

## SQUID MAGNETOMETERS FOR MATERIAL RESEARCH

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### Introduction

Apart from SQUID-magnetometers for biomagnetic and geophysical research, such as multi-sensor systems for brain activity and rock magnetometers for long-core measurements [1], we have also developed SQUID-magnetometers for the study of magnetic properties of electrically conducting and non-conducting materials. In our case this material research concerns the field dependence of the magnetization and the frequency-dependent behaviour of the dynamic susceptibility of magnetic superconductors and magnetic isolators. In this paper we describe a frequency-sweeping SQUID magnetometer for the dynamic measurements and a field-sweeping SQUID system for the quasi-static experiments, both of which are considerably more sensitive than conventional systems. In order to increase the dynamic range of the field-sweeping system we developed a special feedback mode called the reset killer, which will also be described.

### Frequency-sweeping SQUID susceptometer

The frequency-sweeping SQUID susceptometer is equipped with a coil system consisting of a primary coil, which generates an alternating magnetic field, and an astatic secondary coil set, which is part of the superconducting flux transformer circuit coupled to the SQUID (fig. 1). A background field can be applied by means of a superconducting magnet.

The susceptibility  $\chi$  of a sample placed in one of the secondary coils results in a flux change of  $\Delta\phi_S$ , which induces a flux change in the SQUID of

$$\Delta\phi_{SQ} = \Delta\phi_S \cdot M_{SQ}/L_{FT},$$

with  $L_{FT}$  being the total self-inductance of the flux transformer loop and  $M_{SQ}$  the mutual inductance between the SQUID and this loop. The ratio  $\Delta\phi_{SQ}/\Delta\phi_S$  is known as the flux transfer factor  $f$ . The output voltage of the

SQUID system can be related directly to the susceptibility. Because of the

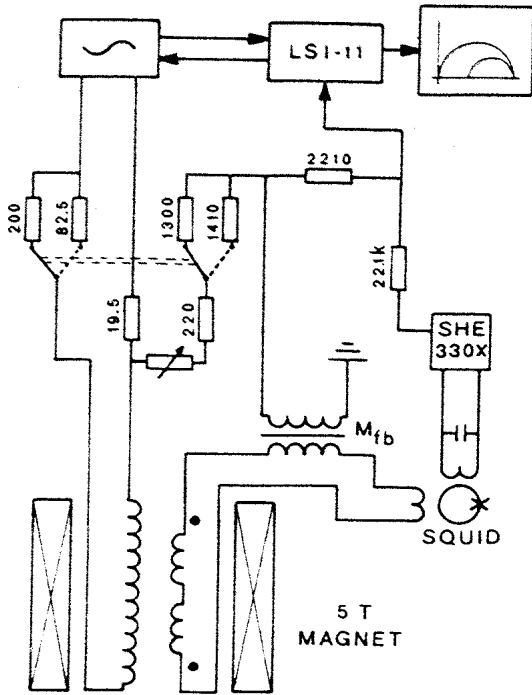


Fig. 1. Schematic diagram of the frequency-sweeping SQUID susceptometer.

frequency-independent response of the system, a frequency-swept operation is possible from about 1 kHz to very low frequencies ( $\approx 2$  mHz). Making use of an additional mutual inductor  $M_{fb}$ , of which the secondary is part of the flux transformer loop and the primary is connected to the external feedback output

