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# The development of a high pressure micro-cell for magnetization and specific heat measurements: the effect of pressure on the magnetism in CeAg

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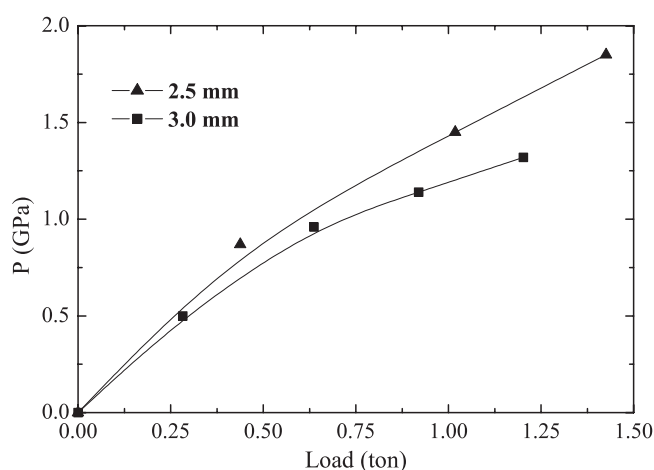
## Abstract

A micro-pressure cell has been developed for measurements of specific heat and magnetization by using a commercial SQUID magnetometer. This small piston-cylinder device can be used up to 2 GPa. In this paper, we report on the efficiency of our micro-cell and present some results of the pressure effect on the magnetic and thermal properties of CeAg.

## 1. Introduction

Many investigations of the electric and magnetic properties of intermetallic compounds have been performed. Magnetization and specific heat measurements are amongst the most important physical experimental methods used. During the last two decades, many investigations of strongly correlated electrons systems such as Ce-, Yb- and U-based intermetallic compounds have been extensively carried out. In these research works, magnetization and specific heat have been often measured to obtain fundamental information for interpretation of their physical properties. Recently, these measurements have been actively attempted under high pressures because such strongly correlated electrons systems (SCES) have ground states which are quite sensitive to external pressure. Therefore, it is very important to advance the high pressure generation techniques for these measurements in order to understand the SCES and discover new interesting physical phenomena such as exotic superconducting states in CePd<sub>2</sub>Si<sub>2</sub> [1], YbInCu<sub>4</sub> [2] and UGe<sub>2</sub> [3]. Thus, there is an urgent need to develop a more convenient pressure cell for these measurements. Several years ago, we developed a long type hydrostatic pressure cell for studying magnetization by using a commercial SQUID magnetometer (Quantum Design) [4]. But this pressure cell is very

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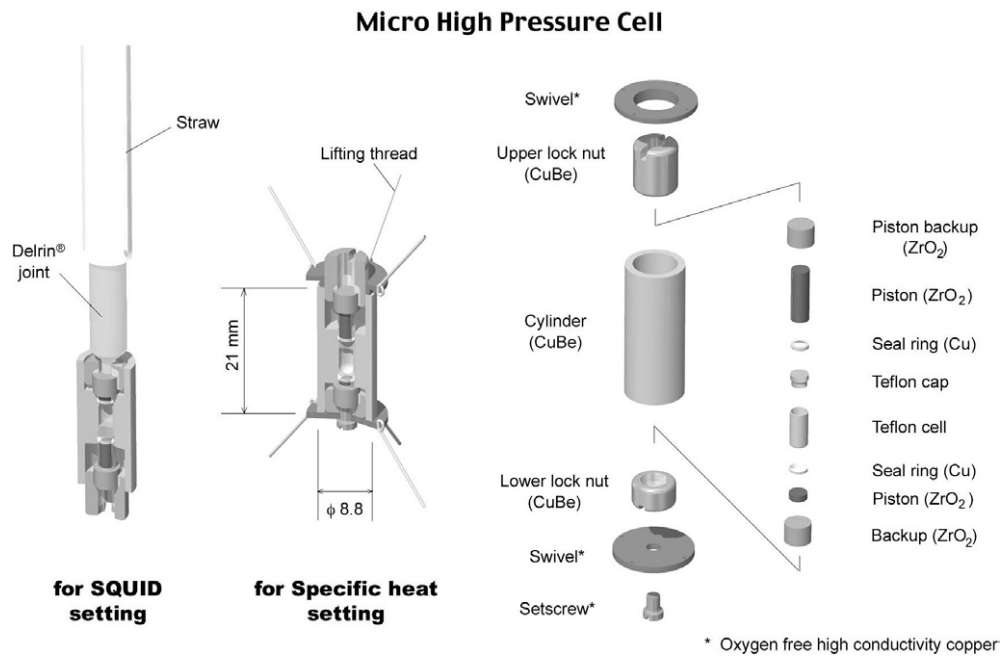
**Figure 1.** The pressure at low temperatures as a function of the applied force at room temperature for the test cell.

