Journal of Magnetics 3(3), 82-85 (1998)

Characteristics of HTS SQUID-based Susceptometer

V. P. Timofeev[1,2], C. G. Kim[1] and V. I. Shnyrkov[2]

[1]*Department of Physics, Sun Moon University, Asan-si 336-840, Korea*
[2]*Inst. for Low Temp. Phys. & Eng., Nat'l Academy of Sciences of Ukraine, Kharkov 310-164, Ukraine*

(Received 27 August 1998)

A portable HTS RF SQUID-based system, weighing less than 20 kg has been built for susceptometry applications in weak magnetic fields. It includes a YBCO sensor for measuring the axial magnetic field component with a resolution of about 7× 10^{-13} T/Hz$^{1/2}$. This is determined by the intrinsic magnetic noise in the quasi-white noise region. There is a relaxation for a sudden increase in field due to magnetic flux creep in HTS. In this instance the time did not exceed 3~5 minutes.

1. Introduction

SQUID (Superconducting QUantum Interference Device) is the most sensitive magnetic field sensor, with a resolution of several femtotesla (10^{-15} T) in 1 Hz bandwidth [1]. This high sensitivity is realized by means of a macroscopic display (change in superconducting current) of the microscopic coherent effects (phase change of the quantum wave function) under the influence of an applied magnetic field. SQUID-based instruments for measuring the variations in the magnetic field, current, voltage and susceptibility play a role on transducers for the different types of input signals applied to the magnetic flux variations $\Delta\Phi$ in a SQUID interferometer.

SQUID applies not only to fundamental physics investigations such as gravitational wave detection, quantum metrology, and biomagnetical research, but also to applications in non-destructive evaluations (NDE), defense and measurement of the magnetic properties in low magnetized materials. The major SQUID applications have been in biomagnetism, but currently there is growing interest in NDE using the SQUID-based system.

Measurements of magnetization and magnetic susceptibility play an important role in materials research. Magnetic susceptibility is commonly measured by applying an oscillating magnetic field to the sample and then measuring the force that is proportional to the magnetic moment induced by this field. Alternatively, using SQUID direct measurements, the DC magnetic moment of a sample can be measured without using an oscillating magnetic field [2]. SQUID-based magnetometers in conjunction with superconducting magnets and shields are now being used for susceptometry, and provide unique combinations in sensitivity, resolution and accuracy [3].

The use of liquid helium as a cooling agent restricts the broad application of this type of magnetometer because of its high cost and costly servicing. We have new opportunities after the discovery of high temperature superconductivity (HTS) in 1986. A number of groups around the world have recently reported HTS SQUID devices with performances of up to 77 K. These devices are comparable with the performance of earlier commercial 4.2 K SQUIDs [4]. In this paper we introduce the constructed HTS SQUID-based system for susceptometry and its main characteristics.

2. SQUID Sensor

Two different construction of SQUIDs cooled by liquid nitrogen (LN) were used in the experimental equipment. Fig. 1(a) and (b) illustrate these SQUIDs, sensitive to axial (B_z) and transverse (B_x) field components, respectively. Both types of sensors are of modular construction shielded by brass to protect against high frequency electromagnetic fields (EMF).

The first one has been made of bulk polycrystalline $YBa_2Cu_3O_{7-x}$ ceramics. The interferometer has an axial cylindrical hole 0.8 mm in diameter and 5 mm high. Its inductance is 1.2510^{-10} H. This value is close to the fluctuation inductance at 77 K determined from relationship

$$L = (1/kT)\times(\Phi_0/2\pi)^2, \qquad (1)$$

where k is Boltzmann's constant and Φ_0 is the magnetic flux quantum; the result equals 2.07×10^{-15} Wb. The weak link of the grain boundary Josephson junction was formed using scribing and high voltage discharge techniques. The resonant tank circuit consists of a coil of $L_T\approx6\times10^{-7}$ H inductance and a capacitor of $C_T\approx70$ pF, resulting in a quality factor $Q\approx100$. The SQUID is operated at a radio

