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Scanning SQUID microscope with an in-situ magnetization/demagnetization field for geological samples

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ABSTRACT

Magnetic properties of rocks are crucial for paleo-, rock-, environmental-magnetism, and magnetic material sciences. Conventional rock magnetometers deal with bulk properties of samples, whereas scanning microscope can map the distribution of remanent magnetization. In this study, a new scanning microscope based on a low-temperature DC superconducting quantum interference device (SQUID) equipped with an in-situ magnetization/demagnetization device was developed. To realize the combination of sensitive instrument as SQUID with high magnetizing/demagnetizing fields, the pick-up coil, the magnetization/demagnetization coils and the measurement mode of the system were optimized. The new microscope has a field sensitivity of 250 pT/$\sqrt{}$Hz at a coil-to-sample spacing of ~350 µm, and high magnetization (0–1 T)/ demagnetization (0–300 mT, 400 Hz) functions. With this microscope, isothermal remanent magnetization (IRM) acquisition and the according alternating field (AF) demagnetization curves can be obtained for each point without transferring samples between different procedures, which could result in position deviation, waste of time, and other interferences. The newly-designed SQUID microscope, thus, can be used to investigate the rock magnetic properties of samples at a micro-area scale, and has a great potential to be an efficient tool in paleomagnetism, rock magnetism, and magnetic material studies.

1. Introduction

Magnetic properties of rocks are the prerequisites for paleomagnetism, environmental magnetism, and magnetic material studies [1]. Conventional methods of measuring geological samples are divided into two categories. The first category involves in measuring the magnetization of the bulk samples. With such methods, the heterogeneity of magnetization within the sample cannot be distinguished [2,3]. Further details on the magnetic properties of samples can be obtained by cutting samples into smaller sizes, which is a difficult and costly task. The second category involves in scanning and mapping the magnetic field distribution using scanning superconducting quantum interference device (SQUID) microscope (SSM). Among all magnetic imaging techniques, SSM is one of the most sensitive techniques in terms of the absolute magnetic field [4–8]. SSM can achieve an ultrafine-scale magnetic distribution, which is a useful tool in many fields, such as deducing deep-sea environmental changes and changes in the Earth's early interior and atmosphere [9–11]. However, SSM cannot generate an in-situ magnetic field for magnetization/demagnetization, which is used to obtain remanent acquisition curves. To resolve these problems, an updated SSM technique equipped with an in-situ magnetization/demagnetization field called the in-situ magnetization SSM was developed in this study. The new technique can map the vertical component of the magnetic field of room-temperature samples and obtain the remanent acquisition curves (and AF demagnetization curves) at each point of the sample.

2. Experimental

The newly-designed in-situ magnetization SSM contains five parts: the cryogenic system, the magnetic field detection system, the sample moving platform, the magnetically shielded room (MSR), and the magnetization and demagnetization device system (Fig. 1).

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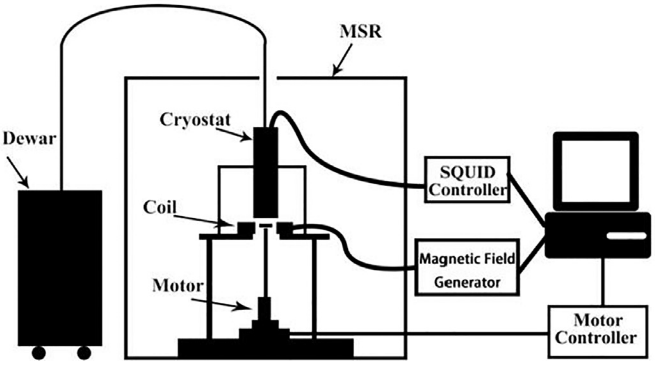

Fig. 1. The schematic of the in-situ magnetization SSM.

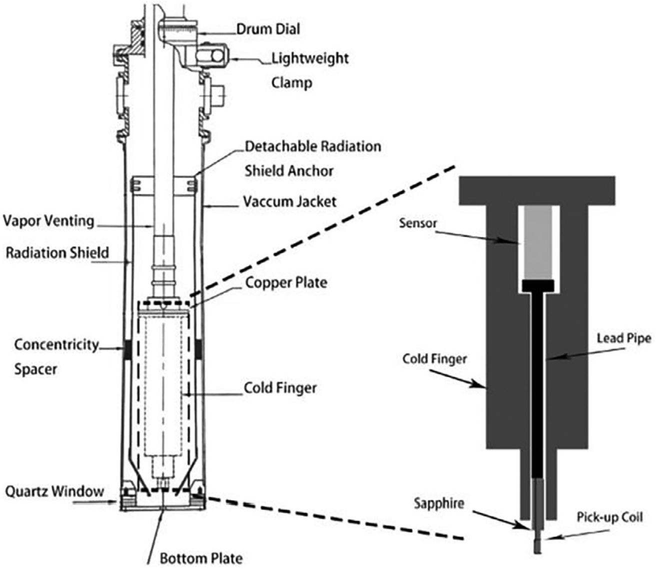

Fig. 2. The schematic of cryostat.

