Development of an extraction type magnetometer under low temperature and high magnetic fields over 20 T by the hybrid magnet

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Abstract. An extraction-type magnetometer has been developed, which is performed under the low temperature of 0.5-0.6 K using a 3He-refrigerator and high magnetic fields up to 18 T using a superconducting magnet (SM) and 27 T using a hybrid magnet (HM). Magnetization curves can be measured with the absolute value over 0.0005 emu using SM and 0.005 emu using HM. We confirmed that the resolution is 0.001 emu for SM and 0.005 emu for HM. For demonstrating the ability of the magnetometer, high field magnetization curves of NdCu₄Ag in 0.6-4.2 K are presented.

1. Introduction
Magnetization is one of the most important properties for investigation of magnetic materials. Especially, the measurements under multi-extreme conditions such as high magnetic field, high pressure and low temperature provide us useful information on the intrinsic properties. Therefore, many research groups have developed magnetometers that are utilized under multi-extreme conditions and showed interesting results so far [1-6]. At the High Field Laboratory for Superconducting Materials (HFLSM), high-field magnetization curves have been measured by a sample extraction magnetometer combined with hybrid magnets (HM). To avoid the influence of large leakage magnetic field from HM, the sample was extracted by an air-piston technique. However it is difficult to precisely control the sample motion using this technique. On the other hand, HFLSM also developed a vibrating sample magnetometer system using a nonmagnetic ultrasonic motor, which is a small system and can be equipped on HM [7]. The result of the development indicated that the non-magnetic ultrasonic motor is very useful for precisely controlling the sample position and motion in the large leakage of the magnetic field generated by HM. Recently, HFLSM developed another extraction type magnetometer using the ultrasonic motor. The magnetometer has been performed under pressures up to 1.2 GPa using a miniature high-pressure cell in magnetic fields up to 27 T and in the temperature range of 1.7-300 K [8,9].

In this work, we have developed a new extraction-type magnetometer so that the magnetization measurement can be carried out under temperature \( T \) of 0.5 K and high magnetic fields up to 27 T. Obtained results show that magnetization curves can be measured with the absolute value over 0.0005 emu using a superconducting magnet (SM) and 0.005 emu using HM. In addition, we confirmed that the resolution is approximately 0.001 emu for SM and 0.005 emu for 28T-HM. For demonstrating the
ability of the instrument, we will present results of the high-field magnetization curves for NdCu₄Ag in 0.6-4.2 K under magnetic fields up to 27 T using 28T-HM.

2. Construction of the magnetometer

In Fig. 1, we show the schematic diagrams of the developed magnetometer in magnetic fields up to 28 T produced by 28T-HM and in the temperature range from 0.5 to 4.2 K. This magnetometer mainly consists of an extraction system driven with the nonmagnetic ultrasonic motor, pickup-coils, a 3He refrigerator, a personal computer (PC) and the hybrid magnet (a water-cooled magnet WM and a superconducting magnet (SM)). This system is designed on the basis of the magnetometer reported by Koyama et al. [2].

The refrigerator consists of a 4He-cryostat with a 4He gas handling system and a 3He insert with a 3He gas handling system. The 4He insert is utilized for the condensation of the 3He gas. The sample holder (Delrin) including the sample is cooled directly with liquid 3He. Removing the 3He insert and 3He gas-handling system, we can also measure the magnetization in a wide temperature range 1.7 K ≤ T ≤ 300 K. In our system, the ultrasonic motor (FUKOKU) controls the sample motion and position precisely by a built-in optical encoder. The sample set in a sample holder is extracted in the pair of pickup coils using the timing belt that connects the ultrasonic motor. The motor can lift a loading weight of 600 g and control the sample position with accuracy of ± 0.1 mm. The maximum speed for the extraction is 70 mm/s. The extraction and the sample position are controlled by PC.

Figure 1. Schematic diagram of the developed magnetometer which is installed in a hybrid magnet consisting of a water cooled magnet and a superconducting magnet.

Figure 2 shows the cross sectional view of axial pickup coils for detecting the magnetization, the sample holder with a driving rod, and the lower port of the 4He-cryostat and the 3He-cryostat. The inner diameters of the 4He cryostat and 3He insert are 44 mm and 26 mm, respectively. The outer diameter of the sample holder is 6.0 mm. The pickup coils are connected in series opposition for compensating a noise signal arising from the fluctuation of the applied magnetic field and located at the center of the magnet. The coil was made by winding an insulated copper wire of 0.06 mm diameter
about 4900 turns on a polycarbonate coil bobbin with the inner diameter of 7.0 mm. The width of each coil is 7.0 mm and the distance between the coil centers is 13.0 mm. In order to obtain the best compensation, the number of turns was carefully adjusted in a uniform ac magnetic field of \( \sim 100 \) Hz. The lower part of the driving rod is connected to the sample holder. A thermometer (Cernox) is at the center of the coils. The thermometer was calibrated in magnetic fields at 0.5 K. For the magnetization measurement, the sample holder including the sample is linearly moved up and down through the pickup coils by the driving rod connected to the timing belt. Usually, the speed of the motion is 55.4 mm/s and the sample is shifted at the distance of 15 mm. The bottom of the magnetometer is fixed using Al foil and a Cu-shield tube in the cryostats.

