Autocalibrating quasistatic *M-H* hysteresis loop tracer with negligible drift

V. Franco
Departamento de Física de la Materia Condensada, Instituto de Ciencia de Materiales, C.S.I.C.-Universidad de Sevilla. Apdo 1065. 41080 Sevilla, Spain

J. Ramos-Martos
Centro Nacional de Microelectrónica. Avda. Reina Mercedes s/n. 41012 Sevilla, Spain

A. Conde
Departamento de Física de la Materia Condensada, Instituto de Ciencia de Materiales, C.S.I.C.-Universidad de Sevilla. Apdo 1065. 41080 Sevilla, Spain

(Received 19 March 1996; accepted for publication 23 September 1996)

A computer-controlled system for measuring *M-H* hysteresis loops of soft magnetic materials is described. It overcomes many of the problems associated with these kinds of measurements of low coercivity materials. The combination of ''low drift'' hardware and measurement procedure makes software correction unnecessary. At every field step, the homemade integrator is reset in order to minimize the drift and to provide a way of separating the sample contribution to the signal from the field contribution and, consequently, to allow *M-H* plots. Furthermore, to avoid the residual drift of the hardware, the method of ''two-way measurement'' is applied, giving drifts of 0.01% per hour. Results obtained for a FINEMET alloy film annealed at different temperatures are presented, illustrating the ability of the system to measure soft magnetic materials. © *1996 American Institute of Physics.* [S0034-6748(96)04112-3]

I. INTRODUCTION

Although different systems for measuring low-frequency hysteresis loops have been reported, there still remain some problems and difficulties; mainly, drift suppression and *H*-field compensation. The former is usually treated in various ways: The systems that employ commercial fluxmeters[1,2] are limited to the drift controls provided by the manufacturer and, as we have verified, they are not sufficient in a medium-resolution loop. In fact, the system's software has to correct the drift by assuming a constant drift rate. However, this is only a first approximation to the problem because drift evolves with time and, in some cases, it is necessary to consider higher-order terms.

The other kinds of systems[3–9] employ homemade integrators instead of fluxmeters. They choose operational amplifiers in order to obtain the least drift and reset the integrator between each H step to minimize the integrating time and, consequently, the drift per step. However, the final accumulation of signals per step makes the total drift important. To solve this, they use a nonlinear $H(t)$ function which is slower in the steepest part of the loop, obtaining a M-dependent resolution and shorter integration time. But this is not enough and the above-mentioned software correction has to be employed.

Our solution to this problem is not only concerned with low drift hardware but consists of a measurement procedure that enables the acquisition of drift values in every H-field step, making software correction unnecessary even in long total integration times. On the other hand, H-field compensation is achieved without a compensation coil by a method of signal deconvolution. This overcomes the problem of equality between the signals coming from pick-up and compensation coils in the absence of the sample, usually accomplished by a balance network or by a variable position of the compensation coil.

The most important characteristics of the system presented here are the following: (i) it requires only standard laboratory instruments, making it a very low-cost system; (ii) it has high sensitivity and a selectable integrating constant, making it capable of measuring low coercivity metallic glasses, such as FINEMET alloys; (iii) the combination of a ''zero-drift'' adjust control and measurement protocol makes the drift negligible (0.01% per hour); (iv) signal deconvolution procedure allows M-H loops presentation without a compensation coil; (v) the measurement procedure makes this system autocalibrating; (vi) the system is capable of measuring M-H loops, first magnetization curves, permeabilities, Barkhausen jumps studies[10] and magnetic disaccommodation (depending on the voltmeter's speed).

II. EXPERIMENTAL ARRANGEMENT

The system's hardware consists of a PC computer including an IEEE-488 bus, a digital output, a voltmeter, a digital-to-analog converter, a bipolar power supply, a homemade integrator, a solenoid, and pick-up coil (Fig. 1).

The solenoid is fed by a bipolar power supply with an output current linearly proportional to an input voltage provided by the digital-to-analog converter. This produces an H-field which provokes a change of magnetization in the sample and, consequently a magnetic flux variation across the pick-up coil. This signal is amplified and integrated by our homemade integrator (described below) controlled by the digital output of the computer. Integrated data are introduced into the computer by the IEEE-488 bus of the voltmeter.

