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Fast Field Mapping of Large Size Bulk Superconductors at Variable Temperatures and Magnetic Fields

Kévin Berger, Santiago Guijosa, Frederic Trillaud, Bruno Douine and Jean Lévêque

Abstract—The present paper deals with the development of a custom-made system and techniques to address the challenges of the magnetization and characterization of bulk superconductors as large as 100 mm in diameter. The system is composed of a dry superconducting magnet capable of producing a field variation up to 18 T and a measurement setup relying on an advanced field mapping arrangement made of a 10×10 Hall sensor matrix for rapid magnetic field acquisition under 0.5 s. To achieve the desired accuracy, the experimental setup incorporates precise thermal management and calibration. Its purpose is to investigate strategies to enhance the stability, structural integrity, and overall performance of superconducting bulk materials magnetized in high magnetic fields. The goal is to promote scientific collaborations through a dedicated experimental facility for forthcoming magnetization and fast field-mapping studies, thereby supporting the development of bulk superconductors.

Index Terms—Bulk superconductor, characterization technique, field mapping, high field cryogen-free magnet.

I. INTRODUCTION

BULK superconductors offer a number of advantages over conventional magnets; the capacity to trap large magnetic flux densities being the most important. In that regard, we can quote striking examples such as two GdBaCuO bulks (25 mm in diameter) trapping 17.6 T at 29 K [1], a stack of two MgB₂ pellets (20 mm in diameter) reaching up to 6.8 T at 12 K [2], or again a stack of two MgB₂/TiB₂ composites (28 mm in diameter) achieving 5.6 T at 12 K [3]. These impressive achievements have opened the path for numerous applications such as magnetic shielding [4], magnetic lenses [5], motors/generators [6], [7], magnetic levitation [8], magnetic bearings [9], flywheel energy storage [10], NRM/MRI [11] and drug delivery [12], among others.

As outlined in [13], it is essential to address the significant challenges associated with magnetization methods, maintaining the stability of trapped fields, improving mechanical properties of the bulk and enhancing characterization techniques. The main interest revolves around large bulks or bulk array patterns for practical applications. To this end, the GREEN laboratory has allocated both financial and human resources to achieve reliable magnetization of large samples and to assess their properties using the well-established field mapping technique. In this context, the present paper introduces recent solutions to make at the disposal of the scientific community

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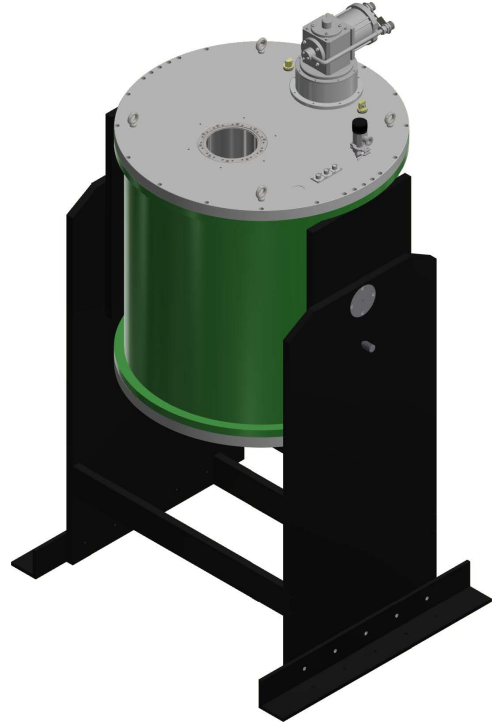


Fig. 1. Computer assisted drawing of the ± 9 T cryogen-free magnet on its rotation stand.

a facility that addresses these obstacles. The second section presents the dry superconducting magnet that can produce variations of up to 18 T (± 9 T) within a 150 mm warm bore. The third section details the sample holder. The fourth section delves into the advanced field mapping system developed at the laboratory, highlighting its rapid data acquisition capabilities and its accuracy in mapping magnetic fields, crucial for evaluating large bulk samples. In the final section, an overview of the samples scheduled for forthcoming study is presented.

