ISSN 1330–0008
CODEN FIZAE4

MULTIPURPOSE MEASURING DEVICE BASED ON AC SUSCEPTOMETER

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Dedicated to Professor Boran Leontić on the occasion of his 70th birthday

Received 30 November 1999; revised manuscript received 1 February 2000
Accepted 28 February 2000

The measuring device based on an AC susceptometer is described and some advantages of particular design are given. The measurements of resistance and hysteresis with the device are described. Some results of AC susceptibility, resistance and hysteresis measurement are shown.

PACS number: 07.55.+x UDC 537.621.4

Keywords: AC susceptometer, new design, measurement of AC susceptibility, resistance and hysteresis

1. Introduction

The AC susceptibility measurement, as an experimental technique, is very useful in studying the magnetic properties of materials, especially of the magnetic phase transitions where the response of magnetic moments in low field is of great importance. Its applications have rapidly increased with the spreading of investigations of high temperature superconductors because a number of parameters can be obtained from AC susceptibility measurements within the critical state model of type II superconductors [1]. Here we discuss some common problems of the method and describe the construction of a very sensitive AC susceptometer together with its modifications that permit the measurement of dynamic hysteresis and resistance.

The method is based on the fact that mutual inductance of two coils (and therefore the induced voltage) changes if a magnetic sample is placed inside them. The AC susceptometer generally consists of the primary coil and two coaxially placed secondary (pick-up) coils which are connected in opposition (their induced voltages subtract) so that without a sample, their joint induced voltage should be zero. If a (magnetic) sample is placed in one of the secondary coils, it causes the disbalance voltages as the consequence of the redistribution of flux lines over the cross-section of that secondary coil. This voltage is related to the susceptibility of the sample [2]. Ideally, if the sample is very long so that the influence of edges on the distribution of flux lines can be neglected, the induced voltage is

$$v(t) = -\mu_0 N_s A_s \mathrm{d}M/\mathrm{d}t\,.$$

Here N_s is the number of turns of either secondary coil and A_s is the cross-sectional area of the sample. The change of the magnetizing field induces the change of the magnetization. If the magnetizing field is harmonic ($H = H_o \exp(\mathrm{i}\omega t)$), we have

$$v(t) = -\mathrm{i}\omega\mu_0 N_s A_s \frac{\mathrm{d}M}{\mathrm{d}H}\,\mathrm{e}^{\mathrm{i}\omega t}\,,$$

i.e., the induced voltage is proportional to the AC susceptibility, $\chi_{\mathrm{AC}} = \mathrm{d}M/\mathrm{d}H$. In reality, the relation between the AC susceptibility and the induced voltage is not so straightforward. It depends on the shape of the sample, its dimensions and inhomogeneities (demagnetizing effect), the dimensions of the secondary coils, etc. Finding out the relation between the induced voltage and AC susceptibility (calibration of the susceptometer) can be a very hard task [3]. But the proportionality between the induced voltage and AC permits the derivation of many valuable data.

2. Design of the susceptometer

The main parts of an AC susceptometer include the coil system and the sample support (commonly called the inset) that are generally home-made, and several other instruments. The design of the inset is determined by the main aim of the application of the susceptometer and a number of other parameters - the temperature, field and frequency ranges, shape and size of samples etc. Here we give a brief review of the questions that have to be considered when the inset is designed together with our solutions of the problems.

The susceptometers are most often used to measure the susceptibility in a wide temperature range. In such devices it is necessary to minimize the temperature dependence of the contributions to the induced voltage of those parts which are close to the sample. That is achieved by the use of a small vacuum chamber for the positioning of the sample, with the coils around the vacuum chamber and immersed in the cryogenic liquid. In devices of this type, the pick-up coil is wider than the sample and, therefore, there is some loss of flux and increase in the induced noise, but more important is that the coils are at constant temperature during the measurement. Since the resistance, R, and the cross-sections of the coils are fixed, the induced voltage can be easily amplified without any distortion.

According to the equation for the induced voltage the sensitivity of an AC susceptometer increases with frequency and field. But there are experimental limits to this way of increasing the sensitivity. The main one is the offset voltage of the secondary coils because the coils can not be made to perfectly match. This offset voltage increases with frequency and field. Consequently, a higher voltage range on voltmeter has to be used which degrades the voltage resolution.