The front-end amplifier is a custom-built circuit which contains an integrator with a selectable integration constant, a second-order low-pass filter with a cutoff frequency of

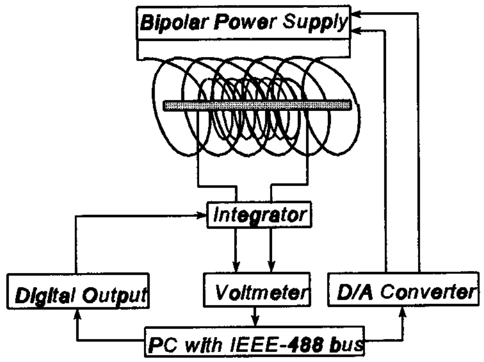

FIG. 1. Block diagram of system's hardware.

5 Hz, and a Track-and-Hold amplifier (Fig. 2). One single-digital input controls the operation of the circuit.

The purpose of the low-pass filter is twofold: first, it attenuates, by at least 40 dB, the 50 Hz interference which is picked up by the coil from nearby instruments and from residual AC ripple in the output of the current source; and second, it reduces the equivalent noise bandwidth and consequently the rms value of the noise sampled by the T&H amplifier.

The pick-up coil is connected between V_{in} and ground. At the beginning of each measurement the *Reset* input is high, so that the integrating capacitor is fully discharged and the output is on *Hold* mode. To start a new measurement, *Reset* is switched to low, leaving the integrator free to operate, and after a short delay D, the T&H amplifier goes on *Track* mode; the D/A converter that controls the current source is then fed a new value that produces a current step, and as a consequence, a voltage at the input of the circuit proportional to the rate of change of the magnetic flux. Steady current is reached approximately 20 ms later; the *Reset* input is switched to high after 150 ms to allow for stabilization of the filter output. This first puts the output on *Hold* mode, and after a delay D, reset the integrator. The voltmeter then takes a reading from the held output, completing one measurement cycle.

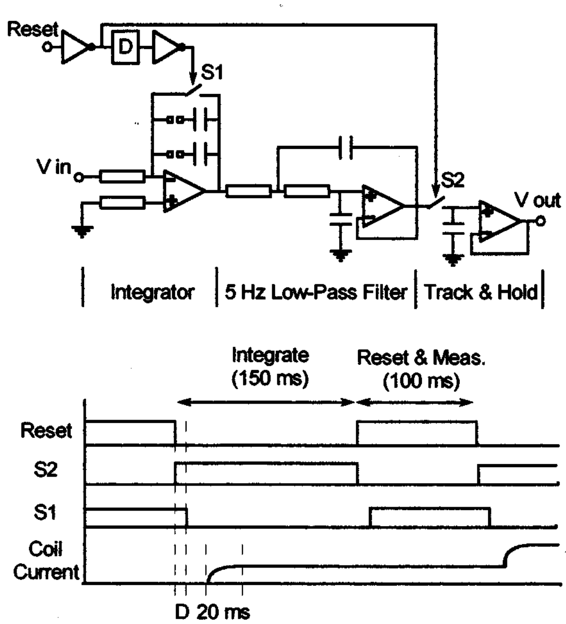

FIG. 2. Scheme of integrator's circuit and control signals.

All common-mode systematic errors present at the output, such as integrator drift and clock-feedthrough of the switches in the integrator and T&H amplifier, are eliminated by the ''two-way'' measurement technique described below, in which the value of the magnetic flux step is obtained subtracting the measured values in two consecutive cycles with current steps of the same amplitude and opposite signs. The value of the integrator drift can be adjusted to less than 5 mV/s, and since the uncertainty in the integration time is less than 1 ms, and after common-mode cancellation, the output error due to drift will be less than 5 μV.

The main causes for random measurement errors are amplifier and current source noise. The output error due to $1/f$ (flicker) noise in the input amplifier has been measured with the input short circuited and it is less than 100 μV. Currently, accuracy of the measurements is limited by flicker noise in the output of the current source, which induces an output error of approximately 500 μV.

