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Ximone Willis, Xiaxin Ding, John Singleton, and Fedor F. Balakirev

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Ximone Willis,[1,a)] Xiaxin Ding,[2,3] John Singleton,[2] and Fedor F. Balakirev[2,b)]

AFFILIATIONS

[1] The University of Texas at Dallas, Richardson, Texas 75080, USA
[2] National High Magnetic Field Laboratory, Los Alamos National Laboratory, Los Alamos, New Mexico 87545, USA
[3] Idaho National Laboratory, Idaho Falls, Idaho 83415, USA

[a)] **Current address:** Raytheon, Waltham, Massachusetts 02451-1449, USA.
[b)] **Author to whom correspondence should be addressed:** fedor@lanl.gov

ABSTRACT

Complex high-precision mechanical devices can be fabricated using a three-dimensional printing technology with the help of computer-aided design. Using 3D stereolithography, we have constructed a cryogenic goniometer for measurements in pulsed magnetic fields of up to 100 T, at temperatures as low as 0.5 K. We review the properties of several materials tested in developing the goniometer and report on its design and performance. The goniometer allows samples to be rotated *in situ* to a precision of 0.2° so that the field can be applied at many different angles to the samples' symmetry directions. Following its success, we establish that 3D printing is now a viable technology for pulsed field and other cryogenic probes.

High magnetic fields are a very important tool for exploring the magnetic and electronic characteristics of novel materials.[1] Such materials frequently have very anisotropic properties, and it is, therefore, very informative to be able to rotate the sample under the study to many different orientations in the magnetic field.[2–5] However, the highest non-destructive fields are provided by using pulsed magnets[1] where very large stresses are ameliorated by minimizing the volume in which the field is provided, leading to very restricted space for the experiment. Hence, cryogenic probes that permit *in situ* rotation of samples within the small bore (corresponding to 6–8 mm diameter within the cryogenic jacket[6]) of a pulsed magnet have thus far been rare. Moreover, pulsed magnetic fields of the order of 100 T place substantial constraints on any sample probe and, in particular, on mechanical actuators.[7] In a pulsed magnetic field, large currents are induced in any metallic components by the rapidly rising and falling magnetic field, leading to heating and mechanical vibration. Catastrophic pulsed-magnet failures, which often result in the complete loss of the probe, have to be factored into the design—the probe should be economical and easy to replicate.

The previous sophisticated designs for pulsed-magnet sample goniometers include piezoelectric rotators.[8,9] These offer high angular precision, but are limited to the ±10° range due to the constraints of the magnet bore. A two-axis differential gear metal rotator was demonstrated in a wide bore mid-field (<40 T) pulsed magnet,[10] but its complexity, large size, and metal construction mean that such a design has not been the progenitor of further probes. Plastic string rotators are the most common design encountered at pulsed-field facilities[11] when the ±90° range is desired and the sample space is limited. The drawbacks of a string rotator of this size are low angular precision, poor reproducibility, and drift. To avoid these problems, a composite worm drive rotator combining stainless steel and plastic components was realized at the National High Magnetic Field Laboratory for high-precision sample rotation in the 100 T magnet. It was successfully employed in a study of the angular dependence of magnetic quantum oscillations that provided key information about the size and shape of the Fermi surface in a high-T_c superconductor.[12] This probe was hand-milled out of Ultem®[13] plastic; despite the advantages of using a worm drive, its manufacture and replication proved to be a lengthy and expensive process.

In this paper, we report the use of the 3D printing technology to produce a cryogenic goniometer for pulsed fields that can be made much more easily and cheaply than in the previous attempts, while preserving the precision and reproducibility associated with worm drives. In contrast to traditionally produced probes, extra degrees of complexity can quickly be added via Computer-Aided Design (CAD).

The success of such a probe hinges on whether or not the materials used for 3D printing can survive the conditions inside the magnets. They must be cold-tolerant, non-metallic, and durable. The tiny measurement volume available at the highest magnetic fields requires high-precision fabrication. Based on the required tolerances for pulsed-field goniometers, stereolithography (SLA) appears to be the most suitable additive manufacturing process. The goniometer prototype parts were printed using several commercially available SLA materials, as well as ABS (acrylonitrile butadiene styrene) and photopolymer materials. We found that only SLA parts met our tolerances (<100 μm). The parts were then subjected to a series of cryogenic thermal cycling and stress tests. We found that the Accura® Xtreme (3D Systems) SLA process meets all our specifications. The details of the tests of 3D-printed materials and techniques are outlined in the supplementary material.

