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Report on the development of low magnetic permeability reference materials

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ABSTRACT

A study has been carried out to select suitable reference materials for the calibration of magnetic permeability measuring instruments over their normal working range of relative magnetic permeability of 1.0025 to 1.4. With the exception of some of the rare earth elements, the permeability of the elements is below the lower limit of this range. It was necessary, therefore, to investigate metallic alloys and composites. As a result, three magnetic alloys were selected on grounds of their stability and uniformity of composition.

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CONTENTS

APPENDIX

1 INTRODUCTION

The determination of the relative magnetic permeability of "non-magnetic" materials is an important requirement in the manufacture of devices and components for space, particle physics and defence applications. As all materials are magnetic to some extent, measurements on raw materials and finished components need to be carried out on a routine basis in the course of production. Work hardening and heat treatments can affect the value and uniformity of the relative magnetic permeability of a material.

A number of types of commercial instruments are now available for determining the relative magnetic permeability of weakly magnetic materials. However, the traceable calibration of such instruments is not straightforward since the relative magnetic permeability is the ratio of the magnetic flux density to the magnetic field strength applied to a medium and it is not possible to generate this quantity directly. As a consequence, magnetic permeability measuring instruments can only be calibrated by reference to materials of known value. Thus reference standards are required for the calibration of these instruments over their normal working range of 1.0025 to 1.4.

Since written commercial standards exist which specify the use of material of maximum relative magnetic permeability of either 1.005 or 1.05, it was aimed to produced materials in the permeability ranges 1.0025 to 1.007 and 1.03 to 1.07. Additionally, to check the higher end of the range of commercial instruments, a material having a relative magnetic permeability in the range 1.2 to 1.4 is required.

Ideally paramagnetic materials should be used for this type of standard since their magnetic permeability does not vary with magnetic field strength, only with temperature which can be controlled. However, the required values of magnetic permeability are higher than those to be found in any truly paramagnetic material or compound. This being so, it is necessary to explore "feebly magnetic" materials which can be obtained in the required permeability ranges. These materials suffer to some extent from the fact that the value of their relative magnetic permeability will vary with the applied magnetic field strength. Since different types of permeability indicator or measuring instruments use probes which generate widely different levels of magnetic field strength, this is a significant problem. To some extent this can be overcome by choosing a material having a low ferromagnetic content and also by measuring the relative magnetic permeability at a number of magnetic field strengths to enable the user to select the appropriate value for a particular probe.

A problem with this solution is the difficulty of determining the effective magnetic field strength acting on the reference material due to the large field gradients produced by the probes. By contrast, the reference materials will be evaluated and calibrated in a uniform field produced in a solenoidal coil system.

2 REQUIREMENTS AND CHARACTERISTICS OF POTENTIAL MATERIALS

The form of supply of the material, e.g. rod, bar, sheet etc., must be such that reference bars of suitable dimensions can be produced to enable absolute measurements to be made according to BS 5884[1]. The standard states:-

The test specimen shall be a round or rectangular bar or a number of strips or wires having a cross-sectional area of at least 100 mm^2; the maximum cross- sectional area will be determined by the diameter of the central aperture of the search coil. To avoid significant errors introduced by self-demagnetizing effects, the length to equivalent diameter ratio of the test specimen shall be not less than 10:1 and the minimum length of the test specimen shall be 200 mm.

An important factor to be taken into consideration when selecting the dimensions of the reference standards is the shape and active area of the probes associated with different types of permeability measuring equipment. To accommodate the three main types of probes currently available, a square section bar of dimensions approximately 25 x 25 x 300 mm having a machined (ground) surface finish is appropriate. Not all potential reference materials will be available in a form of supply which would enable such reference bars to be produced.

The other material characteristics which influence the choice of potential reference materials are:-

a) low ferrous content (low concentrations of iron, nickel etc.)
b) non-porous
c) non-brittle
d) rigid
e) uniformity of permeability throughout their volume
f) stability of permeability with time
g) corrosion resistant (in normal laboratory usage)
h) have no permanent effect of storage at temperatures between 0 and 50 °C
i) low, known temperature coefficient of permeability
j) good surface finish.

3 SELECTION OF POTENTIAL REFERENCE MATERIALS

A list of materials for feebly magnetic reference specimens is given in BS 5884[1], Appendix A, Table 1. The list is reproduced in Appendix 1 of this report. However, the list is by no means exhaustive and some of the materials are not available in a form suitable for the generation of calibrated reference materials. The relative magnetic permeability values quoted in Appendix 1 are for guidance only; the actual value of permeability requires determination by the absolute method given in BS 5884[1].

