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ON THE PRECISION OF A SUSCEPTIBILITY APPARATUS AS DEDUCED FROM MEASUREMENTS ON SUPERCONDUCTING VANADIUM

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ABSTRACT

An apparatus for measuring static magnetic susceptibilities, based on a sensitive (10^{-9} N) vacuum microbalance, has been adapted for measuring magnetic moments of small superconducting samples in small gradients of low fields. Data taken on vanadium at 3.2 K can be analysed for sample moments consisting of two parts: an induced one, proportional to the field; and a permanent one. The analyses of the data show that for a single sample on an undisturbed balance the relative precision on the forces at low temperatures can be better than 0.04 %. For coil positions ranging over 2 cm a good agreement is observed between the experimental and the calculated forces by fitting for one proportionality constant.

INTRODUCTION

An apparatus for the measurement of the static magnetic susceptibilities of small solid samples by the Faraday method has been described previously (ref. 1). The striking part of the set-up is a vacuum microbalance with a sensitivity about 10^{-9} N. From this balance the sample hangs down in a dewar (ref. 2). The sample temperature can be any between 3 and 300 K. A data acquisition and processing system is added to the instrument (ref. 3).

The balance measures the force exerted on a specimen by a non-uniform magnetic field H. The system is chosen in such a way that the z-component of the force

$$F_z = \mu_0 m \,[\chi . H_z + P]\; dH_z/dz \qquad (1)$$

is measured. In equation (1) μ_0 is the free space permeability, m the mass of the sample, χ its susceptibility, P its permanent dipole moment, H_z the z-component of the magnetic field and dH_z/dz the gradient of this field component.

Equation (1) shows that the high sensitivity of the force detector allows for measuring small values of the product $m\,[\chi . H_z + P]\; dH_z/dz$. At the measurement of the magnetization of superconductors the susceptibility values are rather high but the fields and gradients are kept low so that in these measurements the forces also remain small. The aim of this contribution is to report on the generation and the measurement of these low fields and on the accuracy obtained with the apparatus.

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EXPERIMENTAL

The sample lays on the balance pan which hangs in the dewartail. At the sample place the magnetic field and its gradient are generated by a small ironless coil. The copper-coil/sample-pan situation is drawn in Fig. 1. By screwing up or down the holder, the coil position P can be changed with a reproducibility of $<$ 0.02 mm (ref. 4). The four higher sections, each of 125 turns, are electrically connected in series. The fields and gradients have been calculated for a simple model in which the coil has cylindrical symmetry and the sample is assumed to be located on the cylinder axis. For symmetry reason H_x and H_y are put equal to zero. The z-component of the field and the gradient is considered to be the bare effect of current rings, calculable by the Biot and Savart law. The rings build up a simple array, the geometry of which is schematically drawn in Fig. 2. Briefly, the field at coordinate z is approximated by:

$$H_z = (I/2) \sum_{i=1}^{4} \sum_{j=1}^{9(8/13)} \sum_{k=1}^{13} (R + (j-1).b)^2/r_{ijkl}^3 \qquad (2)$$

with:

$$r_{ijkl} = [\,(R + (j-1).b)^2 + ((i-1).a + (k-1).c + l.d)^2\,]^{1/2} \qquad (3)$$

Herein i refers to the number of the section in the coil, j to the number of the layer in the section, k to the number of the ring in the layer and l to the algebraic number of steps with length d, to reach the coordinate z from z = 0. In the coil the sections are measured to be, on the average, a = 7.567 mm apart. In the sections the layers are estimated to be 0.3 mm distant from each other and the rings in the layers are set to be separated by c = 0.2923 mm. The radius of the first layer, at mid-wire, is R = 17.15 mm. The calculation is carried out with the computer programme CFG01 which also approximates, for the same chosen current intensity I, the gradient of the magnetic induction by:

$$G_z = \mu_0\ (H_z - H_{z-\delta})/\delta \qquad (4)$$

with $\delta = 1 \times 10^{-5}$ mm. The programme further calculates:

$$[\Phi_z = \mu_0 H_z G_z]_I \qquad (5)$$

Normalized functions of H_z, G_z and Φ_z are given in Fig. 3 showing their maxima to be situated on different z-coordinates.