heavy and its maximum pressure is not so high. To improve these weak points, we undertook a development of a new high pressure micro-cell. In this paper, we report on the design of this high pressure micro-cell. As typical examples of measurements using this new cell, we will show results of both magnetization and low temperature heat capacity measurements under hydrostatic pressures up to  $P = 1.37$  GPa in the Ce-based ferromagnetic compound CeAg.

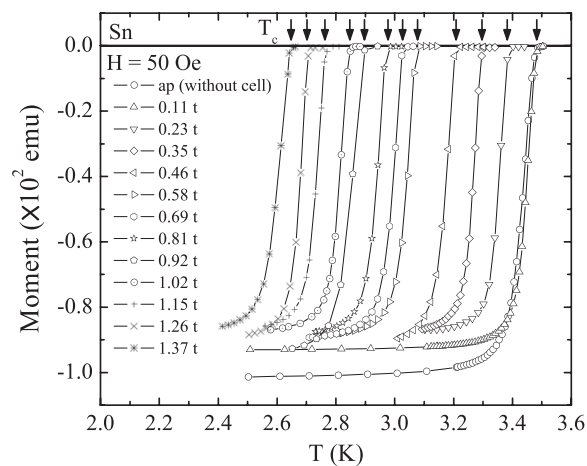
## 2. Experimental details

### 2.1. Design of the high pressure micro-cell

The basic idea of the cell is similar to the former designed stick type high pressure cell [4, 5]. The outer diameter of the new micro-cell is the same as the former one but the length is minimized down to about 21 mm. First, to get information for the minimization of the cell size and the elevation of the highest pressure we manufactured two types of pilot high pressure cells by way of trial. The inner diameters of those two pilot cells are 2.5 and 3 mm, respectively, and the lengths of both cells are 50 mm. To check the delimitation of the minimization of cell size, we estimated the pressure efficiency of each pilot cell (inner size 2.5 and 3 mm). Figure 1 shows the pressures at low temperatures as a function of applied force at room temperature for the pilot cells. The pressures at low temperatures were determined from the superconducting transition temperatures of Sn (for example: see figure 3). The pressure efficiency of the 2.5 mm inner cell was about 1.2 times better than that of the 3.0 mm inner cell at applied force 1.25 ton, as is evident from figure 1. The maximum pressure was 2.0 GPa and 1.4 GPa for the 2.5 and 3.0 mm inner cells, respectively. We adjusted more carefully to elevate the highest pressure and minimize the size of the pressure cell. Finally, the optimum sizes of cylinder are 8.8, 2.7 and 21 mm in outer diameter, inner diameter and length, respectively. A schematic drawing of the new high pressure micro-cell is shown in figure 2. The cylinder, and the upper and lower nuts of the high pressure micro-cell are made of hardened CuBe alloy (C1720B-HT). A nonmagnetic  $ZrO_2$  is used as a piston and a backup. The high pressure sample space is sealed by a Teflon cell technique with Cu seal rings. A mixture of Fluorinert FC70:FC77 = 1:1 or Daphne 7373 was used as the pressure transmitting media. The total weight of this cell is about 9 g. We can measure magnetizations and specific heat under the same pressure condition