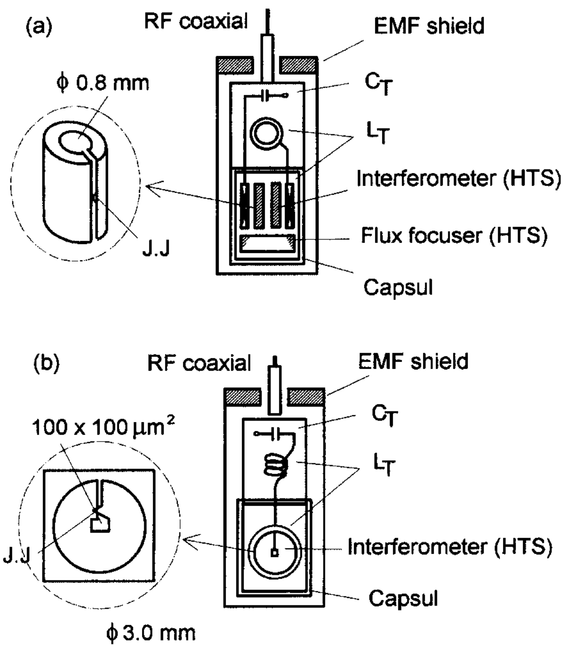

Fig. 1. HTS RF SQUID construction: (a) ceramic bulk YBCO SQUID; (b) thin film YBCO SQUID.

frequency (RF) with a flux bias of 20 MHz. The SQUID flux-to-voltage transfer coefficient reaches a value of 3×10^{10} V/Wb.

The second HTS RF SQUID in our equipment is a $YBa_2Cu_3O_{7-x}/SrTiO_3$ thin film SQUID. The interferometer has a square 100×100 μm^2 hole and a Josephson junction of normal metal-superconductor (S-N-S) step-edge type. Flux focusing can be achieved by increasing the magnetic sensitivity using a 3 mm outer interferometer diameter.

During operation, HTS SQUIDs exhibited some degradation over time due to the absorption of moisture and condensation of atmospheric gases on the cold surfaces during the interferometer thermal cycling. It is important to increase the lifetime of the interferometer by protecting it from these influences. This was achieved by plastic encapsulation construction of the interferometers in conjunction with tank circuit inductances. No degradation in the characteristics of the interferometer has been detected over a period of more than five years.

The system sensitivity is determined mainly by the intrinsic noise characteristic of the sensor, measured within the HTS shield. The results shown in Fig. 2(a) reveal that this level did not exceed 7×10^{-13} T/Hz$^{1/2}$ in the quasi-white noise region for B_z sensitive ceramic YBCO SQUID. The thin film RF SQUID has much lower intrinsic noise, as shown in Fig. 2(b). However, the actual sensitivity of the system is limited by the external low frequency interference in the practical workplace.

3. System Design

A HTS RF SQUID-based magnetometer for investigating the magnetic properties of small samples (up to 1.5 mm in diameter) in the weak DC and AC magnetic fields was designed, constructed and tested.

