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Detection of antiferromagnetic signals in a diamond-anvil cell using a SQUID vibrating coil magnetometer

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Abstract
We present the results of highly sensitive magnetic measurements in a diamond-anvil cell using a SQUID vibrating coil magnetometer, which were made to investigate the possibility of increasing its sensitivity by improving the system. The Néel temperature T_N of $PrSn_3$ ($T_N = 8.4$ K at ambient pressure) was measured with good signal-to-noise ratio up to about 10 GPa using a tungsten gasket, where the magnitude of the anomaly in the temperature dependence of the susceptibility, assigned as T_N, was as small as 10^{-3} emu cm^{-3}. It appeared that elimination of unnecessary motion of the lead wire of the detection coil was needed to increase the signal-to-noise ratio.

1. Introduction

Recent development of the technique of electrical and magnetic measurement in a diamond-anvil cell (DAC) has stimulated us to study a pressure-induced magnetic phase transition at much higher pressures than a piston–cylinder cell can generate. Investigation of magnetic behaviour of the dense Kondo lattice under high pressure is a good example of its application. The dense Kondo effect is observed in many Ce-based compounds in which the Ruderman–Kittel–Kasuya–Yosida (RKKY) interaction competes with the Kondo effect and the magnitudes of the two interactions change with pressure in different ways such that a magnetic–nonmagnetic transition will occur at some pressure when the Kondo effect overcomes the RKKY interaction [1]. For some antiferromagnets, such as $CeCu_2Ge_2$ [2], $CePd_2Si_2$ [3] and $CeRh_2Si_2$ [4], application of pressure suppresses the Néel temperature T_N to zero and induces superconductivity at low temperature. In these measurements, the pressure dependence of T_N was deduced from the shift of the anomaly in the electrical resistivity or its derivative with temperature, since magnetic measurements in a DAC are in general very difficult especially for antiferromagnets. The very poor filling factor of the detection coil together with extremely small sample size results in a large background that changes remarkably with temperature and pressure. Magnetic measurements are, therefore, carried out in a DAC mainly for detecting a

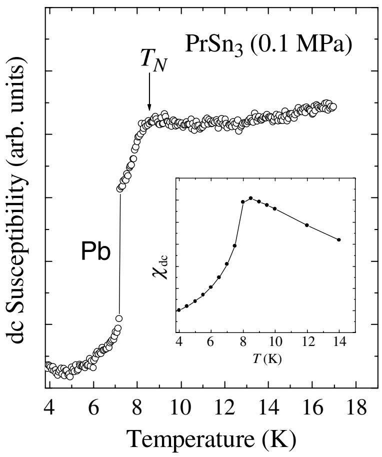

Figure 1. Temperature dependence of the susceptibility of $PrSn_3$ at ambient pressure, measured using the SQUID VCM. Pb shows the superconducting transition of Pb used as a pressure monitor. The inset shows the reported curve [10].

superconducting transition temperature or Curie temperature at which the susceptibility shows drastic change, although much effort has been made to detect signals with good signal-to-noise ratio [5–8]. In view of the assignment of the magnetic transition, magnetic measurements would be preferable to electrical ones and, of course, the latter are not applicable for insulator samples.

For further study of nonmagnetic or antiferromagnetic states under very high pressure, we have to wait for the development of new techniques—such as constructing miniature coils tightly fitted to samples in a DAC. In this paper we will report the results of very highly sensitive measurements for detecting T_N for antiferromagnets in a DAC by using a SQUID vibrating coil magnetometer (VCM), which was originally designed for high-pressure magnetization measurement with high sensitivity and good signal-to-noise ratio [9]. As the VCM detects the gradient of the magnetic flux along the detection coil, the location of the detection coil can be adjusted to have maximum sensitivity above the gasket and the background signal decreases with distance from the detection coil at a higher rate than in the case of magnetic-flux detection. (More details about this system and its performance are given in [9].)

2. Experimental details

We have tried to detect T_N for $PrSn_3$ ($T_N = 8.4$ K at ambient pressure) [10] from the temperature dependence of the susceptibility $\chi_{DC}(T)$. The magnitude of its anomaly at T_N is about 10^{-3} emu cm^{-3}. Figure 1 shows $\chi_{DC}(T)$ for $PrSn_3$, measured at a magnetic field of 3 Oe at ambient pressure using the SQUID VCM. The inset shows the reported curve for $\chi_{DC}(T)$ [10] as a reference. The volume of the sample was about 10^{-3} mm^3 and the sample was placed in a hole of 0.25 mm diameter in a CuBe gasket. The culet diameters of the upper and lower diamond anvils were 0.5 and 0.55 mm, respectively. Using a larger culet for the lower anvil always provides a convex surface of the gasket, above which the detection coil of a SQUID magnetometer vibrates with fixed amplitude [9]. As shown in the figure, the susceptibility increased substantially with temperature and had a maximum at T_N. The sharp change at 7.2 K shows the signal from Pb used as a manometer, superimposed on the data for $PrSn_3$. The gradual increase in χ_{DC} with temperature above T_N was ascribed to the contribution from the background signal. It is shown that the anomaly in χ_{DC} at T_N was clearly observed, although the signal scattered slightly.