Based on manufacturer's specifications and long experience in measuring feebly magnetic materials, the following materials were selected for initial tests:-

Aluminium silicon bronze
Aluminium nickel bronze
High tensile brass
Stainless steel, type AISI 316[2]
Composite of glass impregnated with iron oxide
Composite of polyurethane and iron oxide
Composite of epoxy resin and iron oxide (also with various filler materials).

3.1 ALUMINIUM SILICON BRONZE

Material in a convenient form of supply was available from Delta Metals, alloy type CA12 which generally complied with the specifications of BS 2872[3], BS 2874[4] and NES 834[5]. Extracts from the manufacturer's data state:-

The aluminium bronzes are basically alloys of copper and aluminium often with major additions of both iron and nickel. These display a high mechanical strength (of which a considerable proportion is retained at elevated temperatures), excellent corrosion resistant properties, and excellent resistance to wear, abrasion and shock loading........ The inclusion of iron raises the tensile strength, while nickel improves the proof stress. Both of these improve the corrosion resistance.

Alloy CA12 contains a low concentration of iron and was thought to be a useful alloy for the lower end of the range of relative magnetic permeability. The nominal value of relative magnetic permeability of this alloy is given as 1.005. The quoted nominal percentage chemical composition for the alloy is:-

copper	90.70
aluminium	6.20
iron	0.70
manganese	0.20
silicon	2.20.

3.2 ALUMINIUM NICKEL BRONZE

Material in a convenient form was available from Delta Metals, alloy type CA3 which generally complied with the specifications of ISO 428[6] and NES 833[7]. The nominal relative magnetic permeability of this alloy is quoted by the manufacturer as being 1.40.

The quoted nominal percentage chemical composition of alloy CA3 is:-

copper	82.15
aluminium	9.20
iron	4.20
nickel	4.20
manganese	0.25

The comparatively high content of the ferromagnetic elements is necessary to attain a relative magnetic permeability at the higher end of the required range.

3.3 HIGH TENSILE BRASS

Again, material in a suitable form was available from Delta Metals, alloy type HT1 which conforms generally to BS 2872[3] (CZ114), BS 2874[4] (CZ115) and ISO 426[8].

The nominal relative magnetic permeability of this material is 1.07 making it suitable for use in the middle of the required range.

The quoted nominal percentage chemical composition of alloy HT1 is:-

copper	57.00
zinc	39.00
lead	1.00
tin	0.75
iron	0.75
manganese	1.50

A second batch of material of similar type conforming to BS 2872[3] (CZ114) was later purchased from AALCO (part of Amari Copper Alloys). The relative magnetic permeability of this particular material was thought to be nearer to 1.05, a useful calibration point on the scale of permeability measuring instruments.

3.4 STAINLESS STEEL TYPE AISI 316[2]

Material of this type is available from many metal stockholders. AALCO were able to supply bar stock of the required dimensions. The material conforms to the American Iron and Steel Institute specification 316[2] and has the following nominal percentage chemical composition:-

nickel	10 - 14
chromium	16 - 18
molybdenum	2 - 3
silicon	1
manganese	2
carbon	0.08
phosphorus	0.045
sulphur	0.030
iron	balance, approx 65%

In the annealed state the magnetic permeability is quoted as being typically 1.003 - 1.004 making it a possible material for the low end of the range.

3.5 COMPOSITE OF GLASS IMPREGNATED WITH IRON OXIDE

Discussions were held with Dr R Morrell in the Division of Materials Measurements at NPL whose work includes the production and study of ceramic composites with a view to dispersing iron oxide in a ceramic powder to form a feebly magnetic ferrite material. Due to the brittle nature of the ceramics they would have been unlikely to withstand even moderate usage as reference materials. Glass with a natural iron impurity was suggested as a possible material.

Preliminary measurements on samples of silica glass and plate glass obtained from the Optical Workshop indicated that these materials have far too low a magnetic permeability to be of interest. Discussions with Pilkington Glass revealed that comparatively high concentrations of iron oxide can be fused with glass. A paper on the subject had been published by C R Bamford[9] and this was discussed with Dr Morrell, DMM, who undertook to prepare a glass sample loaded with 10% gamma Fe_30_4.

