PAPER *Special Issue on Superconductive Devices and Systems*

Development of a High-T_c SQUID Cryo-System for the Measurement of a Remanent Magnetic Field of Rock

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SUMMARY A portable cryo-system using a high-T_c SQUID for the measurement of the remanant magnetic field of a rock specimen was designed and fabricated. The sensing surface of the SQUID faces upward in our system, although the system for biomagnetics faces down. The SQUID is cooled by liquid nitrogen via a sapphire heat transfer rod. The total heat transfer of the system was measured by means of a boiling-off method and was found to be 1.65 W. It was demonstrated that the system can be operated for more than 17 hours without any maintenance such as filling with liquid nitrogen. The system was applied to the measurement of the remanent magnetic field distributions of rock samples cored from deep underground. We have successfully measured the distributions.

key words: *High-T_c, SQUID, superconductor, microscope, rock, remanent magnetic field*

1. Introduction

There are many advantages to the use of high-T_c superconducting quantum interference devices (SQUIDs) such as easy thermal insulation, easy handling and low running costs, although a high-T_c SQUID is typically an order of magnitude less sensitive than a 4.2 K low-T_c SQUID [1], [2]. Recently a SQUID microscope was developed and used in biology and metallurgy, among other fields [3]–[11]. For these applications, portability is an important factor. The key element of such a system is the development of a small cryostat, which keeps the SQUID sensor at the same temperature for a long time. Random telegraph signals on the high-T_c SQUID resulting from the hopping of a single flux vortex are sometimes observed. In that case, the temperature of the SQUID is raised up to more than its transition temperature to eliminate the trapped flux. Conventionally, the elimination is performed by having a 77 K SQUID come into contact with a room temperature vacuum window so that the temperature of the SQUID can be increased. However, an installation of a trap elimination heater provides us with an easier trap elimination. We have developed a cryostat with a heater and evaluated its performance. This high-T_c SQUID cryo-system was applied in the measurement of a remanent magnetic field of rock core specimens. Since a SQUID magnetometer has a high magnetic field resolution, it is suitable for the measurement of a weak magnetic field from some specimens within the granite category in the area of paleomagnetic measurements. The design and the thermal properties of the cryostat, and the results of the measurements of the rock specimens are described in this paper.

Manuscript received May 24, 1999.
Manuscript revised July 13, 1999.

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2. Experimental Set Up

A cross-sectional view of the cryostat is shown in Fig. 1. This cryostat design concept is based on the system developed at U.C. Berkeley and modified here [4]. The size of the cryostat is 250 mm in diameter and 280 mm in height. Most of the parts of the cryostat are made of G-10 fiberglass. They are nonmetallic except for a copper reservoir and stainless steel tubes for filling liquid N_2 (LN_2). The volume of the LN_2 copper reservoir, which is suspended from a 30 mm thick upper disc by four 10 mm dia.- fiberglass columns, is 0.64 liters. The side and the bottom of the reservoir are superinsulated by layers of a single-sided aluminum evaporated my-

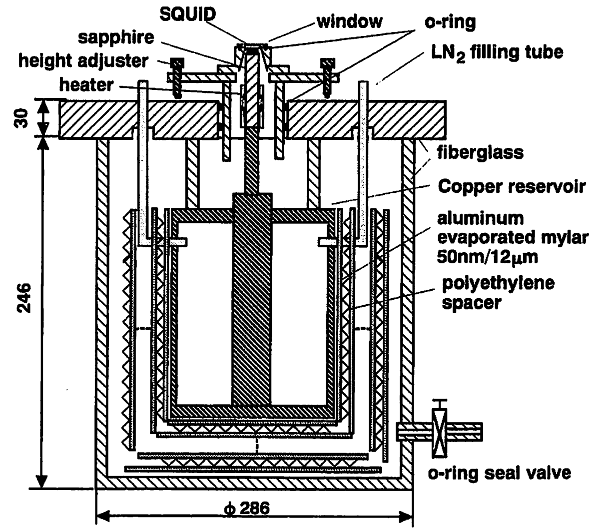


Fig. 1 Sectional side view of the cryostat. The size of the cryostat is 250 mm in diameter and 280 mm in height. Most of the parts of the cryostat are made of fiberglass.