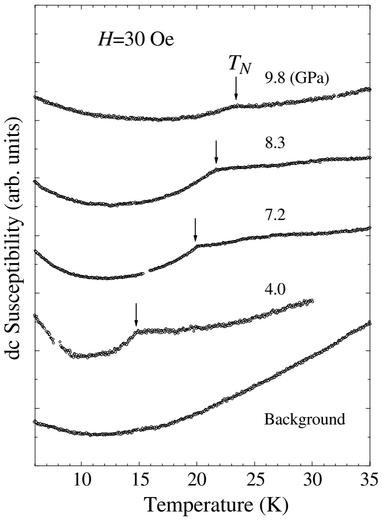

Figure 2. Temperature dependence of the susceptibility of $PrSn_3$ at various pressures up to about 10 GPa.

3. Results and discussion

Figure 2 shows the result of the high-pressure measurements of $\chi_{DC}(T)$ up to about 10 GPa [11]. Subtraction of the background signal, which is shown in the lower part of the figure was not made for any of the curves. The experimental conditions were almost the same as for the case shown in figure 1 except for a gasket. As the size of the sample was large, in view of the present culet diameter, a tungsten gasket was used so as to ensure a large sample chamber under high pressure, in which a mixture of 4:1 methanol–ethanol was introduced as a pressure medium. In fact, the gasket was found not to flow out in this pressure range and, also, the background signal did not change so much with increasing pressure, as shown in figure 2. It should be mentioned that at temperatures below about 6 K, a large tail of the diamagnetic contribution due to the superconductivity of tungsten degrades the signal, so a tungsten gasket is difficult to use for magnetic measurements at low temperatures. As shown in figure 2, T_N was certainly observed up to 9.8 GPa, although the magnitude of the anomaly at T_N decreased with pressure, probably due to the pressure inhomogeneity over the sample. These results show that an anomaly as small as 10^{-3} emu cm^{-3} can be well detected in this pressure range and at temperatures above 6 K.

It has been known that performance of the VCM depends on the degree of cancellation of the unwanted signals induced in the detection coil and the uniformity of its vibration. We used the detection coil of a coaxial first-derivative type. The coils were designed to compensate the uniform component of the magnetic field, and good signal-to-noise ratio was actually obtained for highly balanced ones.

For the present system, the DAC had to be removed from the cryostat together with the detection coil whenever the pressure was changed, so fixing of the lead wire of the detection coil to the cryostat was effected using Scotch tape for practicality. It appeared that this process required great care to suppress the noise—that is, elimination of fluttering of the lead wire was needed to increase the signal-to-noise ratio, as a guidebook for the SQUID magnetometer states. As shown in figure 2, the magnitude of the noise was not the same for all the data and the noise level for 4.0 GPa was large compared with other cases, although the detection coil and the actuator were the same for all the measurements. Such noise was found to come mainly from small but irregular motion of the lead wire of the detection coil, excited by the vibration

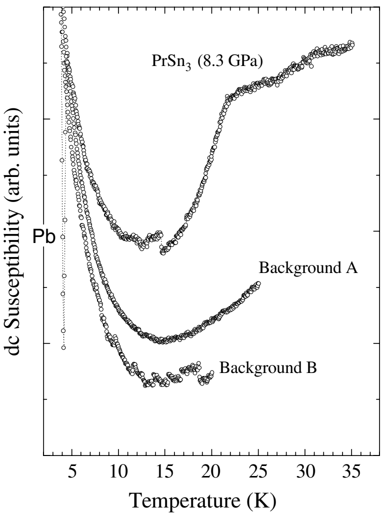

Figure 3. Comparison of the backgrounds. A description is given in the text.

of the detection coil, as a result of insufficient holding of the lead wire. Figure 3 shows the comparison of the noise levels for two backgrounds, A and B, together with the result for $PrSn_3$ at 8.3 GPa as a reference, where the lead wire was fixed with and without great care, respectively. Apparently, a very low noise level was observed in background A. The magnitude of the noise when securely holding the lead wire was found to be almost independent of the applied magnetic field up to 30 Oe, which was the maximum value of the magnetic field for the present system, suggesting higher sensitivity at larger magnetic field.

As regards the background signal, a slight deviation from the original one appeared sometimes, as observed in the data for 8.3 GPa at temperatures around 31 K (figure 3). This may be due to the unexpected movement of the position of the detection coil although the actuator was thermally anchored to the helium bath so as to maintain its vibrational properties during the measurements. The cause of such a change in the vibration is not clear at present. In addition to the compensation for the large temperature dependence of the background signal especially at low temperatures, improvement of the stability of the actuator is a subject for future study, to obtain further increase in the sensitivity of the magnetic measurements in the DAC using the SQUID VCM.

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