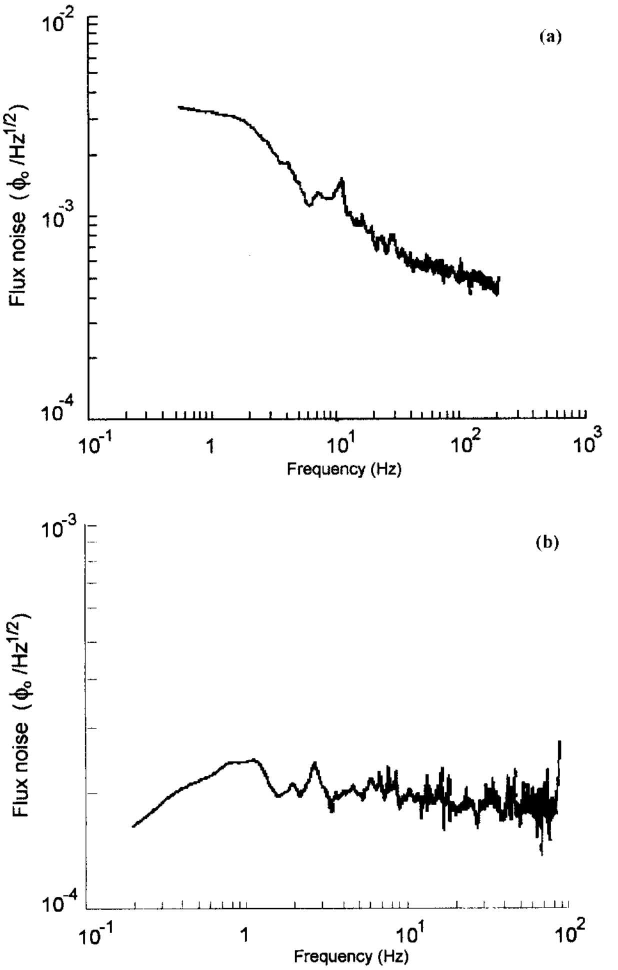

Fig. 2. Spectral noise characteristics of (a) bulk and (b) thin film SQUIDs measured in HTS shield.

Figure 3 shows the main components of the susceptometer:

- Nonmagnetic fiberglass-reinforced plastic (FRP) dewar for LN; 4.2 L volume in two-layer mu-metal magnetic shield (about 60 dB suppression for the earth's magnetic field).
- Cryogenic measuring probe with an inner glass tube-like dewar and solenoid, gas flowing thermo-exchanger with quartz measuring chamber and differential-type thermocouple for thermometry.
- Quartz holder for moving a sample along the solenoid axis.
- YBCO HTS SQUIDs fixed near the solenoid in parallel and standard type RF SQUID electronics.

Space homogeneous DC and AC magnetic fields in the measuring chamber were generated by a copper wire solenoid of 150 mm in length and a diameter of 18 mm. The current supplied from a controlled battery voltage or from a standard low frequency generator was fed to the solenoid through a low frequency filter to prevent external interference. The measuring chamber temperature, and

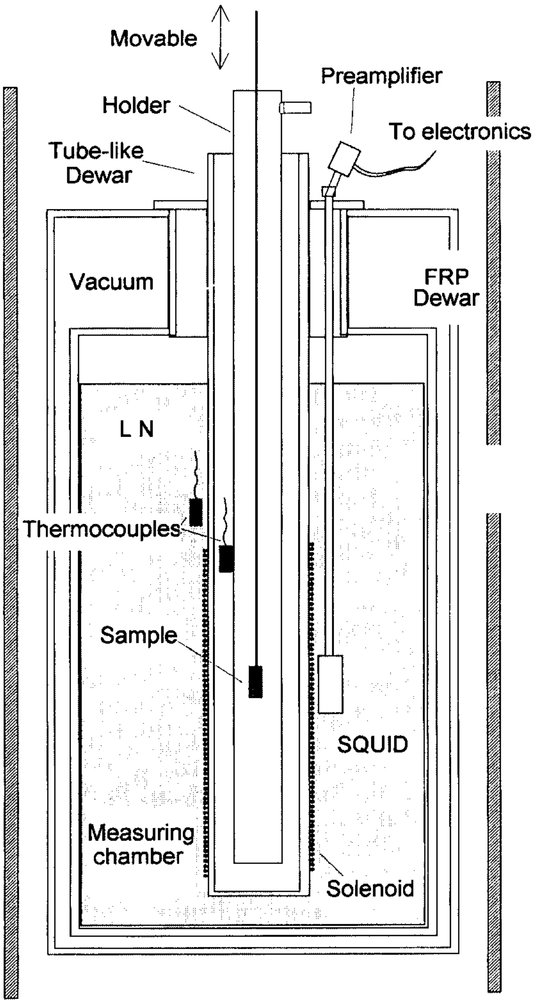

Fig. 3. Construction of HTS RF SQUID-based susceptometer.

therefore the sample temperature, was controlled in the range from 77 K up to 150 K (and above) by filling the thermo-exchanger with LN or by flowing heated nitrogen gas through the thermo-exchanger.

