Sample holder for neutron scattering in high magnetic fields

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A type of sample holder for neutron scattering experiments in high magnetic fields has been developed and tested, which is based on the fixation of the sample by a compressed powder of the cryostat material, in our case aluminum. It has been tested up to a field of 13 T in a measurement of the transversal magnetostriction of a $PrCu_2$ single crystal. © *2005 American Institute of Physics.*
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I. INTRODUCTION

When doing scattering experiments in high magnetic fields the anisotropy of a magnetic sample may lead to significant forces and torques, which can move or turn the sample and change its orientation during the measurement. Therefore, the sample has to be fixed properly. In addition, the fixation of the sample should not contain any material which might lead to an artefact in the measured signal. In the case of neutron scattering a sample fixation is preferred, which is made out of the same material as the parts of the cryomagnet in the beam.

However, in most circumstances it is difficult if not impossible to shape a solid metal sample holder exactly in the form of the sample. We solved this problem by a type of sample holder, which is based on the fixation of the sample by a compressed powder of the cryostat material, in our case aluminum.

II. DESCRIPTION OF THE SAMPLE HOLDER

The cross section of the sample holder is shown in Fig. 1 and a photograph in Fig. 2. It consists of an aluminum tube (h) with two pistons (b,j). Initially the sample is a mounted well oriented onto a sample rod (d) using aluminum wire or a small amount of glue. The sample rod is mounted on the upper piston (b) and orientation is checked by neutron diffraction. If the orientation is satisfactory, the upper piston is introduced into the tube. The piston thread is sealed with Teflon before screwing in order to avoid damage of the thread and to prevent moisture from entering the sample space. Aluminum powder of grain size 125 μm (purity 99.5%, in our case provided by Goodfellow, UK) is used to fill the tube. In order to be able to compact the powder the tube is filled such as to allow for about 5 mm of compression when the second piston (j) is attached. Finally the thread of the second piston is covered with Teflon tape and the piston is screwed firmly into the tube. In this way the powder is compressed resulting in a firm attachment to the sample which prevents any movement of the sample.

III. MAGNETOSTRICTION MEASUREMENT ON $PrCu_2$

A promising candidate for the sample holder testing was $PrCu_2$. The compound shows a Jahn-Teller transition below T_Q=7.5 K, which is associated with a monoclinic distortion of the orthorhombic unit cell.[1] The magnetic easy axis in this system is the (100) axis with a magnetic moment of 2.5 μ_B/f.u. at a temperature T=2 K and a magnetic field $\mu_0 H$=10 T (Ref. 2).

A $PrCu_2$ single crystal was grown by a modified Czochralski technique in the tetra-arc furnace (IMR, Tohoku University-Sendai, Japan). The compound forms congruently at T=841 °C, therefore an initial polycrystalline sample (mass ~9 g) was easily obtained by direct melting of high-purity elements (Pr-3N, Cu-4N) in stoichiometric composition under Ar protective atmosphere. The bulk was remelted several times to obtain a good homogeneity and the single crystal was subsequently grown. We used antiparallel rotation of both the seed and the water-cooled Cu crucible (~8 rotations per minute). The pulling speed was set to 15 mm/h, then decreased to 10 mm/h (growth of the ingot main part)

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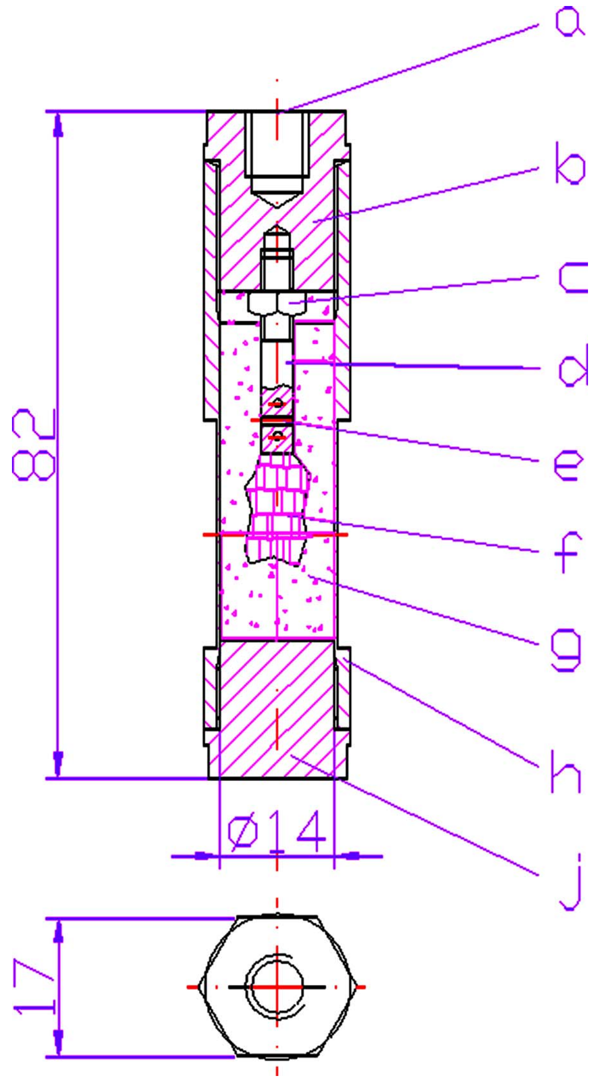

FIG. 1. Engineering detail drawing of the sample holder labeled as follows: (a) thread for attachment to sample stick; (b) upper piston; (c) counter nut; (d) inner sample rod; (e) holes for sample fixing; (f) sample; (g) aluminum powder; (h) aluminum tube; (j) bottom piston.

