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Magnetic properties of samples 1E and 2J (EUROMET Project 324)

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Introduction

The following is a report of measurements made at the BIPM in accordance with instructions sent to participants in the comparisons organised as part of EUROMET Project 324. The two 1 kg mass standards (samples 1E and 2J) arrived at the BIPM in good condition on 1996-01-09. They were brought by M. Mosca of the IMGC. Various measurements were carried out at the BIPM on ten different days during the following two weeks. Although the main purpose of these measurements is to determine the volume magnetic susceptibilities of the samples, a number of auxiliary measurements are also reported below.

All uncertainty components are given at a confidence level of approximately 68% (1 standard deviation, in the case of Type A uncertainties). Uncertainties are combined in accordance with ISO recommendations.

Measurements with a gaussmeter

Magnetic susceptibility cannot be measured without exposing the sample to a magnetizing field. For samples of poor magnetic quality, the magnetizing field may itself induce permanent magnetization. In contrast, the measurement of permanent magnetization by use of a gaussmeter is passive and thus leaves the magnetic properties of the sample unchanged.

For this reason, I began with a survey of the magnetic fields of the samples. These measurements make it clear that both mass standards arrived at the BIPM in a magnetized condition.

The instrument used was a Hall probe gaussmeter (LakeShore model 450; transverse probe model MMT-6J08-VH). It was calibrated by the manufacturer on 12-12-95 and certified to be within its specifications

On the most sensitive range, the instrument has a readability of 10 nT and a repeatability (based on one standard deviation of a large sample) of about 75 nT. For differential measurements, the instrumental standard uncertainty of this range is stated by the manufacturer to be 0,05% of the reading. The manufacturer also states that the combined standard uncertainty of a measurement of magnetic field is unlikely to be better than 0,13 % of the instrument reading. *

A square aluminium plate with a slot running down the middle of the upper surface was constructed to hold the Hall probe. The slot has the same width as the probe and a depth

* As established through personal communication, the manufacturer's published specifications for "accuracy" are stated with a 95% confidence level. I have therefore divided the manufacturer's uncertainties by 2 in order to arrive at approximate standard uncertainties.

slightly greater than the probe thickness. Thus the probe fits snugly into the slot and its active area is just below the surface of the plate. The aluminium plate serves three purposes. First, it immobilises the probe. If this were not done, changes in the orientation of the probe would make it impossible to measure fields smaller than the background field of the Earth. Second, the plate defines a coordinate system that places the sensitive axis of the probe perpendicular to the plate's surface. Third, the plate provides a thermal anchor thereby minimising changes in instrument reading due to the thermal coefficient of the Hall probe.

A piece of graph paper was taped to the aluminium plate so that the principal axes were centred directly on the active area of the Hall probe.

The probe calibrated according to the manufacturer's recommendation. That is, from time to time the probe was removed from its mount, placed in a zero-field chamber and run through its autocalibration mode. Only a small part of the most sensitive scale of the device was used in the measurements.

Two types of measurement were made. First, a single sheet of clean lens tissue was placed over the graph paper and the test mass was placed directly on the lens tissue. By moving the mass, a map could be made of the magnetic induction perpendicular to each face (Fig. 1a). With the mass placed horizontally on the lens tissue, the induction pependicular to the curved surfaces could also be mapped.

In a second series of measurements, the mass was supported directly above the probe so that its axis intersected the active area of the probe (Fig. 1b). This was done at heights of 40 mm and 45 mm above the probe so that the average gradient of induction in this region could be estimated. The results were then be compared with with those derived from susceptometer data, described below.

For the results summarized in Table 1, the measured induction had approximate azimuthal symmetry and so only an average value of readings taken at 90° intervals are given. As a sign convention, fields coming from the north pole of a magnet are considered positive. The averages shown were reproducible to about 5% for measurements at the centre of each flat surface. Thus the instrumental uncertainty of the gaussmeter is neglible for these measurements.

The surfaces of 2J have a relatively strong, negative magnetic induction in the centre. Its strength falls off towards the edge and even changes sign in the cases of the top. The induction changes by about a factor of two between the top and bottom of 2J.

For 1E, the central induction minimum is much stronger and the change in induction from the centre to the edge is more pronounced than for 2J.