**Figure 2.** Cross sectional view of axial pickup coils for detecting the magnetization, the high-pressure cell assembly with a driving rod, and the lower port of the \(^4\)He-cryostat.

The voltage signal induced in the pickup coils by the movement of the sample is amplified by 10-10,000 times using a low noise pre-amplifier (DL INSTRUMENTS) that has a band pass filter. The amplified signal is directly acquired in the memories of the PC though a 16 bit analog-to-digital (A/D) converter (TEAC). The magnetization is calculated from the integration of an output voltage by the computer. The calculation method for magnetization was described in the previous paper [2]. The magnetic field is taken to the PC by GPIB and RS-232C standards.

3. **Performance test**

3.1. **Magnetization measurement of Pd**

In order to calibrate the pickup coil, the magnetization of a standard Ni sample (60.92 mg) was measured at 4.2 K using a superconducting magnet. In addition, we measured the magnetization of Pd (271.9 mg) at 4.2 K using the superconducting magnet (18T-SM) in order to estimate the ability of the magnetometer. Figure 3 shows the magnetization curve of Pd at 4.2 K in field range of 0 - 18 T (a), 0 - 0.3 T (b) and 17-18T (c). As seen in these figures, the magnetization can be measured for the absolute value over 0.0005 emu and the resolution is 0.0003 emu using the superconducting magnet (SM).
Figure 3. Magnetization curves of Pd in field range of 0 -18 T (a), 0 - 0.3 T (b) and (c) 17-18T (c) at 4.2 K.

3.2. Magnetization measurements of NdCu₄Ag
Intermetallic compound NdCu$_4$Ag with the cubic MgCu$_4$Sn-type crystal structure is an antiferromagnet [10]. Recently, Adachi and Koyama reported that the Néel temperature $T_N$ is 4.3 K using the single crystal of NdCu$_4$Ag [11]. Their measurements show that the magnetization was not saturated even a magnetic field of 18 T at 2 K. In this work, we carried out high-field magnetization measurements of a NdCu$_4$Ag single crystal with the weight of 10.35 mg in magnetic fields up to 27 T to examine the ability of the present apparatus. We show only some of the data to demonstrate the ability of the magnetometer. Details of the experimental results and discussions will be presented elsewhere.

![Graph](image)

**Figure 4.** High-field magnetization curves of the single crystal of NdCu$_4$Ag at 4.2 K, 1.7 K and 0.6 K in fields up to 27 T (a) and 15 T (b) using 28T-HM (a).

Figure 4(a) shows the high-field magnetization curves of the single crystal of NdCu$_4$Ag at 4.2 K, 1.7 K and 0.6 K using 28T-HM. The single crystal was prepared by Kitai [12]. For measuring the magnetization at 4.2 K, we utilized only WM in magnetic fields up to 15 T. The temperature decreases down to 0.6 K for 28T-HM though it reaches to 0.5 K using 15T-SM. The sample-extraction rate was controlled for keeping the temperature 0.6 K.

At 4.2 K, the magnetization curve shows a paramagnetic behavior and we cannot see the field-induced phase transition in $B < 15$ T. On the other hand, we clearly observed metamagnetic transitions in the vicinity of 5 T and 13 T at 1.7 K, which increases with decreasing temperature. At 0.6 K, the
metamagnetic transitions occur in the vicinity of 6 T and 14 T. The magnetic moment increases with increasing a magnetic field over 15 T, which is not saturated even in 27 T. Figure 4(b) shows the magnetization curves of NdCu$_4$Ag at 1.7 K in $0 \leq B \leq 5$ T using HM. Figs 4 (a) and 4(b) indicate that magnetization is measured with the absolute value over 0.005 emu and the resolution is 0.005 emu in $B < 24$ T using present HM.

It should noted that any big noise due to a ripple field of WM is not seen even in a maximum field of 15 T, as seen in Fig. 4(a). However, when we generated WM under the HM mode, the data are scattered in magnetic fields over 24 T. This is due to that the magnetometer detects noise field from WM of 28T-HM. Therefore we believe that high-resolution data is obtained if the present magnetometer is utilized in high-quality magnetic field even using HM. Consequently, we succeeded in the development of high-sensitive magnetometer that is utilized under low temperature of 0.6 K and high magnetic fields over 20 T.

4. Summary
We developed the extraction-type magnetometer that is performed under the temperature of 0.5-0.6 K using the $^3$He-refrigerator, and high magnetic fields up to 18 T using the superconducting magnet (SM) and 27 T using the hybrid magnet (HM). Magnetization curves can be detected with the absolute value over 0.0005 emu using SM and 0.005 emu using HM. We confirmed that the resolution is 0.001 emu for 18T-SM and 0.005 emu for 28T-HM. Using this magnetometer, we observed the metamagnetic transitions of NdCu$_4$Ag in 0.6-1.7 K.

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