

* Manufactured by CIBA Products Corp., Fairlawn, N. J.

Fig. 11. Sample chamber with sample rod assembly, associated connectors, and electrical leads.

the wires are brought up the center of the fiberglass-epoxy rod and leave the sample chamber through a piece of surgical rubber tubing. A hose clamp is used to obtain a vacuum seal on the wires. The sample tube is connected to the transducer via a collet-type arrangement that firmly grips the stainless steel section of the tube. A fully assembled tube weighs about 15 g and has no effect on the mean position of the bronze cantilever springs.

The pick-up coils consist of about 20,000 turns of No. 50 triple Formvar covered copper wire wound on fiber spools. The coils are 5/16 inch o.d., 1/16 i.d., $\frac{1}{2}$ inch long, and have a room temperature resistance of about 10,000 Ω . They are potted into plexiglas plates, Fig. 12, with a silicone potting material to prevent damage. The magnet used with this magnetometer has axial holes to accommodate a microshim attachment. The coil mounting plates and hence the coils are easily centered in the magnet with a 0.010-inch raised aligning plug on the plates that fits tightly into the axial holes. The remainder of the pick-up coil mount serves to firmly press the mounting plates against the pole pieces of the magnet and prevents unwanted motion. It has been observed that the signal induced in the coils is dependent of the alignment of their axis with the direction of vibration. This alignment is easily performed with the off-center adjustment screws shown.

Fig. 12. Pick-up coil mount.

IV. OPERATING PROCEDURES

A. Sample Preparation and Mounting

By far the most simple sample geometry to deal with from the preparation point of view is the sphere. When the samples under study are in the powdered form, the powder is simply loaded into hemispherical cavities that have been milled in two high purity copper slugs with a ball-end mill of the desired size. General Electric adhesive No. 7031 diluted with alcohol is then dripped into each cavity. When the glue has dried, the two sections are in turn glued together and the entire assembly attached to the sample tube.

For solid samples there are two lines of approach that have been employed. For those samples where induced strains are of no particular consequence or where these strains can be removed by annealing, irregular pieces are roughly spherodized in an air operated grinder. The grinder consists of a 2-in. circular race lined with 320-grit contact grinding paper. Air enters the race tangentially through one or more small holes and exits through axial ports located in plexiglas end plates. The depth of the race can be designed to suit the immediate requirements and can be compartmented to facilitate grinding of several materials at the same time. Efficient operation of the grinder requires at least one air entrance port every $\frac{1}{4}$ inch of race depth.

Once the rough spheres have been prepared in the air grinder, they are finished by hand with a technique commonly used by lapidarists. In this method the rough sphere is forced to skid in a random manner between two copper pipes in the presence of 5 μ diamond paste, Fig. 13.

Fig. 13. Arrangement used to obtain spherical samples.

One of the pipes rotates under power and the other is hand manipulated and turned in the opposite direction. Varying the pressure and the inclination of the hand held pipe in addition to the counter rotation increases the randomness of the motion and greatly aids in obtaining an accurate sphere. Spheres approximately 1/8 in. in diameter with tolerances of ± 0.0001 in. can be produced in about 30 min. depending upon the material. Sphericities of better than 20 millionths of an inch and surface finishes of one micro-inch can be obtained from a custom polisher.*

In metal single-crystal work it is necessary to use a less drastic method than the air grinder to obtain rough spheres. This is most easily accomplished by spark cutting. A roughly cylindrical sample containing the single crystal desired is mounted in a motor driven chuck submerged in the oil bath. A tantalum tube with an i. d. somewhat larger than the final desired sample diameter is used as the cutting tool and advances perpendicular to the sample rotation axis. This process yields a rather exact sphere with only one or two small protuberances at the points where the sphere is attached to the main body of the sample. These are easily removed and the sphere is finished as described above. X-ray examination of the surface shows little evidence of strain induced by the final polish.

Once the spherical samples have been prepared, they are glued to copper sample mounts with General Electric adhesive No. 7031 and attached to the heater and thermocouple mount, Fig. 11. In order

* Joseph A. Corriveau, 18421 Ardmore Avenue, Detroit, Michigan.

to prevent erroneous temperature measurements the thermocouple bead is usually glued directly onto the sample. It has been found that thermal equilibrium can be established more quickly by gluing a slotted copper foil can over the sample and thermocouple. This same effect can be obtained by pinching the thermocouple bead between the lip of the heater-thermocouple mount and the sample mount and painting the sample and the lower portion of the sample mount with silver emulsion paint.