Care has to be taken when using controlled-current supplies to excite the solenoid. Normally these supplies have a feedback loop where the measured variable is the output current, converted to voltage by a precision resistor, and the control variable is the output voltage. If the voltage error amplifier is fast, inductive loads may create instability at the output of the power supply due to the lag introduced in the feedback path. For inductance load values above a certain limit, this will produce an oscillation at the supply output. This effect can be corrected by modification of the time-constant of the error amplifier, or in a simpler way, placing a compensating capacitor in parallel with the solenoid. The capacitor introduces a phase-lead correction in the transfer function from the output voltage to output current that compensates the inductance phase lag.

III. MEASUREMENT PROCEDURE AND SOFTWARE DESCRIPTION

As mentioned above, to obtain low drift measurements the hardware used is not only important, but the way you employ it is essential as well. The block diagram of the measurement procedure is presented in Fig. 3. At program start, parameters such as the section of the sample, waveform of the exciting signal (and, consequently, number of points per cycle and time duration) and output files are asked for.

The one-way measurement is only concerned with hardware requirements and has already been described. The principle of two-way measurement is presented in Fig. 4 and its objective is to remove the hardware's residual drift by measuring it at every field step. Once we have defined a field increment ΔH, we apply it, covering curve a and obtaining a signal composed of: integral of flux variation due to $\Delta H +$ integral of flux variation due to change of magnetization of the sample+residual drift. At this point, we apply ΔH again with the opposite sign and the system will go over b. The magnitude of this signal will be different from the previous one because ΔM is different, but the drift will be the

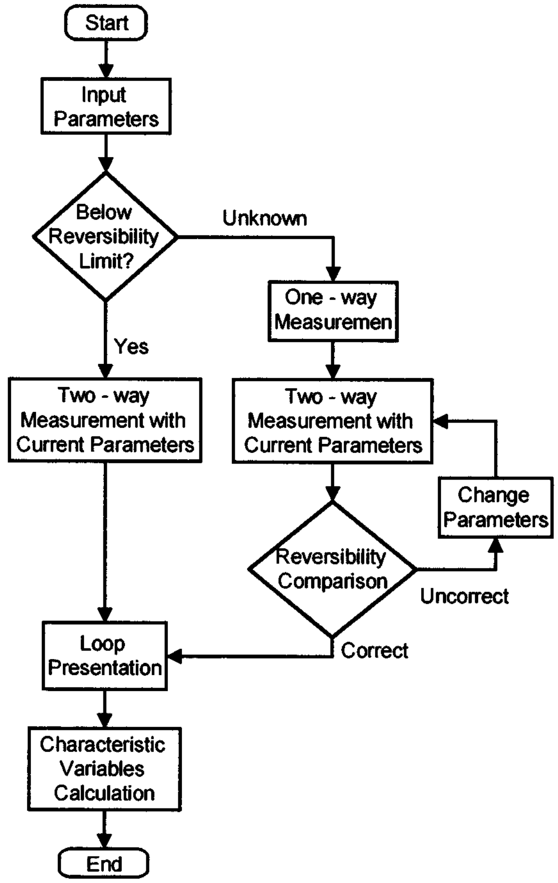

FIG. 3. Flux diagram of measurement procedure.

same (as these measurements are only a few milliseconds apart and the evolution of drift with time is slow). Finally, we go over c. If ΔH is small enough, the cycle bc will be reversible and the drift at this ''measurement block'' can be obtained by just adding the two measured signals and dividing by two. To obtain the ''real signal'' (i.e., the sum of the sample and field contribution) over a, we must only subtract the drift from the measured signal. Furthermore, we can obtain the reversible permeability from loop bc. When two-way measurement is applied, the system speed is slowed down to 750 ms per point.

In the case of an unknown sample type, we must check if the field steps we have defined so far are reversible in order to apply the two-way measurement. This is accomplished by making two measurements, one of both types, and comparing them. In fact, these two measurements cannot be equal because one of them (one-way measurement) will have more drift than the other. However, this must be the only difference between them. This can be assured by taking only a branch of each signal, fitting them to a function and observing that they have the same parameters but different offsets (due to the drift). Drift effect can also be observed on different offsets on both branches of the same signal. The fitting function can be a series of Gaussians but it is irrelevant, as the only thing we want is to compare the two signals and no information will be extracted from the parameters.