II. MAGNET SYSTEM

Cryogenic Ltd [14] designed and manufactured the rotatable cryogen-free magnet according to our specifications. Fig. 1 presents an overview of the magnet system. It consists of a $\Phi 150$ mm ± 9 T NbTi superconducting coil, conduction-cooled by a SHI RDE-418D4 two-stage Gifford-McMahon cold head connected to an F50-SHV compressor unit able to achieve 1.8 W at 4.2 K. Without current, the magnet temperature can drop to approximately 2.7 K. The magnet is powered by an SMS120C-H-4Q-T 10 V four-quadrant power supply that supplies ± 120 A with an output stability of 25 ppm/K. The entire magnet system including the cryocooler is 1.85 m high with a footprint of 1.11 m \times 0.85 m. The field homogeneity

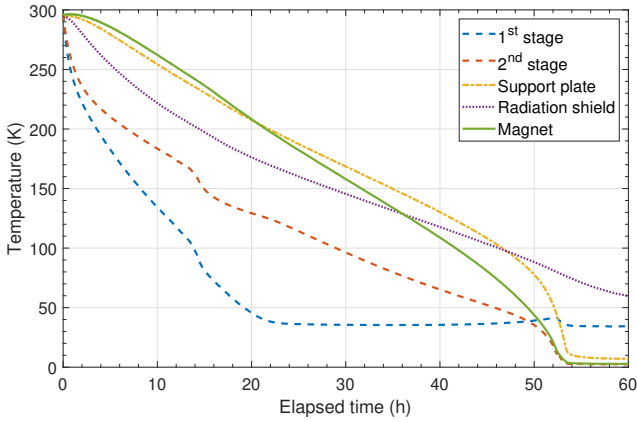


Fig. 2. Evolution of some cryogen-free magnet temperatures during cool down. The magnet is ready for operation after 54 h.

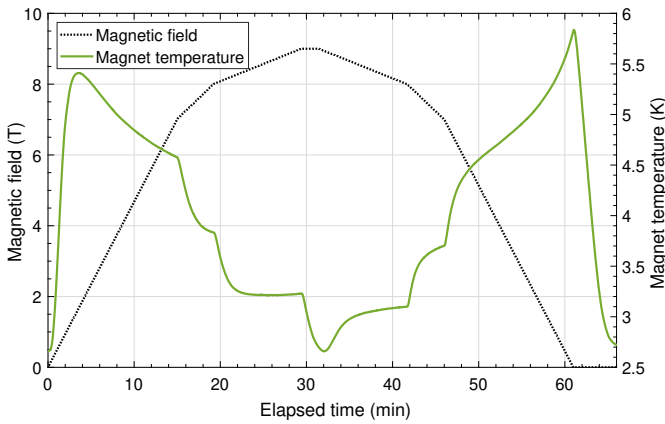


Fig. 3. Magnetic field and temperature evolution using the default ramp-up and down profile, from 0 T to 9 T and back. The temperature minimum at 9 T results from the reduced ramp rate limiting AC losses.

is greater than 98% within a disk of 100 mm in diameter at the middle plane of the coil, while the magnetic field variation is less than 1.5% over a length of 60 mm along the coil axis. This allows uniform magnetization for large bulk materials with a diameter up to 100 mm and a thickness of 60 mm. For typical samples of 30 mm in diameter and 10 mm thick, the field homogeneity over the bulk sample exceeds 99.9%.

Following a 54 h cool-down shown in Fig. 2, the magnet is ready for operation. Setting the output voltage of the power supply to 10 V under an inductance of the NbTi coil equal to 63.6 H, the fastest ramp rate to generate a variable high magnetic field is 0.15 A/s, which corresponds to about 0.7 T/min with a field constant equal to 0.077105 T/A. To prevent heat generation and consequently quenches at high field, the default ramp profile is the following: 0.5 T/min until 7 T, then 0.25 T/min until 8 T and finally 0.1 T/min until 9 T. A complete ramp-up and down to 9 T is shown in Fig. 3. The magnet can stably reach 9 T in less than 30 min with a maximum temperature below 6 K. The minimum magnet temperature observed at 9 T results from the reduced current ramp rate near maximum field, which limits AC losses while the cryocooler maintains its cooling power. Temperatures across the magnet system are recorded with a LakeShore Model 218 cryogenic temperature monitor.