Once the sample is mounted on the sample tube, the sample rod is inserted into the sample chamber and the electrical lead coupling is connected to the chamber. The transducer-reference system and elevator table are pivoted around the vertical axis of the compound vise table to allow the sample chamber assembly to be lowered into the dewar. The transducer-reference and elevator table are then rotated back over the dewar and the table is then locked at a predetermined "zero" position. The electrical coupling is then detached from the sample chamber assembly and connected to the mating coupling extending from the stainless steel axle. The electrical lead exit pipe is aligned with one of the slots in the collet connector. The sample tube is then inserted into the collet connector so that the top of the tube is even with the electrical lead exit pipe. A nut with a tapered hole is then screwed onto the slotted aluminum collet, thus firmly gripping the sample tube. The sample chamber is then raised and connected to the electrical lead coupling. The surgical rubber tubing is then pinched off and the system evacuated and back filled with helium gas.

The entire operation described above takes only a few minutes to perform and can be done with liquid helium in the dewar. In this case a plastic bag is attached to the underside of the elevator table. Gaseous He is blown through the transducer-reference chamber and out the electrical exit pipe. When the bag has been thoroughly purged by the He, the connecting operation described earlier is simply reversed. Before reinserting the sample chamber into the liquid He, the chamber must be evacuated and stoppered. The connection procedure is then carried out in the He atmosphere provided by the plastic shroud remembering that the chamber should only be opened in a nitrogen- and oxygen-free dry He atmosphere.

B. Centering the Sample

Before any meaningful measurements can be made all samples must be centered between the sensing coils. This is a very simple process in which the screws on the compound vise table provide accurate X and Y positioning and the elevator table provides the Z positioning. Position sensitivity of this apparatus is shown in Fig. 14. The overall positioning procedure can be done to within 0.1% and is reproducible to within 0.1%.

C. General

Once the magnetic characteristics of the sample have been investigated at the bath temperature, either liquid nitrogen or liquid helium, the sample chamber is evacuated to thermally isolate the sample. A few mA of dc current from a 12 V source are passed through

Fig. 14. Position sensitivity of VSM.

the heater to obtain the desired temperature above the bath. This procedure is repeated for the various temperatures and magnetic fields necessary to obtain an understanding of the material. By using a liquid helium bath it is possible to perform an isofield study of a material from 4.2 to 300K in 10K intervals in about $1\frac{1}{2}$ h. Several such runs can be performed with the liquid helium from one transfer (about 1.5 liters). It is also possible to make two or three sample changes with the liquid helium from one transfer if only one iso-field heating study is performed.

V. CALIBRATION

A. Saturation Method

The method most commonly used to calibrate magnetometers utilizes a saturated ferromagnet with a known saturation magnetization at a specified temperature and magnetic field. Pure nickel is the accepted standard for this type of calibration, mainly because of its availability. It has been pointed out,¹³ however, that although many researchers use nickel as a standard there have been only a few investigators who have actually measured the absolute value of the magnetization of nickel and among these measurements there exists a variation of several percent in the determined values. Other problems such as purity and the presence of residual stresses commonly encountered in the use of nickel as a magnetic standard are discussed in detail by Case and Harrington.¹³

Annealed spheres of reagent grade (99.8+ %) nickel have been

used to obtain the calibration constant of the system described in this report. This constant, κ , is related to the dial reading (DR) on the mixer potentiometer at null and the total moment being sensed by the relation

$$DR \times \kappa = \sigma_{8000,293} \times \text{sample weight (g)}.$$

A saturation value of 54.55 emu/g at 8000 Oe was used for the nickel.¹⁴

B. Initial Slope Method

The following relation can be written for an isotropic magnetic material in a uniform magnetic field:

$$B = H_I + 4\pi M = \mu H_I, \quad (1)$$

where B is the magnetic induction, H_I is the internal magnetic field, M the magnetic moment per unit volume and μ the magnetic permeability. Solving Eq. (1) for the magnetization, we obtain

$$M = H_I \frac{(\mu - 1)}{4\pi}. \quad (2)$$

The demagnetizing field is taken into account in the usual manner through the relation,

$$H_I = H_{\text{ext}} - DM, \quad (3)$$

where H_{ext} is the external applied magnetic field and D is the demagnetization factor which is equal to $4\pi/3$ for a spherical sample.

