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Development of a high-field ESR system under high pressure

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Abstract
We developed a new high-field ESR system under pressure. A clamped-type pressure cell with sapphire pistons is used to transmit the electromagnetic wave. The first measurements on the spin–Peierls system $CuGeO_3$ under pressure and our development of the new standard of pressure for high-field ESR are presented.

1. Introduction

Temperature, magnetic field and pressure are the major important parameters in studying condensed matter physics, and combined experiments on these parameters are becoming especially important nowadays. On the other hand, high-field ESR measurements turned out to be a very powerful means to study quantum spin systems [1] and other physical or chemical properties [2]. However, there has been no high-field ESR measurement under high pressure to our knowledge. Therefore, the aim of our study is to develop a compact pressure cell which can be used in our high-field ESR system. A compact pressure cell already exists and is widely used for SQUID measurement [3] or magnetization measurement [4]. However, millimetre and submillimetre waves have to transmit through the pressure cell in the case of high-field ESR. Therefore, we developed a clamped-type pressure cell made of Be–Cu alloys using sapphire for the pistons, which can transmit the millimetre and submillimetre waves. And we also need a new standard of pressure at low temperature which can be observed by our high-field ESR. In this paper we will show our new high-field ESR system under pressure, and our high-field ESR results on the spin–Peierls system $CuGeO_3$ under pressure. Our search for a new standard of pressure at low temperature using our high-field ESR will be also presented.

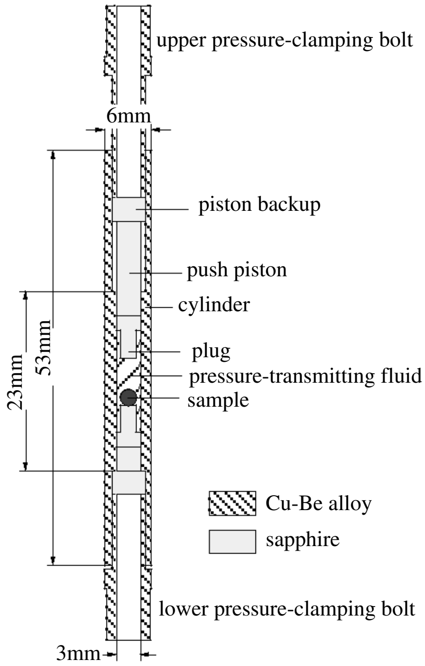

Figure 1. The pressure cell developed for our high-field ESR.

2. Experimental details

Our pressure cell is of clamped type and it is made of non-magnetic Be–Cu alloys except for the sapphire pistons, as shown in figure 1. Flourinert is used as the pressure transmitting medium. The calibration of the applied pressure at low temperature was done by observing the superconducting transition temperature of Sn with a SQUID magnetometer. As we designed the outer diameter of the pressure cell to be 6 mm in order to fit into our pulsed magnet [1] and the inner diameter of the sample space to be 3 mm, as shown in figure 1, the highest pressure achieved was 3.5 kbar. We think that higher pressure could be achieved by making the Be–Cu wall of the cell thicker or using other stronger metallic alloys.

The details of our high-field ESR system can be found elsewhere [1, 5, 6]. The millimetre and submillimetre waves from the light sources are transmitted through the pressure cell placed in the centre of the pulsed magnet. The transmitted light is detected by a liquid-He-cooled InSb detector. The frequency region 30–160 GHz is covered by Gunn oscillators and the higher-frequency region is covered by backward-travelling-wave oscillators (BWO). For the measurements under pressure, a pulsed magnetic field up to 16 T is used and the temperature was 4.2 K.

3. Results and discussion

First the results for $CuGeO_3$ are shown in figure 2. $CuGeO_3$ is in the spin–Peierls phase (SP phase) with the singlet ground state at 4.2 K, while there is a magnetic phase transition at around $B_c = 12.5$ T from the SP phase to the magnetic phase (M phase). As shown in figure 2(a) we can see the change of ESR around B_c at 4.2 K under ambient pressure. This result is consistent with our previous result [7]. On the other hand, the magnetic field where the ESR changes