The coil current

The current is fed by a John Fluke 382A constant current source (CCS in Fig. 5). The current intensity I is defined by measuring the voltage over a reference resistor (see Fig. 4). In this case in which the susceptibility of the surroundings can be neglected, a constant sample-versus-coil geometry results in:

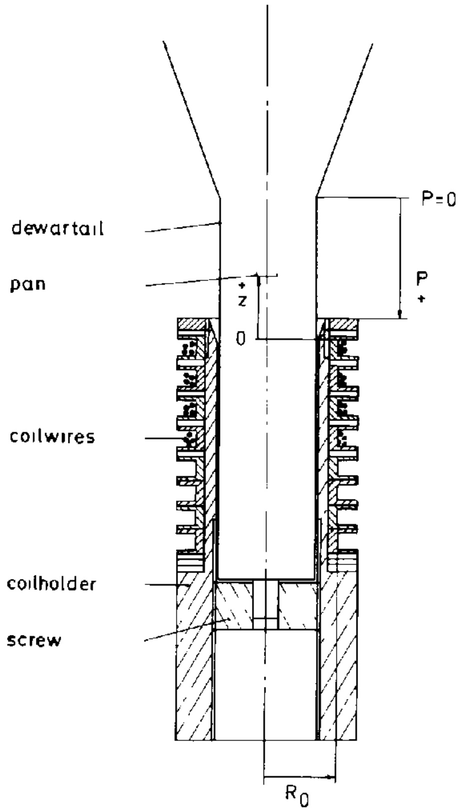


Fig 1 Coil/sample situation

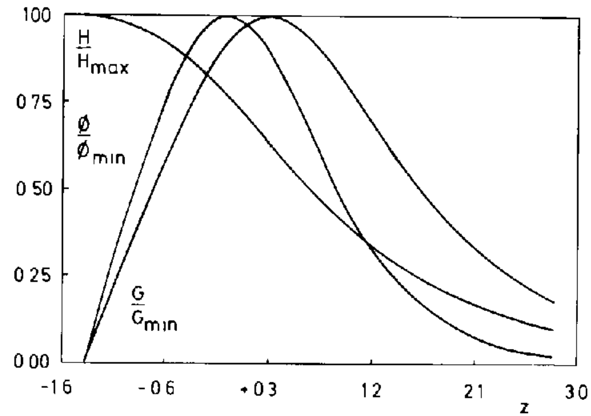


Fig 3 Field, gradient and their product, normalized, vs z

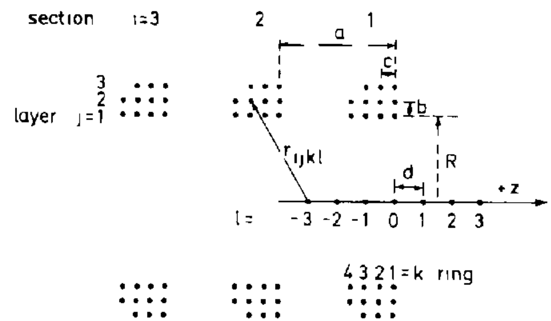


Fig 2 Ring array for field calculations

$$H = H_1 . I \qquad (6)$$

and:

$$G = G_1 . I \qquad (7)$$

where H_1 and G_1 are proportionality constants the values of which are those of H and G calculated for I = 1 ampere. For the sake of simplicity, the subscript z is omitted here and in what follows.
With the above relations, equation (1) becomes:

$$F = AI^2 + BI \qquad (8)$$

with:

$$A = m\chi . H_1 G_1 \qquad (9)$$

and:

$$B = mPG_1 \qquad (10)$$

A is proportional to $\chi . H_1$, the induced moment at unit current intensity and B to the permanent moment.

The force, exerted on the vacuum microbalance, is compensated by the negative feed-back control system, described in ref. 5. The output voltage of the system is related to the force by:

$$V = \psi . F \qquad (11)$$

As V is, within close limits, linearly related to the compensating force and a large fraction (> 0.99) of the force to be measured is compensated, ψ becomes, to a good approximation, the proportionality constant:

$$\psi = C = 5.14 \times 10^{+5} \ VN^{-1} \qquad (12)$$

With these relations equation (1) yields:

$$(V(+) + V(-))/(2|I|^2 C) = A \qquad (13)$$

$$(V(+) - V(-))/(2|I|C) = B \qquad (14)$$

$$(V(+) + V(-))/(V(+) - V(-)) = A|I|/B \qquad (15)$$

The signs (+) and (-) refer to the polarity of the current. The possibility of reversing the current direction in the coil allows for differentiating between induced and permanent moment.