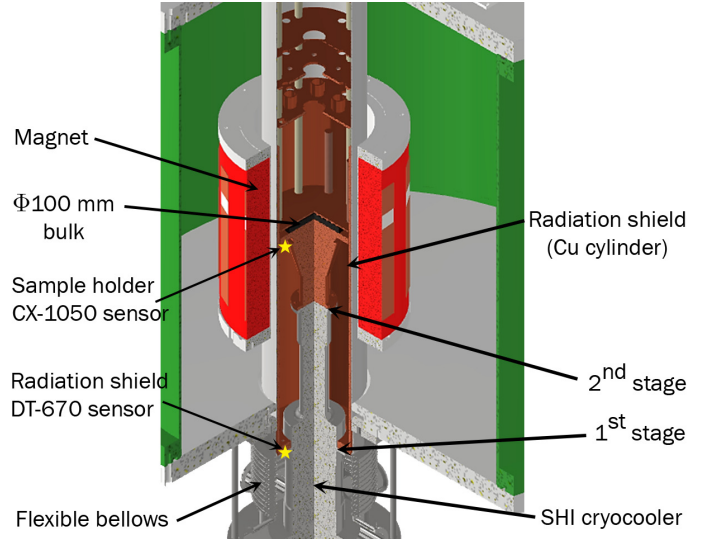


Fig. 4. Three-quarter view of the sample holder inside the magnet system with its associated cryocooler at the bottom.

III. SAMPLE HOLDER

The sample holder was designed in-house to accommodate superconducting samples with a diameter of 100 mm inside the warm bore of the cryogen-free magnet. The design can be easily adapted to the need of the user. For now, it enables precise positioning of the sample at the magnetic center of the magnet, efficient and rapid cooling to low temperatures, and easy and quick mounting and removal of the samples. The cooling is guaranteed by a second SHI RDE-418D4 two-stage GM cryocooler connected to the lower flange of the magnet using a flexible bellow. To ensure a seamless connection between the second stage of the cold head and the sample holder, a conical copper fitting was specifically manufactured, as shown in Fig. 4. This feature ensures optimal centering and maximum surface contact. The first stage of the cold head is fastened to a radiation shield that consists of a cylindrical copper tube having a thickness of 4 mm, a length of 546 mm and an outer diameter of 148 mm. The clearance with the warm bore is about 1 mm (see section II for more details).

The sample holder was optimized to cool large bulks of 100 mm in diameter and 10 mm in thickness to 4 K in less than 4.5 h. The design optimization was carried out using non-linear thermal finite element simulations. A no-load test was performed to check the viability of the proposed solution. The results are reported in Fig. 5. The cool-down time to reach 4 K is about 3.8 h thereby complying with the design specification. At this stage, the radiation shield temperature was approximately 30 K. Noteworthy, the sample holder could be quickly warmed up to room temperature in roughly 2.5 h.

The temperature of the sample holder is measured with a LakeShore CX-1050-CO-HT-1.4M-QT Cernox sensor. The temperature is controlled by a LakeShore Model 336 cryogenic temperature controller, whose 100 W output is connected to two OMEGA KHLVA-203/10-P polyamide flexible heaters in series, giving a resistance of 26 Ohm. The dependence of temperature on output power has been measured and shown in Fig. 6. It provides a more direct control of the sample holder's temperature.

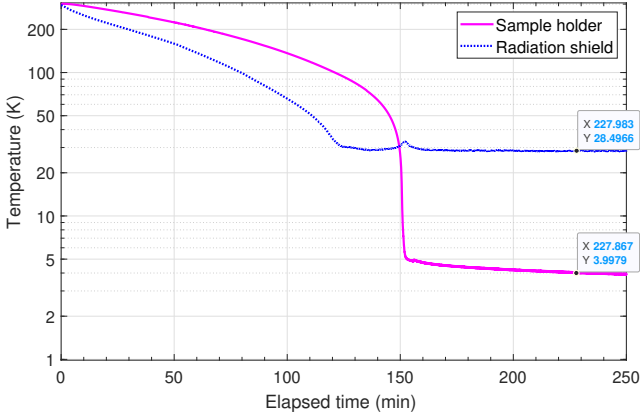


Fig. 5. Evolution of the sample holder temperature under no-load conditions. A temperature below 4 K is reached after 3.8 h (228 min).