Upon substitution of Eq. (3) into Eq. (2), we obtain,

$$M = (H_{\text{ext}} - \frac{4\pi}{3} M) \left(\frac{\mu - 1}{4\pi} \right), \quad (4)$$

which, after simplification, becomes

$$M = \frac{3}{4\pi} \left(\frac{\mu - 1}{\mu + 2} \right) H_{\text{ext}}. \quad (5)$$

Since the dial reading on the mixer is proportional to the sample moment, we can write

$$DR \cdot \kappa = VM, \quad (6)$$

where DR is the mixer dial reading, κ is a calibration constant, and V is the sample volume.

From Eqs. (5) and (6) we find that in the initial magnetization region where the permeability is constant

$$\frac{dM}{dH_{\text{ext}}} = \frac{3}{4\pi} \left(\frac{\mu - 1}{\mu + 2} \right) = \frac{\kappa}{V} \frac{dDR}{dH_{\text{ext}}} \quad (7a)$$

$$\kappa = \frac{3}{4\pi} \left(\frac{\mu - 1}{\mu + 2} \right) \frac{V}{\frac{dDR}{dH_{\text{ext}}}}. \quad (7b)$$

Thus we see that a calibration constant can be readily obtained from the slope of a plot of DR vs H_{ext} if the initial permeability and the volume of the sample are known, Fig. 15. Two limiting values of the permeability exist that allow the calibration constant to be determined without a knowledge of the exact value of the permeability.

1. High Permeability Materials

By using a ferrite material such as Ceromag 24* with $\mu \cong 2500$,

* Trade name for a ferrite material manufactured by Stackpole Carbon Company, St. Marys, Pennsylvania.

Fig. 15. Typical calibration curve for nickel showing the regions that can be used to obtain K .

the $\frac{\mu-1}{\mu+2}$ term becomes essentially one. The accuracy of the calibration is then almost solely dependent upon the sphericity of the sample and the accuracy to which the diameter of the sphere can be determined. Using the procedures described in the section on sample preparation it is possible to obtain a sphere of which the volume can be determined with an accuracy of 0.2%. More elaborate sphere polishing techniques greatly reduce this uncertainty.

2. Zero Permeability Materials

The other limiting case of interest occurs for those materials where $\mu=0$; i.e., for a perfect diamagnetic material. Such perfect diamagnetic behavior is exhibited by materials in the superconducting state. For a spherical sample of such a material

$$M = -\frac{3}{8\pi} H_{\text{ext}}$$

and

$$\kappa = -\frac{3}{8\pi} \frac{V}{\frac{dDR}{dH_{\text{ext}}}}$$

To perform this calibration it was necessary to reverse the leads of the pick-up coils to compensate for the negative susceptibility of the superconductor. It has also been found necessary to cool the superconductor through its critical temperature under zero field conditions. Only then is it possible to obtain DR vs H_{ext} data where the slope is the same from run to run. Niobium, a Type II superconductor, with $T_c = 9.5\text{K}$ has been used successfully many times to perform this calibration.

C. Comparison of Methods

Although the saturation method is definitely the quickest to perform, the resulting calibration constant has been found to be consistently lower than those constants determined by the initial slope methods by about 1%. Hence it is strongly recommended that the initial slope method as described by the American Society for Testing and Materials¹⁵ be used for calibration purposes. This method employs a sphere of high-permeability material such as the one mentioned earlier, i. e., Ceromag 24. Once a sphere with the desired accuracy has been obtained, the actual calibration process is quite straight forward.

The material used should be checked for hysteresis effects. If noticeable hysteresis exists, then the calibration must be made with decreasing external field, Fig. 15, to insure that the proper magnetization curve is being used for the calibration. The Ceromag 24 used for calibration had negligible hysteresis, i. e., the magnetization curve obtained with increasing field matched the curve obtained with decreasing field to within experimental error.

The use of a material with zero permeability affords an opportunity to obtain an absolute calibration of the magnetometer. However, it necessarily requires somewhat elaborate dewar systems and associated paraphernalia that would eliminate its use in many instances.

Once a spherical sample of a suitable material such as Nb has been obtained and carefully annealed to relieve residual stresses, it is cooled through its critical temperature to 4.2K in zero external

field. Moment measurements are made with increasing field until the critical field is reached and the superconductor is driven normal. The calibration constant determined in this manner is very reproducible as long as the sample is cooled under zero field conditions. It also agrees with the constant determined using the high permeability ferrite to within $\pm 4\%$.

All of the calibration studies were performed on samples that were attached directly to a quartz rod. This information was later compared with calibrations made with the samples connected to the copper sample holder. No appreciable change in the calibration constant was observed. The small contribution due to the sample holder will be discussed in a later section.