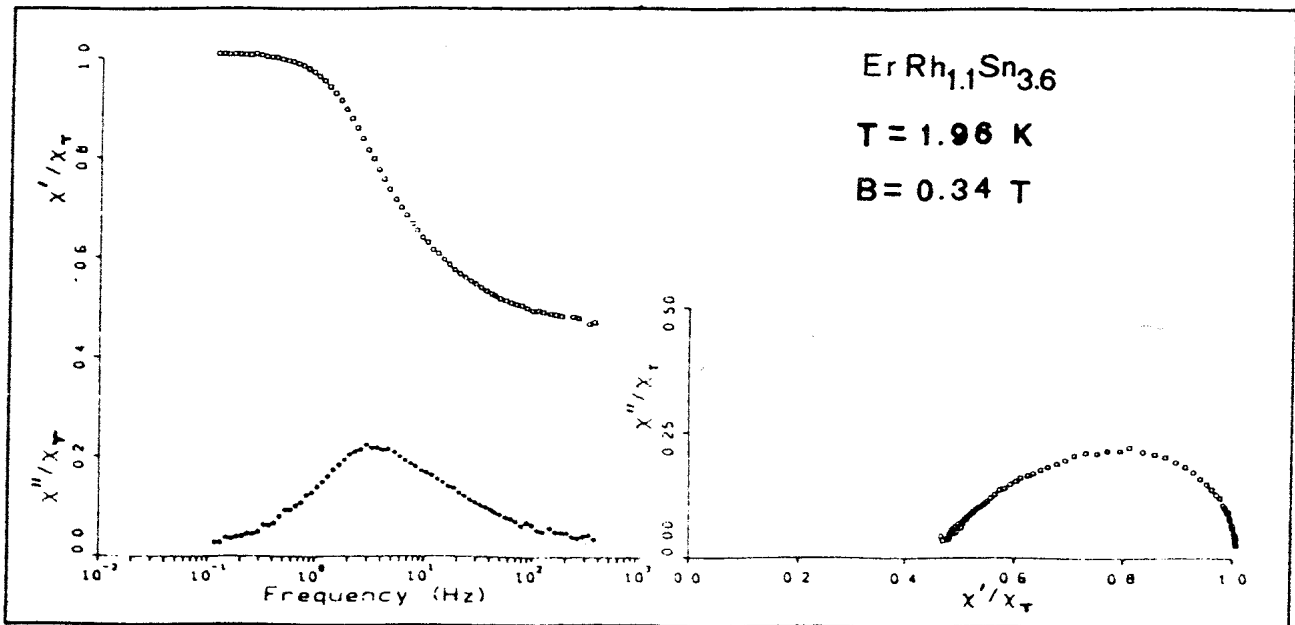


Fig. 2. Susceptibility of  $ErRh_{1.1}Sn_{3.6}$  at  $T = 1.96$  K and  $B = 0.34$  T.

of the SQUID electronics, the bandwidth of the system can be extended up to about 3 kHz. The inductor  $M_{fb}$  is primarily incorporated in order to compensate for imbalance effects of the primary-secondary coil set [2].

The frequency-sweeping technique proved to be very successful especially for the measurement of spin-lattice relaxation phenomena in thermally isolated samples and relaxation at sharp magnetic phase transitions such as is the case in spin-flop systems. As an example we present the frequency-dependent behaviour of the magnetic superconductor  $\text{ErRh}_{1.1}\text{Sn}_{3.6}$  at  $T = 1.96$  K and  $B = 0.34$  T in the paramagnetic region (fig. 2). The isothermal limit  $\chi_T$  is reached at about  $10^{-1}$  Hz and the adiabatic susceptibility at about 500 Hz.

#### The field-sweeping SQUID magnetometer

Field-sweep experiments with a SQUID magnetometer are, in practice, severely hampered by the fact that the mutual inductances between the superconducting magnet and each of the two pick-up sections of the sensing coil cannot be made exactly equal. When the applied field is varied, the current change in the flux transformer due to the imbalance will usually be considerably larger than the current change, which reflects the field-dependent behaviour of the magnetization of the sample. This imbalance effect can be eliminated in the same way as in the frequency-sweeping system by introducing an additional mutual inductor in the flux transformer circuit (fig. 3). The current  $i_c$  through the primary of this inductor is derived from the power supply of the superconducting magnet and satisfies the condition  $\Delta M_{ps} i_p = -M_c i_c$ , where  $\Delta M_{ps}$  represents the imbalance of the pick-up coil set,  $M_c$  the mutual

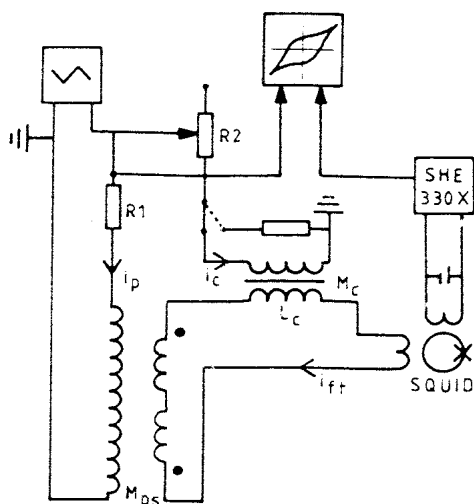


Fig. 3. Schematic diagram of the field-sweeping SQUID magnetometer.

inductance of the additional inductor and  $i_p$  the current through the magnet. In this way the imbalance is perfectly compensated.