To obtain the M-H loop we must remember that the signal measured is proportional to the emf induced on the pick-up coil due to magnetic flux variations during the integration time.

$$\varepsilon = -\frac{\partial \phi}{\partial t} = -\frac{\partial}{\partial t}\int_{S_{\mathrm{coil}}} B\,dS$$
$$= -\mu_0 \frac{\partial}{\partial t}\left(\int_{S_{\mathrm{coil}}} H\,dS + \int_{S_{\mathrm{sample}}} M\,dS \right), \qquad (1)$$

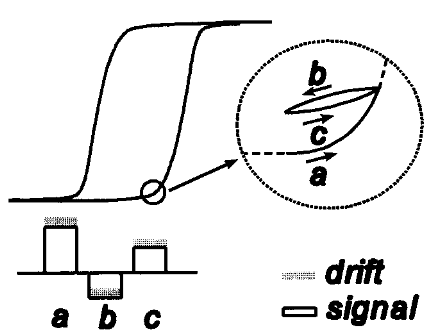

FIG. 4. Principle of ''two-way'' measurement.

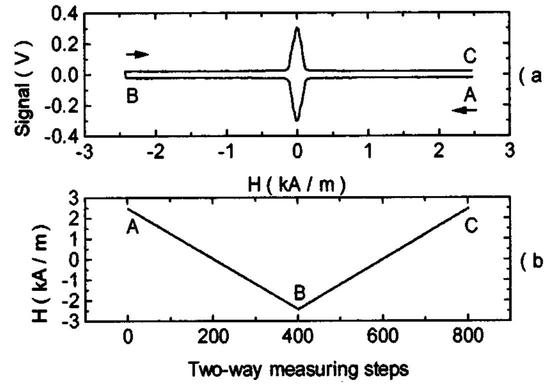

FIG. 5. (a) A typical integrator's output signal, (b) applied field vs time.

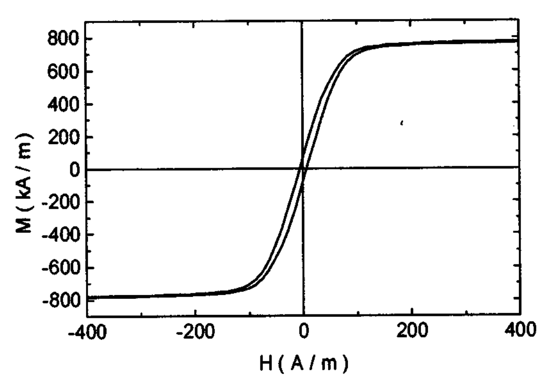

FIG. 6. M-H loop of a FINEMET alloy annealed for 1 h at 825 K.

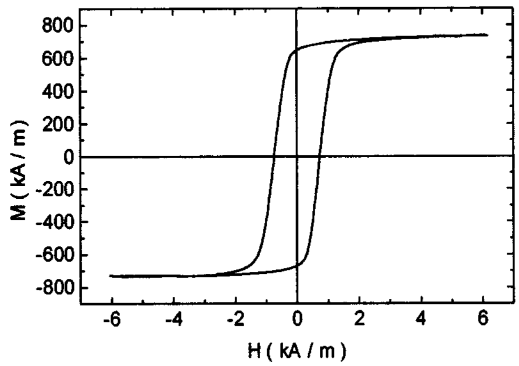

FIG. 7. M-H loop of a FINEMET alloy annealed for one hour at 875 K.

$$V_{\mathrm{meas}} = \kappa \cdot \int_0^{\tau} \varepsilon\, dt = \kappa_1 \cdot \Delta H + \kappa_2 \cdot \Delta M$$

$$= \kappa_1 \cdot \Delta H + \kappa_1 \frac{S_{\mathrm{sample}}}{S_{\mathrm{coil}}} \Delta M. \quad (2)$$

A typical output signal is shown in Fig. 5(a) produced by a piecewise linear $H(t)$ function [Fig. 5(b)]. It can be seen that the initial part of the signal is only due to H-field variations. As we decrease the field, variations of M produce a decrease in measured voltage and, after them, the only signal is again H variations. When the minimum value of H is reached and the field begins to increase, a change in the sign of the signal can be seen. From the horizontal parts of the signal we can obtain κ_1, and as the applied field is known, the corresponding part of M can be obtained easily and the use of a compensation coil is not necessary, nor is calibration necessary because all the values required for this operation are the coil area, the sample section (which are known) and the constant κ_1 which is obtained from the measurement itself. This makes this system autocalibrating.

