AIP | Review of Scientific Instruments

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Citation: Review of Scientific Instruments **85**, 026112 (2014); doi: 10.1063/1.4865406
View online: http://dx.doi.org/10.1063/1.4865406
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/85/2?ver=pdfcov
Published by the AIP Publishing

Note: Versatile sample stick for neutron scattering experiments in high electric fields

M. Bartkowiak,[1,a)] J. S. White,[2,3] H. M. Rønnow,[3] and K. Prša[3]
[1]Laboratory for Developments and Methods, Paul Scherrer Institut, CH-5232 Villigen, Switzerland
[2]Laboratory for Neutron Scattering, Paul Scherrer Institut, CH-5232 Villigen, Switzerland
[3]Laboratory for Quantum Magnetism, Ecole Polytechnique Fédérale de Lausanne (EPFL), CH-1015 Lausanne, Switzerland

(Received 13 December 2013; accepted 29 January 2014; published online 12 February 2014)

We present a versatile high voltage sample stick that fits into all cryomagnets and standard cryostats at the Swiss Spallation Neutron Source, Paul Scherrer Institut, and which provides a low effort route to neutron scattering experiments that combine electric field with low temperature and magnetic field. The stick allows for voltages up to 5 kV and can be easily adapted for different scattering geometries. We discuss the design consideration and thermal behavior of the stick, and give one example to showcase the abilities of the device. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4865406]

For studying phenomena in solid-state physics, temperature, magnetic field, and pressure are the most common control parameters. With the resurgent interest in multifunctional materials such as magneto-electric (ME) multiferroics, electric (*E*-) field emerges as a necessary additional control parameter too. The requirements for neutron scattering experiments involving *E*-fields can be quite diverse, and depend on both the properties to be studied (e.g., surface or bulk effects), and the chosen neutron technique. Considerations include both the *E*-field strength and its relative orientation with respect to the scattering plane, and furthermore the ability to combine *E*-field, temperature, and magnetic field control in a single experiment.

To apply a well-defined *E*-field to a sample, usually the potential applying electrodes are in intimate contact with the sample surface. Typical values for electric fields at which physical phenomena are reported are of order kV/mm. Another important consideration is that to obtain a reasonable scattered neutron intensity, the thin dimension of the sample across which the E-field is applied should not be much thinner than 1 mm.

Instead of designing a dedicated cryostat to achieve applied E-fields across a sample, we have built a sample stick that integrates into our suite of cryomagnets and standard flow cryostats. These cryogenic systems are all top loading, with the sample and stick cooled by He exchange gas. While this simplifies sample changes and assures good thermal contact between the sample and the cryostat, in general it hampers the application of large voltages. As described by Paschen,[1] the maximum *E*-field that can be applied between two electrodes is limited by the break down voltage of the gas. For He at a typical exchange gas pressure of 10 mbar, the breakdown voltage between electrodes 1 mm apart is as little as 150 V.[2] To overcome this, we have designed a stick with a sample containing vacuum chamber. To permit multiple neutron scattering geometries with respect to the *E*-field direction, we rejected the approach to use readily installed electrodes. Instead, the electrodes are either sputtered onto the sample, or painted on with silver paint during sample preparation.

The sample vacuum can is made from Al and is sealed at the cold part with an In seal against the Cu flange (see Fig. 1(f)). The stick shaft is used as the pumping tube and incorporates all wiring thus avoiding low temperature electric feed-throughs (see Fig. 1). The room temperature side of the shaft terminates with a NW-16 flange. This permits the use of standard components for the vacuum feed-through of all wiring and pump connections. The stick is sealed to the sample container with a NW-50 flange (see Fig. 1(b)). The position of this flange can be altered to tune the stick length for a specific cryostat, and it is sealed against the stick by a radial seal with the position fixed with a split ring clamp. There are three baffles each of outer diameter 49 mm that serve as radiation shields. They are made from aluminum and are clamped to their position with only a side screw. Two baffle sets are placed inside the pumping tube; one affixed at the centering ring of the NW-16 flange at room temperature (see Fig. 1(c)), and a second one fixed at the low temperature flange (see Fig. 1(e)) to reduce the effect of thermal radiation on the vacuum space. The baffles are made from 0.1 mm thick aluminum foil which allows them to flex when pushing against the HV cable.