In Table 2, it is interesting to note that the sign of the induction is reversed at 40 mm from the top of 2J (but not from the bottom) and at 40 mm from the bottom of 1E (but not from the top). The measurements are reasonably consistent with the susceptometer results described in the next section.

The magnetic induction perpendicular to the curved surfaces was also measured and differ by almost a factor of 100 between the two samples. For 2J, fields ranging from + 8 μT to −8 μT were found and their distribution did not have azimuthal symmetry.

For 1E the permanent fields were azimuthally symmetric, but otherwise unusual: the induction perpendicular to the curved surface passed through zero at a height about 12 mm above the base of the sample. The induction perpendicular to the side reached a strong maximum of +60 μT at a height of about 22 mm above the base. The variation of the induction was approximately linear between these two points.

Clearly both samples are permanently magnetized in a complex way that cannot be entirely determined from the measurements made with the gaussmeter (see Appendix 2). In addition, since magnetization is not an intrinsic property of the alloy, we have no way of knowing how permanent is the "permanent" magnetization measured during our study.

Measurements with BIPM susceptometer

Some measurements were carried out with the BIPM susceptometer. The construction and calibration of this instrument have been described in detail elsewhere [1,2]. Since the samples were already quite magnetized (especially 1E), we made measurements only at relatively low magnetic intensities (< 1 mT) in order to avoid further degradation of the samples. As verified by the gaussmeter, the permanent magnetization previously observed did not change significantly as a result of the susceptibility measurements. However, working at low induction reduced our signal and caused the readings to be greatly influenced by the permanent magnetization of the samples (in the formalism of [2], $F_a \approx F_b$).

The results, based on equations given in [2], are shown in Table 3 and Figs. 2 and 3. The equations are correct to first order in the susceptibility, that is to about 20% in the case of 1E. This problem is considered in more detail in the discussion.

In [2] I define a quantity M_z which I refer to as the "effective magnetization" of the sample along the vertical axis. This parameter has a simple interpretation only for uniformly magnetized samples which is evidently not the case here. Here, this quantity has been multiplied by μ_0 to give an effective polarization J_z(eff) as shown in the table. Since the susceptibility of sample 1E exceeds the acceptable range of the susceptometer [1], I have placed an asterisk in Table 3 next to the parameters calculated for 1E.

For the measurements made at the distance $Z_0 = 42{,}3$ mm it should be possible to infer the value of the field gradient in the vertical direction at the position of the susceptometer magnet due to the presence of the sample. This is because Z_0 is then much larger than the magnet dimensions (height = diameter = 5 mm). Thus the field gradient due to the presence of the sample should be approximately constant throughout the volume occupied by the susceptometer magnet. In this case, the measured force F_b (see [2] and Appendix 1) takes the simple form:

$$F_b = -m\left(\frac{\Delta B_z}{\Delta z}\right)_{z=0} , \qquad (1)$$

where m is the magnetic moment of the susceptometer magnet (0,089 Am^2). Results from this calculation are given in Table 2, where they may be compared with gaussmeter measurements of the same quantity. Remember that F_b is the <u>sum</u> of the permanent magnetization of the sample and the induced magnetization of the sample due to the presence of the Earth's field. An attempt to remove the contribution of induced magnetization is made in order to calculate J_z(eff).

Discussion

Uncertainty of susceptibility measurements

In order to assign a realistic total uncertainty to the susceptibility measurements, it is useful to consider contributions from four distinct sources :

1. <u>Reproducibility of the measurements</u>. The reproducibility is computed by routine statistical analysis. It is the only component appearing in Table 2 and Figs. 2 and 3.
2. <u>Susceptometer calibration</u>. If the susceptometer were used to measure samples which were:

- $\mu_r < 1{,}01$;
- linear, isotropic and homogeneous (LIH),

  a component of systematic uncertainty would apply to all reported measurements of susceptibility. In [2] we estimate the standard uncertainty of this component as 3% of the susceptibility for samples the size of 1E and 2J. This estimate was confirmed in 1992 by good agreement in a double-blind comparison between the BIPM and the PTB. The measurements were made on the same 1 kg mass standard which had a susceptibility of about 0,0035. The PTB apparatus operates on a principle different from that applied in the BIPM susceptometer.