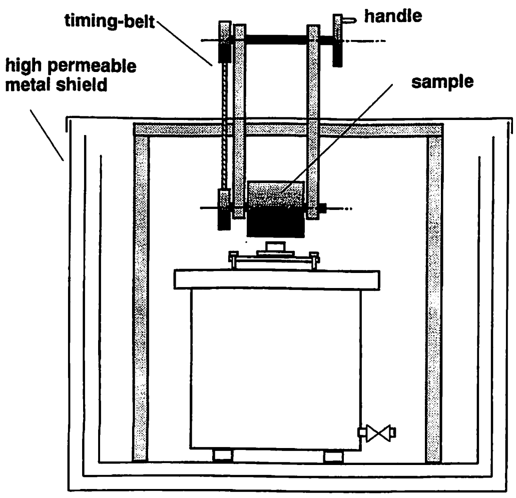


Fig. 2 Schematic drawing of the system. The cryostat was located inside a three-layered highly permeable metallic cylinder. The rock core samples can be rotated manually.

lar sheet (50 nm thick Al on 12 μm thick mylar) with a spacer. The inside of the cryostat can be evacuated by a high vacuum pump and sealed off by an o-ring valve. An adjustable-height sapphire window assembly is connected to the top disc via two o-rings. These o-rings are lubricated with silicon grease to make the assembly slide smoothly. The height can be adjusted by three height-adjusters with stainless-steel micrometer heads. A sapphire window with a thickness of 200 μm is sealed by an o-ring.

We used a $YBa_2Cu_3O_{7-y}$ (YBCO) SQUID magnetometer for the system. This SQUID is a product of Sumitomo Electric Ind., Ltd. [13]. The junctions utilized in the SQUID are of the step-edge type. The washer size of the SQUID is about 5.0×5.0 mm^2 and the effective area is 0.2 mm^2. The typical field noise $S_B^{1/2}$ of the SQUID is 400 to 500 fT/Hz$^{1/2}$ at 10 Hz. The SQUID chip is silver pasted on top of a sapphire rod (8 mm-dia. × 50 mm), which is thermally anchored with the LN_2 reservoir. Electrical wiring to the SQUID chip are made by applying conductive silver paint to the bonding pads and the side of the SQUID chip. A 6-turn modulation coil for SQUID operation in a flux-locked loop is installed just below the top of the sapphire rod. In our system, a heater for eliminating any trapped flux is installed. The heater wire made of CuNi alloy is non-inductively wound on the sapphire rod in order to avoid the generation of a magnetic field. The nominal resistance is 40 ohms/m and its diameter is 0.15 mm. The temperature of the SQUID can be raised up to more than its transition temperature and lowered to 77 K again.

Figure 2 shows the schematic drawing of the system. The cryostat was located inside a three-layered highly permeable metallic cylinder with an inner diameter of 380 mm, an outer diameter of 480 mm and a height of 550 mm. Rock core samples (50–80 mm-dia. × 50 mm) can be rotated by a handle connected to the sample housing via a timing-belt. The handle has an angular index showing the measuring point of the sample. This design enables us to measure the radial component of the magnetic field of the sample. The distance between the sample surface and the SQUID magnetometer is approximately 6 mm. The SQUID is operated in a flux-locked loop with a flux modulation frequency of 100 kHz [3]. The output signal was directly measured by a voltmeter without a band-pass filter. A bias reversal scheme, which dramatically reduces $1/f$ noise in the lower frequency range, was not used here [12].