The current is chopped, which means that it remains 51 seconds "on" and 51 s "off" (see ref. 3). The reason for chopping is the instability, on the long term, of the zero point of the balance. During a single cycle the zerodrift remains rather small. The zero position at the "on" period, therefore, can be calculated to a good approximation by interpolation from the zero point values measured during the "off" periods. The chopping circuit is designed, see Fig. 5, to

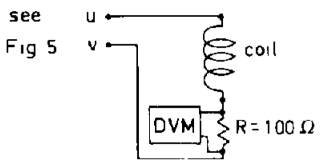
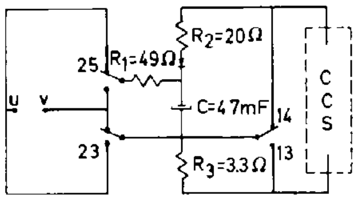
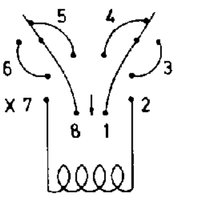


Fig 4 Coil current circuit



(1Y and 2Y refer to contacts on S1 and S2 in Fig 7)

Fig 5 Reversing and chopping circuit



Schrack relay SX non energized

Fig 6

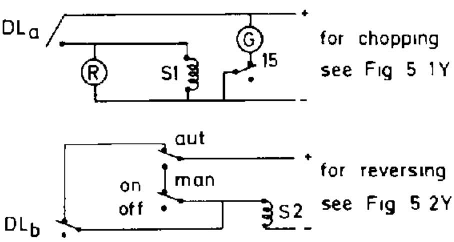


Fig 7 Relay driving circuit

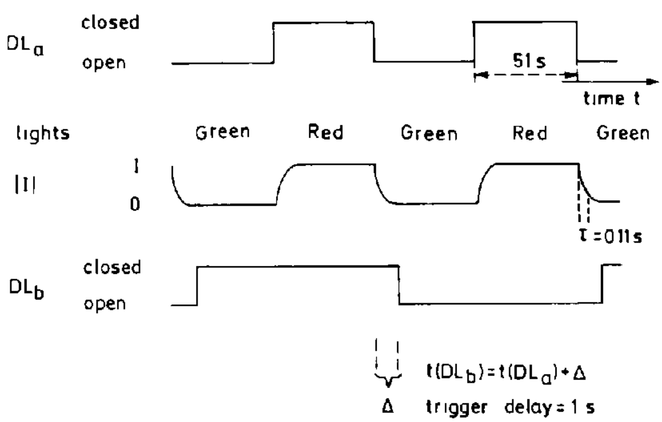


Fig 8 Time diagram

avoid fast transient effects for the reason explained in ref. 5. For the same reason the current direction is reversed during the "off" periods. The chopping and reversing are carried out by Schrack relays (see Figs. 5-7). These are driven by the data logging system to which is referred in ref. 5. The driving circuits are given in Fig. 7 in which DL refers to contacts which open and close at a constant rate. The time diagram of the switching and the evolution of the absolute value of the coil current are given in Fig. 8.

The comparison of forces to ϕ_1

At the comparison of measured forces to calculated ϕ_1-values, in this first approximation, the dimensions of the sample are not taken in account. The fields and gradients, at all places in the sample, are considered to be practically equal to their value at the coil axis at the nominal z-coordinate of the specimen. Consequently, the sample should be small but its reduction is limited by the limited sensitivity of the force meter. A compromise consisted in considering a 0.2 mm thick, 3.0 mm diameter, superconducting pure vanadium disc at 3.2 K. The disc is placed on the balance pan, its plane perpendicular to the z-axis. The force measurements, in a first experiment, have been carried out at coil positions $P > -0.2$ cm; for coil currents in the chopping mode alternating the current direction. From the output voltages of the force meter, the values of A are calculated following equation (13). The maximum value of A, for a certain current intensity I, is found at $P = +1.10$ cm. The values of A, gathered with the coil unchanged at this position in a single day experiment i.e. with one dewar-filling of liquid helium, are plotted in Fig. 9 versus the current intensity. The data can be analysed as consisting of a constant $\bar{A}_{1.10} = 4.0752 \times 10^{-5}$ (NA^{-2}) on which a random deviation is superposed. The standard deviation, for the results taken with a current larger than 0.2 A, is $\sigma = 0.0014 \times 10^{-5}$ (NA^{-2}) and the variation coefficient $\zeta = 0.035$ %. At lower coil current intensities, the deviations can be attributed to fluctuations on the force measurements, the uncertainty being about 1×10^{-9} N. The over all precision on the measurements which have been carried out on the superconductor at 3.2 K, gives faith to the low temperature force measurements.