FIG. 2. Casing (right) and insert (left) of the sample holder with mounted $PrCu_2$ sample. The sample is orientated such that the angle between the easy axis (100) and the magnetic field is 60°. In order to achieve this it was necessary to place the sample on an inner sample rod with a surface tilted by 30°.

and finally increased up to 25 mm/h (finishing stage); the current was varied from 5 to 15 A per one arc. The final ingot had 0.5 cm maximum diameter and length 65 mm (including the neck ~10 mm). The ingot was cut and a sample of ~15 mm length with a mass of 2.9 g was prepared and used in our neutron scattering experiment. The quality of the sample was checked by neutron Laue technique. It was found that the sample consists of a big single crystal and a small fraction of disordered crystallites. The mosaicity of the big part was 0.6°.

Our neutron diffraction measurements in magnetic field have been performed using both the 12 and 15 T cryomagnets (Oxford Instruments) subsequently on the IN12 three-axis spectrometer of the Institute Laue Langevin (Grenoble). The crystal was mounted in our sample holder as shown in Fig. 2. The (−103) direction was parallel to the vertical magnetic field, i.e., the field was applied at an angle of 60° with respect to the (easy) a axis. After inserting the crystal into the sample tube, the tube was filled with Al powder as described in Sec. II. Neutron diffraction experiments were done and the field dependence of the (101) and (020) reflections was measured at a temperature of $T=2$ K (i.e., well below T_Q) up to a magnetic field of 13 T. At zero field the lattice constant $b=0.69885$ nm was determined.

From the magnetic moment[2] we estimate a turning moment of about 200 N cm at an applied magnetic field $\mu_0 H=10$ T. Figure 3 shows a comparison of the (020) Bragg peak at zero field $\mu_0 H=0$ and at an applied field of $\mu_0 H=11$ T. The intensity loss observed during the measurement was less than 5% for both the (020) and (101) reflection, indicating that the sample was stabilized very well by the aluminum powder matrix.

The profiles of the (020) reflection were fitted by Gaussians. From the shift of the center the magnetostriction $\Delta b/b$ as a function of applied magnetic field was obtained (see Fig. 4). From this measurement we conclude that the crystal contracts in the b direction by $\Delta b/b=-3.6\times 10^{-4}$ when applying a magnetic field of 10 T. The resolution of the method is about 3×10^{-5} (see error bars in Fig. 4) and thus cannot

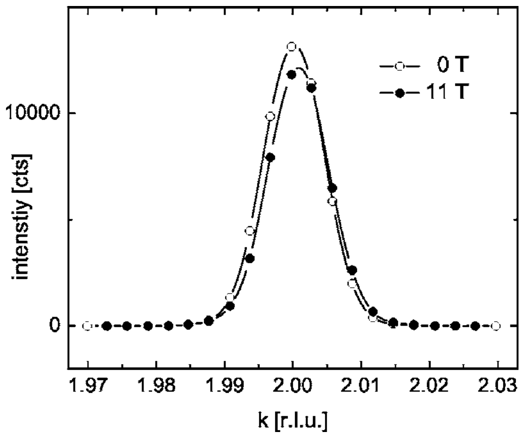

FIG. 3. Field dependence of the (020) reflection of $PrCu_2$ measured at $T=2$ K using the 12 T vertical magnet at the three axis spectrometer IN12 (ILL, Grenoble). Error bars are smaller than symbols.

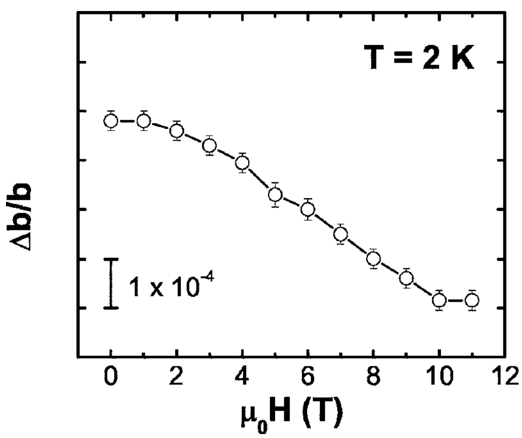

FIG. 4. Magnetostriction $\Delta b/b$ of a $PrCu_2$ single crystal at the temperature $T=2$ K as a function of applied magnetic field in the *ac* plane along (−103), i.e., at an angle of 60° with respect to the (easy) *a* axis.

compete with high resolution methods such as capacitance dilatometry.[3] However, the determination of magnetostriction by means of neutron diffraction provides excellent data on the intrinsic magnetostrictive strains, because it is much less sensitive to error sources such as torque on or small cracks in the sample.

IV. DISCUSSION

We believe that this type of sample holder may be of great value when doing neutron scattering experiments in magnetic fields. It is easy to use and is made of the same material as the cryomagnet. Therefore, it may provide high quality results and prevent loss of beam time.

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