High Pressure Instrumentation

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Significant discoveries in experimental physics are very often associated with advances in instrumentation, and high pressure research is one area where this is particularly true. The development in the SPSMS of measurements using diamond anvil cells is a long term effort which started more than 20 years ago. Our transport and calorimetry measurements with continuously variable pressure are still unique worldwide, and we have recently added ac susceptibility. We have also started to extend them to extreme conditions of low temperatures and high magnetic field. In parallel, developments of large volume cells for neutron scattering and thermal expansion measurements have made a new generation of experiments possible in the 0 – 3 GPa range. Finally, for studies where neither the diamond cell nor the large volume cell are suitable, a modified Bridgman anvil cell allows experiments up to 8 GPa in much more hydrostatic conditions than previously possible.

The diamond anvil cell is the backbone of high pressure research in the SPSMS. Our existing set-up allows resistivity and calorimetry measurements up to about 20 GPa, with a noble gas (Ar, He) pressure transmitting medium ensuring hydrostatic conditions, and a home-built bellows system allows in-situ tuning of the pressure. The ANR funded project ECCE (Extreme Conditions Correlated Electrons) will extend this technique to dilution fridge temperatures (50mK) and high field (14T). As a first step we have started to perform ac calorimetry measurements at fixed pressure in these conditions, to test the feasibility of these measurements. The temperature limit of these measurements is about 100 mK, mainly due to the reduced sensitivity of the Au:AuFe thermocouple below this temperature. The heating of the sample can be either optical, by using a stabilized laser diode, or resistive, with and additional wire soldered on the sample. These measurements, in addition to breaking the ground for extending the technique to extreme conditions, have also produced several important results, including the determination of the re-entrance of magnetic order inside the superconducting state in $CeRhIn_5$, and the determination of the high pressure phase diagrams of $YbRh_2Si_2$ and $CeCoGe_3$.

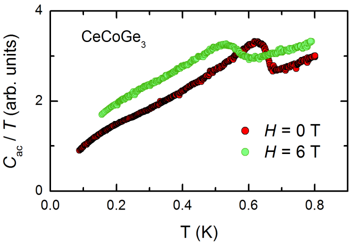

Fig. 1: C/T versus T (down to 200 mK) of $CeCoGe_3$ at 5.7 GPa for different magnetic fields showing that the pressure induced superconductivity is a bulk property.

Another valuable addition to the diamond cell is the implementation of ac susceptibility measurements in collaboration with Cambridge University. The technique adopted is to insert a tiny pick-up coil (350 µm diameter made from 10 turns of 12 µm wire) into the pressure chamber. This ensures a good filling factor and high sensitivity allowing superconducting and magnetic transitions to be detected. This was particularly successful for investigating the magnetic phase diagrams of $CeFe_2$ and $YbCu_2Si_2$, and the superconducting phase diagrams of FeSe and $Sr_{14-x}Ca_xCu_{24}O_{41}$ up to 15 GPa.

Resistivity measurements in the diamond cell rely on making spot welded contacts to the sample. On some compounds this is not possible. For example on most oxides the spot welding technique does not work well. We have therefore developed a modified version of the standard Bridgman cell which uses the usual tungsten carbide anvils, but a composite pyrophyllite/nylon gasket allows loading with a liquid medium (Fluorinert). This makes resistivity measurements possible in relatively hydrostatic conditions up to 8 GPa with samples up to 1mm in length, in a compact pressure cell. This technique was successfully used to explore the phase diagram of the spin ladder superconductor NaV_6O_{15}.

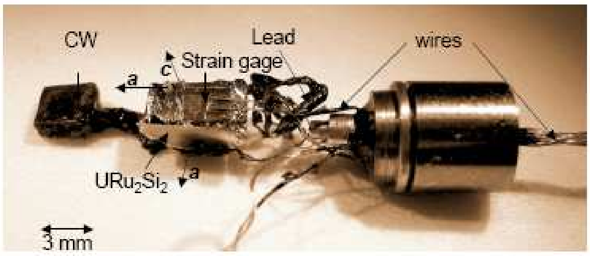

Fig. 2: Pressure cell setup with strain gage glued to an URu_2Si_2 sample for simultaneous thermal expansion and neutron scattering experiments.

The principle new development for large volume cells is the introduction of thermal expansion measurements under high pressure and magnetic fields using a strain gage technique. This allows the measurement of the thermal expansion coefficient with an accuracy of 5 x 10^{-7}. This technique has been successfully used to prove the first order nature of the collapse of antiferromagnetic order under high pressure. Furthermore, we showed that this technique can be used under high magnetic fields, e.g. to determine the (p,T,H) phase diagram of MnSi. A new design of cell for neutron scattering has also introduced several improvements, including the use of a Pb manometer. Finally the simultaneous use of thermal expansion and neutron scattering under high pressure and magnetic field, allowed us to investigate the inelastic response of URu_2Si_2 in the successive paramagnetic, hidden order, and antiferromagnetic phases.

Selected publication(s): E. Colombier et al., Rev. Sci. Instrum. **78**, 093903 (2007), G. Knebel et al., Phys.Rev. B **74**, 020501 (2006)	
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