Figure 1 shows the drawings and a photograph of the finished goniometer assembly. The goniometer assembly was drawn in the Autodesk Inventor® CAD software. The 3D-printed platform that holds the sample is suspended in the frame via a pair of sapphire sliding bearings (a sapphire tube sliding inside a sapphire cup) on each side. The magnetic field is applied along the vertical axis of Fig. 1. The rotational movement of the platform about the horizontal axis is achieved by turning the worm thread. The hemispherical wheel formed by the platform can rotate within ±110°, while remaining in full contact with the worm. Taken with the reversible direction of the magnetic field, a full 360° circle coverage can be achieved.

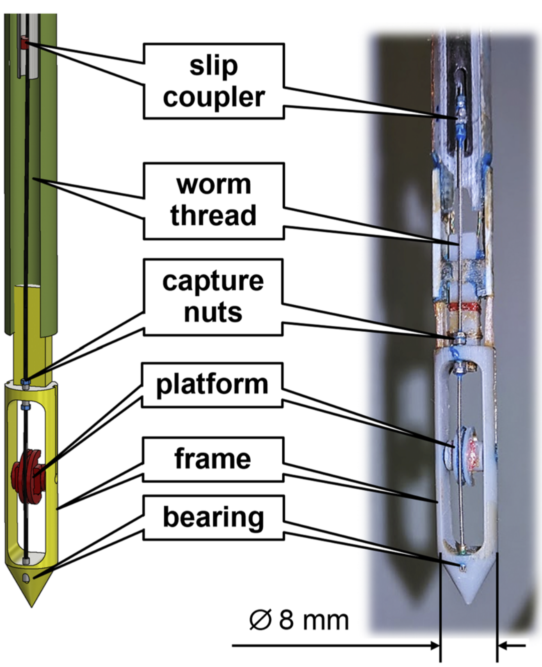

FIG. 1. The modeled (left, Autodesk Inventor drawing) and assembled (right) goniometer. The 3D-printed sample platform can rotate within the 3D-printed frame around the axis supported by two sapphire bearings on each side. The position of the platform is set by turning the thread until the desired angle is reached.

The control knob, which rotates the worm, hermetic mechanical and electric feedthroughs, and other essential components at the top of the 2.5 m-long probe are attached to the bulkhead body of the complex geometry, which was printed using Acura Bluestone® SLA nanocomposite (3D Systems) (Fig. 2). The 3D-printed bulkhead provides both the structural support and the hermetic seal, allowing a controlled sample atmosphere, such as ^{3}He or ^{4}He exchange gas. The hermetic seal is achieved via rubber o-rings compressed between the bulkhead surface and the commercial hermetic feedthroughs, as well as a custom mounting flange on an aluminum enclosure (not shown). The total length of the probe is ~2.5 m, defined by the need to reach the field center of the National High Magnetic Field Laboratory's 100T magnet. The details of the design are provided in the supplementary material.

The angular position of the sample is measured using a mechanical rotary counter at the top of the probe (Fig. 2). One full turn of the counter changes the platform's angular position by 2.6°. The position is checked using a pick-up coil attached to the platform (Fig. 3). A changing magnetic field induces a voltage $V = \mathrm{d}B/\mathrm{d}t * A * \cos(\theta)$ in this coil, where dB/dt is the rate of change of magnetic field, A is the effective area of the coil, and θ is the angle between the coil's axis and the field direction. A second, stationary, pick-up coil is mounted on the frame in a fixed position. The voltage traces from the fixed and tilt coils are recorded simultaneously during the magnet pulse. The integral of the fixed coil signal over time provides the magnetic field as a function of time, while the ratio of the tilt coil

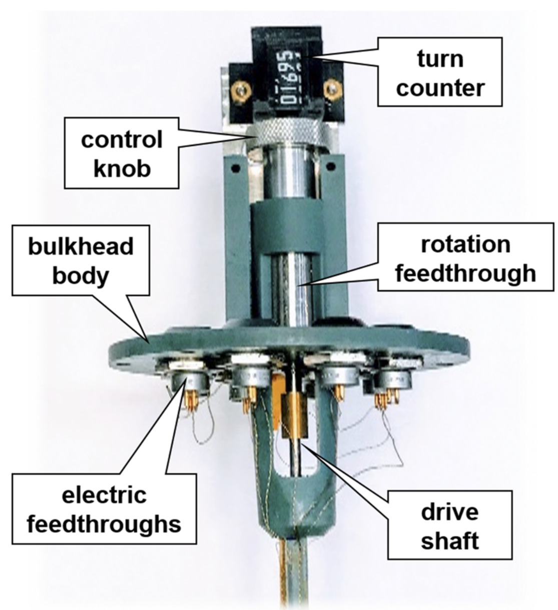

FIG. 2. Hermetic bulkhead assembly. The 3D-printed bulkhead body provides a vacuum-tight structural support for the rotational and electric feedthroughs, and other essential goniometer components.