The observation that $A_{1.10}$ is practically independent on $|I|$ indicates that the factor $m.\chi$ in equation (9) is a sample constant. Consequently, the quotient

$$Q = (A/\phi_1) = m.\chi/\mu_0 \qquad (16)$$

is expected to remain constant too for different coil positions. To check this statement, another single day experiment has been carried out with the coil at different positions, from $P = +4.30$ to $P = -0.20$ cm. In the region $P \leq 2.1$ cm measurements have been performed every 0.1 cm. To become able to locate more precisely the maximum of the field by interpolation the coil positions

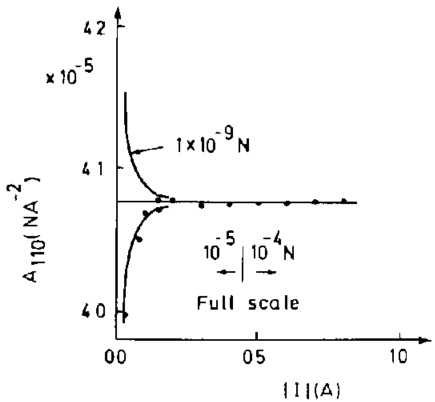


Fig 9 Induced moment parameter vs coil current intensity

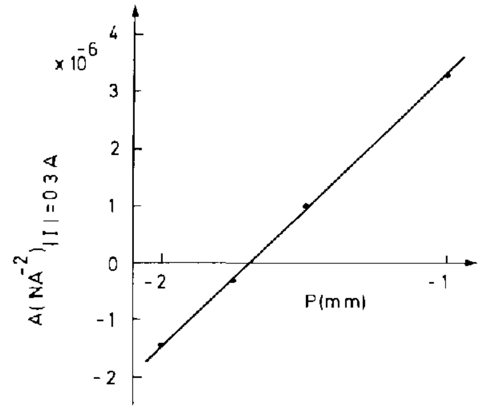


Fig 10 Induced moment parameter vs coil position

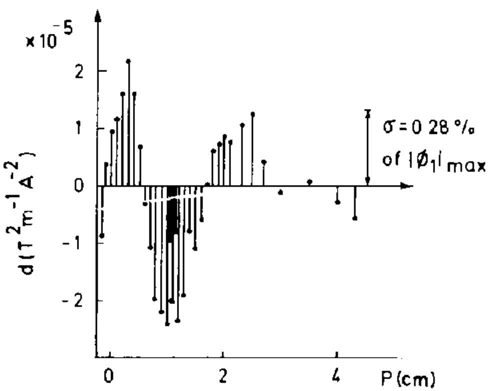


Fig 11 Deviations of the experimental from the calculated ϕ_1 values

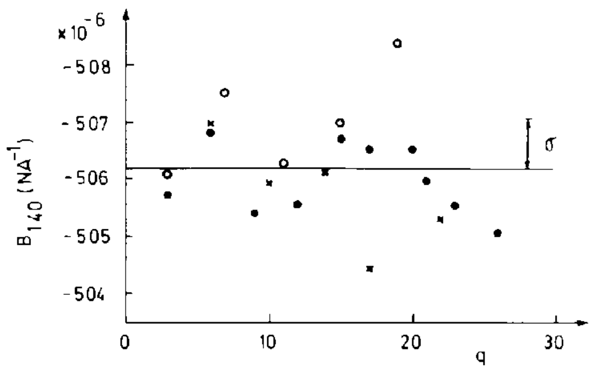


Fig 12 Permanent moment parameter vs measuring number

$P = -0.175$ and $P = -0.150$ cm have been considered next. Finally the positions $P = +1.10$, $P = +1.05$ and $P = +1.15$ cm allowed the location of the maximum of A at $P_{top} = +1.10$ cm. All 36 measurements that day have been carried out with $|I| = 0.300$ A. Part of the results is plotted in Fig. 10 versus P, locating the maximum of the magnetic field at $P_{max} = -0.169$ cm. From Fig. 1 it can be deduced that the measured P values are related to the calculation z-coordinates by:

$$z = P + DP \tag{17}$$

DP being constant. With the calculated z coordinate for maximum field and with P_{max}, obtained from Fig. 10, equation (17) results in $DP = (-1.306) - (-0.169) = -1.137$ cm.

