

Fast cycle, wide temperature range a.c. susceptometer

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An a.c. susceptometer of simple design, operating from 4 K to room temperature is described. The sample can be easily replaced and cooled to 4 K in less than 1 h with low helium boil-off. Measuring coils are immersed in the cryogenic bath, so that a serious source of thermal drift voltage is avoided.

Keywords: a.c. susceptibility; cryostats; design considerations

The a.c. susceptibility technique has been widely used in recent years to characterize new superconducting samples, probably because it has the advantage that no electrical contacts are required on the sample. Bad quality samples (multiphased or weakly linked) can be easily detected¹ and good samples can be selected for future studies, which consist mainly of critical current², reversibility line³ or flux dynamics⁴ determinations.

We describe below a simple cryostat based on a previous design⁵ of a low dead time insertion cryostat that minimizes warm-up and cool-down times, and that maintains primary and secondary measuring coils at constant temperature after the initial cooldown, reducing the temperature drift signal. The dimensions allow the use of storage Dewars or superconductor magnets with only 15 mm diameter clearance.

We shall first briefly describe the technique, the cryostat and its operation, and then show, as an application, the determination of the resistivity of a normal metal over wide range of temperature using the skin depth parameter.

Description and operation

A.c. susceptibility

The measurement of a.c. magnetic susceptibility by mutual inductance techniques is well documented¹. In the present case, the mutual inductance consists of two coaxial secondary coils connected in series opposition and a common concentric primary coil. An alternating magnetic field is applied to the sample by means of an a.c. current through the primary coil. If an a.c. bridge is used, the bridge is balanced with no sample present, and the change in magnetic flux created by the sample when positioned in one of the secondary coils gives rise to an

unbalanced voltage which provides the input signal to a phase sensitive detector (PSD). The output voltage of the PSD is proportional to the a.c. magnetic susceptibility, χ^{ac} , of the sample. If a bridge is not used, then it is the change in the output voltage of the secondary coils that is proportional to χ^{ac} .

The a.c. magnetic susceptibility is defined as $\chi^{ac} = \chi' + j\chi''$, where χ' is the magnetic response which is out of phase with the excitation field and χ'' represents a.c. losses in phase with the excitation field⁶. It is therefore necessary to determine the phase of the excitation relative to the reference. This can be done either by measuring the potential drop on a resistor in series with the primary coil (which is assumed to be ideal), or by fixing the relative phase to obtain zero χ'' at temperatures at which one knows the dissipation is zero¹. One serious drawback is the fact that, as both secondary coils are never identical, there is a signal produced by the coils, even without a sample, that depends on the coil temperature and whose phase relative to the reference may vary with temperature and time¹.

As pointed out elsewhere⁷, one frequently encounters data showing negative χ'' below the critical superconducting transition temperature T_c or finite positive χ'' about T_c , even when no dissipation is expected. This could be due to improper phase setting or because the phase angle may drift with temperature. This situation can be avoided by keeping the primary and secondary coils at a fixed low temperature, and the easiest way to do so is to keep the coils immersed in a constant temperature liquid helium bath.

On the other hand, it is necessary to be able to vary the sample's temperature in a convenient and known way over the whole temperature range. In another design⁸ this is achieved by supporting the sample in a helium filled glass sample holder which is itself suspended in an evacuated glass tube. We have avoided glass tubes,

located the sample in high vacuum and thermally anchored it to a sample holder whose temperature is controlled with a heater and thermometer, in a similar way to Couach *et al.*⁹.

Cryostat

Measurements down to liquid helium temperatures usually require a day to cool down the sample and warm it up again to room temperature and replace it by another sample. An immersion cryostat reduces this dead time by an order of magnitude⁵. The cryostat is housed in a 1 m long non-magnetic stainless steel tube which is inserted in a storage Dewar through a suitable O-ring seal. Figure 1 shows schematically the experimental set-up. The sample is located in a high vacuum chamber and thermally anchored to the copper sample holder whose temperature is controlled with a heater and thermometer. The sample holder, which must be at least 1.5 cm away from the top of the primary coil to avoid magnetic perturbations, is connected to the bath by means of two stainless steel pins soldered to a copper thermal exchanger, which is in contact with the cryogenic bath. Electrical connections are thermalized first at the copper heat exchanger and then at the sample holder, and wired from a feed-through. The sample is connected to the sample holder by means of a foil coil or sapphire¹, to guarantee the absence of any magnetic or electrical contribution.

