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Experimental report

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Neutron spectroscopy from low-d spin S=1/2 quantum magnets to pressures of 15kbar and use of lead capsules as pressure calibration tool on TASP

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We successfully performed inelastic neutron scattering on a low dimensional spin S=1/2 quantum magnet under high pressure and used the thin lead sample capsule as a pressure calibration tool.

In quantum magnets one interesting experimental challenge is to tune the interactions between the magnetic ions in order to induce a quantum phase transition. To achieve this, chemical substitution and hydrostatic pressure are the favored techniques.

In the present experiment, performed on TASP in December 2005, we succeeded in obtaining good quality inelastic signal from a low-dimensional spin S=1/2 quantum magnet. The sample, grown by traveling solvent floating zone at PSI by E. Pomjakushina, M. Stingaciu and K. Conder, had cylindrical shape, was 40mm long and its diameter was reduced to less than 7 mm to fit into the pressure cell. The large sample volume allowed a reasonable counting rate during the experiment. The sample was first measured outside the pressure cell, giving signal of 27cnts/min over a background of 2cnts/min. Mounted inside the pressure cell, the signal reduced to 12.5cnts/min and the background increased to 3cnts/min. These conditions make detailed inelastic studies up to 15kbar possible at PSI.

In this report we will focus on the use of the lead capsules containing the sample inside the pressure cell as a pressure calibration tool for inelastic neutron experiments.

Hydrostatic pressure is applied on the sample with Fluorinert\textsuperscript{TM} (FC77) liquid as pressure transmission medium. The sample and Fluorinert are inserted into the lead capsule with 7mm inner diameter, walls of 0.35mm, and mass of 8.6g. The pressure cell itself consists of an aluminium cylinder inside which the lead capsule is squeezed by two pistons hold by a two large screws, it is designed to stand pressures up to 15kbars.

A first estimate of the pressure inside the cell can be made, while applying it, using the manometer on the pressure pump and the conversion table for the piston’s diameter. However there is still a need for a more precise measurement and for the verification of the actual pressure during the neutron experiment itself at low temperature. Using the Birch-Murnaghan equation of state (BM EOS) for lead \(^{(1)}\) where \(a\) is the lattice parameter and calculating the Bragg reflection conditions, we were able to monitor the pressure in the sample during the experiment with an uncertainty of less than 1kbar.

\begin{equation}
\begin{align*}
P &= \frac{3}{2} B_0 (x^3 - x^5) \left(1 + \frac{3}{4} (B_1 - 4) (x^3 - 1)\right) \\
B_0 &= 43.2 \text{ GPa} , B_1 = 4.9 , x = V(P=0)/V , a^3 = V
\end{align*}
\end{equation}

The count rate for neutrons scattered by the lead capsule is good enough for fast measurements: a clear Bragg peak with a signal to noise ratio between 2 and 4 is reached with scans of about 6 seconds per point (see figure 1 inset).

The lead capsule and sample first resisted pressures of about 10 and 12kbar. When releasing pressure from 12kbar, the lead capsule leaked and the sample broke. However the present experiment has demonstrated that existing sample containers, in our case thin lead capsules, can directly and efficiently be used for pressure determination in high-pressure inelastic neutron scattering experiments.

Figure 1: Pressure calibration curves: full and dotted lines, BM EOS. Symbols: lattice parameters from (002) and (111) Bragg reflections. Inset: Pb Bragg (111) at 12kbar. T= 1.5K.

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