The value of the quotient $Q = (A/\Phi_1)$ is expected to be determined most precisely from data taken at P_{top}. The two measurements, which have been carried out at this coordinate during the day, yield for A_{max} the values 4.0676×10^{-5} and 4.0775×10^{-5} (NA^{-2}) respectively. Their relative deviation 0.244 % is about seven times the ζ-value of the forementioned experiment. The probability that this deviation is due to a fluctuation on the balance-output, therefore, is accepted to be small. Consequently, it is believed that the coil manipulations always disturb the apparatus in a certain way and reduce the reproducibility of the measurements to about 0.25 %. This precision is expected to hold also for the quotient $Q_{1.10} = (A/\Phi_1)_{1.10} = -8.732 \times 10^{-3}$ (NmT^{-2}), which figure is obtained by dividing the average of the values of A, measured the latter day at $P = +1.10$ (± 0.03) cm, by the related value of $\Phi_1 = -4.664 \times 10^{-3}$ $T^2m^{-1}A^{-2}$ calculated for $z_{dip} = -0.06$ (± 0.01) cm. The uncertainty on the coordinates is due to the step-wise gathering of the data. It should be remarked that the average value $\bar{A}_{1.10} = 4.0726 \times 10^{-5}$ (NA^{-2}), here considered, happens to deviate only by 0.065 % from the average of the forementioned experiment.

When the quotient remains constant at all coordinates then its value should be identical with the ratio:

$$R = \frac{\sum_{i=1}^{n} \frac{A_{(i-1)}+A_i}{2} \Delta P_i}{\sum_{i=1}^{n} \frac{\Phi_{1,(i-1)}+\Phi_{1,i}}{2} \cdot \Delta P_i} \tag{18}$$

wherein $\Delta P_i = P_i - P_{(i-1)}$, $A_o = 0$, $\Phi_{1,0} = 0$, $P_o = P_{max}$ and where the subscript i refers to the i-th measurement in a list of n measurements which are ordered in a way that the related P-coordinates always increase for increasing i. To calculate R the computer programme CFG02 has been written. One part of it calculates, with Q_c, a chosen value for the quotient Q, the $\Phi_{1,i}$ value for each of the related P_i coordinates. It further calculates the standard deviation:

$$\sigma = [(\sum_{i}^{n} d_i^2)/(n-1)]^{1/2} \tag{19}$$

wherein the deviation is:

$$d_i = [(A/Q_c) - \Phi_1]_i \tag{20}$$

The same programme minimizes the standard relative deviation σ_r by putting:

$$[(d\sigma_r)/(dDP)] = 0 \tag{21}$$

with:

$$\sigma_r = [(\sum_{i}^{m} d_{r,i}^2)/(m-1)]^{1/2} \tag{22}$$

and:

$$d_{r,i} = [1 - Q_c \cdot \Phi_1/A]_i \tag{23}$$

on the m measurements for which A is larger than $A_{max}/3$. The calculation yielded DP = - 1.138 (± 0.002) cm, in good agreement with the value obtained from the matching of the measured - to the calculated - field maximum. The programme CFG02 also does the summations to be used in equation (18). The result is $R = - 8.698 \times 10^{-3}$ (NmT^{-2}). The introduction of this R value as the quotient Q_c in the programme yields $\sigma_r \leq 0.44$ %. This figure can be considered as an order of magnitude of the expected precision on R. The R value deviates by 0.39 % from the above mentioned quotient $Q_{1.10}$. The agreement is rather good when it is remembered that the relative precision on a single A determination becomes smaller with the coil position more distant from P = 1.10 cm. The agreement indicates, because R is a kind of an average, that systematic deviations are small. The standard deviation over the whole spectrum is $\sigma \leq 13 \times 10^{-6}$ ($T^2m^{-1}A^{-2}$), about 0.28 % of the maximum value of $|\Phi_1|$. Although the small value of σ indicates that the shape of the spectrum of A fits nicely that of Φ_1, the deviations, which are given in Fig. 11, show correlation with the coil position P. The systematic behaviour indicates the presence of a minor effect which has not been considered in the model relating the A- to effective Φ_1-values.

