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Susceptometry Application of Portable HTS SQUID-Based System

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A portable RF HTS SQUID-based susceptometer was used for small size magnetized sample testing in weak DC (up to 200 A/m) and AC (up to 4 A/m) magnetic fields. The system resolution for the magnetic moment is of the order of 1.6×10^{10} A·m^2. The measured DC susceptibility of a tested sample agrees well with the value obtained by using a commercial liquid helium susceptometer.

1. Introduction

The SQUID sensor has been used for measurements on magnetic samples almost from the time of the inception of this sensor [1]. After a relatively general-purpose SQUID-based susceptometer was developed for a wide range of temperature measurements, the first commercial general-purpose susceptometers were produced in 1974 [2]. It provided continuous variation of temperature samples from below 4 K to room temperature by passing temperature controlled helium gas through the sample chamber [3].

From the theoretical estimations it is known that the sensitivity of a SQUID-based susceptometer $\delta\chi$ is given as function of the solenoid magnetic field H, the volume of tested sample V, and the SQUID intrinsic energy noise $\delta\varepsilon$ [4].

$$\delta\chi = 1/H\sqrt{(2/\delta\varepsilon/(\mu_0 V))} \qquad (1)$$

where μ_0 is the magnetic permeability of free space. For liquid helium-cooled systems, the sensitivity of a helium-cooled system can be improved up to a level of 10^{8} EMU, and even more in medium magnetic fields (of about 200 G) [5]. The equipment is large and heavy and therefore immobile. Consequently it has a high capital cost. A successful attempt has been made to design a portable liquid helium-cooled SQUID-based susceptometer [6]. However, no literature is available for liquid nitrogen-cooled systems. In this paper we present the first application of a portable high temperature superconductive (HTS) SQUID-based susceptometer [7] and report on its performance.

2. Calibration Procedure

The commonly used calibration procedure for this type of susceptometers is based on the information recorded using a small, magnetized sample and a very small coil with precisely known dimensions and current [4]. If a small calibration coil approximating a point magnetic dipole m_z of known current is moved near the SQUID sensor sensitive to axial field B_z, the output signal will be as shown in Fig. 1. The amplitude of the output voltage V_m of the system resulting from the change in interferometer magnetic flux is directly proportional to the magnetic moment of the calibration coil. The magnetic moment of this coil is equal to $m_z=S\cdot I\cdot N$, where S is the current loop area, I is the current, and N is the number of turns in the coil. The approximation of the magnetic dipole point used is reasonable because the diameter of 1.5 mm and axial coil length of 1.5 mm are much less then the minimum distance between this dipole and the SQUID sensor. In construction of the cryogenic probe used in this research, the length was 15 mm. To minimize the influence of external magnetic interference, the average output signal during the calibration was over six cycles of up and down motion of the coil. The determined magnetic moment-to-voltage transfer coefficient $k_m=m_z/V_m$ is equal to 8.2×10^{8} Am^2/V for our SQUID-based susceptometer.

3. Results and Discussion

3.1 DC measuring field

When a very small cube of $Gd_{88}La_{12}$ sample (volume $V=3\times 10^{9}$ m^3) is passed up or down near the flux-locked SQUID in a homogeneous DC magnetic field of H=43 A/m, the output signal is as shown in Fig. 2. The result of several repeated procedures of this motion gives an average value of the output signal amplitude. The total magnetic moment of the sample is measured as $m_z=k_m V_m$ in a known DC magnetic field at a fixed temperature. The magnetic moment per unit volume (magnetization) is calcu-

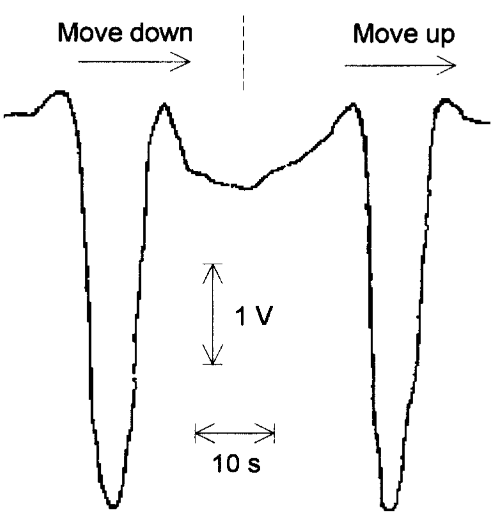

Fig. 1. HTS SQUID-based susceptometer output signal during calibration procedure by using a very small coil with current m_z= 2.55×10^{7} Am^2.