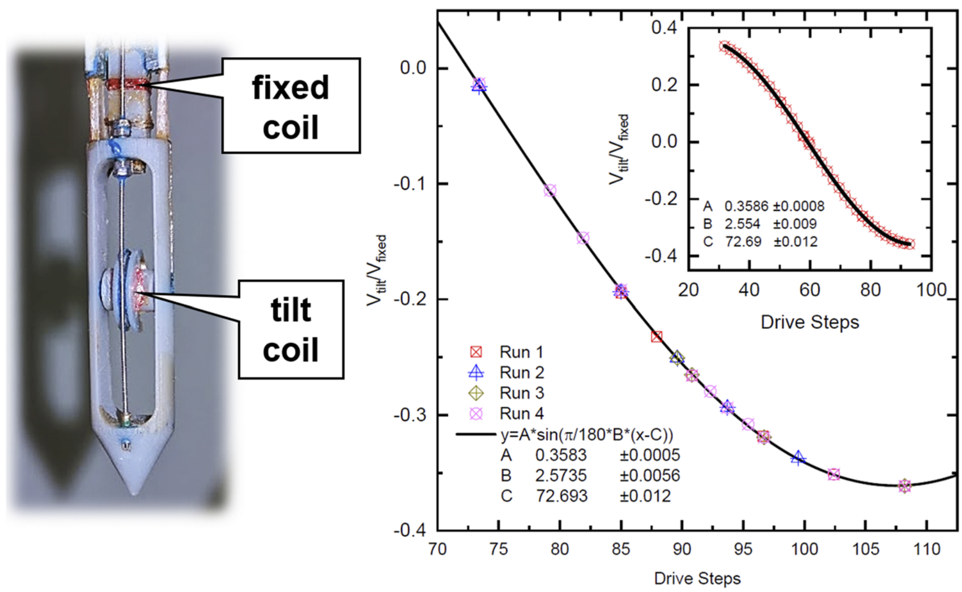

FIG. 3. Test of the goniometer's angular position readout, and its accuracy and reproducibility. The angular position is determined from the ratio of voltage values induced in the tilt to fixed coils by changing the magnetic field. The signal ratio is plotted as a function of the worm rotation counter readings. Different symbols correspond to several different platform rotations. The solid line is a best fit to a cosine function. Insert: ratio values obtained in a wide angular-range sweep with a fit to the cosine function.

signal to the fixed coil signal provides a measure of the platform's angular position.

The accuracy and reproducibility of the goniometer's sample positioning were characterized by recording the ratio of tilt to fixed coil signals over several consecutive platform rotations (Fig. 3). The variation of the ratio as a function of the rotary counter reading was subsequently fitted to a cosine function. The observed angular position reproducibility is ≈0.2°, with no detectable degradation over the 1–2 year probe life cycle.

The goniometer has been used successfully in a variety of pulsed-field experiments: studies of Dirac fermions in heavy-Fermion superconductors,[14] mapping of the Fermi-surface topologies of skutterudites,[15] and measurements of the angular dependence of magnetostriction.[16]

In summary, a cryogenic goniometer has been constructed using 3D stereolithography. The goniometer is suitable for measurements in pulsed magnetic fields of up to 100T. *In situ* sample rotation is realized with ≈0.2° precision at temperatures as low as 500 mK, achieved using a simple ^{3}He vacuum jacket of similar design as in Ref. 6. With recent advances in additive manufacturing,[17,18] 3D printing is fast becoming a viable and affordable technology for pulsed-field and other cryogenic and vacuum applications. Some of the possible applications of the technology developed for cryogenic goniometers under consideration include sample strain actuators and optical assemblies for pulsed-field measurements.

See the supplementary material for details of the testing of additive manufacturing materials and techniques as well as the detailed description of the design of the pulsed field goniometer.

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