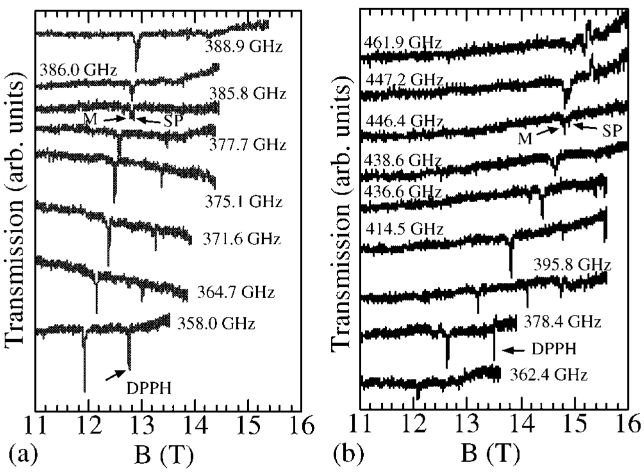

Figure 2. The frequency dependence of the ESR of $CuGeO_3$ for $\boldsymbol{B} \parallel \boldsymbol{a}$ at 1 bar (a) and at 3 kbar (b).

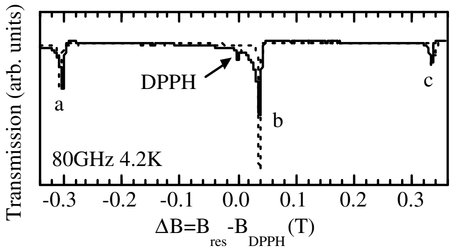

Figure 3. Absorption curves of ruby at 80 GHz and 4.2 K. DPPH is the standard for $g = 2$. The solid and dashed curves are the absorption curves for 1 bar and 2 kbar, respectively.

shifts to 14.8 T when we apply pressure up to about 3 kbar, as shown in figure 2(b). This result is also consistent with the recent magnetization measurement under high pressure [8]. In summary, we succeeded in observing the high-field ESR under high pressure for the first time.

The search for a new standard of pressure using our high-field ESR is very important because the high-field ESR measurements of the sample and the standard material at the same time will enable us to determine the correlation between the ESR of the sample and pressure easily and precisely under the same experimental conditions. We focused on ruby as the standard of pressure in the high-field ESR measurement. The magnetic ion in ruby is Cr^{3+} ($S = 3/2$) and shows three ESR absorption curves, a, b and c as shown in figure 3 due to the existence of the D-term ($D = -5.7$ GHz [9]). The applied magnetic field is tilted from the c-axis by about 20° in the case of figure 3. As the molar concentration of Cr^{3+} ions is determined as about 0.18% for the ruby which we used, no exchange interaction between Cr^{3+} ions is expected. If we apply pressure up to 2 kbar, it is clear that absorption curves a and c shift to lower and higher field, respectively, while the absorption curve b remains unchanged. This result can be easily understood if the absolute value of D is increased by the pressure and g-value is not affected by pressure. The absorption curves a, b and c correspond to the transitions between $S_z = -3/2$ and $-1/2$, $S_z = -1/2$ and $1/2$, $S_z = 1/2$ and $3/2$, respectively. The frequency dependences of absorption curves a and c are shown in figure 4. As the linewidths of a and c are about 10 and 5 mT, respectively, we have enough resolution to distinguish the

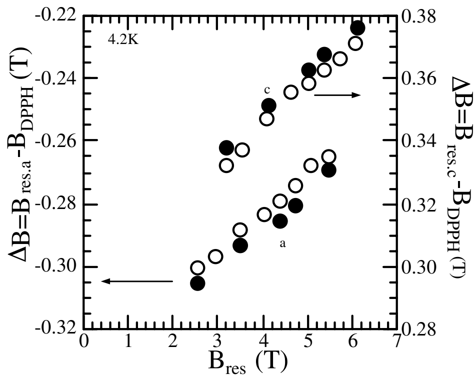

Figure 4. Pressure dependences of resonance fields for absorption curves a and c at 4.2 K. Open and solid circles correspond to 1 bar and 2 kbar, respectively. The resonance field is changed by changing the frequency from 80 to 160 GHz.

shift of resonance under the pressure of 2 kbar. The parallel shifts of resonances a and c in figure 4 by pressure also suggest that the g-value is not affected and the D-term is affected by the pressure. Our results clearly show that ruby can be used as the standard of pressure in the high-field ESR system under pressure.

4. Conclusions

A new high-field ESR system under high pressure up to 3.5 kbar is developed. At 4.2 K we succeeded in observing the shift of the magnetic phase transition in $CuGeO_3$ under high pressure for the first time. We also showed that ESR of ruby can be used as the standard of pressure in the high-field ESR system under high pressure.

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