The main idea in the design and construction of our susceptometer was to remove the limit of the resolution arising from the offset voltage. This has been done by adding the third secondary coil to the inset and using it to cancel the offset voltage of the usual two–pick-up–coil setup. Our inset consists of a non-magnetic stainless steel thin-wall tube-like sample chamber, with coil system mounted around it. Each of the three secondary coils is 3 mm long, with approximately 700 turns in 14 layers of the copper wire 0.05 mm in diameter. They are wound on the round teflon former, which fits the outside diameter of the vacuum tube. The primary coil is 20 cm long, 2 cm in diameter and produces the field of 11140 A/m (140 Oe) for the current of 1 A. Two of the secondaries are positioned symmetrically in respect to the center of the primary coil and are 7 cm apart. The voltages induced in these two coils are balanced to 1 part in 10^4 (which corresponds to better than 1/10 of a single turn). The balance of the induced voltages to better than one turn has been achieved by changing slightly the distance between the pick-up coils. Small out-of-phase component of the offset voltage which appears dominant after the in-phase balancing is canceled by a very small capacitor connected in parallel to one of the secondaries. Third secondary coil is placed close to the edge of the primary coil. It is connected to a variable autotransformer which has a resolution of 1 part in 111100 and which has a negligible phase shift between the input and output voltage in a wide frequency range. The importance of the use of this autotransformer is explained later. The primary coil provides the magnetizing fields of up to about 8000 A/m (100 Oe) (with appropriate current sources) and a linearity with frequency up to a few kHz. The whole system is immersed into the cryogenic liquid.

The sample holder is made of a sapphire rod, 3 mm in diameter and 3 cm long. One can easily move it up and down within the sample chamber tube. That is very important for the proper adjustment of the phase of the induced voltages. One half of the sapphire rod is flattened and the sample is glued with silicon grease on that flat surface. Over the rest of the sapphire, a nonmagnetic resistive wire is bifilarly wound which serves as the heater. The thermocouple (AWG 40) is glued on the other side of the flattened surface. Two thermocouples were used: gold-iron/copper for temperatures below 100 K and copper/constantan for temperatures above 77 K. The contribution of the sample holder designed in this way to the induced voltage is minimal and temperature independent. The holder permits the use of rectangular samples with the maximum dimensions of 12mm $\times$ 3mm $\times$ 1mm. The holder is equipped with a four-contact connector for the resistance measurements. Because of a good thermal contact of the holder, sample and thermocouple and fast response of the holder to the change of heating power, it is possible to carry out the measurements in the temperature drift regime which greatly facilitates the measurements.

3. *Experimental assembly*

The AC susceptometer setup can be seen from the block diagram in Fig. 1. The voltage induced in the pick-up coils is amplified by a passive amplifier (isolation transformer). This voltage is compared with the voltage of the variable autotrans-