3. Permanent magnetization. One may not exclude *a priori* the possibility that the relatively large "permanent" magnetization of the samples may interfere with the measure of intrinsic susceptibility. From Table 2 it can be seen that F_a/g, the balance reading due to susceptibility, in some cases is considerably smaller that F_b/g, the balance reading due to a combination of permanent magnetization of the sample and induced magnetization from the Earth's field. It is conceivable that correlations exist between χ , B_z(max) and J_z(eff). Perhaps the fact that the same susceptibility is found for the top and bottom of 2J despite their different degrees of magnetization indicates that permanent magnetization has been properly dealt with. In any case, I am unable to estimate an additional uncertainty so I have not included it.
4. Violation of condition that $\mu_r < 1{,}01$. The calculated result for sample 1E is $\mu_r \approx 1{,}2$. This violates condition 2. As shown in [1] for a special geometry, the calculated value of χ is actually the first term of a power series in χ. (The second term is of order χ^2). Thus the susceptibilities given in Table 2 for 1E are subject to an unknown correction of order 20%. For 2J, the unknown correction is of order 1%. No corrections have been made but the added uncertainty for 2J has been estimated as 1%. Susceptibility results obtained for 1E are considered to be unreliable (they are probably systematicaly low). For this reason, the results given in Table 2 are marked with an asterisk.

For 2J we must therefore add an uncertainty of 0,00028 to the standard uncertainties of χ stated above. Under the assumption that no field dependence or difference between top and bottom is observed, I therefore find the following result for the susceptibility and combined standard uncertainty of 2J :

$$\chi = 0{,}00900(32)$$

As explained above, I suspect my measured susceptibilities for 1E are low by about 20% but this bias is itself uncertain. The only sure comment one can make regarding 1E is that it does not meet the OIML criteria for mass standards of classes *E* or *F*.

Additional comments

The agreement between the last two columns of Table 2 is satisfactory. The uncertainty component shown is simply the reproducibility of the data. No account has been taken of the various simplifications used in deriving the gradient.

One striking feature of the permanent magnetization deserves additional comment. Table 1 shows that the surface magnetization perpendicular to each sample at the axis is negative (directed into the surface). Table 2 shows that, at a distance of 40 mm from each sample along the axis, the sign of the field has reversed for 1E(bottom) and 2J(top). Qualitatively, these phenomena might be explained by the presence of a "small" magnetized volume located near the surface of each sample and on the axis. The field produced from this source dies away quickly with distance so that a second field, due to a large volume of material magnetized in the opposite direction, is revealed. A semi-quantitative model is presented in Appendix 2. If this hypothesis is valid, we could be seeing the imprints of two different types of apparatus used to measure the magnetic susceptibility.

References

(note: a photograph of the susceptibility apparatus appears in [1] and more detailed drawings of each component are given in [2]).

1. R.S. Davis, *Meas. Sci. Technol.* **4** (1993) 141-147.

2. R.S. Davis, *J. Res. Natl. Inst. Stand. Technol.* **100** (1995) 209-225.

Appendix 1
Demonstration of the validity of eq. (1).

The force between two magnetized bodies is given in [2] as :

$$F_z = -\mu_0 \frac{\partial}{\partial z} \int \boldsymbol{M} \bullet \boldsymbol{H} \mathrm{d}V ,$$

where M is the magnetization of one of the bodies and H is the field due to the second body. The integral is taken over the volume of the first body. If we consider the first body to be the permanent magnet of the susceptometer, then $MV = M_z V = m$ where m is the moment of the magnet and V is its volume. The magnet is aligned parallel or antiparallel to the vertical (z) axis. Let the origin of the coordinate system be located at the centre of the magnet, as in [2].

Now expand H_z in a Taylor series in z about $z = 0$. If H_z varies slowly over the volume of the magnet, then variation in the x-y plane may be ignored and only the leading term retained from the Taylor series. The above equation simplifies to :

$$F = -m \left(\frac{\Delta \mu_0 H_z}{\Delta z} \right)_{z=0} .$$

Since H is defined as the field in free space due to the second body, $\mu_0 H_z = B_z$.

Appendix 2
A qualitative model of magnetization

Referring to Figure 4, imagine that the volume of the weight contains two spherical regions which have become permanently magnetized in opposite directions along the vertical (Z) axis. For the dimensions and magnetizations shown in the figure, it is not hard to calculate that the inductions along Z axis are as follows :

40 mm above top of weight	+0,14 μT
At top of weight	−4,2
At bottom of weight	−6,0
40 mm below bottom of weight	−0,22 .