The comparison of forces to G_1

Persistent currents have been introduced in the superconducting disc, generating a magnetic dipole. The terms B, which contain the permanent dipole, have been related to the calculated G_1-values by the same kind of data fitting which was used in the foregoing section. The persistent currents are induced in the superconducting disc in the following way. The coil is placed at P = - 0.20 cm and the current set at I = 0.900 A. The temperature of the vanadium disc then is brought down from 7 to 3.0 (± 0.2) K. At the latter temperature the force measurements have been carried out for coil currents in the chopping mode alternating the current direction. Immediately after cooling to 3 K a few data have been taken at P = - 0.20 cm with $|I|$ = 0.100 A but the bulk of the measurements were performed with $|I|$ = 0.060 A at different coil positions. The related B factors have been calculated following equation (14). To point out the persistence of the persistent currents, one in two to six measurements has been carried out with the coil positioned at P = + 1.40 cm, the position for maximum B value. The time spent, for changing the coil position over relative large distances and for allowing the balance to settle for a relative high precision, limited the number of measurements that could be performed with one single dewar-filling of liquid helium. Consequently this B to G_1 relating experiment concerns data taken in three days. Each of these days can be seen as a partial experiment which started by cooling the dewar and filling the liquid helium vessel. The liquid helium then is pumped off to reduce its temperature below 3 K. After this operation the persistent currents are induced in the sample. The force measurements then are carried out. Three different P_{max} values have been measured, one on each day: P_{max1} = - 0.170, P_{max2} = - 0.173 and P_{max3} = - 0.171 cm. The average value $\bar{P}_{max}$ = - 0.171 cm compares well with the value - 0.169 cm obtained in the foregoing experiment fitting A to ϕ_1. It means that, for a sample which remains undisturbed on the balance pan, the coordinate can be reset to within 0.004 cm.

At P_{top} = 1.40 cm, the B-values are rather insensitive for small coil displacements. The amplitudes of B, therefore, can be compared to each other without considering the fine-adjustment of the coordinates. The values $B_{1.40}$ are plotted in Fig. 12 versus q, the number indicating the q-th measurement taken that day. The values can be analysed as consisting of a constant main term, $\bar{B}_{1.40}$ = - 5.062 x 10^{-5} (NA^{-1}), on which a random deviation is superposed. The standard deviation then is σ = 0.009 x 10^{-5} (NA^{-1}) with σ = 0.18 %. When $\bar{B}_{1.40}$ is divided by the value of the maximum gradient, - 0.4978 $Tm^{-1}A^{-1}$ calculated for z_{top} = + 0.30 (± 0.01) cm, the quotient is $Q = (\bar{B}/G_1)_{1.40}$ = 1.0169 x 10^{-4} (NmT^{-1}). This figure, introduced in the computer, the programme CFG02 yields: 1) DP = - 1.138 (± 0.003) cm in good agreement with the value obtained from the foregoing experiment, and 2) the value Q_c = R = 1.0183 x 10^{-4} (NmT^{-1}) which lead to a minimum standard relative deviation σ_r < 0.32 %. The standard deviation over the whole spectrum of B is σ = 9.1 x 10^{-4} ($Tm^{-1}A^{-1}$) which is less than 0.19 % of the maximum-gradient value of G_1. The deviations $d_i = |(B/R) - G_1|_i$ show a similar although less systematic correlation with the coil position than that observed at the fitting of A/R to ϕ_1 in the foregoing experiment.