4. HTS SQUID Response for Changed Magnetic Field

A non-shielded HTS SQUID sensor is sensitive to any change in magnetic field because the signal is detected by interferometer inductance directly, rather than by special pick-up circuits as used for helium-cooled shielded SQUIDs. When the solenoid current is turned on or changed, there is a response in the output voltage of the SQUID electronics. Fig. 4 shows the output voltage when the solenoid field is changed smoothly from zero to H=43 A/m. Each peak appeared when the output voltage reached the full dynamic range of the SQUID electronics. In the experimental work this was usually equal to 10 Φ_0. The reset circuit adjusted the output voltage to zero.

The known magnetic hysteresis in the YBCO SQUID interferometer [5] essentially complicates the effect of the change in magnetic field. When the magnetic field is turned on abruptly, the magnetic flux is caused to creep within the HTS materials and a "zero" output voltage drift results. Time is required to achieve a state of equilibrium. Fig. 5 shows the relaxation of the output voltage due to changes in solenoid fields (currents were applied). In the $1-e^{1/\tau}$ approximation the dependence of the time constant τ, typical for this magnetic field system, is shown in Fig. 6. For a magnetic field changing up to ΔH=130 A/m, the

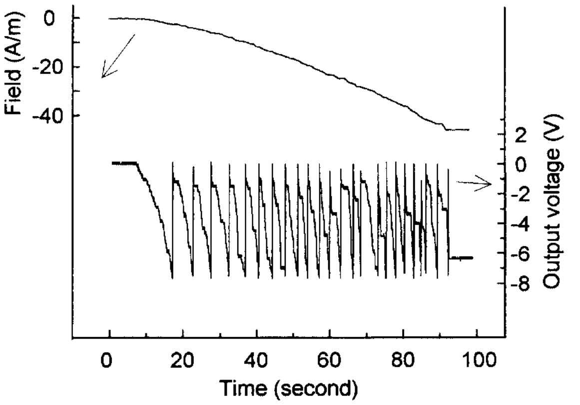

Fig. 4. System output voltage for a solenoid field change from zero to H=43 A/m.

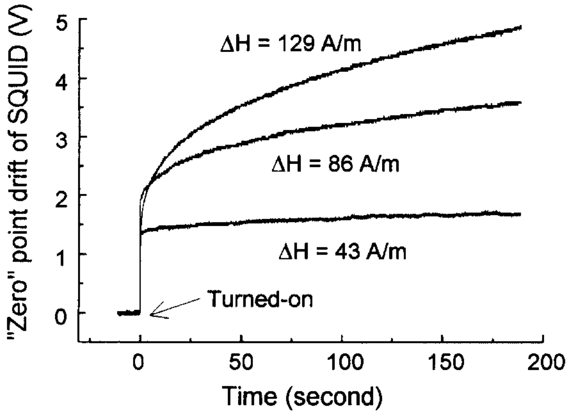

Fig. 5. Drift of "zero" point of output voltage when magnetic fields are abruptly turned on.

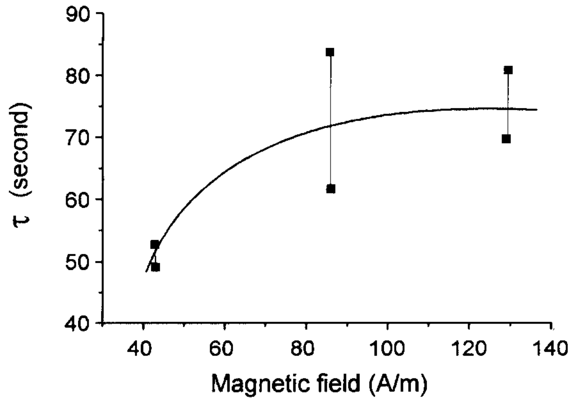

Fig. 6. The time constants for system relaxation for different magnetic field changes (fields are turned on). Vertical lines illustrate the estimated possible deviations.

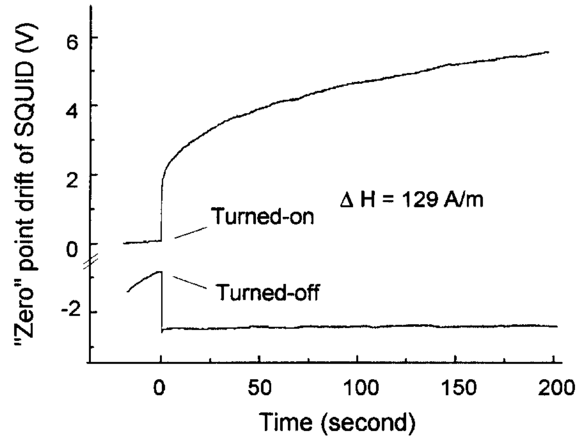

Fig. 7. The system output voltage when solenoid field is turned on and turned off.

