Development of an extraction type magnetometer under high pressure and high magnetic fields over 200 kOe in the hybrid magnet

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Abstract. An extraction-type magnetometer has been developed, which is performed under pressures up to 12 kbar using a miniature high-pressure clamp-cell, in magnetic fields up to 270 kOe using our hybrid magnet and at the temperature range from 1.5 to 300 K. Magnetization curves can be measured for absolute value over 0.04 emu. We confirmed that resolution is about ±0.01 emu under high pressures and high magnetic fields if a sample has the magnetic moment of about 3 emu. For demonstrating the ability of the instrument, high field magnetization curves for SmMn$_2$Ge$_2$ under high pressures are presented.

1. Introduction
Magnetization is one of the most important properties for investigation of magnetic materials. Especially, measurements under multi-extreme conditions such as high magnetic field, high pressure and low temperature provide us useful information on intrinsic properties. Therefore, many research groups have developed magnetometers that are utilized under multi-extreme conditions and showed interesting results so far [1, 2, 3, 4, 5, 6]. At the High Field Laboratory for Superconducting Materials (HFLSM), high field magnetization has been measured by the sample extraction magnetometer combined with the hybrid magnet (HM). To avoid influence of a large leakage magnetic field from HM, the sample was extracted by an air-piston technique. However it is difficult to precisely control sample motion using this technique. On the other hand, HFLSM also developed a vibrating sample magnetometer system using a non-magnetic ultrasonic motor, which is a small system and can be equipped on HM [7]. The result of the development clearly showed that the non-magnetic ultrasonic motor is very useful for precisely controlling the sample position and motion in large leakage of the magnetic field generated by HM.

In this paper, we present construction of a new extraction-type magnetometer that is utilized in HM at HFLSM, and typical results of high field magnetization measurements for SmMn$_2$Ge$_2$ under pressures up to 12 kbar.

2. Construction of magnetometer
In Fig.1, we show the schematic and block diagrams of the developed system for magnetization measurement under magnetic fields up to 280 kOe produced by HM, high pressures up to 12
Figure 1. Schematic (a), (b) and block (c) diagrams of the developed magnetometer.

kbar and in the temperature range from 1.5 to 300 K. This magnetometer mainly consists of an extraction system with a nonmagnetic ultrasonic motor, pickup-coils, a nonmagnetic high-pressure cell, and HM. This system is designed on the basis of the magnetometer reported by Koyama et al. [2]. The construction of the ultrasonic motor used in our system was described in the previous paper [7]. In our system, the ultrasonic motor (FUKOKU) controls the sample motion and position precisely by a built-in optical encoder. The sample in the pressure cell is extracted in the pair of the pickup coils using a timing belt that connects the ultrasonic motor (Fig. 1(a)). The motor can lift loading weight of 600 g and control the sample position with precision of ±0.1 mm. Maximum speed for the extraction is 70 mm/s. The extraction and sample position are controlled by a personal computer (PC).

Figure 1(b) shows the cross sectional view of axial pickup coils for detecting magnetization, the pressure cell assembly with a driving rod, and the lower part of a cryostat. The pickup coils are connected in series opposition for compensating a noise signal arising from fluctuation of applied magnetic field and located at center of HM. The coils were made by winding an insulated copper wire of 0.06 mm diameter about 6000 turns on a polycarbonate coil bobbin with an inner diameter of 10.5 mm. The width of each coil is 8.0 mm and the distance between the coil centers is 10 mm. Thermometers (Cernox) were set just on the pressure cell and at the center of the coils. For magnetization measurement, the cell 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 intervals of 25 mm. A brass tube with a heater covers the coil bobbin. The bottom of the magnetometer is fixed using Al foil and a Cu-shield tube in the cryostat. The temperature of the sample can be controlled using the heater, and the data are taken to the PC (Fig. 1(c)).

The voltage signal induced in the pickup coils by the movement of the sample is amplified from 10 to 10,000 times using a low noise pre-amplifier (DL INSTRUMENTS) that has a band pass filter. The amplified signal is directly acquired in memories of 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 PC. 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.