These numbers are in qualitative agreement with the measurements using 2J (Tables 1 and 2). Such a crude model, though suggestive, should not be pushed too far.

Table 1. Summary of gaussmeter measurements of surface magnetism. The tabulated measurements of B_z are given in units of μT. The magnetism is approximately symmetric about the central axis, so these results represent averages taken at 90° intervals. The reproducibilities of results at the centre (distance = 0 mm) are about 5% of the values. Values obtained at greater distances from the centre are less reproducible. This is due to imperfect azimuthal symmetry and the fact that, in some cases, the field varies more rapidly with distance

Distance from centre / mm	0	5	10	15	20
Sample	B_z / μT				
2J (top)	− 4,1	− 3,9	− 1,24	0,74	1,20
2J (bottom)	− 8,1	− 7,9	− 5,6	− 3,0	− 2,3
1E (top)	−60	−41	3,8	41	34
1E (bottom)	−92	−66	13,8	56	56

Measurements made at surface.

Table 2. Summary of gaussmeter measurements of B_z along axis at a distance Z_0 from surface. The gradient calculated from these data may be compared with the same quantity inferred from eq. (1). The only uncertainty component given is the reproducibility of the measurements.

			$(\partial B_Z / \partial z)$ / (μT/m)	
Sample	Z_0 / mm	B_Z /μT	from these data	from (1)
1E (top)	40	−0,36		
	45	−0,38	− 4(5)	−15(7)
1E (bottom)	40	6,12		
	45	5,10	−204(6)	−241(13)
2J (top)	40	0,24		
	45	0,19	− 10(5)	− 6,8(1)
2J (bottom)	40	− 0,50		
	45	− 0,41	+ 18(3)	+ 21,1(6)

Table 3. Summary of measurements made with susceptometer. The uncertainty component given for each χ is based only on the reproducibility of the measurement. Additional uncertainties are discussed in the report.

2J (top)

Z_0/mm	B_z(max)/μT	(F_a/g)/μg	(F_b/g)/μg	χ	J_z(eff)/μT
27,3	880	202	− 2	0,00905(7)	+ 0,38
32,3	530	92,1	− 47,5	0,00902(7)	+ 0,58
37,3	340	46,5	− 59,9	0,00913(25)	+ 1,1
42,3	240	24,6	− 61,8	0,00903(50)	+ 2,7

2J (bottom)

Z_0/mm	B_z(max)/μT	(F_a/g)/μg	(F_b/g)/μg	χ	J_z(eff)/μT
27,3	880	205	524	0,00919(7)	−10,3
32,3	530	90,6	357	0,00886(7)	− 9,4
37,3	340	45,6	254	0,00896(25)	− 8,9
42,3	240	24,4	192	0,00898(50)	− 7,9

1E (top)

Z_0/mm	B_z(max)/μT	(F_a/g)/μg	(F_b/g)/μg	χ*	J^*_z(eff)/μT
27,3	880	3652	684	0,164(7)	− 5,3
32,3	530	1172	528	0,166(7)	− 5,5
37,3	340				
42,3	240	492	138	0,181(24)	− 2,3

1E (bottom)

Z_0/mm	B_z(max)/μT	(F_a/g)/μg	(F_b/g)/μg	χ*	J^*_z(eff)/μT
27,3	880	3506	3674	0,157(5)	− 62
32,3	530	1658	3366	0,166(5)	− 77
37,3	340				
42,3	240	470	1329	0,173(14)	− 13