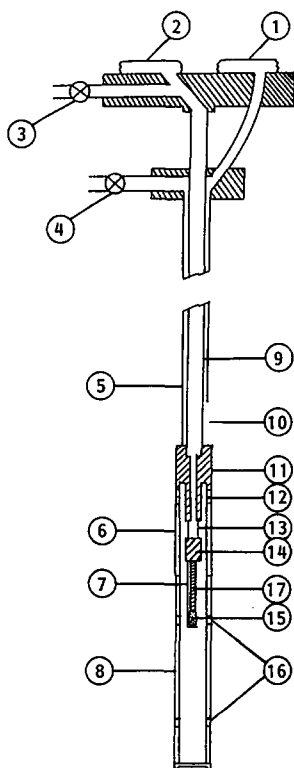


Figure 1 Experimental set-up: 1, feed-through for connections to coils; 2, feed-through for internal connections; 3, high vacuum and security valve; 4, helium boil-off recovery valve; 5, external stainless steel tube; 6, stainless steel vacuum shroud; 7, foil coil; 8, assembly of coils; 9, pumping line; 10, orifice on external tube; 11, thermal exchanger; 12, Wood's solder; 13, stainless steel pins; 14, sample holder and thermometer; 15, sample; 16, slots provided for secondary windings; 17, sapphire support used to centre sample

Primary and secondary coils are wound on a stainless steel vacuum shroud which is a 15 cm long, 13 mm outer diameter tube of 0.5 mm wall thickness, and therefore presents a negligible skin effect at audio frequencies. The two secondaries consist of 60 turns each oppositely wound on two slots 0.2 mm deep, 2.5 mm long and 35 mm apart, provided on the vacuum shroud. The primary coil, 7 cm long, was wound in two layers resulting in 9440 turns per metre. Enamelled copper wires 0.06 mm and 0.15 mm in diameter were used for the secondary and primary coils, respectively. This configuration resulted in a sensitivity of 5×10^{-7} e.m.u.

The vacuum shroud is soft soldered with Wood's metal so that the sample can be easily replaced with no damage to the coils, which are immersed in water when the shroud is soft soldered, or to the sample, whose temperature never exceeded 40°C above room temperature during the soldering procedure. Electrical connections from the primary and secondary coils are wired from a second feed-through and guided through the space between the central pumping line and the external stainless steel tube for protection. The wires are available for connection to the coils through an orifice in the external tube via gold plated pins.

The cryostat may be operated from room temperature down to 4 K. For the cool-down procedure, it is partially introduced into the Dewar so that its bottom remains at the liquid nitrogen cooled neck of the Dewar. The temperature falls rapidly if the vacuum can is evacuated to ≈ 1 torr ($\approx 133.322 \text{ N m}^{-2}$). The cryostat is lowered slowly until the bottom reaches ≈ 50 K. At this temperature, the residual air still conducts heat and speeds the cooling rate provided by cold helium gas. Then the cryostat is lowered into the liquid helium bath, and cryopumping establishes a high vacuum in the can, with a total liquid helium boil-off of $\approx 0.3 \text{ dm}^3$.

Application

As an application, the a.c. susceptibility of a normal metal was measured for temperatures between 10 and 100 K. If a metal cylinder is placed with its axis parallel to an homogeneous alternating magnetic field $\mathbf{H}(t) = \mathbf{H}_0 \exp(j\omega t)$, an azimuthal current $\mathbf{J}(\mathbf{x}, t) = \mathbf{J}(r)\exp(j\omega t)$ will be induced. This situation may be also described as a magnetostatic equivalent problem where the equivalent currents generate an average magnetization

$$\mathbf{M}_{\text{eq}} = \frac{1}{2} V \int \mathbf{x} \times \mathbf{J}_{\text{eq}}(\mathbf{x}) d\mathbf{x}$$