$$M(H_t) = \sum_{n=1}^{n_t} \Delta M_n = \sum_{n=1}^{n_t} \frac{V_{\mathrm{meas}}^{(n)} - \kappa_1 \cdot \Delta H}{\kappa_1 S_{\mathrm{sample}} / S_{\mathrm{coil}}}. \quad (3)$$

This method of obtaining $\kappa_1$from the horizontal part of the signal can only be applied if the sample is well saturated. If it is not, all the signal will have a non null slope, indicating that the sample is on a minor loop. In this case, H field contribution is measured by extracting the sample from the coils without changing their positions and measuring the signal provoked by the field step.

In the case of another $H(t)$ function, the procedure should be the same, except that ΔH is not constant with time. However, the usual motivations to apply a nonlinear $H(t)$ function is to shorten integration time due to drift, but as mentioned above, the system being described has a typical drift of 0.01% per hour, which is negligible for our purposes. Once the loop is obtained in this way, the calculation of the parameters which describe it is trivial and the saturation magnetization, remanence, coercive field and core losses are displayed when the analysis software is run.

IV. ILLUSTRATIVE RESULTS

This system has been employed for studying magnetic properties of FINEMET films annealed at different temperatures. Samples of composition $Fe_{77}Si_{10}B_9Cu_1Nb_3$, 100 mm long, 9.4 mm wide, and 25 μm thick have been isochronally annealed at different temperatures. The primary coil used has 330 turns and the secondary 750.

It is already known that appropriate heat treatment of some amorphous alloys (among them, the FINEMET alloys) makes possible the creation of nanocrystalline materials with coexistence of different magnetic phases.[11,12] The variation of size, volume fraction, and composition of the crystallites embedded in the remaining amorphous matrix provokes changes in the magnetic properties of the material.

The $M(H)$ loop of the sample annealed for one hour at 825 K is shown on Fig. 6. It is evident that the sample is softer ($H_c = 6$ A/m) than the amorphous one ($H_c = 17$ A/m) due to nanocrystallization of Fe(Si). However, when the sample is annealed for 1 h at 875 K, the appearance of boride phases,[13] with bigger magnetocrystalline anisotropy and short magnetic exchange length, makes the coercive field increase abruptly (see Fig. 7).

ACKNOWLEDGMENTS

This work was supported by the CICYT of the Spanish Government (Project MAT95-961-CO2-01) and the PAI of the Regional Government of Andalucía. V. Franco wishes to thank the ''Fundación Cámara'' of the University of Sevilla for the fellowship.

[1] T. Kulik, H. T. Savage, and A. Hernando, J. Appl. Phys. **73**, 6855 (1993).
[2] D. C. Jiles, J. Appl. Phys. **63**, 3946 (1988).
[3] P. T. Squire, S. M. Sheard, C. H. Carter, and M. R. J. Gibbs, J. Phys. E **21**, 1167 (1988).
[4] R. Cammarano, R. Street, P. G. McCormick, and M. E. Evans, J. Appl. Phys. **69**, 5100 (1991).
[5] T. H. O'Dell, IEEE Trans. Magn. **MAG-17**, 3364 (1981).
[6] D. C. Gall and J. D. Watson, J. Sci. Instrum. **33**, 265 (1956).
[7] J. A. Baldwin,Jr, Rev. Sci. Instrum. **41**, 468 (1970).
[8] H. Fay, Rev. Sci. Instrum. **43**, 1274 (1972).
[9] P. Mazzetti and P. Soardo, Rev. Sci. Instrum. **37**, 548 (1966).
[10] R. M. Bozorth, *Ferromagnetism* (Van Nostrand, Princeton, NJ, 1951).
[11] C. F. Conde and A. Conde, Nanostruct. Mater. **6**, 457 (1995).
[12] C. F. Conde, M. Millán, and A. Conde, J. Magn. Magn. Mater. **138**, 314 (1994).
[13] T. Kulik and A. Hernando, J. Magn. Magn. Mater. **138**, 270 (1994).