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Absolute magnetic moment measurements of nickel spheres

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The preparation and measurement of nickel spheres for use in the calibration of magnetometers are described. The absolute value of the magnetic moment of a set of these spheres near room temperature was measured using the Faraday method. The variations of moment for temperatures near room temperature and for applied fields between 0.3 and 5 T were also determined.
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I. INTRODUCTION

Because of its well characterized saturation magnetization and relative chemical inertness, pure nickel is often used as a calibration sample for magnetometers. This has the advantage of simplicity but, for precise interlaboratory comparisons, it has the problem that the saturation behavior of Ni depends not only on purity, but also on thermo-mechanical history and geometry. Further, the response of most magnetometers will depend to some extent on the geometry of the sample being measured. From this point of view, an advantageous shape for a calibration sample is a sphere because the magnetic field perturbation external to a uniformly magnetized sphere is equivalent to that from a point dipole.[1] Once calibrated with a point dipole, corrections can be estimated for other geometries.[2] In addition, the demagnetization factor for a sphere is known exactly, and a polycrystalline sphere may be mounted in any orientation. We briefly describe here the preparation and absolute calibration of a set of Ni spheres for use as a Standard Reference Material (SRM).

II. EXPERIMENT

Nickel spheres were prepared from annealed Ni wire with a purity of 99.999. The wire was first formed into balls 3.0 mm in diameter, ultrasonically cleaned in acetone and alcohol, and then annealed in dry hydrogen at 1000 °C for 1 h. A lot of 1000 was selected and ground into spheres with diameters of 2.382 mm (3/32 in.), ultrasonically cleaned in alcohol and acetone, and then given a final anneal at 950 °C in dry hydrogen for 2 h. A micrograph of one of the annealed spheres is shown in Fig. 1. Due to the greater amount of cold work at the surface, annealing gave more grain growth near the surface than near the center. The average grain size is approximately 100 μm. Twenty annealed balls were selected at random and their masses measured. The average mass was 63.16 mg with a standard deviation of 0.02 mg. A mass variation of 0.02 mg corresponds to a diameter variation of 0.24 μm.

A magnetometer was assembled to measure the absolute value of the magnetic moment in a magnetic field. The components of this magnetometer included (1) a 250 mm pole diam, variable gap electromagnet. The poles were shimmed to give the greatest field uniformity at the center of the gap at a field of 398 kA/m (5000 Oe). The field uniformity was better than 1 ppm over a 5 cm diam sphere at the center of the gap; (2) a digital recording balance calibrated with a set of mass standards. The balance was covered with a temperature controlled magnetic shield fabricated from soft iron; (3) a glass Dewar to enclose the sample and a gold chain hanging down to which a quartz sample bucket was attached. The Dewar was given a thin gold plating to eliminate static electricity problems. A temperature controlled copper shield using a calibrated Pt resistance thermometer surrounded the sample chamber; (4) a laser calibrated motion control stage capable of reproducing position to better than 1 μm; (5) a working Hall effect teslameter calibrated using a nuclear magnetic resonator (NMR) Teslameter; (6) a set of gradient strips, similar to those described by Spal[3] were used to produce a magnetic field gradient. The strips were held in a fixture which also held the teslameter and helped maintain a homogeneous field by holding the pole caps parallel. The current through the strips was monitored by measuring the voltage drop across a standard resistor in parallel with the strips; (7) a personal computer was used to control the in-

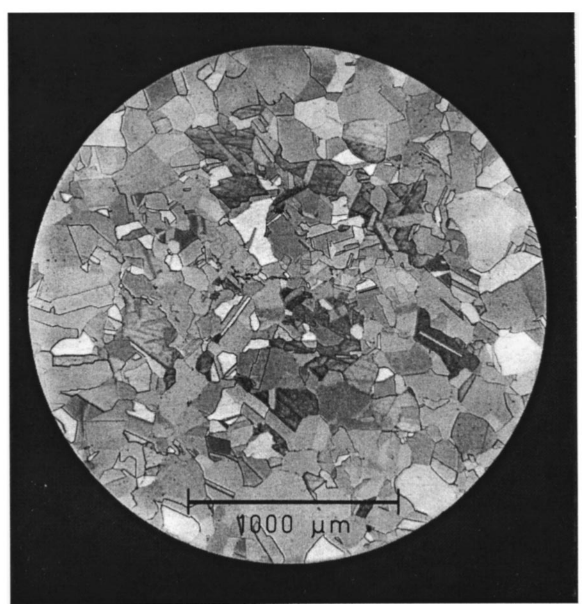

FIG. 1. Micrograph of a typical Ni sphere after final anneal at 950 °C for 2 h.