Running all wiring inside the pumping tube simplifies the assembly but increases the effort required to thermalize the cables. Commercial high voltage (HV) cables are usually made of good conductors and require a thick insulation layer to avoid electric breakdown. To reduce the effort required to thermally anchor such cables in vacuum, we manufactured our own. We used phosphor-bronze wire (Lakeshore SL-32) and strengthened the insulation utilizing PTFE capillaries with an inner diameter of 0.25 mm and a wall thickness of 0.23 mm. Although the use of low electrical conducting wire reduces the initial heat load by a factor 100, the loose fit of the wire inside the capillary tube, and the insufficient thermal coupling of the tube inside the sample stick can

a)Author to whom correspondence should be addressed. Electronic mail: marek.bartkowiak@psi.ch.

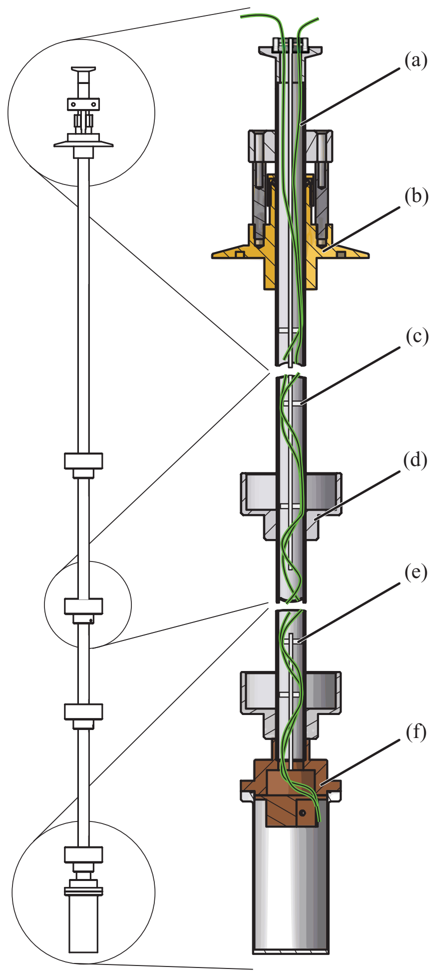

FIG. 1. This figure shows the stick assembly with detailed cuts where labels correspond to (a) the HV cables, (b) the NW-50 flange to the sample chamber, (c) the upper, inner baffle set, (d) the outer baffles, (e) the lower, inner baffle set, and (f) the low temperature flange. The usable stick length can be 1340 mm maximum with the stick as shown and 1600 mm with the magnet extension.

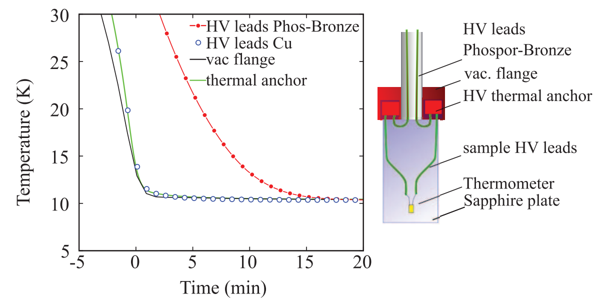

FIG. 3. Left: Measurement of the sample thermalization time. Open blue circles (filled red circles) correspond to Cu (phosphor-bronze) leads labeled as "sample HV leads" in the sketch of the setup (shown right). With Cu leads the sample is always in thermal equilibrium with the flange, while a time lag of more than 10 min is observed for phosphor-bronze wires.

nevertheless prolong the thermalization time of the wire. Therefore, a thermal sink for the wires was made by clamping a 0.1 mm Cu foil (8 × 8 mm^2) between two 10 × 10 × 2 mm^3 sapphire plates, which itself is pressed against the Cu body of the flange (see Fig. 2). The wires coming from room temperature are soldered to the Cu foil and a short wire connected to a different side of the foil extends to the sample position. Two of these cables are placed inside the stick to enable polarization current measurements, but most commonly just one wire is used and the ground is provided by the stick. The manufactured cables were individually tested for their break down resistance prior to installation and no signs for material fatigue have been observed for voltages up to 5 kV (the maximum of our voltage supply).