3.6 COMPOSITE OF POLYURETHANE AND IRON OXIDE

Further discussions were held with BNF Fulmer Research Laboratories at Redhill, Surrey, to explore the possibility of loading plastic or epoxy resin materials with concentrations of iron oxide to produce materials of controlled magnetic permeability. It was suggested that a composite of polyurethane rubber loaded with iron oxide would be practical to produce in the form we require and BNF Fulmer agreed to produce prototype samples for our evaluation.

3.7 COMPOSITE OF EPOXY RESIN AND IRON OXIDE

Concurrent with the production of samples of glass and polyurethane composites, samples of epoxy resin loaded with various concentrations of gamma Fe_30_4 were produced in the Magnetics Section. An easily pourable form of epoxy resin was obtained from RS Components (Stock No. 561-628, with hardener Stock No. 561-628). About 10% by weight of gamma Fe_30_4 was evenly dispersed in the resin by careful stirring. The mixture was then placed in a vacuum de-gassing oven, heated to 80 °C, and allowed to set.

A problem was encountered with air bubbles being trapped in the resin causing a non-uniformity of the magnetic properties. A less viscous type of epoxy resin (Ciba-Geigy type CY1303 with hardener HY1300) was obtained after seeking advice from Ciba Geigy Plastics. In addition various filler materials were obtained. Composites were made up as follows:-

a) Approximately 7% Fe_30_4, 93% Cu Ni Brass alloy (no binder)
b) Approximately 15% Fe_30_4, 52% Kaolin, with 33% epoxy resin binder
c) Approximately 13% Fe_30_4, 45% Fullers earth, with 42% epoxy resin binder.

4 MAGNETIC MEASUREMENTS ON SELECTED MATERIALS

Test specimens of each type of material were produced by machining or casting, as appropriate. The dimensions were chosen to conform with those given in section 2 of this report, length 300 mm, cross-section 25 x 25 mm.

The average cross sectional area of the test specimens was determined from a number of measurements of the width and thickness of the specimens, measured at intervals along their length. The relative magnetic permeability of the test specimens was determined by the solenoid method described in BS 5884[1], clause 3. A circular search coil of 1500 turns of length approximately 40 mm and with a central aperture of diameter 60 mm was mounted at the centre of a solenoid of length 1 m and diameter 100 mm. A measured current was passed through the solenoid, the field to current constant of which was known. The current was adjusted to produce magnetic field strengths of 5 and 10 kA/m. The test specimens were demagnetised and then each in turn was inserted into the search coil. The output from this search coil was connected to an integrator, calibrated in terms of the magnetic polarisation, and the change in magnetic polarisation measured when a test specimen was removed from the search coil. The measurement procedure was then repeated with the test specimen reversed end for end and the average of the two magnetic polarisation values was calculated.

The relative magnetic permeability, μ_r, of the test specimens was then calculated from the average value of magnetic polarisation using the following relationship:-

$$\mu_r = 1 + \frac{J}{\mu_o H}$$

where: J is the magnetic polarisation, in tesla

μ_o is the magnetic constant (=$4\pi.10^{-7}$ henry/metre)

H is the magnetic field strength, in ampere/metre.

A correction was applied for the self demagnetization effect of the bars. The correction is in two parts, firstly the correction to the value of the magnetic field strength due to the self demagnetizing field calculated from the following relationship:

Correction to field strength, H

$$\delta H = \frac{2JA}{\mu_o 4\pi (0.4l)^2}$$

$$\frac{JA}{\mu_o l^2}$$

where: J is the magnetic polarisation, in tesla

A is the cross sectional area of the test specimen, in metre^2

μ_o is the magnetic constant (= $4\pi.10^{-7}$ henry/metre)

l is the length of the test specimen, in metres.

Secondly, a correction to the value of J is made to allow for the correction due to the self demagnetizing field strength according to the relationship:-

Correction to magnetic polarisation, J:

$$\delta J = \frac{A_s}{A}\mu_o \delta H$$

where: A_s is the cross-sectional area of the search coil, in metre^2.

5 RESULTS OF MEASUREMENTS

The values of relative magnetic permeability obtained in the above series of measurements on representative samples of the materials are given in the following table.