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struments and to store and analyze the data. The entire system is similar to that described by Candela and Mundy.[4]

To measure a moment, the sample is placed in a quartz bucket attached to the balance by a gold chain. The sample is then lowered to the precise position where the value of the field gradient has been calibrated. The desired field, H, is applied and the moment, m, calculated from

$$m=(W-W_0)\frac{g}{KI}, \tag{1}$$

where W is the measured weight when a current I is flowing through the gradient strips, W_0 is the measured weight when a current I is flowing through the gradient strips but with no sample in the quartz bucket, K is a calibration constant in units of field gradient per unit of current, and g is the acceleration of gravity [the value of g at the National Institute of Standards and Technology (NIST) site is $9.80103\,\mathrm{m\,s^{-2}}$]. Each value of W is actually $(W_+ + W_-)/2$, where W_+ is the increased weight with the current flowing in the positive direction, and W_-, is the decreased weight with the current flowing in the negative direction. For our system, and for samples with a moment greater than about $0.1\,\mathrm{mA\,m^2}$ (0.1 emu), the dominating source of error in the determination of m is the error in K, which is almost an order of magnitude greater than the other errors.

The value of K was determined by three methods: direct measurement using a teslameter, the Thorpe-Senftle method,[4,5] and an iron sphere method. The iron sphere method uses the fact that, well below saturation, the magnetization of a high permeability sphere is determined by the demagnetizing factor. In each method, a K value is determined by averaging the value of K determined with the current flowing in the positive and negative directions. The average values obtained for K at a current of 50–80 A by the three methods were $0.1195\,\mathrm{cm^{-2}}$ by the direct method, $0.1196\,\mathrm{cm^{-2}}$ by the iron sphere method, and $0.1194\,\mathrm{cm^{-2}}$ by the Thorpe-Senftle method. Statistical analysis[6] of these determinations of K, along with the measurements on the Ni spheres, was used to determine the moment value and the expanded uncertainty for the set of 1000 spheres.

III. RESULTS

For the set of annealed Ni spheres investigated here, the moment, at 298 K in an applied field of 398 kA/m (5000 Oe) was found to be $3.47\pm0.01\,\mathrm{mA\,m^2}$ (3.47 ± 0.01 emu). The uncertainty given is the expanded uncertainty for a coverage factor at the 95% level of confidence. The stated emu value corresponds to a specific magnetization of $54.97\,\mathrm{mA\,m^2\,g^{-1}}$ (54.97 emu/g). This set was compared with a set of Ni spheres prepared previously[7] at NIST which gave a specific magnetization of 55.03 at the same temperature and field when measured in the above described magnetometer, a difference well outside the precision (although not the accuracy) of the two numbers. The difference is attributed to the different thermo-mechanical history of the two sets of spheres.

The variation of the moment, m, with fields between 0.24 MA/m (3000 Oe) and 4 MA/m (50 000 Oe) was measured. The results of the measurement are plotted in Fig. 2. The solid line in Fig. 2 is a plot of $m=3.47[1+0.0026\ln(H/398)]$ where H is the field in kA/m and m is the moment in $\mathrm{mA\,m^2}$. The dotted line is a plot of $m=3.48(1-0.96/H)$, again with m the moment in $\mathrm{mA\,m^2}$ and H the field in kA/m. At fields up to about 800 kA/m (10 000 Oe) both equations describe the data well within $0.01\,\mathrm{mA\,m^2}$. The behavior of Ni at high fields seen for this Ni sphere has been noted previously[8] for pure Ni and attributed to a paramagnetic susceptibility term and a classical rotation term. The 1/H term has been used[7] in the past for NIST SRM 772. The log term used above is empirical and is used only for convenience in fitting the data over an extended range of fields.

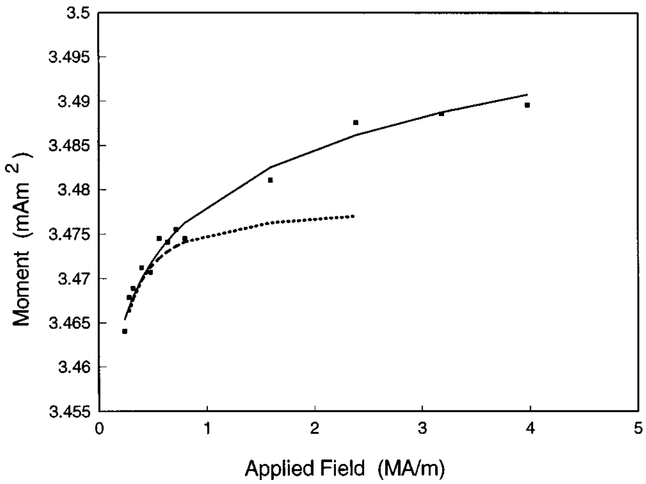

FIG. 2. Plot of the Ni sphere moment measured at 298 K as a function of applied magnetic field strength. The solid line and dotted lines are fits to the data as described in the text. Error bars were not included because statistical errors are much smaller than the systematic error of ±0.01.

The temperature dependence of m was measured between 280 and 320 K. A plot at several fields is shown in Fig. 3. From these data the moment as a function of temperature for $H=398$ kA/m and T between 280 and 320 K is $m=3.47[1+0.00047(T\text{-}298)]$.

