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High-pressure apparatus for magnetic neutron diffraction beyond 3 GPa at low temperature

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Abstract

A new high-pressure apparatus for magnetic neutron diffraction studies are described. Maximum pressure generated by the anvil-type high-pressure cell with 2.5 mm anvil tip and Cu–0.3% P gasket is about 6 GPa at present. The neutron-focusing device with many supermirrors, which is useful for the experiments with tiny sample, is also shown.

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Keywords: High-pressure; Magnetic neutron diffraction; Sapphire-anvil; Neutron-focusing device

On the transport and magnetic properties of 3d, 4f, and 5f electron systems, interesting phenomena, such as the magnetic phase transition, insulator–metal transition, and superconductivity, appear by pressurization up to about 10 GPa. Therefore, if neutron scattering experiments can be performed at low temperature, putting pressure to the range of about 10 GPa, it can make a great contribution to the research of the magnetism of these systems. However, the highest attainment pressure in almost all magnetic neutron scattering experiments has stopped at about 3 GPa generated by piston-cylinder-type high-pressure cells. In this paper we report recent development of the high-pressure apparatus for magnetic neutron diffraction experiments beyond 3 GPa in JAERI. The most suitable method for generating pressure beyond 3 GPa is using anvil technique. Fig. 1 shows the schematic drawing of the newly developed anvil-type high-pressure cell. We adopted a pair of sapphire (single crystal of Al_2O_3) anvils because sapphire is a hard material (Knoop hardness: ~2000) and hardly absorbs neutrons. We also use the moissanite (single crystal of SiC) anvils [1] or Ti-doped sapphire anvils, which are harder (Knoop hardness: ~3000) than the sapphire anvils. A gasket sandwiched between the anvils is made of Cu–0.3% P alloy, aluminum alloy (7075T6) or Cu–Zn alloy. We usually use the 4:1 deuterated methanol–ethanol solution, some kinds of oil (Daphne oil 7373, Silicon oil KF96-50cs, Fomblin oil Y140/13) or Fluorinert FC70-77 mixture as the pressure-transmitting medium. The pressure is measured by the standard ruby-fluorescence method. Maximum pressure generated by the sapphire-anvil cell (SAC) with 2.5 mm anvil tip and Cu–P gasket is about 6 GPa at present. Fig. 2 shows the temperature dependence of (1, −2, 0) and ($\frac{1}{2}$, −2, 0) magnetic Bragg intensities of CeSb in the SAC at 4.6 GPa [2]. The peaks correspond to the type-I and type-IA antifferomagnetic orders of 2 μ_B magnetic moments of Ce ions. In spite of the tiny sample, the S/N ratio was nearly the same as that of the standard piston-cylinder cell. From this result, it is expected that, when magnetic moments are 1 μ_B or more, the magnetic signal is observed.

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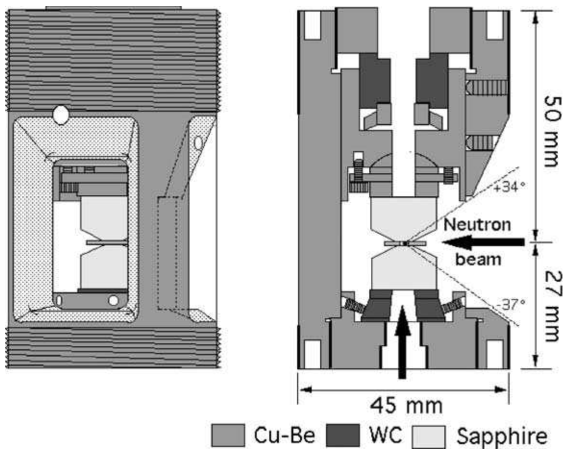

Fig. 1. Schematic drawings of the sapphire-anvil high-pressure cell.

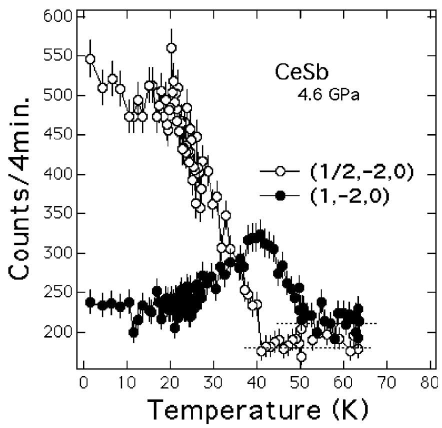

Fig. 2. Temperature dependence of the magnetic Bragg intensities of CeSb in the SAC at 4.6 GPa.

The best method for compensating weak magnetic signals from a tiny sample in SAC is focusing the incident neutron beam on a sample position. We are now developing the focusing device with a lot of curved supermirrors according to the proposal of Mildner [3].

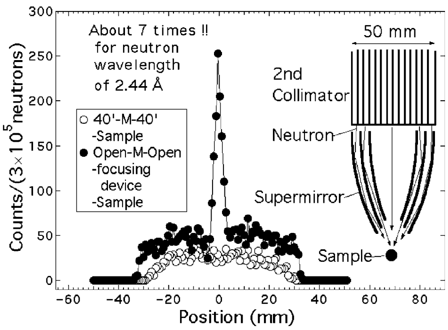

Fig. 3. Calculated distribution of the neutron intensities.

The inset of Fig. 3 shows the schematic drawing of the device. The shape of the cross section of each supermirror is a circle arc. The extension of the end of each mirror meets at a focal point (sample position). Neutrons are repeatedly reflected with the same angle by the inner side of the circle and arrive at the sample position. Fig. 3 shows results of Monte Carlo simulation for a triple-axis neutron spectrometer. The closed circles indicate the neutron intensity distribution at the sample position. This is the case of placing the device of 450 mm length that has 16 supermirrors with Q of 3.0 in the space of 600 mm between the 2nd collimator and the sample. Here, Q is the critical wave number of the total reflection of the mirror relative to that of natural nickel. The open circles indicate the distribution for the general experiment condition without the device. It is clear that the considerable gain of about 7 is obtained using the device. Therefore, it is expected that the device is very effective not only in the high-pressure experiments but also in the neutron inelastic scattering experiments with very weak signals.

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