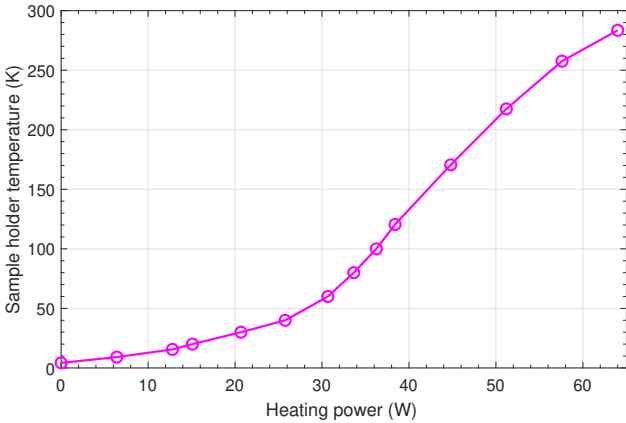


Fig. 6. Dependence of sample holder temperature on LakeShore 336 controller output power.

When measuring the magnetization of large bulks at low temperatures and high fields, it is essential to take into account the forces that can be exerted on the sample. If the sample is offset from the center of the magnetic field, it will be subjected to an upward or downward force depending on the offset. Finite element analyzes were conducted in COMSOL Multiphysics 6.2 to understand the impact of this offset on the mechanical response of samples. The case study is a 100 mm bulk in diameter with a thickness of 10 mm. The $E(J)$ power law is assumed with $J_c = 800 \text{ A/mm}^2$ and an exponent $n = 21$. Figure 7 reports on the resulting axial force experienced by the bulk during a field cooling process at 9 T. The initial field cooling is followed by a full magnetic cycle (9 T \rightarrow 0 T \rightarrow -9 T \rightarrow 0 T) for which different magnetic alignment offsets are considered. For instance, if the center of the bulk is off by +10 mm compared to the magnetic center of the magnet system, a resulting maximum axial force of 3.15 kN (320 kg) is reached near -9 T. This force is rather large and would lift the bulk from its support if no precautions are taken. Hence, mechanical features were implemented to ensure that the sample is tightened to the sample holder aligning magnetic centers and that the sample holder is itself mechanically stable.

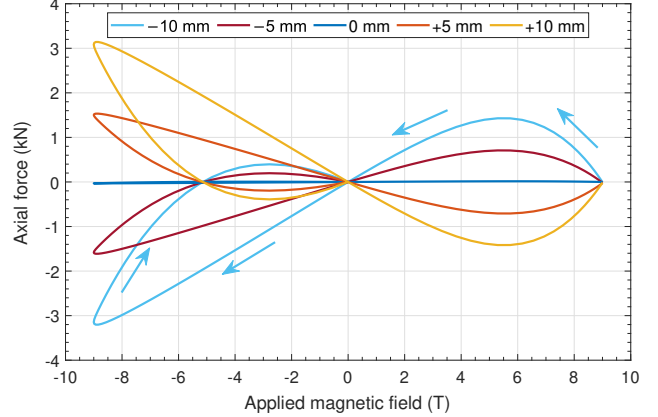


Fig. 7. Results on the axial force generated on a 100 mm diameter bulk during a field cooling process followed by a full magnetic cycle 9 T \rightarrow 0 T \rightarrow -9 T \rightarrow 0 T. Different offsets from the magnetic center are considered.