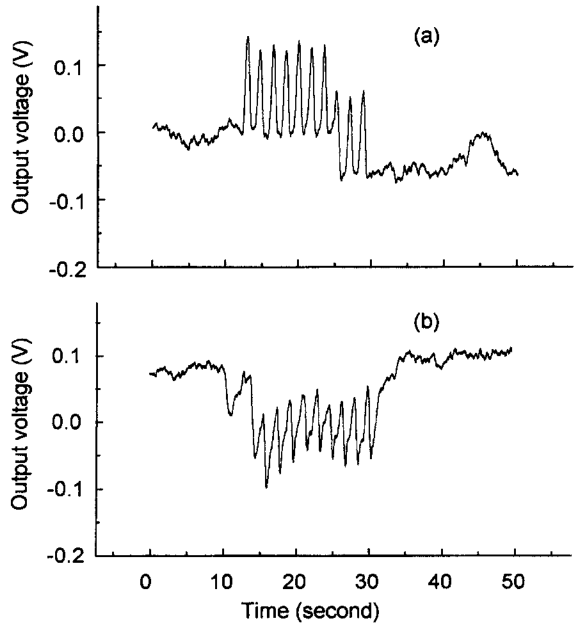

Fig. 8. Typical magnetic noise interference in the laboratory: (a) the rotation of a small piece of permanent magnet at a distance of 4 m, (b) the swing of telephone receiver at a distance of 2 m.

typical relaxation time did not exceed 3-5 minutes. The drift of the SQUID zero point output voltage for a solenoid field turned off is compared with that of the one turned on, shown in Fig. 7. There is no relaxation in the output voltage for a non-excited field. To minimize this drift, the intensity of the solenoid magnetic field should be controlled step by step, starting from zero and increasing the intensity to a maximum.

Even though two layers of μ-metal magnetic shielding have significant magnetic noise suppression (about 1000 times), the SQUID sensor is highly sensitive to external low frequency magnetic disturbances. Fig. 8(a) and (b) illustrate some influence of the external interference on the system output signal. One is the effect of the rotation of a small permanent magnet at the distance of 4 m and the other a swinging telephone receiver at a distance of 2 m, respectively. Hence, the choice of an optimal laboratory is essential.

5. Summary

A portable HTS RF SQUID-based system, weighing less than 20 kg has been built for susceptometry applications in weak magnetic fields. It includes a YBCO sensor for measuring the axial magnetic field component with a resolution of about 7×10^{-13} T/Hz$^{1/2}$. This is determined by the intrinsic magnetic noise in the quasi-white noise region. There is a relaxation for a sudden increase in field due to magnetic flux creep in HTS. In this instance the time did not exceed 3-5 minutes. Even though two layers of μ-metal magnetic shielding have significant magnetic noise suppression (about 1000 times), the SQUID sensor is highly sensitive to external low frequency magnetic disturbances, indicating the choice of an optimal laboratory is essential.

Acknowledgments

The author V. P. Timofeev wishes to acknowledge the financial support of STEPI during this work. This work was partly supported by Sun Moon University under a 1997 research program.

References

[1] T. Ryhanen and H. Seppa, J. of Low Temp. Phys., **76**(5/6), 287 (1989).
[2] R. E. Sager, A. D. Hibbs, and S. Kumar, IEEE Trans. Mag., **28**, 3072 (1992).
[3] J. S. Philo and W. M. Fairbank, Rev. Sci. Instrum., **48**, 1529 (1977).
[4] D. A. Konotop, V. I. Shnyrkov, V. P. Timofeev, S. S. Khvostov, and G. M. Tsoi, Cryogenics, **33**(6), 632 (1993).
[5] J. W. Purpura and R. F. Wieger, IEEE Trans. Appl. Supercon., **7**(2), 2549 (1997).