In addition to HV cables, wiring for thermometry is also provided inside the sample can. Furthermore, there are two different vacuum can sizes available. One has an inner diameter of 36 mm and length 70 mm, and can be used in cryostats and magnets with a sample space diameter of 50 mm (Figs. 2(a)) and 2(b). In the standard configuration, the sample is mounted on either aluminum or a sapphire plate of 63 × 23 × 1 mm^3. However, enough space exists to mount the sample on a Huber goniometer head (type 1001). A swing

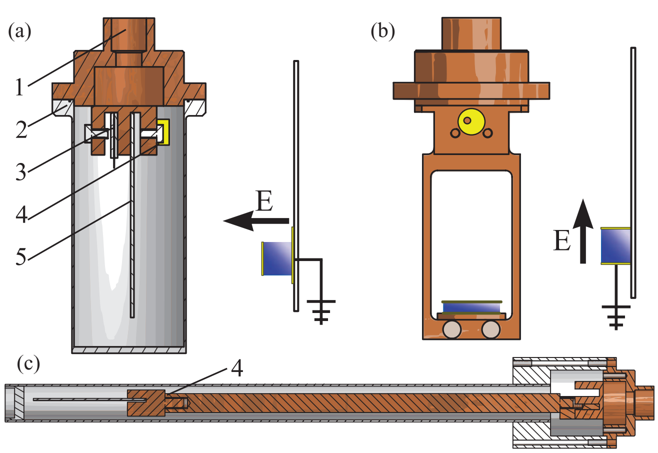

FIG. 2. Three possible sample holder configurations are shown. (a) The standard setup, with (1) Cu flange, (2) Indium seal, (3) HV thermal, anchor, (4) sample thermometer, and (5) aluminum/sapphire sample, plate. (b) A holder suitable for reflectometry. In (c), we show the extension for use within a cryomagnet, and with a sample space, diameter < 50 mm.

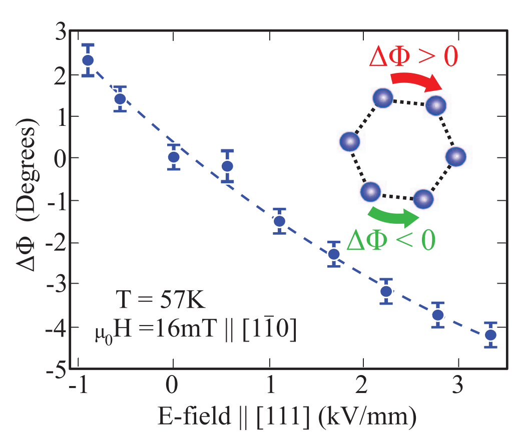

FIG. 4. Azimuthal rotation angle $\Delta\Phi$$ of the entire hexagonal skyrmion lattice in Cu_2OSeO_3 as a function of applied poling *E*-field. The inset sketch illustrates that $\Delta\Phi>0$ ($\Delta\Phi<0$) corresponds to a clockwise (counter clockwise) collective rotation of the skyrmion lattice. The voltage was applied across a 0.9 mm thick sample.

shaped holder can also be fitted that is suitable for neutron reflectometry (Fig. 2(b)). A second thin tail vacuum can is available so that the stick can be used in magnets with a smaller sample space. The can tail has an outer diameter of 20 mm and inner diameter of 18 mm, and an extra Cu cold finger extension allows the sample to be located at the magnet center. The samples are mounted on sapphire plates of size 60 × 14 × 1 mm^3. With this option, the stick can be used in all our cryomagnets including 15 T vertical and 11 T horizontal field options (Fig. 2(c)).

We tested our thermal design by mounting a thermometer (Cernox 1050-SD) at the sample position on the sapphire holder, and using the HV cables to connect the sensor to a temperature controller. Figure 3 shows the last few minutes of a cool down of the stick after insertion into a sample chamber at a temperature of 100 K. The temperature of the stick flange is observed to closely follow the VTI heat exchanger. The sample (thermometer) temperature agrees very well with the flange temperature, which evidences the expected functioning of the HV heat sinks. We expected that the sample is mainly cooled by the sapphire sample holder. However, the thermalization time is strongly dependent on the conducting material used for the HV cable between the sink and the sample. When using a Cu wire, the sample temperature follows the flange temperature closely. However, when phosphor-bronze wire is used (the same as used within the shaft) there is a time lag of 10 min until the sample reaches the flange temperature. In both cases, the wires were strengthened with Teflon. This shows the HV wire to provide an important thermal path for cooling the sample and care has to be taken to ensure good thermalization of this conductor.

The stick has been used successfully in multiple neutron scattering experiments on ME multiferroic materials where the ME coupling allows the direct electric field control of magnetism.[3,4] By way of example, Fig. 4 shows new small-angle neutron scattering data obtained from the magnetic skyrmion lattice in the ME insulator Cu_2OSeO_3. The measurements show the precise azimuthal orientation of the skyrmion lattice to be intrinsically dependent on the poling *E*-field applied. Further details can be found in Ref. 4.

We thank W. Latscha for technical support, and A. A. Omrani, I. Živković and J. L. Gavilano for support with the experiments on Cu_2OSeO_3.

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