VI. CORRECTIONS

A. Nonzero Origin of the System

The voltage generated in the pick-up coils has a complex nature, being the vector sum of a resistive component and an inductive component. The essentially pure resistance nature of the mixer section dictates therefore that the approximately 180° phase relationship between the signal channel and the reference channel remain unchanged during the course of the measurements. This criteria is generally satisfied. However, when samples with very low resistivities are used, the eddy currents generated within the sample alter the phase relationship between the inductive and resistive portions of the emf generated in the coils. This in turn appears as a phase change between the signal

and reference channel which leads to erroneous results.

It is believed that the above shortcoming in the signal mixing procedure is also responsible for the systems' lack of a zero origin. That is, when known moments of different magnitudes are studied and the results displayed as dial readings at null vs size of moment, the relation should extrapolate to the common zero point. It has been observed, however, that in all cases studied there is a reproducible offset in this relationship that indicates the presence of positive system errors which cannot be cancelled. For a measured moment of 4 emu this error is less than 0.1% and rises to 0.75% for a moment of 0.4 emu. This error is corrected by subtracting a constant number of DR units from each DR before processing. This constant being determined, of course, by the change in DR required to force the known moment data to have a zero origin.

B. Correcting for the "Image"

When an isolated magnetic dipole, Fig. 16a, is placed near a high permeability material such as the pole pieces of an electromagnet, some of the flux lines find that the reluctance of their completion path can be considerably reduced by linking with the magnet and closing their loop via the magnet housing or yoke, Fig. 16b. When this dipole is vibrated, as is the case in the method described here, these flux lines do not contribute to the voltage induced in the cylindrical pick-up coils used with this instrument since the flux lines are tangent to the area formed by each turn. Hence these flux lines are essentially "shorted out" and their presence goes undetected.

a) MAGNETIC DIPOLE IN FREE SPACE

b) MAGNETIC DIPOLE IN CLOSE PROXIMITY
TO HIGH PERMEABILITY MATERIAL

Fig. 16. Origin of magnetic images.

The tendency for magnetic dipole flux lines to link with the magnet has been observed by several investigators.^{16, 17} Since this flux linkage induces compensating poles of opposite character in the pole pieces of the magnet, the term "image effect" is usually associated with this phenomena. It is generally assumed that the character of the effect can be described in a manner similar to that used for the image problem encountered in electrostatics.¹⁸

Several workers have observed experimentally that the "image effect" is small, $< \pm 1\%$, over a wide range of moments and magnetic fields. This is interpreted as resulting from the compensation of the induced magnetic pole by the associated eddy currents induced in the pole pieces by the oscillating magnetic pole.

Experiments performed with the system described here, using small coils carrying dc current as the measured moment, indicate a 5 to 7% decrease in the apparent moment when the measurement is made at a location removed from the magnet and then repeated in the magnet gap. However, once in the gap, the observed moment is constant to within $\pm 1\%$ for fields up to 20,000 Oe. This indicates that the image effect is present below 20,000 Oe but is nearly constant.

Above about 20,000 Oe the signal sensed by the pick-up coils is greatly enhanced, Fig. 17. This reproducible signal enhancement is believed to arise since, in the 20,000 Oe region, portions of the pole pieces, (e.g., the regions around the periphery of the truncated sections of the tapered pieces) saturate. As this saturation proceeds through the pole pieces, hence decreasing their permeability toward

Fig. 17. Signal enhancement at high magnetic fields.

one, fewer of the flux lines of the sample dipole link with the magnet and more complete their paths within the gap. This increased number of complete flux circuits in the gap of course increases the voltage induced in the pick-up coils and gives rise to the effect shown in Fig. 17.

In order to correct for the signal enhancement observed above 20,000 Oe, it has been found convenient to assume that the saturation level established below 20,000 Oe for such materials as iron and nickel would continue to higher fields if it were not for the saturation of the pole pieces. The ratio at the extrapolated DR_{ext} to the observed DR_{ob} at any field of interest then serves as a correction factor that must be applied to all raw data obtained above 20,000 Oe. Table I presents the correction factors that have been determined for the system described here. The reproducibility of these factors is better than 1%.

C. Correcting for the Sample Holder

It was anticipated that the copper sample holder and heater assembly might influence the measurement of the magnetic moment of the attached samples even though the materials constituting the holder are only feebly magnetic. Of primary concern was the low resistivity of the copper holder and the extent to which eddy currents would be induced in the holder as a result of the field gradients produced by the attached sample. Such eddy currents, if large and properly oriented would produce magnetic moments that would be superimposed upon that of the material under study.