In our system the flux transformer circuit and superconducting magnet are placed in a dilution refrigerator in order to have the appropriate temperature range for the research on magnetic superconductors at our disposal. The two sections of the pick-up coil each consist of 20 turns of 0.15 mm NbTi wire (CuNi-matrix) and have a diameter of 5.5 mm. The effective self-inductance of one section is 1.96  $\mu\text{H}$ . The superconducting magnet has a bore of 35 mm and a coil constant of 0.115 T/A. The mutual inductance between the magnet and one of the pick-up sections is approximately 60  $\mu\text{H}$ .

The imbalance effect can be illustrated by the following practical example. Assuming an imbalance of the two sections of 1% and a current change in the superconducting magnet of 1 mA, a signal of about 1000  $\phi_0$  will be coupled to the SHE RF-SQUID, as can be calculated by using a flux-transfer factor of  $3.1 \times 10^{-3}$ . The SQUID signal due to the sample's magnetization is, of course, dependent on the size and susceptibility. A single crystal of  $\text{ErRh}_{1.1}\text{Sn}_{3.6}$  with a volume of  $1.1 \times 10^{-9} \text{ m}^3$ , having a susceptibility of 0.28 at 4.2 K, will result in a SQUID signal of about 100  $\phi_0$  per mA current change. This means that only 9% of the SQUID signal results from the sample.

As mentioned above the imbalance effect can be eliminated by means of an extra inductor. In our case we have chosen a toroidally shaped balance inductor placed inside a lead shield in order to minimize the interfering effect of external magnetic fields. The secondary is wound on an araldite core with a diameter of 9 mm and consists of 30 turns of 5 mm diameter. The primary also

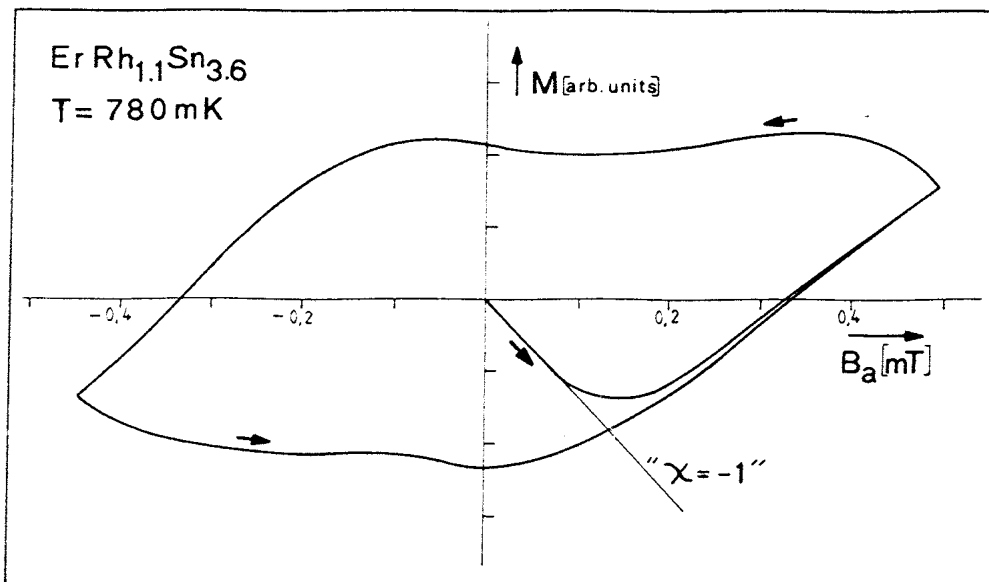


Fig. 4. DC-magnetization versus applied field  $B_a$  for  $\text{ErRh}_{1.1}\text{Sn}_{3.6}$  at  $T = 780 \text{ mK}$ .

consists of 30 turns with a diameter of 5.5 mm. Primary and secondary are decoupled for high-frequency interference signals by placing an electrically conducting shield between them. The mutual inductance is 0.99  $\mu\text{H}$  whereas the self-inductance of the secondary is 0.86  $\mu\text{H}$ , so that the flux transfer factor is only slightly reduced. In practice balancing could be obtained by making  $i_c$  about three times larger than  $i_p$ .

As an example the DC-magnetization versus the applied field is presented in fig. 4 for the magnetic superconductor  $\text{ErRh}_{1.1}\text{Sn}_{3.6}$  at  $T = 780$  mK. At this temperature the material behaves as a superconductor with  $H_{c1} \approx 80$  A/m. The hysteretic behaviour of the compound is shown in this figure.