2.1. *Cryogenic system*

2.1.1. *Cryostat*

To provide a working temperature for the low-temperature (LT) SQUID, a cryostat was used to decrease the temperature and insulate the thermal exchange (Fig. 2). A commercial cryostat (ST-200, Janis Research Company LLC., USA), which is a continuous-flow research cryostat, was improved for the system demand in this study. The liquid helium reservoir was placed outside the MSR, and the cryostat continuously transferred liquid helium through a high-efficiency, super-insulated line from the reservoir to a copper plate inside the cryostat vacuum jacket. A cryostat consumes about 0.5 L/h when the system works stably [12]. A cold finger moved vertically and precisely to modify the distance between the pick-up coil and samples by adjusting the linear motion manipulator of the cryostat. The scanning distance is ~350 μm, including that the thickness of the sapphire window between the sample and the pick-up coil is 100 μm, the distance from the pick-up coil to sapphire window is ~100 μm, and the distance from the sample to the sapphire window is ~150 μm.

2.1.2. *Cold finger*

A copper cold finger was designed to house the sensor and the pick-up coil. The cold finger was fixed on the plate in the cryostat by four copper screws. The sensor and pick-up coil were thermally anchored on the cold finger. Before assembling the cold finger on the cryostat plate, Apiezon N grease was brushed on the junction surface and infused into the interior of the cold finger to improve thermal contact. The temperature detected at the coldfinger is achieved to 4.648 K, which measured by the diode thermometer installed on the coldfinger. The low temperature could make the superconducting state of the SQUID and the pick-up coil by means of conduction.

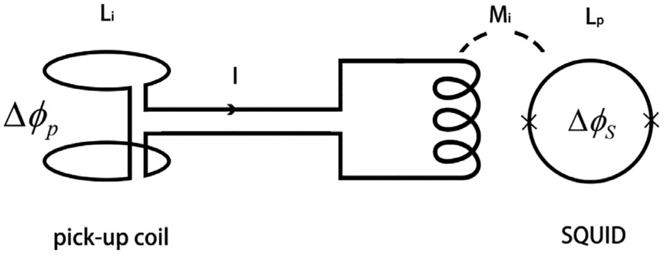

Fig. 3. The schematic diagram of the flux transmission loop.

2.2. *Magnetic field detection system*

2.2.1. *DC-SQUID sensor*

The SQUID microscope is based on a commercial SQUID sensor (Quantum Design Inc., USA). This sensor was housed in a niobium container, which can block interferences from the ambient environment. This container was also thermally anchored on the cold finger.

2.2.2. *Pick-up coil*

A pick-up coil, which is a crucial component that affects the sensitivity of the proposed the in-situ magnetization SSM system, can couple the external magnetic flux to the SQUID sensor. A one-order gradiometer, which has two coils connected reversely in series and can lower the ambient interference, was designed as the pick-up coil. The pick-up coil was wound on a sapphire bobbin with a 66 μm diameter niobium–titanium (Nb–Ti) wire. The diameter of the bobbin is 1 mm, and the baseline is 2.6 mm.

The pick-up coil is connected to the input coil on the SQUID chip (Fig. 3). The magnetic flux applied to the pick-up coil $\Delta\emptyset_p$ has a relationship with the output voltage: $\Delta\emptyset_S = \frac{M_i \times \Delta\emptyset_p}{(L_i + L_p)} = \frac{M_i \times U}{(L_i + L_p) \times 0.7}$, $L_i = 0.5\ \mu H$ is the inductance of the input coil. $L_p = 1.86\ \mu H$ is the inductance of the pick-up coil, and $M_i = 10.9\ nH$ is the mutual inductance between the input coil and the SQUID. U is the output voltage. The transfer function of our SQUID circuit is 0.7. The sensitivity of the pick-up coil is given by $B^{(p)} = \frac{\sqrt{S_f^{(p)}}}{n \times \pi \times r^2} = \frac{M_i \times U}{(L_i + L_p) \times 0.7 \times n \times \pi \times r^2}$, where $r = 0.5$ mm is the radius of the pick-up coil and $n = 10$ is the number of turns. We use a 1 kHz low pass analog filter and a digital 50 Hz notch filter in software. The system noise signal, whose amplitude is −2 mV to 2 mV, is shown in Fig. 4(a). The power spectrum of noise is shown in Fig. 4(b), and the field sensitivity is approximately 250 pT/$\sqrt{\text{Hz}}$.

2.3. *Sample moving platform*

The sample moving platform has three parts: Three motors, one controller, and one sample stage. This platform is installed inside the MSR (Fig. 5).

2.3.1. *Piezoelectric motors*

Three motors were utilized to move a sample in three directions simultaneously. Two of these motors were used to scan the sample on the horizontal plane to obtain the magnetic field distribution. The third motor was responsible for vertical movement which caused the magnetic field change for obtaining the