IV. FIELD MAPPING SETUP

Magnetic field characterizations of bulks are usually done using Hall probes. They can be either fixed on the sample at different locations [15]–[17] or moved around using a three-axis positioning system [18], [19]. The first solution offers the possibility of tracking the instantaneous evolution of the magnetic field in a limited number of locations, whereas the second solution can map the magnetic field over the whole surface of the bulk at the end of the magnetization. Depending on the spatial resolution of the positioning system, typically 1 mm, acquiring the map over a 30 mm diameter bulk may last about 20 min. This time span may introduce variation of the trapped field due to flux relaxation. To the best of our knowledge, a unique two-dimensional technique for dynamic mapping of entire bulks was developed by T. Ida [20]. The system made of a 5×5 Hall probe matrix (each probe spaced 10 mm apart) demonstrated its capability to acquire the magnetic field every 0.5 ms. The aim was to develop a 15×15 matrix using the same setup. The details of the setup were recently published in 2024 [21]. It enables the magnetic field to be acquired every 1 ms by a 15×15 Hall probe matrix with probes spaced 5 mm apart. Based on the same principle, we have developed three PCB holding a 10×10 LakeShore HGT-2101 Hall sensor matrix. The probes are spaced either 3 mm, 5 mm or 7 mm apart to cover bulk diameters ranging from 20 mm up to 90 mm. The configuration needs 200 wires for voltage pick-ups and 11 wires for current supply as the ground is shared. Heat load and wire management posed significant challenges, leading to the development of a specialized design utilizing LakeShore WQT-32 phosphor bronze Quad-Twist wire (32 AWG). The wires are 1.5 m long connecting 300 K to 4 K. Joule losses in the wires are approximately 0.7 mW, while conduction losses amount to roughly 80 mW. All of these losses are therefore negligible for the cryogenic system.

The PCB are designed to be easily interchangeable and reconfigurable, with a minimum adjustable pitch of 3 mm as shown in Fig. 8. A nominal current of 1 mA is applied sequentially to each row of 10 Hall probes connected in series. The voltage of each probe is recorded through a multiplexer

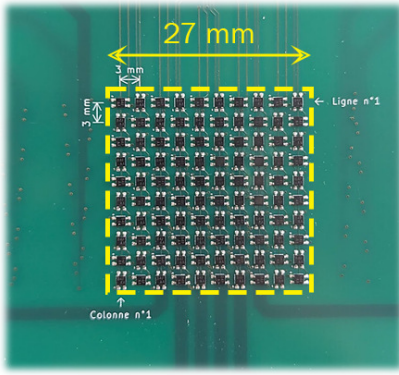


Fig. 8. Photograph of the 10×10 Hall probe matrix with a pitch of 3 mm on its PCB. Two additional PCB are available with pitches of 5 mm and 7 mm.

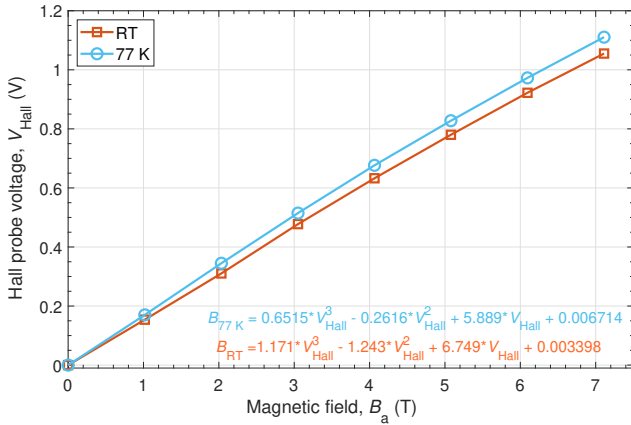


Fig. 9. Average voltage of eight HGT-2101 Hall probes as a function of the applied magnetic field at room temperature (RT) and 77 K.

system. This process is repeated for each row until all the data have been acquired. The multiplexer system consists of a Keithley 3706A Switch/Multimeter, which offers a $7\frac{1}{2}$ -digit resolution at 5 power line cycles (PLC) for precision and a $6\frac{1}{2}$ -digit resolution at 0.5 PLC to enhance speed. For mapping the 10×10 Hall sensor matrix, the multiplexer is equipped with one Model 3761 10-channel low current multiplexer card and two Model 3723 dual 1×30 high-speed multiplexer cards. According to the datasheet, when operating at 0.5 PLC with a 1 V DC range, the noise level is maintained below $10 \mu\text{V}$. Additionally, within this rate, all data is successfully transferred to the computer in 0.5 s.