#### Reset killer

In some experiments the signal of the sample is much larger than the full scale deflection of the standard SQUID electronics. In principle the range of a SHE-SQUID system is unlimited because of the autoreset at  $\pm 500 \phi_0$  ( $\Delta I_S > 50 \mu\text{A}$ ) in the sensitivity \*1 mode. However, the reset does not take place instantly but requires a time of 100 ms in which the output is disabled and at the end of this period the output will go to the nearest lock point. If the signal changes at a rate greater than  $1 \phi_0/100$  ms the system will lose an integral number of flux quanta. This effect is especially serious in field-sweep experiments if the magnetization is a rapidly changing function of the applied field. As a result it is difficult to reconstruct the magnetization curve.

The above problem can be solved by extending the dynamic range of the SQUID system. This can be achieved by introducing an additional integrator in an extra flux-feedback loop (fig. 5). The integrator, time constant  $\tau$ , results in an output voltage  $V_{\text{out}_2}$  and together with a Voltage-to-Current Converter (VCC), transfer function  $A_2$ , and the mutual inductance  $M_{fb}$  the flux feedback is realized. The following expressions can be derived for the two signal outputs  $V_{\text{out}_1}$  and  $V_{\text{out}_2}$ ,

$$\frac{V_{\text{out}_1}}{\phi_S} = f A_1 \frac{p\tau_{\text{eff}}}{1 + p\tau_{\text{eff}}} \quad \text{with} \quad \tau_{\text{eff}} = \frac{\tau}{fA_1A_2M_{fb}},$$

$$\frac{V_{\text{out}_2}}{\phi_S} = \frac{1}{M_{fb}A_2} \frac{1}{1 + p\tau_{\text{eff}}},$$

$\phi_S$  being the signal flux,  $f$  the flux transfer factor and  $A_1$  the transfer function of the SQUID electronics in the internal feedback mode.

The high-frequency signals are still available at the normal SQUID output having the dynamic range of  $\pm 500 \phi_0$ . There is no effect from the additional

feedback circuit.

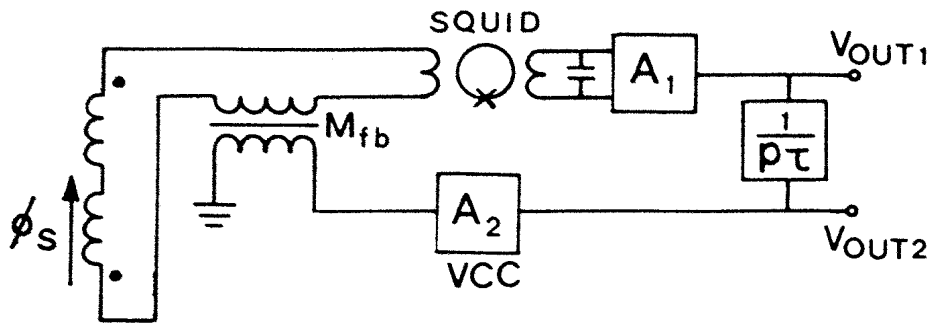


Fig. 5. Schematic diagram of the reset killer

The low-frequency signals are present at output 2 and the dynamic range is determined by the feedback circuit and the maximum output voltage of the integrator. The dynamic range can be easily varied by changing  $A_2$ .

The integrator consists of a unity gain differential amplifier input stage, a variable attenuator to provide for a continuous adjustment of the time constant and an inverting integrator with a switchable time constant. The time constant can be varied between 6.8 ms and 680 s.

The VCC consists of an inverting amplifier with variable gain  $\beta$  ( $0 < \beta < 1$ ) used for calibration purposes and a voltage-regulated current source, including a booster in order to have a maximum output current of 200 mA available. It is possible to change the transfer function of the VCC by means of an attenuation factor  $F = 10^{-n}$  with  $n = 0, 1, 2, 3$  or 4 so that the total transfer function becomes  $A_2 = \beta \cdot F$ .

The mutual inductor  $M_{fb}$  can be the same as the one used in the field-sweeping SQUID magnetometer. Thus the reset killer can be easily implemented in this magnetometer system. The dynamic range of the system is equal to

$$V_{out_2} \Big|_{max} \cdot M_{fb} A_2.$$

In our situation the dynamic range increases by a factor  $F \cdot 10^3$  so that an extension of a factor of 1000 is possible. In experiments the reset killer proved to be a useful instrument, greatly facilitating the field-sweep magnetometer in case of signals outside the dynamic range of normal SQUID-systems. We thank Mr. A.J. van Dalfsen for his work on the reset killer.

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