so that the magnetic field in the cylinder will be given by

$$\mathbf{B} = \mu_0(\mathbf{H}_0 + \mathbf{M}_{\text{eq}})$$

where the equivalent magnetization is proportional to the applied field: $\mathbf{M}_{\text{eq}} = \chi \mathbf{H}_0$ (reference 10). Solving this problem¹¹ one obtains

$$\chi = - \left(1 - \frac{2J_1(ka)}{kaJ_0(ka)} \right)$$

where: a is the cylinder radius; $k = (1 + j)/\delta$; $\delta = \sqrt{2\rho/(\mu_0\omega)}$ is the skin depth; ρ is the electrical resistivity of the cylinder; $\omega = 2\pi f$, where f is the frequency of the

alternating field; and μ_0 is the vacuum magnetic permeability. J_0 and J_1 are the ordinary Bessel functions of zero and first order.

The resistivity of a 99.5% purity polycrystalline lead cylinder of $a = 2.5$ mm and 10 mm length was determined at a frequency of $f = 1000$ Hz for $H_0 = 0.35$ Oe. The complex measured voltage V is related to the complex susceptibility by

$$\chi = \frac{\alpha V}{2\pi f A_m N_s H_0}$$

where: α is a calibration constant; A_m is the sample's cross-section; N_s is the number of turns of the secondary; and H_0 is the field amplitude. The system was calibrated

by normalizing to the theoretical maximum $\chi'' = 0.377$ (reference 11); demagnetization corrections were not taken into account.

The temperature at which the maximum occurs depends on the sample, its radius and frequency because at the maximum a/δ is equal to 1.78.

There is a range of frequencies that can be used to obtain a maximum in χ'' somewhere between 4 and 100 K. Once α is known, one can calculate $\rho(T)$ for each value of χ' and/or χ'' , because $a/\delta(T)$ as a function of χ' or χ'' can be tabulated and therefore $\rho(T)$ evaluated.

Other calibration procedures are described elsewhere¹². Figure 2 shows the calculated resistivity obtained from magnetic and dissipation data, in good agreement with results published¹³ for polycrystalline lead with less than 0.05% impurities.

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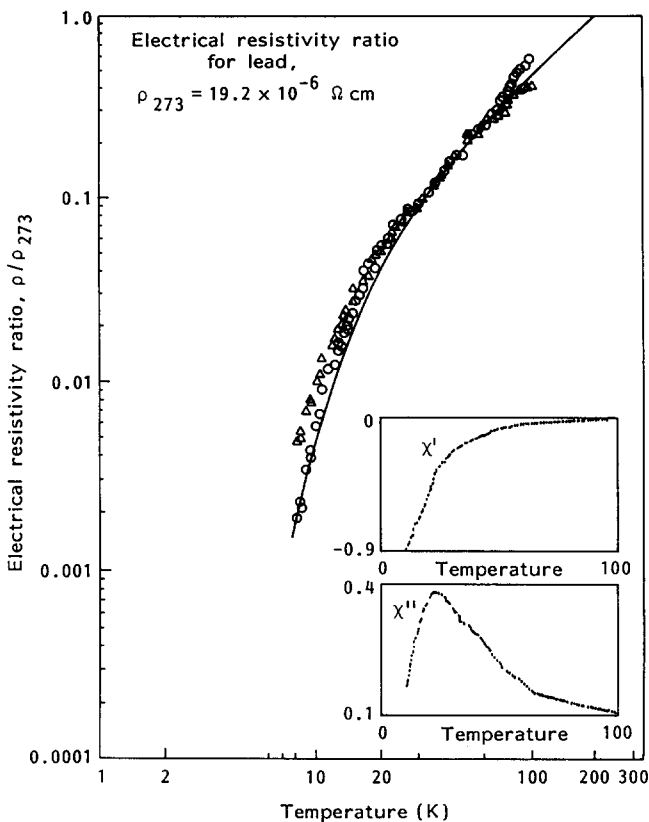


Figure 2 Electrical resistivity of lead versus temperature. $\circ$, Values calculated from χ' data; $\triangle$, values calculated from χ'' data; —, tabulated values¹³. χ' and χ'' are shown in inset