To guaranty measurement accuracy, all 100 LakeShore HGT-2101 Hall probes were calibrated for both field strength and temperature. Figure 9 shows how the average voltage of the eight sensors varies with the magnetic field at room temperature and 77 K. The reciprocal third-order polynomial function used to determine the magnetic field at both temperatures is also reported. It should be noticed that using a linear approximation for calibrating the Hall probes results in errors of $\pm 5\%$ on the magnetic field. Similarly, it was found that the use of the calibration curve at room temperature to obtain the field at 77 K showed an artificial increase in the estimated magnetic field of 10%. It was then concluded that calibration of the sensors at different temperatures and, in particular, at the operating temperature of the sample, is necessary to reach the best achievable accuracy.

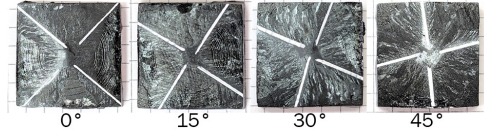


Fig. 10. Square YBCO bulks with a side length of 27 mm having their seed orientation tilted by 15° . Samples from Barış Güner, Recep Tayyip Erdoğan University, Turkey.

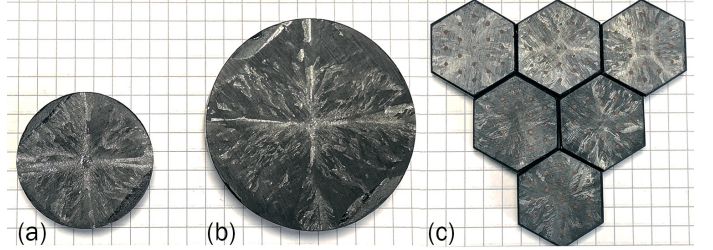


Fig. 11. YBCO bulks from Can Superconductors [24]: (a) CSYL-35, (b) CSYL-56 and (c) five hexagonal-shaped bulks with artificial holes filled with epoxy and copper arranged in a hexagonal pattern.

V. SAMPLES TO BE INVESTIGATED

The first batches of samples to be magnetized and studied with this new setup are shown in Fig. 10 and Fig. 11. The initial batch to undergo magnetization and mapping is four square YBCO bulks shown in Fig. 10, each with a side length of 27 mm and an approximate thickness of 11 mm, with their seed orientation tilted by 15° . The second batch of samples (Fig. 11) are commercial REBCO bulks from Can Superconductors: two sets of three cylindrical samples, each with diameters of 35 mm and 56 mm, and a set of seven hexagonal-shaped YBCO bulks with artificial holes filled with epoxy and copper, arranged in a hexagonal pattern. Other REBCO materials to be investigated include Eu-123, Gd-123, and (Nd, Eu, Gd)-123 samples. In addition, a MgB_2 pellet fabricated through the reactive Mg-Liquid infiltration process [22] with a diameter of 50 mm and a height of 20 mm and a large high-density MgB_2 produced by Spark Plasma Sintering [23] with a diameter of 100 mm and a height of 10 mm will be next for field mapping.

VI. CONCLUSION

A detailed description of a magnetization system designed for studying large HTS bulks has been introduced. This system can produce rapid field variations up to 18 T at different operating temperatures. Cooling times, maximum field ramp rates, operating temperatures, and the precision of devices that enable the quick acquisition of magnetic field map in 0.5 s have been reported. The measurement setup uses a 10×10 LakeShore HGT-2101 Hall sensor matrix. Some thorough numerical studies have been carried out to ensure the safe and reliable operation of the sample holder inside the warm bore of the magnet system. The measurement system is calibrated to achieve the best possible accuracy. The system is now ready for operation and intended for forthcoming magnetization and fast field-mapping experiments on large bulk superconductors, including REBCO and MgB_2 materials. Our aim is to promote the platform as a collaborative facility, enhancing accessibility for the global scientific community to an up-to-date experimental setup.

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