The comparison of forces to ϕ_1 and G_1

The computer programme CFG02 has been written to be able to calculate either

A or B directly from the force measurements at different P. The input data, P, |I|, V(+) and V(-), together with an indication for the dipole term looked for, starts the programme to fit the amplitude of the measured term to that of the calculated term for all values in the region wherein the amplitude does not depend much on the coil position; i.e. practically between the maximum and the maximum divided by 1.3. The programme then makes the computer adjust DP by putting $\Sigma d_1 = 0$ for the high P region wherein the values of A or B depend rather steeply on P, i.e. practically between the maximum / 1.3 and the maximum / 3. Because DP depends on the magnitude of the quotient, say Q_e, the best fit is obtained by an iterative process. The result of this part of the programme is the starting point for the formerly discussed part in which, with $Q_e = Q_c$, σ_r is minimized by changing DP and the sums are made for the determination of R. This procedure is applied, for induced moment information, to the data taken on the superconductor containing the relatively large permanent dipole. Because it concerns a relatively small effect the precision is expected to be rather poor. The calculated DP value is by 0.047 cm too negative as compared to the value obtained by the more precise fitting of B to G_1. The misfit is due to the small signal to noise ratio. Therefore DP has been kept constant in the programme. With DP = - 1.137 cm, CFG02 yields $R = -8.79 \times 10^{-3}$ (NmT^{-2}) with $\sigma_r = 4.8$ % and $\sigma = 1.24 \times 10^{-4}$ ($T^2m^{-1}A^{-2}$) which is ≃ 2.7 % of the maximum of ϕ_1. The value of R is 1.0 % more negative than the value obtained in the experiment, in which no permanent dipole has been purposely induced in the disc and in which the measurements have been carried out with |I| = 0.300 A. Because the maximum force related to |I| = 0.300 A is 3.7×10^{-6} N, which is well suited for being measured by the balance, the related R value is more precise. With the lower current |I| = 0.060 A the maximum force only is $|3.7 \times 10^{-6}$ N x (0.060 A / 0.300 A$)^2|$ ≃ 0.15×10^{-6} N. A precision of 1.0 % on this force means an uncertainty on the force of ≃ 1.5×10^{-9} N. This is practically the sensitivity limit of the balance. Consequently, to within the precision of the apparatus in these experiments no difference is revealed in the A value, whether or not the permanent dipole was present in the superconducting disc of vanadium. Because the uncertainty 1.5×10^{-9} N is related to a maximum force ≃ 3.0×10^{-6} N due to the permanent dipole, the precision on the force measurement is 0.05 %. This precision could be achieved because the balance system has been kept undisturbed during the measurements at each z-coordination point.

The programme CFG02 has been applied, for B information, to the data taken on the disc in which no permanent dipole had been induced purposely. Again DP is too negative, here by 0.067 cm, indicating that the signal to noise ratio is even less than in the above case. Fixing DP = - 1.137 cm the programme yields $R = -2.38 \times 10^{-7}$ (NmT^{-1}) with $\sigma_r \simeq 7.4$ % and $\sigma = 2.3 \times 10^{-2}$ ($Tm^{-1}A^{-1}$) which is ≃ 4.7 % of the value of the maximum gradient G_1. The R value in this case is only - 0.24 % of that, obtained on the disc with the permanent dipole purposely induced. The change in sign indicates that the direction of the parasitic permanent dipole vector points to the-z-direction, the direction of the earth magnetic field. When the trapped flux is proportional to the field, the generating parasitic field should be (- 0.24 x 10^{-2} x 119 x 10^{-2} T =) - 2.9 x 10^{-5} T. This figure should be compared to the earth magnetic field, ≃ - 4.5 x 10^{-5} T. The maximum force, due to the permanent parasitic dipole, was 3.6 x 10^{-8} N. It is about 1 % of the maximum force due to the induced moment. When it is assumed that the relative precision on the total force is about 0.05 %, as in the former case, then the relative precision on the parasitic permanent moment should be about 5 % in good agreement with σ_r.

CONCLUDING REMARKS

The experiments with the superconducting vanadium disc on the vacuum microbalance reveal that, with the described system, magnetically induced forces on the sample can be measured, at temperatures as low as 3.2 K, with a reproducibility better than 0.04 % when the position of the fieldcoil remains unchanged. The induced moment parameter A, in this case, is independent of the coil current, indicating that m_A is a sample constant. The introduced persistent moment, measured by the parameter B, remained constant, to within one percent, during the experiments. For different coil positions the values of A are observed to be proportional to the calculated product of the field times its gradient; these of B to the gradient. The proportionality not only indicates that the assumptions on A and B are sound but also that the field versus z-coordinate, as calculated for the simple coil model, are precise to within 0.5 %. The absolute value of the proportionality constant is not discussed here, for lack of space, although measurements have been carried out which yield a field accuracy better than one percent.

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