**Figure 2.** A schematic drawing of the high pressure micro-cell.

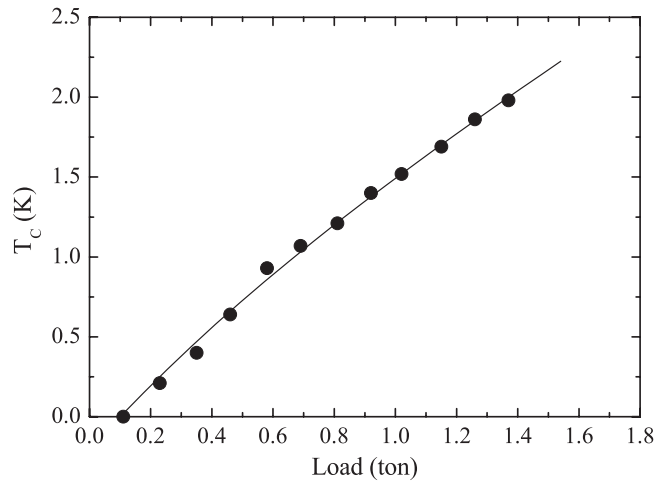


**Figure 3.** The temperature dependences of magnetization of Sn under various pressures.

by using this micro-pressure cell. The magnetization was measured by a commercial SQUID magnetometer, and the specific heat by a conventional adiabatic method.

## 2.2. High pressure micro-cell calibration

The pressures at low temperatures are determined from magnetization measurements of Sn applied on several forces. Sn has a well-known pressure dependence of the superconducting transition temperature,  $T_C$  [6]. The temperature dependence of the magnetization at  $H = 50$  Oe in Sn applied on several forces is shown in figure 3. The superconducting transition temperature