3. Experiment

3.1 Performance of the System

We have measured the total heat transfer by measuring the boil-off rate of LN_2. The heat transfer was calculated by the following equation,

$$Q_t = \frac{VDL_v}{t}. \quad (1)$$

Here, Q_t is the total heat transfer per second, V is the volume of an N_2 gas bag, D is the density of N_2 gas 1.15 g/l (at 300 K, 1 atm), L_v is the latent heat of vaporization of the nitrogen 199.1 [J/g] and t is the collection time. Theoretical values Q_{th} are given by

$$Q_{th} = \frac{1}{(N+1)} \frac{S_h \sigma (T_h^4 - T_c^4)}{\frac{1}{\varepsilon_h} + \frac{S_h}{S_c}\left(\frac{1}{\varepsilon_c} - 1\right)}, \quad (2)$$

where ε_h and ε_c are the emissivities of hot and cold surfaces, respectively, S_h is the area of the hot surface 0.27 m^2, S_c is the area of the cold surface 0.061 m^2, T_h is the hot surface temperature 300 K, T_c is the cold surface temperature 77 K, σ is the Stefan Boltzmann constant 5.67×10^{-8} W/m^{2}K^4, and N is the number of aluminum mylar sheets [14].

We put aluminum foil on both the hot and cold surfaces with good thermal contact and used 0.056 for both of the radiation coefficients [14]. Figure 3 shows the average heat transfer as a function of the number of aluminum mylar sheets. The cryostat was evacuated to 1.0×10^{-6} Torr before filling with liquid nitrogen. The heat transfer is saturated when the number of layers is more than 20 and is 1.65 W. This value corresponds to a consumption of liquid nitrogen of 0.037 liter per hour. This means that this cryo-system can be operated for more than 17 hours without refilling with liquid nitrogen. The theoretical value of the heat transfer is also depicted in Fig. 3. The value is saturated at 20 layers and is 0.04 W. There is a difference of about 0.6 W. The additional heat transfer comes from the sapphire rod and the thermal conduction of the inter-mylar sheets.

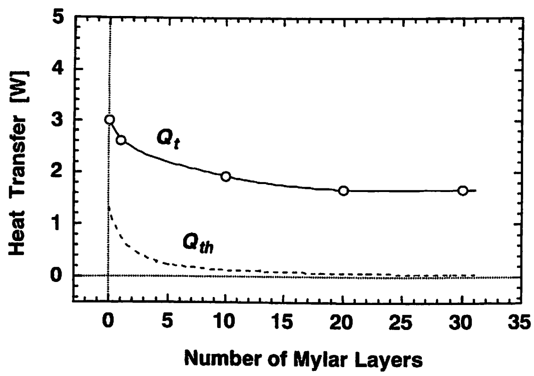


Fig. 3 Average heat transfer as a function of the number of aluminum mylar layers. The heat transfer is saturated when the number of layers is more than 20 and is 1.65 W. The theoretical value is saturated at 20 layers and is 0.04 W.

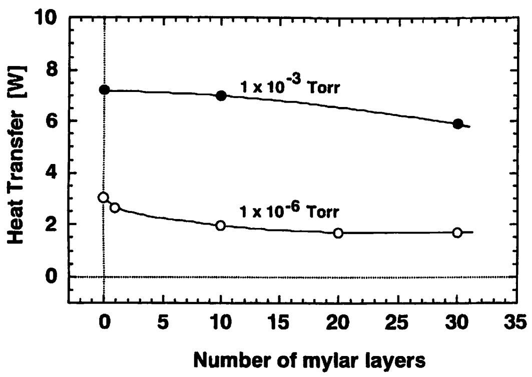


Fig. 4 Average heat transfer as a function of the number of aluminum mylar layers. The heat transfer of the cryostat evacuated to 1.0×10^{-3} Torr is a factor of two or three larger than that evacuated to 1.0×10^{-6} Torr.

We evaluated how the vacuum pressure inside the cryostat affects the heat transfer. The cryostat was evacuated to 1.0×10^{-3} Torr before filling with liquid nitrogen in this experiment. Because at the time the liquid nitrogen is filled the moisture inside the cryostat should be condensed at the surface of the LN_2 reservoir, therefore, the pressure must be below the measured value. We only measured the pressure at a point near the high vacuum pump, about 1 m away from the cryostat, before filling with the liquid nitrogen because of difficulties. The heat transfer was evaluated by the same boil-off method. The results are shown in Fig. 4. The results at 1.0×10^{-6} Torr are also depicted for comparison. The heat transfer of the cryostat evacuated to 1.0×10^{-3} Torr is a factor of two to three larger than that evacuated to 1.0×10^{-6} Torr. This suggests that the heat transfer due to the motion of gas molecules and conduction among the contact surfaces was greatly increased. Under these conditions the SQUID could not show a superconducting properties because the temperature was higher than its transition.

