Development of hybrid-anvil technique and its application to single crystal magnetic neutron diffraction studies

Toyotaka Osakabe[1]

[1] QuBS, JAEA

Magnetic neutron scattering is a powerfull tool for studies on interesting magnetic phenomena under pressure from microscopic point of view. However, the highest attainable pressure in almost all single-crystal magnetic neutron diffraction experiments has been below about 3 GPa. It is well known that the most suitable method for generating pressure beyond 3 GPa is using anvil technique. Recently, we have developed the hybrid-anvil device composed of an opposed pair of a sapphire anvil and a WC anvil. Figure shows the hybrid-anvil device. The sizes in the figure show the actual sizes of the device used in the experiments. The essential point of the device is a hollow in WC anvil. The hollow highly suppresses deformation of the gasket, since a part of the gasket surrounding the sample chamber is strongly caught by the edge of the hollow when the gasket is pressed. This gives three important effects. One is preventing the opposite sapphire anvil from cracking. Cracks are often caused by using the hard gasket in order to avoid the large deformation. Another is enlarging the available sample size. The sample size is determined by the initial diameter of the sample chamber and the final distance between two anvils. In comparison with a sapphire-anvil device which has the opposed flat culets, the sample size of the hybrid-anvil device amounts to more than 4 times. The other is improving the pressure generation efficiency. The efficciency is often reduced by sliding of the gasket in the pressing process. For example, the efficciency of the hybrid-anivil device is about 1.5 times larger than that of the sapphire-anvil device at the same load of two tons. As a matter of course, we can observe the sample directly by a microscope and measure the pressure value accurately by a standard ruby-fluorescence method through the transparent sapphire anvil. We could generate the pressure up to 7 GPa in a test experiment and 6.2 GPa in an actual magnetic diffraction experiment. In order to expand the pressure range up to 10 GPa, we plan to use a pair of a sinterd diamond anvil and a moissanite anvil.

Pressure transmitting medium has an extremely important role in single crystal diffraction experiments. Low hydrostaticity or uniaxiality of applied pressure highly depraves the crystallinity of the sample and as a result diffraction intensity lowers further. Empirically the crystallinity of the pressed samples in clamp-type high-pressure cells is not depraved during cooling. Thus, hydrostaticity during pressurizing at room temperature is crucial importance. In order to investigate hydrostaticity of the mediums, we measured the mosaic spread of NaCl under pressure using the hybrid-anvil device with some kinds of the mediums: the mixture of Fluorinate FC70 and FC77, Daphne 7373 oil, Si oil (KF96-50SC), Fombrin oil (Y140/13), Glycerin and solid Graphite. The results are as follows. The mosaic spread starts to increase steeply at about 1 GPa in the mixture of Fluorinate. In Si oil and Fombrin oil, those start to increase at about 1 GPa and 2.5 GPa. In Graphite, the mosaic spread at 1.2 GPa reaches more than 10 times compared to that at ambient pressure. On the other hand, in Glycerin, the mosaic spread increases very gently with increasing pressure and is only 1.5 times at 7 GPa compared to that at ambient pressure. This indicates that Glycerin transmits the nearly hydrostatic pressure even at 7 GPa.

Using the hybrid-anvil device, we have so far performed single-crystal magnetic neutron diffraction experiments on Mott-insulator $TbVO_3$ up to 4.3 GPa, filled skutterudite compound $PrFe_4P_{12}$ up to 3.8 GPa and valence transition compound $YbInCu_4$ up to 6.2 GPa. In particular, in spite of the small magnetic moment of about 0.8 m_B /Yb in $YbInCu_4$, we could detect distinct magnetic signals. This was accomplished by using the thermal neutron focusing device with the hybrid-anvil device.