Table 1 Values of relative magnetic permeabilities obtained by measurements

Material	μ_r	Observations
Aluminium silicon bronze	1.000 7	Too low a value for a standard
Aluminium nickel bronze	1.2 -1.4	The permeability changed during heat treatment
High Tensile Brass	1.05	-
Stainless steel type 316	1.003	-
Composite of glass and 10% Fe_3O_4	< 1.001	Fe_3O_4 decomposed at the temperature required to fuse with the glass
Composite of polyurethane and: 2% Fe_3O_4	1.015	occluded air bubbles
8% Fe_3O_4	1.052	
16% Fe_3O_4	1.089	
Composite of epoxy resin and 15% Fe_3O_4	1.162	occluded air bubbles

6 DISCUSSION OF OBSERVATIONS ON RESULTS

6.1 ALUMINIUM SILICON BRONZE

This was found not to be a suitable material since the value of relative magnetic permeability of the test sample was well below the lower end of the range of magnetic permeabilities required.

6.2 ALUMINIUM NICKEL BRONZE

The heat treatment process reduced the relative magnetic permeability from 1.4 to 1.2. From preliminary measurements on the bars it is not clear if the magnetic permeability is uniform throughout their thickness; work is continuing to investigate the problem. However, this does seem to be a possible material for the higher end of the permeability range.

HIGH TENSILE BRASS

The permeability of this material was found to be close to 1.05 which is one of the "cut off" values used in a number of materials specifications. (Material above this level would be rejected according to the specifications). However, its permeability was found to vary slightly with the level of applied magnetic field strength. Provided measurements were made at the values of magnetic field strength likely to be encountered during usage, this material should prove to be very useful in the mid-permeability range.

6.4 STAINLESS STEEL TYPE AISI 316

Despite its apparent high content of ferromagnetic elements, this material had a relative magnetic permeability of 1.003 which is close to the low end of the permeability range. Provided it is a truly austenitic type of stainless steel it should be stable with time and change only marginally with the level of magnetic field strength. Therefore it appears to be a very suitable material for use at the low end of the permeability range.

6.5 COMPOSITES OF GLASS IMPREGNATED WITH IRON OXIDE

These proved to be unsuccessful due to the change in state of the Fe_3O_4 to Fe_2O_3 at the temperatures required to fuse the iron oxide with the glass.

COMPOSITES OF POLYURETHANE WITH IRON OXIDE

These proved to be disappointing since, although it was possible to mix given concentrations of Fe_3O_4 with the polyurethane to produce pre-determined values of magnetic permeability, the resulting composite suffered from occluded air bubbles throughout its volume. The resulting material was therefore of non-uniform density and hence of non-uniform magnetic permeability.

COMPOSITES OF EPOXY RESIN AND IRON OXIDE

Similar results were found to those obtained with polyurethane composites rendering them unsuitable as reference materials.

7 SELECTION OF REFERENCE MATERIALS

From the seven types of material evaluated, three were selected as possible reference materials for the required magnetic permeability ranges as follows:-

μ_r = 1.0025 to 1.007, Stainless steel type AISI 316[2]
1.03 to 1.07, High tensile brass
1.2 to 1.4, Aluminium nickel bronze.

These materials already complied with most of the requirements set out in section 2 of this Report. Although the type 316 stainless steel has a comparatively high content of iron and nickel, the concentrations and heat treatment combine to produce a material in the non-magnetic austenitic phase. All of the selected materials were non-porous, non-brittle, rigid and were machined to have a good surface finish.

Measurements over a period of two years indicated that their magnetic permeability was stable with time and measurements on all faces indicate that the material is uniform. The uncertainties in the measurement of the relative magnetic permeability of these bars at a magnetic field strength of 10 kA/m are estimated to be:

μ_r	uncertainty (±%)
1.0025 to 1.007	0.01
1.03 to 1.07	0.1
1.2 to 1.4	0.5

The above uncertainties are for a confidence level of at least 95%.

A sample bar from each type of material was selected to determine the resistance to corrosion and the temperature coefficient of permeability. Each bar was placed in an enclosure where the relative humidity was maintained at 95% for 3 weeks. No significant change in the surface condition or of the relative magnetic permeability of any bar was observed.

Each bar was then successively maintained at temperatures of 15, 20 and 25 °C and the relative magnetic permeability measured whilst the bar was at each of these temperatures. No significant difference in the relative magnetic permeability was observed over this temperature range for any bar.

8 CONCLUSIONS

Bars of low magnetic permeability reference materials have been produced with values in three ranges, 1.0025 to 1.007, 1.03 to 1.07 and 1.2 to 1.4. The materials conform to the essential requirements of being uniform, stable with time, and have a low temperature coefficient of permeability, good resistance to surface corrosion under laboratory conditions, they are non-porous, non-brittle, rigid and have a good surface finish.