We have tested the performance of the installed

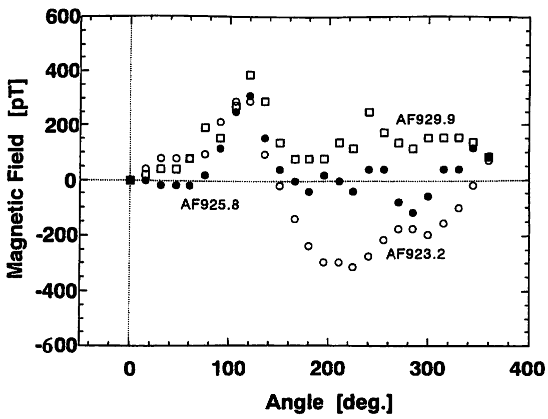


Fig. 5 Angular dependence of the remanent magnetic field of core samples. The number following the "AF" indicates the vertical depth in meters at the cored location. All the samples have a peak at around 120 degrees.

trap elimination heater. The temperature of the rod was estimated by measuring the resistance of the YBCO thin film. As a result, it was found that the temperature was elevated up to 100 K by having a current of 500 mA flow to the heater for 40 sec.

3.2 Measurement of Remanent Magnetic Field of Rock Samples

We measured the remanent magnetic field of rock samples. The core samples were taken from underground at a depth of 900 to 1000 m in 1990 in Ogachi, Akita Prefecture in Japan. The place is famous for being a geothermal area. The samples had been kept in a wooden box with each at a random direction since 1990. The samples are in the granite category. The shape of all of the core samples is 50 mm-dia. × 50 mm. They have an orientation index showing the relative orientation among the samples. We named the samples AF923.2, AF925.8 and AF929.9, where the number following the "AF" shows the vertical depth in meters at the cored location. The differences in depth among the samples is approximately 3 m. No magnetic treatment such as degaussing was performed before measurement. The sample was manually rotated, stopping at intervals of 15 degrees, so that the SQUID output signal could be recorded. It took about 120 sec. for one cycle of measurement from 0 to 360 degrees. The results are shown in Fig. 5. The base magnetic field at zero degrees was subtracted to indicate the angular dependence of the remanent magnetic field. The base magnetic field was on the order of 1000 pT. All the samples have a peak at around 120 degrees although they were kept in a box at random directions. From this fact we can assume that the specimens have kept their magnetic field almost unchanged since they were cored out. The measured fields at 360 degrees were not equal to those at zero degrees. We found that this phenomenon is due to a signal drift. When there were no samples, no drift could be seen. The drift comes from the thermal radiation from the sample which is at ambient temperature and emits heat flow to the 77 K SQUID. The insertion of a thermal shield between the sample and the SQUID may be helpful.

It is worthy of note that we sometimes observed random telegraph signals resulting from a single flux hopping when we opened and closed the lid of the magnetically shielded cylinder. In these instances, activating the installed heater successfully eliminated the trapped flux.

4. Conclusion

A portable cryo-system using a high-T_c SQUID with a flux trap elimination heater was designed and fabricated. The total heat transfer of the system was 1.65 W. The magnetic field distribution of rock samples cored from an underground location in Ogachi, Akita Prefecture was measured by using the SQUID system. All the samples had a similar distribution of the magnetic field around them with the peak in the same direction. This indicates that the remanent magnetic fields of the samples were kept nearly unchanged from the time that they were cored out.

Acknowldgments

We would like to thank Dr. Thomas Lee of the University of California Berkeley (now he is working for SYMYX Technologies, Inc.) for his advice on the cryostat design. This work was partially supported by a Grant-in-Aid for Scientific Research on Priority Area "Vortex Electronics."