The pressure cell is also utilized in a commercial SQUID magnetometer for magnetization measurement under high pressure as well as the present magnetometer (Fig. 1(b)). The original design of this cell was presented in the previous paper [8]. The outer and inner diameters of the cell cylinder are 8.5 and 3.0 mm, respectively. The clamp cylinder and locking screw are made
of CuBe alloy. The piston and piston cap are made of zirconia. Sample is compressed in a Teflon capsule filled with liquid pressure-medium, mixture of two types of fluorinert (FC70 + FC77), in the cylinder. The produced pressure at low temperatures was calibrated by measuring the pressure dependence of a superconducting transition temperature of Sn [9].

In order to calibrate the pickup coil and estimate data quality, magnetization measurements for standard Ni samples were performed. Figure 2 shows the typical result of high field magnetization curves of Ni with and without the pressure cell at 285 K. The influence on the magnetization data from ripple noise due to the water-cooled magnet (WM) is negligibly small even in \( H > 150 \) kOe if we use the sample having the magnetization of 3 emu. The resolution is about ±0.01emu under high pressure and high magnetic field.

![Figure 2. Magnetization curves of Ni with and without the pressure cell at 285 K.](image)

![Figure 3. Temperature dependence of magnetization of SmMn\(_2\)Ge\(_2\) at various pressures.](image)

3. Application of the magnetic measurements of SmMn\(_2\)Ge\(_2\)

The magnetic state of SmMn\(_2\)Ge\(_2\) is ferromagnetic (FM) in the temperature range 150 K (\(= T_{t1}\)) \(< T < 350 \) K (\(= T_C\)), antiferromagnetic (AFM) at 100 K (\(= T_{t2}\)) \(< T < 150 \) K, and it becomes a re-entrant ferromagnetic (RFM) state below 100 K [10]. Figure 3 shows the \( T \) dependence of magnetization \( M \) of polycrystalline SmMn\(_2\)Ge\(_2\) under 1 kOe at various pressures \( P \) up to 12 kbar using the pressure cell and a SQUID magnetometer (Quantum Design). By applying pressure \( P \), \( T_{t1} \) increases, whereas \( T_{t2} \) decreases, which was consistent with the reported data [11, 12, 13]. As seen in this figure, the magnetic state transforms from the RFM to AFM by applying \( P \) at 77 K.

Using our extraction-type magnetometer, we measured magnetization curves of SmMn\(_2\)Ge\(_2\) at 77 K and at various pressures up to 12 kbar in magnetic fields up to 270 kOe generated by HM, as shown in Fig.4. Since the sample weight is very small (8.28 mg), the data are scattered in higher fields. This is due to ripple noise from WM. In this system, the magnetization cannot be detected below 0.04 emu.

However, we clearly observed the metamagnetic transition with a small hysteresis under high pressure, as shown in Figs. 4 and 5. The sample space in the pressure cell is 2.4 mm in diameter and 4 mm in length. Therefore, precise data of magnetization value can be obtained even in 270 kOe if we use the sample as much as possible (~50 mg). At present, the magnetometer is also utilized in other 200 kOe-class superconducting magnet in HFLSM, in order to measure the detailed magnetization process under high pressure. Static high magnetic field has the advantage of suppressing eddy current effect on a bulky and metallic sample, compared with
pulsed fields. Therefore, we believe that the magnetometer system developed in this work will provide important information about high-field and high-pressure effect for metallic materials.

4. Conclusion
We present the extraction-type magnetometer that is performed under pressures up to 12 kbar using the pressure cell, in magnetic fields up to 270 kOe using our hybrid magnet. Magnetization can be measured for the absolute value over 0.04 emu. We confirmed that the resolution is about ±0.01 emu under high pressure and high magnetic field if a sample has the magnetic moment of about 3 emu. Using this magnetometer, we detected the pressure-induced metamagnetic transition in SmMn$_2$Ge$_2$.

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