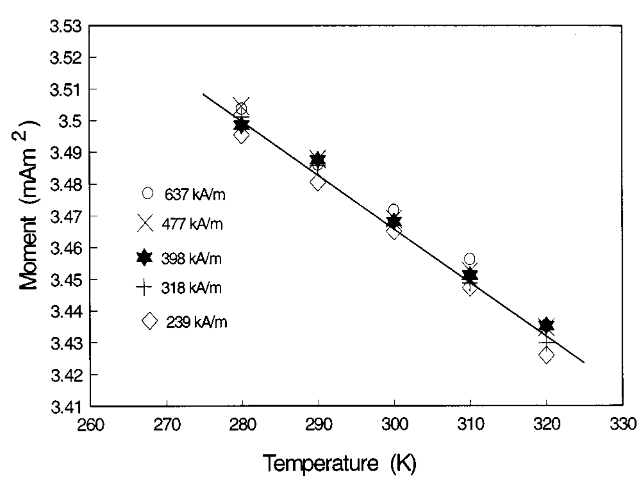

FIG. 3. Plot of the Ni sphere moment as a function of temperature for several applied magnetic field strengths.

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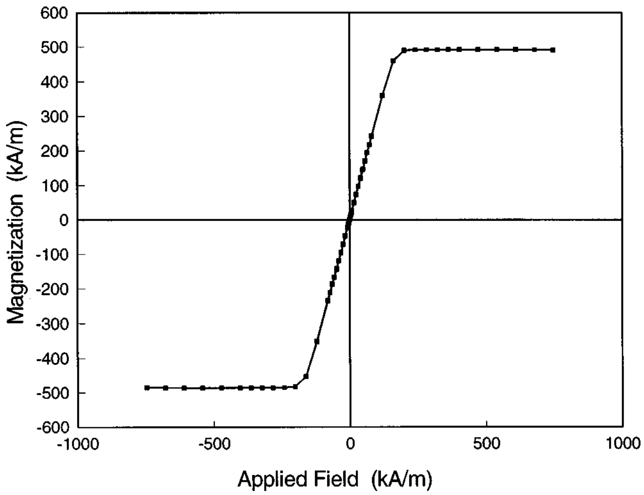

FIG. 4. Hysteresis loop at 298 K of a Ni sphere sample measured in a VSM.

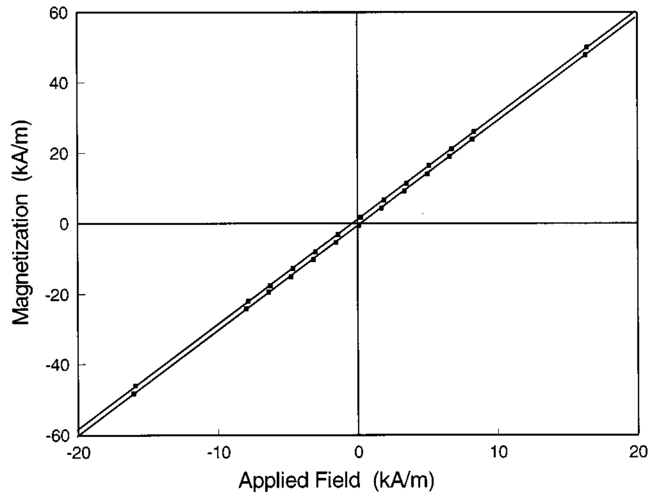

FIG. 5. Central part of the hysteresis loop shown in Fig. 4.

After final grinding and before any annealing, the coercive field of the Ni spheres was determined to be approximately 1400 A/m (18 Oe). After the final anneal at 950 °C for 2 h, the coercive field was reduced to approximately 440 A/m (5.5 Oe). Annealing at 1000 °C did not further reduce the coercive field.

Figure 4 shows a hysteresis loop taken at 298 K with a vibrating sample magnetometer (VSM) and plotted as magnetization in kA/m versus the applied field in kA/m. Figure 5 is the central region of the loop. The slope of the line in this central region is dominated by the demagnetizing factor. For a perfect sphere with high permeability, the slope should be slightly less than 3. The slope determined for the lines of Fig. 5 was 2.98. Possible factors influencing the deviation of this slope from the value 3 include (1) finite value of the permeability, (2) nonuniform magnetization of the sphere at low applied fields, (3) errors at low field in the calibration and linearity of the gaussmeter used, (4) inhomogeneity of the magnetic field causing the field at the sample to be slightly different from the field at the gaussmeter probe, and (5) error in the volume used to convert the moment to a magnetization (the volume was computed from the mass of the sample divided by a density of $8914\,\mathrm{kg\,m^{-3}}$).

The above described measurements show that the set of Ni spheres studied here could be useful for calibrating magnetic measuring instruments such as vibrating sample magnetometers. A number of these Ni spheres has been issued[9] by NIST as SRM772a. The calibration is limited to a small range of temperatures near room temperature. Further measurements to extend their usefulness to lower temperatures is desirable.

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[9] For information on the availability of Standard Reference Materials, contact the NIST SRM Program Office, 100 Bureau Drive, Gaithersburg, MD 20899-2322; tel: 301-975-6776.

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