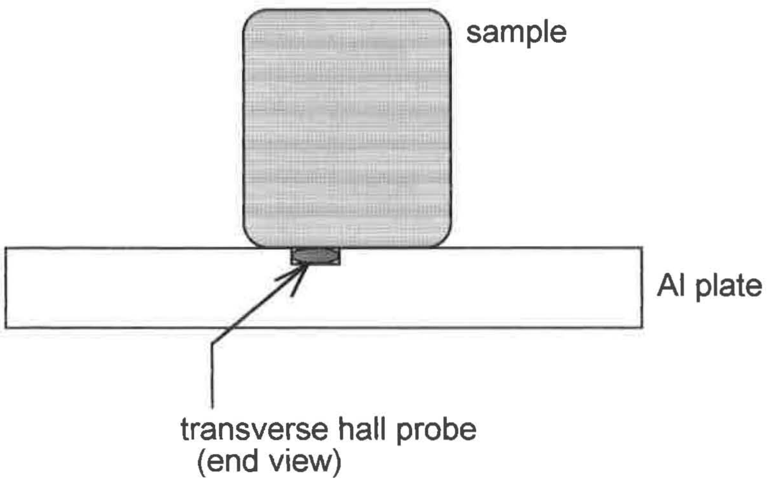

Fig. 1a. Measurement of magnetic induction perpendicular to the base of the sample. The drawing is not to scale. The active area of the probe is about 1 mm^2 . The top of the aluminium plate is covered with a piece of graph paper whose axes are centred on the active area of the probe. The paper is covered by a single sheet of lens tissue to protect the sample. The sample is displaced relative to the graph paper in order to map out the induction.

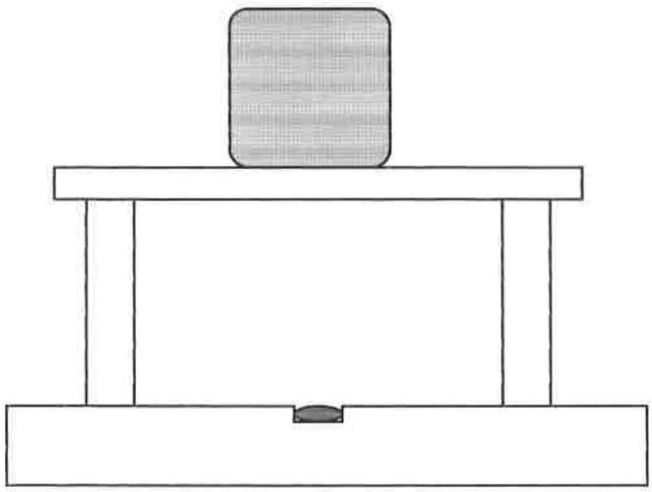

Fig. 1b. Schematic view of induction measurements along the axis of the sample. The distance from the base is set by the height of the bridge.

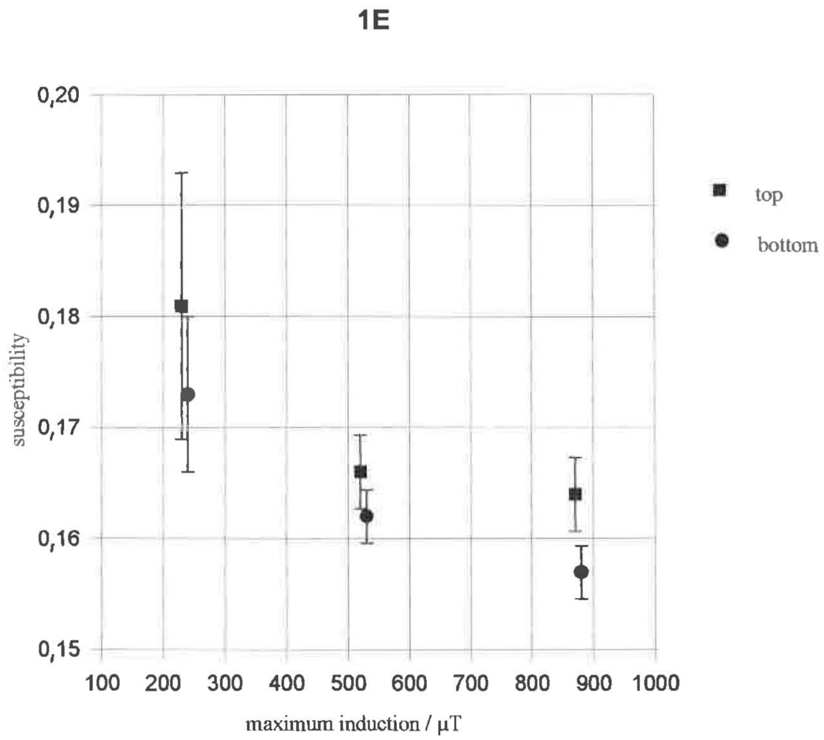

Fig. 2. Susceptibility measurements taken of the two faces of sample 1E. The error bars represent the reproducibility of the measurements. Additional, very large, uncertainties are discussed in the text. For clarity, a small offset has been given to the fields of the bottom measurements.