The dimensions of the bars enable both an absolute determination of their relative magnetic permeability (and hence the possibility for recalibration at intervals) and usage as reference materials for the calibration of commercial instruments at three fixed points over their range of operation.

Guidance notes on the use of these reference materials have been produced and have been published as NPL Report DES 121[16].

9 REFERENCES

1 *British Standard, BS 5884:1987, Determination of relative permeability of feebly magnetic materials.*

2 *American Iron and Steel Institute, AISI 316:1974, Steel Products Manual No 13, Stainless heat resisting steels, p18 and p51.*

3 *British Standard, BS 2872:1989, Specification for copper and copper alloy forging stock and forgings.*

4 *British Standard, BS 2874:1986, Specification for copper and copper alloy rods and sections (other than forging stocks).*

5 *Naval Engineering Standards, NES 834:1991, Requirements for aluminium silicon bronze.*

6 *International Standards Organization, ISO 428:1983, Wrought copper aluminium alloys chemical composition and forms of wrought products.*

7 *Naval Engineering Standards, NES 883:1988, Requirements for nickel aluminium bronze.*

8 *International Standards Organization, ISO 426:1983, Wrought copper zinc alloys - chemical composition and forms of wrought products.*

9 *Bamford C R. A study of the magnetic properties of iron in relation to its colouring action in glass. Physics and Chemistry of Glasses, Vol 1, No 5, October 1960.*

10 *British Standard, BS 2870:1980, Specification for rolled copper and copper alloys: sheet, strip and foil.*

11 *British Standard, BS 2873:1969, Specification for copper and copper alloys: wire.*

12 *British Standard, BS 1400:1985, Specification for copper alloy ingots and copper alloy and high conductivity castings.*

13 *British Standard, BS 970:Part 1:1991, General inspection and testing procedures and specific requirements for carbon, carbon manganese, alloy and stainless steels.*

14 *British Standard, BS 3100:1991, Specification for steel castings for general engineering purposes.*

15 *British Standard, BS 1504:1976(1984), Specification for steel castings for pressure purposes.*

16 *Ager, C.I. and Drake, A.E., Guidance notes for use with NPL low permeability reference materials, NPL Report DES 121, August 1993, National Physical Laboratory.*

APPENDIX 1

Materials for feebly magnetic reference specimens are detailed in table 2.

Table 2. Materials for feebly magnetic reference specimens

Description	British Standard		Relative magnetic permeability	Approximate composition
60/40 brass duplex phase, hot-working alloy	BS 2874[4]	CZ109	1.005	60 Cu, 40 Zn
5% Tin phosphor bronze, single phase cold working alloy	BS 2870[10] BS 2873[11] BS 2874[4]	PB102 PB102 PB102	1.005	95 Cu,5 Sn
Cast phosphor bronze	BS 1400[12]	PB1	1.005	90 Cu,10 Sn
Nickel-iron-chromium non-heat-treatable alloy	-	-	1.005	40 Ni,21 Cr,3 Mo, 1.75 Cu,bal.Fe
Austenitic flake graphite cast iron	-	-	1.03-1.05	11 Ni,6 Mn,bal.Fe
Austenitic stainless steel, fully softened	BS 970:Pt 1[13]	316S33	1.05	18 Cr,10 Ni,3 Mo, bal.Fe
Austenitic stainless steel, stabilized	BS 970:Pt 1[13]	320S31	1.05	18 Cr,10 Ni,3 Mo, 0.5 Ti,bal Fe
9% Aluminium bronze	-	-	1.08-1.18	9 Al,4 Fe+Ni, bal.Cu
Nickel aluminium bronze	BS 2872[3]	CA104	1.18-1.50	10 Al,5 Fe,5 Ni, bal.Cu
Cast austenitic stainless steel	BS 3100[14] BS 1504[15]	317C16 304C15	1.5-2.5 1.5-2.5	19 Cr,10 Ni,3.5 Mo bal.Fe 19 Cr,8 Ni,bal.Fe
Copper manganese aluminium alloy as cast	BS 1400[12]	CMA1	2.2-15	75 Cu,12 Mn,8 Al, 3 Fe,2 Ni

Note: This Appendix is reproduced by permission of the British Standards Institution from BS 5884[1].