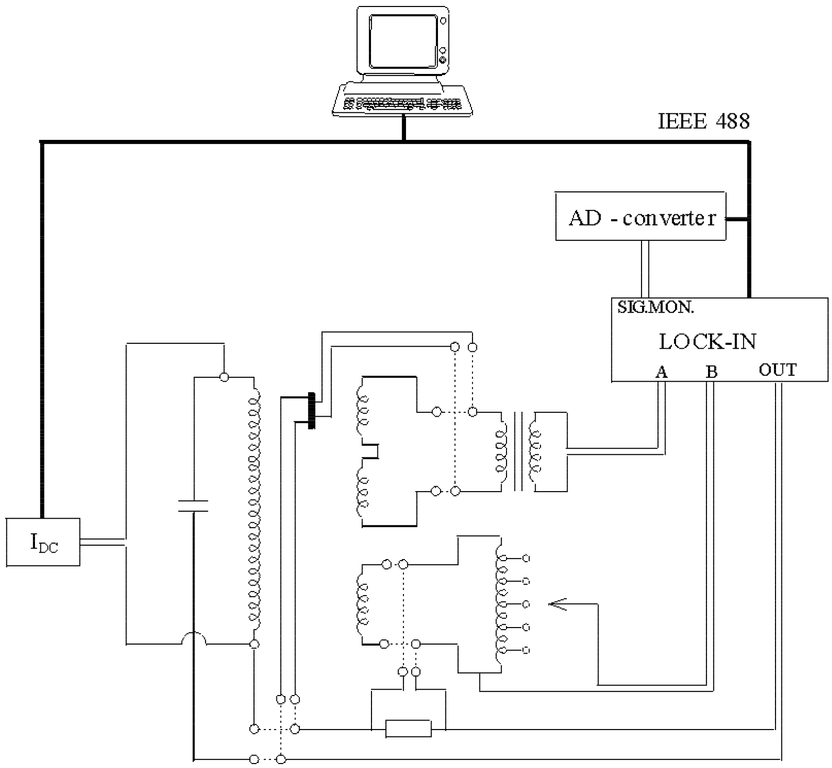

Fig. 1. Block diagram of the AC susceptibility setup. Dotted lines show the optional connections for susceptibility or resistance measurement.

former in a differential preamplifier of the lock-in detector. Without the sample, the part of the voltage of the third secondary coil is adjusted by the autotransformer setting to perfectly cancel the offset voltage of the pick-up coils. After inserting the sample, a disbalance of the voltages is caused by the sample only. Autotransformer can be used not only to balance the offset voltage of empty coils but also to compensate a part of the in-phase component of susceptibility so that the rest of the signal can be measured on a more sensitive scale. In fact, the limit of the sensitivity is determined only by the noise level of the electronics.

The possibility of a partial signal compensation is used in the resistance measurement. It is the standard four-contact method with the isolation transformer (the same as the above) connected to the voltage leads. A standard resistor is connected in series with the sample, and voltage from that resistor feeds the autotransformer. A small condenser can be connected in parallel to the standard resistor for a fine adjustment of the phase. A large part of the sample's voltage can be compensated and its slight changes can be measured.

The above setup can be used to measure the dynamic hysteresis curve by the help of an AD converter. On trigger, the converter samples the signal and puts the measured values in the buffer. When the buffer is full, the data are transferred to the computer by a DMA. This procedure can be quickly repeated, so the averaging of the signal can be done. The voltage accross the resistor in the primary circuit, which is proportional to $H(t)$, is taken first. The voltage from the pick-up coils is proportional to $\mathrm{d}M(t)/\mathrm{d}t$. If the integral of latter is plotted against the former, one obtains the hysteresis curve. In this way the hysteresis measurement can be done in the temperature drift regime and there is no need for temperature stabilization which is extremely time consuming in the usual point by point measurement.

4. Experimental results

To illustrate the performance of our susceptometer, three measurements are presented.

AC susceptibility of a Nb sphere (volume ≈ 1.5 mm^3) on the superconducting transition is shown in the upper part of Fig. 2, and in the lower part of Fig. 2,

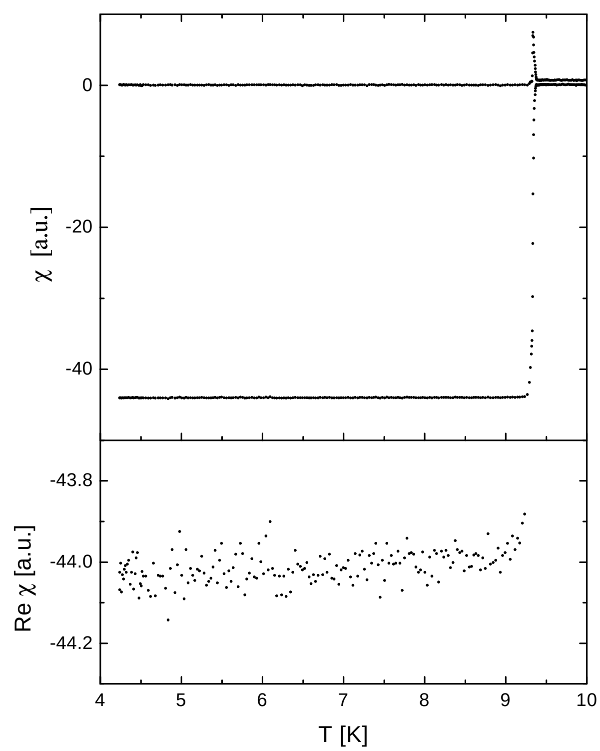

Fig. 2. Up: real and imaginary part of susceptibility on the transition to superconducting state of a Nb sphere. Down: Real part of susceptibility of the superconducting phase on a very expanded scale.