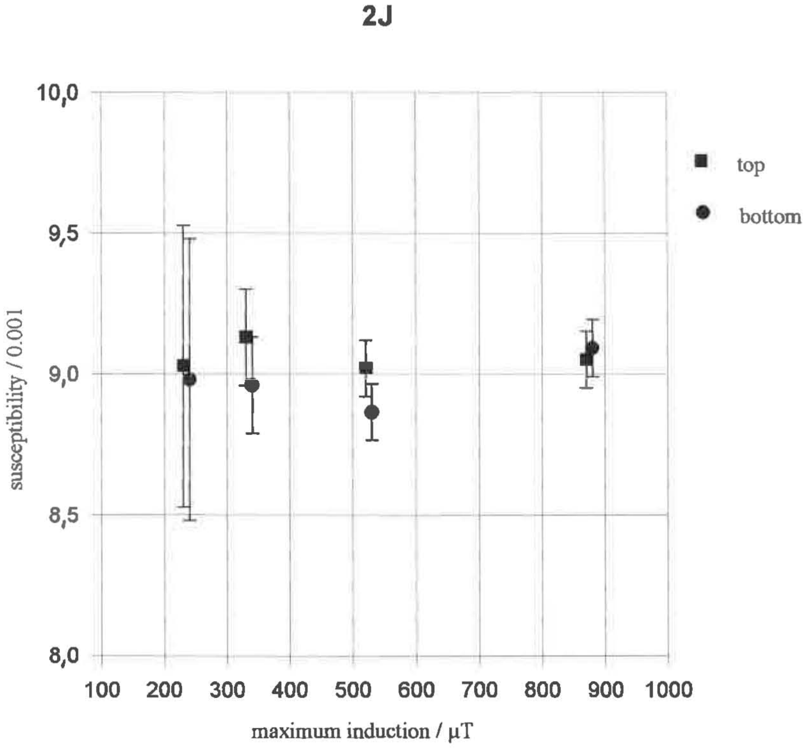

Fig. 3. Susceptibility measurements taken of the two faces of sample 2J. The error bars represent the reproducibility of the measurements. Additional uncertainties are discussed in the text. For clarity, a small offset has been given to the fields of the bottom measurements.

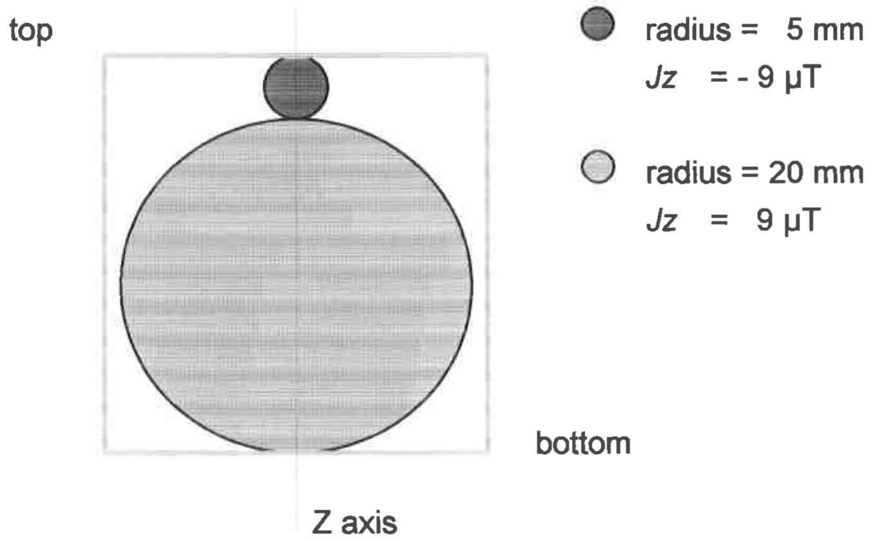

fig. 4. Model of permanent magnetization that accounts qualitatively for induction measurements made on sample 2J. The model assumes that the sample has two regions of permanent magnetization oriented in opposite senses.