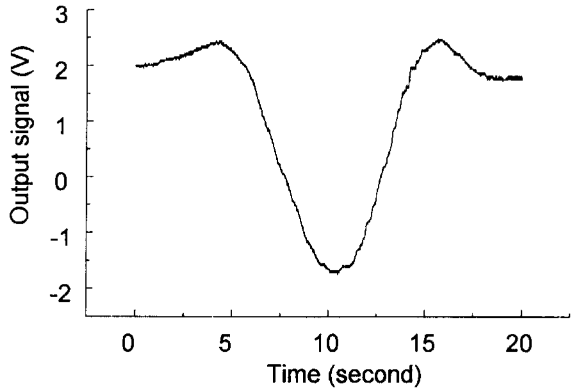

Fig. 2. System output signal when the small Ga$_{88}$La$_{12}$ polycrystalline sample was tested in a DC magnetic field.

lated as 2.9 from the relationship of $\chi=m_z/(VH)$.

The temperature dependence of DC susceptibility for this Gd$_{88}$La$_{12}$ sample averaged over a series of measurement procedures is shown in Fig. 3. The results of these measurement procedures agreed well with the measured results obtained from a stationary commercial helium-cooled QUANTUM DESIGN susceptometer (KRISS).

A HTS SQUID-based susceptometer was used for investigations into HTS material (ceramic) in order to demonstrate the different applications. The temperature transition characteristics of an HTS YBCO sample at two different initial conditions were observed, that is, cooling in the presence of a magnetic field (non-zero field cooling) as shown in Fig. 4(a) and cooling without a magnetic field (zero field cooling) as shown in Fig. 4(b).

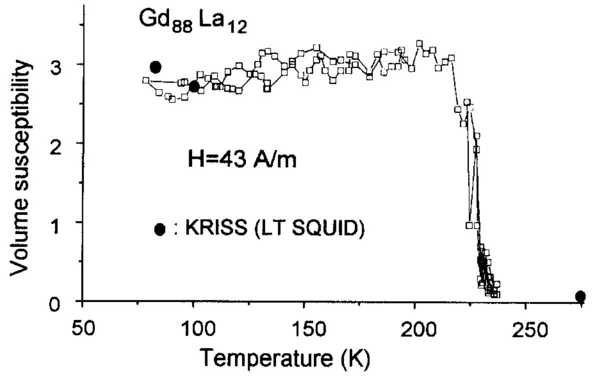

Fig. 3. The temperature dependence of susceptibility measured in a 43 A/m DC magnetic field for Gd$_{88}$La$_{12}$ sample averaged over a series of measurement procedures. Solid circles show the result of comparative measurements by QUANTUM DESIGN commercial helium cooled susceptometer.

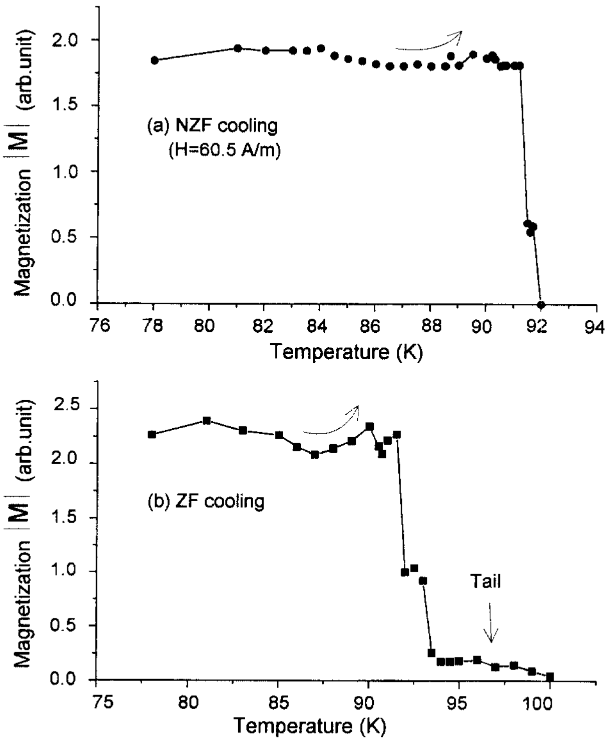

Fig. 4. The temperature transition characteristics of YBa$_2$Cu$_3$O$_{7-x}$ ceramic small sample: (a) Cooling in the presence of a magnetic field (non-zero field cooling); (b) Without cooling in the presence of a magnetic field (zero field cooling).

The working solenoid DC magnetic field was 60.5 A/m for both measurements. It can be clearly seen from the experimental curve that when the temperature increases from 77 K to about 84 K, structural change in the HTS begins, suppressing the weak intergrain links. In this state, HTS ceramics can be considered as Josephson media. Substantial superconductive grains are connected by a weak Josephson junction type link. For a further increase in temperature, the total magnetic moment of the sample increases slightly because of thermal activation of the magnetic vertex motion. The superconductivity suppression inside the grain results in an output signal drop at about 91 K, but the presence of a *tail* in Fig. 4(b) indicates that the magnetic flux is strongly trapped by a small amount of grains inside the sample.