the data in superconducting phase are shown on a very expanded scale. Since the susceptibility is constant in the superconducting phase, the scatter of the points shows directly the resolution of the susceptometer. The resolution is commonly expressed as the equivalent magnetic moment that can be undoubtedly resolved by the apparatus (it is usually given in CGS unit emu). From the relations $\Delta\chi/\chi \sim \Delta v/v$ and $\Delta\chi = \Delta m/VH$ follows

$$\Delta m = \chi HV \frac{\Delta v}{v},$$

where the absolute value of χ is equal to 1 ($1/(4\pi)$ in CGS), V is the volume of sample and H is the magnetizing field. The driving field was 1.20 A/m (15 mOe) at 231 Hz. The voltage resolution Δv is estimated to be less then 100 nV (tick interval in the lower part of Fig. 2 which corresponds to approximately three standard deviations). The relative error of the voltage measurement is $\Delta v/v \approx 100\text{nV}/44\mu\text{V} = 0.0023$. It follows that Δm is approximately 4×10^{-12} Am^2 (4×10^{-9} emu).

The hysteresis measurement was performed on a single, 12 mm long ribbon of amorphous ferromagnet $Fe_{91}Zr_9$ ($T_c \approx 205$ K). The amplitude of the driving field was 450 A/m and the frequency 196 Hz. The time dependence of the field and induced voltage at 82 K are shown in Fig. 3 and the integrated voltage against the field, giving a typical ferromagnetic hysteresis curve, is shown in Fig. 4.

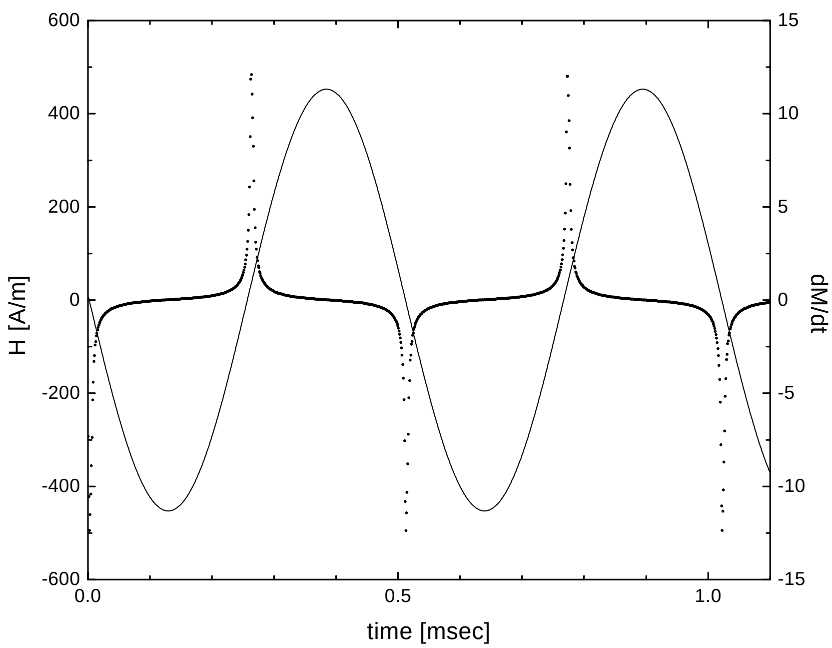

Fig. 3. Time dependence of the field (full line - scale at left) and the derivative of magnetization (dots - scale at right). $T = 82$ K.

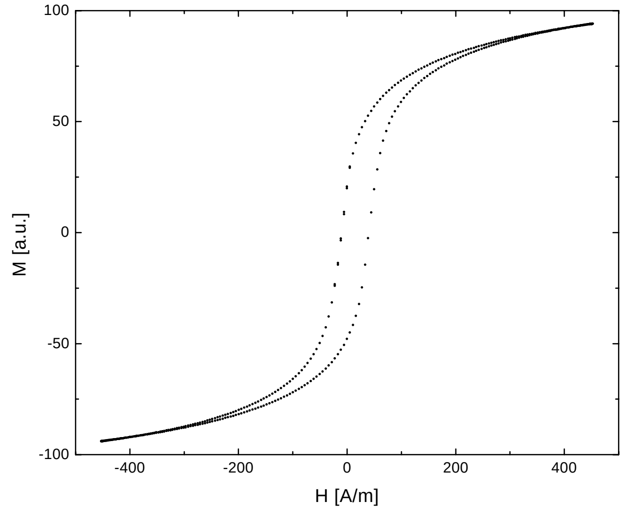

Fig. 4. The hysteresis curve.