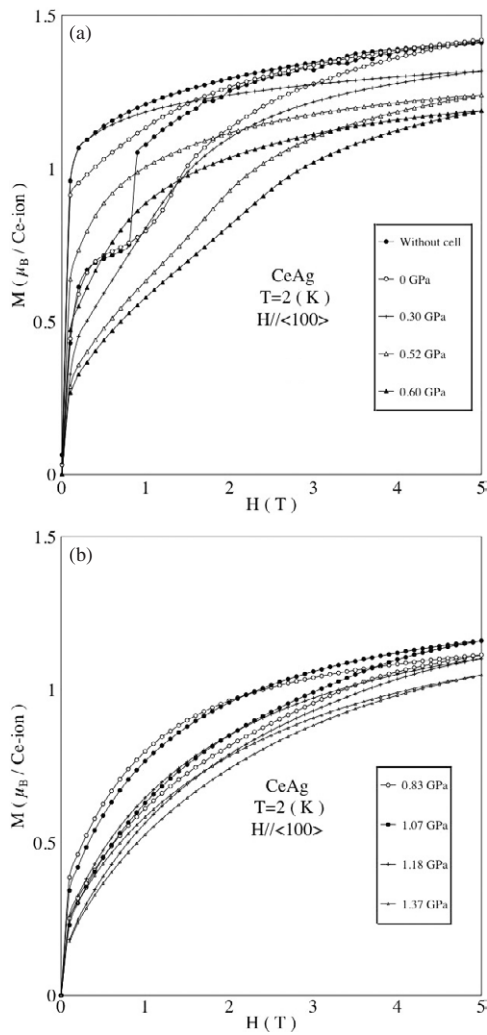


**Figure 4.** Pressures at low temperatures versus applied forces at room temperatures.

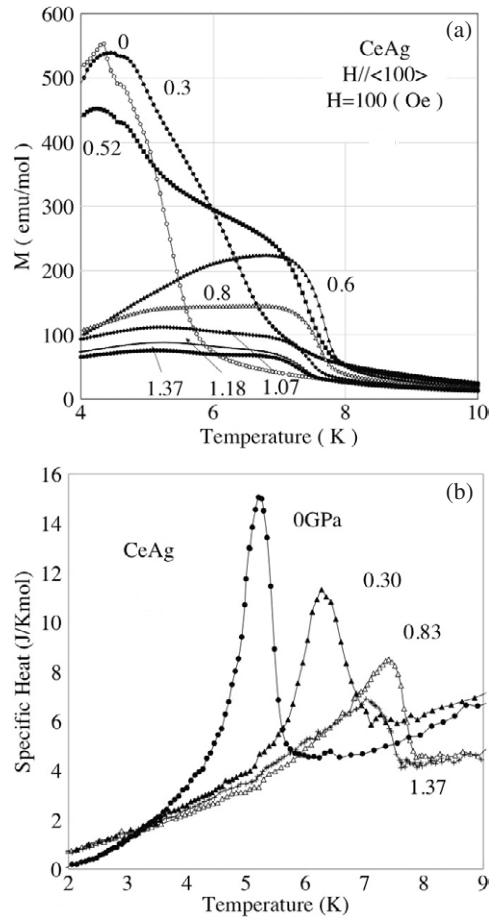
was estimated by extrapolating the magnetization curves near  $T_C$  to zero. The values of  $T_C(P)$  decrease with increasing applied force. The real cell pressures around liquid He temperature which were obtained from the data in figure 3 are plotted as a function of applied force at room temperature in figure 4. There is an almost linear variation of the pressure slope at low temperature.

### 3. Measuring example for CeAg single crystal

As an example we show the magnetization and specific heat measurements as a function of temperature for single crystal CeAg under high pressures. CeAg is a well known material which has ferromagnetic order at 5.5 K and ferroquadrupole ordering at 16 K [7]. Single crystals of CeAg were grown by the Czochralski pulling method. CeAg is sensitive to pressure and stress and shows a Kondo-like behaviour under pressure [8, 9]. We present the results of the high pressure measurements of magnetization and specific heat for CeAg using our high-pressure micro-cell. A single crystal of the CeAg compound was oriented with the easy magnetization direction, i.e. [100], along the magnetic field. The magnetization curves at  $T = 2$  K under several pressures are presented in figures 4(a) and (b). At ambient pressure (without cell), a sharp and large magnetization jump is observed in an increasing field due to ferroquadrupole ordering. This sharp jump, however, broadens with sample setting in the cell and then increasing pressure. The values of saturation magnetization decrease with increasing pressure monotonically from  $1.4 \mu_B/\text{Ce-ion}$  at ambient pressure to  $1.05 \mu_B/\text{Ce-ion}$  at  $P = 1.37$  GPa. The temperature dependences of magnetization and specific heat measurements under different hydrostatic pressures are shown in figures 6(a) and (b). A magnetic field of 100 Oe was applied along the  $\langle 100 \rangle$  direction as in cubic notation at room temperature. The Curie temperature 5.5 K at 0 GPa increases with increasing pressure below around 0.6 GPa and then decreases with increasing pressure up to 1.37 GPa. In the case of specific heat measurements we first measured the heat capacity of the micro-pressure cell with a mixture of Fluorinert under several pressures to obtain the net heat capacity of samples under pressure. The weight of the sample in the micro-cell was about 80 mg. This value is less than 1% compared to that of the cell. However, the heat capacity of the CeAg sample at around 8 K



**Figure 5.** Temperature dependences of magnetization at 2 K for CeAg under various pressures: (a)  $0 < P < 0.7$  GPa, (b)  $0.7 < P < 1.4$  GPa.



**Figure 6.** Temperature dependences of (a) specific heat and (b) magnetization for CeAg under various pressures.

approaches about 20% of total heat capacity. The change of the values of the ferromagnetic ordering temperature from specific heat and magnetization are in excellent agreement with each other, and also with previous resistivity measurements [8, 9]. The ferromagnetic Curie temperature is suggested to disappear roughly above  $P = 3$  GPa. We note that we succeeded to achieve high accuracy magnetization and specific heat measurements under pressures up to 1.4 GPa at low temperatures for CeAg by using the same micro-cell.

#### 4. Conclusions

We have designed a piston-cylinder-type high pressure micro-cell. As final results, it has been found that the maximum working pressure could be raised constantly up to 2.0 GPa at  $T = 2$  K without any trouble. We also measured the temperature dependence of electrical resistivity

under high pressure in the temperature range down to 50 mK and found that the ferromagnetic Curie temperature would disappear under pressures above  $P = 3$  GPa in CeAg.

### Acknowledgment

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