References

[1] S. Tanaka, H. Itozaki, H. Toyoda, N. Harada, A. Adachi, K. Okajima, and H. Kado, "Four-channel $YBa_2Cu_3O_{7-y}$ dc SQUID magnetometer for biomagnetic measurement," Appl. Phys. Lett., vol.64, p.514, 1994.

[2] H. Weinstock, "Prospects on the application of HTS SQUID magnetometry to nondestructive evaluation (NDE)," Physica C, vol.209, p.269, 1993.

[3] S. Tanaka, O. Yamazaki, R. Shimizu, and Y. Saito, "Windowless high T_c superconductig quantum interference devices," Jpn. J. Appl. Phys., vol.38, no.L505, 1999.

[4] T.S. Lee, G. Dantsker, and J. Clarke, "High-transition temperature SQUID microscope," Rev. Sci. Inst., vol.67, p.4208, 1996.

[5] T. Shaw, K. Schlenga, R. McDermott, J. Clarke, J.W. Chen, S.-H. Kang, and J.W. Morris, Jr., "High-T_c SQUID microscope study of the effects of microstructure and deformation on the remanent magnetization of steel," IEEE Trans. Appl. Supercond., vol.9, p.4107, 1999.

[6] R.C. Black, A. Mathai, F.C. Wellstood, E. Dantsker, A.H. Miklich, D.T. Nemeth, J.J. Kingston, and J. Clarke, "Magnetic microscopy using a liquid nitrogen cooled YBCO SQUID," Appl. Phys. Lett., vol.62, p.2128, 1993.

[7] T. Morooka, S. Nakayama, A. Odawara, and K. Chinone, "Observation of superconducting device using magnetic imaging system with a micro-DC superconducting quantum interference device magnetometer," Jpn. J. Appl. Phys., vol.38, no.L119, 1999.

[8] N. Kasai, N. Ishikawa, H. Yamakawa, K. Chinone, S. Nakayama, and A. Odawara, "Nondestructive detection of dis-locations in steel using a SQUID gradiometer," IEEE Trans. Appl. Supercond., vol.7, p.2315, 1997.

[9] L.N. Vu, M.S. Wistrom, and D.J. van Harlingen, "Imaging of magnetic vortices in superconducting networks and clusters by scanning SQUID microscopy," Appl. Phys. Lett., vol.63, p.1693, 1993.

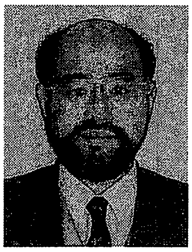
[10] G. Donaldson, S. Evanson, M. Otaka, K. Hasegawa, T. Shimizu, and K. Takaku, "Use of SQUID magnetic sensor to detect aging effects in duplex stainless steel," British Journal of NDT, vol.32, p.238, 1990.

[11] C.C. Tsuei, J.R. Kirtley, C.C. Chi, L.S. Yu-Jahnes, A. Gupta, T. Shaw, J.Z. Sun, and M.B. Ketchen, "Pairing symmetry and flux quantization in, a tri-crystal superconducting ring of $YBa_2Cu_3O_7$," Phys. Rev. Lett., vol.73, p.593, 1994.

[12] R. Koch, W.M. Goubau, J.M. Martinis, C.M. Pegrum, D.J. van Harlingen, and J. Clarke, "Flicker (I/f) noise in tunnel junction dc SQUIDs," J. Low Temp. Phys., vol.51, p.207, 1983.

[13] Catalog on web page: http://squid.sei.co.jp, E-mail: squid@info.sei.co.jp

[14] W. Obert, "Emissivity measurements of metallic surfaces used in cryogenic applications," Advances in Cryogenic Engineering, vol.27, p.293, Plemum Press, New York, 1982.



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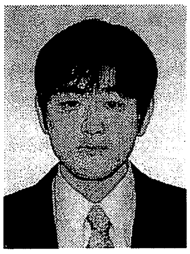

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