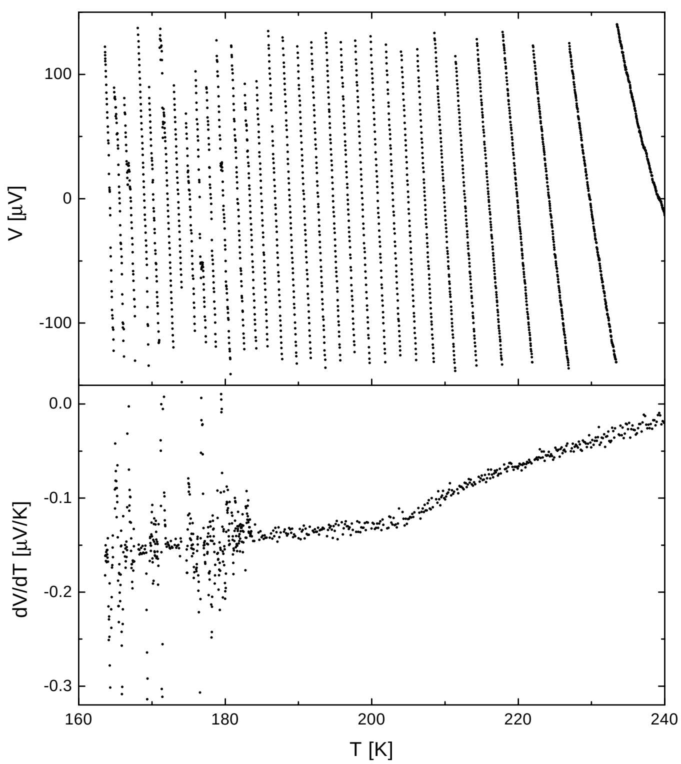

Fig. 5. Up: Scans of the voltage drop accross the voltage contacts. Down: The derivative of the above voltage with temperature.

The resistivity of same sample monotonously decreases with temperature around T_c and has a wide minimum above T_c. The as-measured data of voltage drop accross the voltage contacts are shown in the upper part of Fig. 5. The current of 1 mA was passing through the sample (1 Ohm resistance between the contacts) and the total voltage (proportional to the resistance of the sample) of approximately 1 V (100× transformer setting and 10× lock-in amplification) was compensated by the autotransformer up to the value of 140 μV (saturation value of the 100 μV scale). The change of the voltage with temperature was then recorded down to – 140 μV and then the compensation was corrected again. The voltage changes of less than 1 μV can be recorded, so the total resolution of the resistivity measurement was better than 1 part in 10^6. The derivative of these data is shown in the lower part of Fig. 5. The anomaly at T_c is clearly visible. The great scattering of derivative below 185 K is a consequence of the Hopkinson effect. In fact, the measurement was performed in the DC magnetizing field of 220 A/m. This field is strong enough to shift the Hopkinson contribution far below T_c [4]. Without the magnetic field, the scattering of derivative exists above T_c covering completely the knee of the derivative.

5. *Conclusion*

The method of improvement of sensitivity of an AC susceptometer is presented. The additional advantages of this technique are given. The illustrative examples of susceptibility, hysteresis and resistance on superconducting and ferromagnetic transition are shown. This non-destructive technique is ideal for the combination of the measurements of different properties on the same sample.

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VIŠENAMJENSKI MJERNI UDREĐAJ ZASNOVAN NA AC SUSCEPTOMETRU

Opisujemo mjerni uređaj zasnovan na AC susceptometru i prednosti njegove izvedbe. Opisujemo metode mjerenja otpora i histereze tim uređajem. Prikazujemo ishode nekih mjerenja AC susceptibilnosti, otpora i histereze.