3.2 AC measuring field

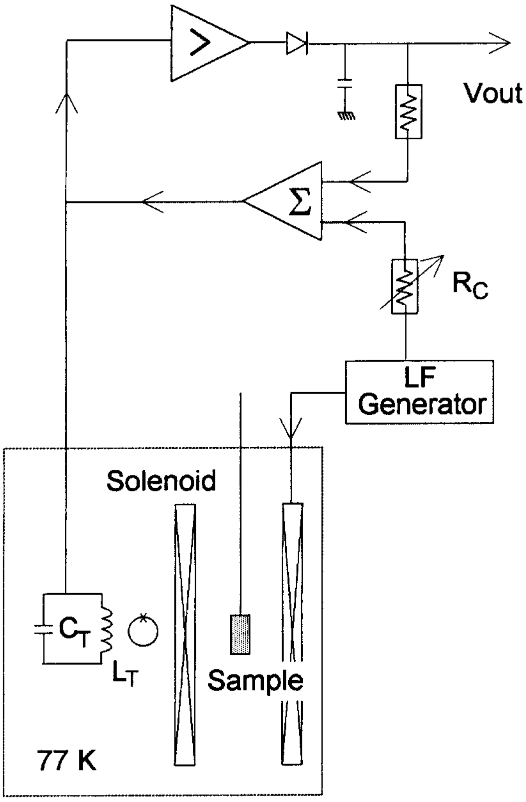

Fig. 5. Compensation electronic circuit for the susceptibility measurement in AC magnetic fields.

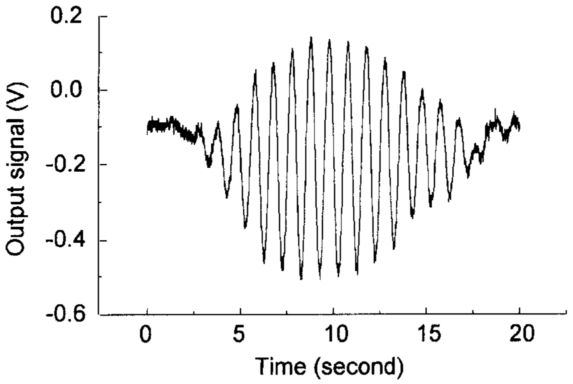

Fig. 6. System output signal when the small $Ga_{88}La_{12}$ polycrystalline sample is moved in the AC magnetic field of 1 Hz frequency and 4 A/m amplitude.

AC magnetic susceptibility measurements can provide valuable information about magnetic structures, especially when they are frequency dependent as, for example, in ferromagnets [5]. The SQUID sensor is sensitive to any change in magnetic field. Therefore, the system design must prevent any effect due to the presence of a homogeneous AC magnetic field of the solenoid appearing in the output signal. In a commonly used helium-cooled susceptometer, this would be achieved by gradiometer-type superconducting pickup coils and shielding of the SQUID by a superconductive capsule. The result is that the magnetic sensor is not sensitive to uniform AC fields generated by the solenoid, but it is sensitive to magnetic fields caused by the magnetizing sampling.

Because of the absence in practice of suitable HTS wire for the d*B*z/dz pickup coil, this task is very difficult using liquid nitrogen-cooled systems. However, for measurements in AC magnetic fields, a special electronic circuit was used for compensation of the output signal when the tested sample is in either the up or the down position, as shown in Fig. 5. The exciting current from the standard low frequency generator was passed through the solenoid, and part of the current was fed to the resonant circuit coil. This current produced the magnetic flux inside the SQUID interferometer in the opposite direction to the solenoid field. The amplitude of this compensation current can be finely adjusted by means of a special potentiometer R_c in order to minimize the output signal, ideally to zero. When the magnetic sample is moved near a SQUID sensor, the compensation is discontinued. The amplitude of the output signal is proportional to the sample magnetization. The response is shown in Fig.6, where an AC magnetic field of 1 Hz frequency has an amplitude of 4 A/m. Both methods (DC and AC magnetic field excitation) gave the same results for Gd-based sample volume susceptibility$\approx$2.9 ($\pm$0.1) in the temperature range from 120 K to 200 K.

4. Summary

The portable HTS RF SQUID-based magnetic susceptibility measurement system was applied to investigate low magnetized materials and different types of magnetic materials in weak DC and AC magnetic fields with accuracy compatible with previously used commercial liquid helium units. We plan in the future to design a model for measuring the magnetic properties for NDE applications.

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