A study of the temperature and field dependence of the magnetic

Table I. Correction factors for the high field image effect.

External magnetic field (H_{ext}) (Koe)	Correction factor
	<u>DR extrapolated</u> DR observed
20.020	0.9970
20.940	0.9950
22.020	0.9901
22.990	0.9756
23.980	0.9452
25.000	0.9099
26.110	0.8873
26.970	0.8726
27.900	0.8628

character of the holder showed that its moment is negligible in comparison to that of most materials studied (≤ 0.002 emu at 10,000 Oe and 4.2 K). However, when a magnetic sample is attached to the holder, a slightly larger diamagnetic moment can be attributed to the holder. This moment originates from the eddy currents induced in the copper holder by its motion in the distorted field produced by the presence of the strongly magnetic sample.

An exact calibration of this effect would be very difficult if not impossible since the contribution should depend upon temperature, sample moment, and magnetic field in a complex interrelated fashion. However, it is instructive to examine this effect in a simple manner.

The moment of the holder as a function of temperature and magnetic field was determined by comparing the reported magnetic information for Gd_2O_3 (Ref. 19), Dy_2O_3 (Ref. 19), and Tm_2O_3 (Ref. 20) with experimental data obtained for these materials. It was assumed that the total moment measured was a sum of the moment due to the sample as calculated from the reported data, σ_{sample} , and that due to the holder, σ_{holder} . The relation

$$\sigma_{\text{sample}} + \sigma_{\text{holder}} = \kappa DR$$

was then solved for σ_{holder} at various temperatures and internal magnetic fields for each of the materials selected. The values obtained for σ_{holder} by this method agreed to within $\pm 3\%$ for the three reference materials. This agreement is quite good considering the uncertainty in the published data, the fact that the effect is small, and that significant figures are lost in the subtraction process. An average of the values of σ_{holder} obtained by this method is displayed in Fig. 18 and tabulated in Table II.

Two equations of the type mentioned above, e.g., one for Gd_2O_3 and one for Tm_2O_3 , were solved simultaneously for σ_{holder} and κ at various temperatures and internal fields. The values obtained for σ_{holder} by this method agreed well with the values displayed in Fig. 18. A variation in κ of less than 2% was observed with the average agreeing to within 1.5% of the values determined by other methods.

VII. CONCLUSION

In the foregoing presentation an attempt was made to provide the

Fig. 18. Moment of sample holder.

TABLE II. Moment of sample holder (emu).^a

Temperature °K	External magnetic field (Oe)		
	3055	6090	20020
4.2	0.0362	0.0902	0.1823
27.0	0.0070	0.0175	0.0354
53.4	0.0018	0.0045	0.0091
82.5	0.0018	0.0045	0.0080
103.6	0.0010	0.0028	0.0068
126.1	0.0000	0.0015	0.0043
152.8		0.0010	0.0068
176.7		0.0000	0.0050
200.3			0.0045
226.1			0.0048
249.6			0.0045
273.2			0.0031
299.2			0.0028

^a The last digit in each of the above values of σ_{holder} should have been rounded off prior to obtaining σ_{holder} and hence is tenuous at best.

detailed information required to construct a vibrating-sample magnetometer capable of analyzing moments from about 200 emu to 1×10^{-4} emu over a wide range of temperatures and fields with an accuracy of better than 1% for large moments and about 4% for the smallest moments.

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X. APPENDIX

VERTICAL CROSS SECTION

Fig. A1. Vertical cross section of transducer-reference unit.

SECTION B-B

Fig. A2. Horizontal section of transducer-reference unit.

PHOSPHOR BRONZE SPRING

.020 THICK

Fig. A4. Phosphor bronze flexure spring for transducer-reference unit.

Fig. A5. Mounting table and cover for transducer-reference unit.

Fig. A6. Cross sectional view of electrical slip rings.

Fig. A7. Top view of outer electrical slip ring track and car.

A8. Top view of inner electrical slip ring track and car.

Fig. A9. View of elevator for transducer-reference unit.

Fig. A10. Top view of transducer-reference unit elevator.

USE 12-24 BRASS MACHINE SCREWS EXCEPT WHERE INDICATED

Fig. All. Mounting bracket.

Fig. A12. Top view of mounting bracket.

Fig. A13. Bolt used to attach mounting bracket to magnet.

Fig. A14. Glass dewar.

Fig. A15. Top view of glass dewar.