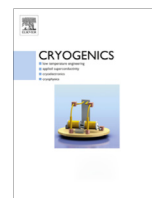


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Research paper

Forced flow He vapor cooled critical current testing facility for measurements of superconductors in a wide temperature and magnetic field range

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ABSTRACT

As superconducting materials find their way into applications, there is increasing need to verify their performance at operating conditions. Testing of critical current with respect to temperature and magnetic field is of particular importance. However, testing facilities covering a range of temperatures and magnetic fields can be costly, especially when considering the cooling power required in the cryogenic system in the temperature range below 65 K (inaccessible for LN₂). Critical currents in excess of 500 A are common for commercial samples, making the testing of such samples difficult in setups cooled via a cryocooler, moreover it often does not represent the actual cooling conditions that the sample will experience in service. This work reports the design and operation of a low-cost critical current testing facility, capable of testing samples in a temperature range of 10–65 K, with magnetic field up to 1.6 T and measuring critical currents up to 900 A with variable cooling power.

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1. Introduction

The current a given superconductor can carry depends on a multitude of variables including temperature, magnitude of the magnetic field and its direction. These data are important when designing and optimizing conductor materials for applications, and can serve as inputs for computer models [1,2] used to validate new designs of superconducting devices.

Critical current, I_c , is often measured for a sample immersed in a cryogenic liquid such as liquid nitrogen (77.4 K) or liquid helium (4.2 K). Close contact with the cooling medium provides cooling for non-superconductive parts of the testing rig, i.e. current leads and sample contacts. However, for intermediate temperatures, choices of cooling system are limited. Cryocoolers are increasingly popular, but their cooling power is still fairly modest, cool-down to testing temperature can take hours and a substantial capital investment is required, making them unfeasible for smaller institutions. On the other hand, flow cryostat facilities tend to be bulky and require a magnet to be integrated into the system, increasing the cost [3,4].

We propose a new, low-cost system for critical current testing in a wide range of temperatures from 10 to 65 K and magnetic

fields of up to 1.6 T, which in this case was limited by the electromagnet used. Due to the low achievable base temperature, the system is suitable for critical current measurements of materials such as MgB₂, rare-earth cuprates and iron-based superconductors. Samples of up to 16 cm in length can be measured. The system allows for testing of a variety of materials in a cost-effective manner. The design and construction of the critical current measurement system is outlined in Section 2, and initial data collected with the system are presented in Section 3.

2. Design of the critical current measurement system

The design of a critical current measurement system must take into account a number of design considerations and constraints, including cryogenic insulation and temperature control, very large current handling capability, applied magnetic field and forces on the sample and the system components due to said field. The cryogenic environment and the presence of large magnetic fields also dictates the choice of materials and components. Each of these aspects will be considered in the following subsections.

2.1. Overall design

A schematic outline of the system is provided in Fig. 1. The simplicity of the concept allows the system to be modular and easy to

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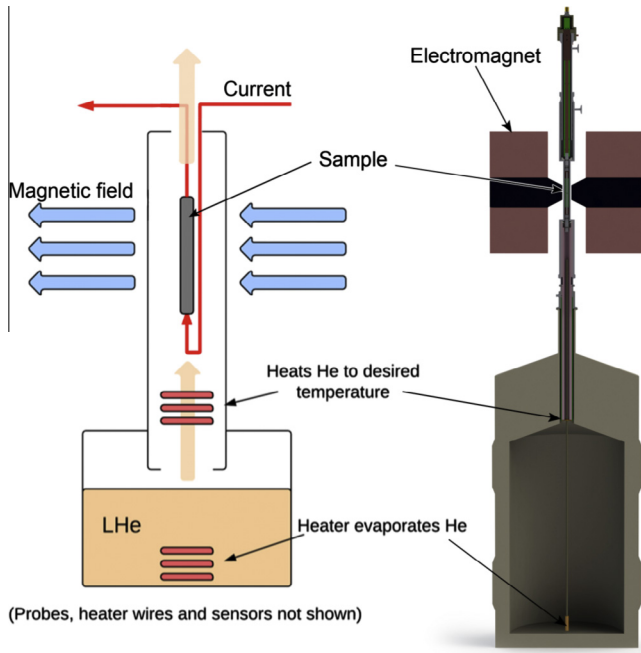


Fig. 1. A schematic sketch of the critical current measurement system. Probes, heater wires and sensors are not shown. Blue arrows indicate the applied magnetic field, while LHe denotes liquid helium.

maintain. A vacuum walled sample tube is placed on the liquid helium transfer dewar, without tampering with the safety features and pressure release valves of the dewar. A heater inside the dewar is used to evaporate the helium (evaporation heater) that controls the gas flow rate of helium vapor, whilst another heater in the neck of the dewar is used to adjust the temperature of the vapor (gas heater), and hence control the sample temperature. The gas heater is controlled via a three term proportional-integral-derivative control loop (PID loop) to set the sample temperature. Having two heaters allows the helium flow rate and sample temperature to be set independently. The resulting helium boil-off can, in principle, be collected for re-liquefaction.

The magnetic field was applied externally by an electromagnet, but any other source of magnetic field can be used, e.g. a permanent magnet. In each case, fields approaching 2 T can be achieved, even without the use of superconducting magnets.

Lastly, as soon as the desired temperature is stabilized and the magnetic field applied, current is ramped through the sample until the critical current criterion is met. The procedure is repeated at multiple temperatures and magnetic field values in a sequential manner to characterize the sample with minimum liquid helium usage. At this point the system can be warmed up and another sample mounted.

2.2. Construction and materials used

The design of the system is modular. The system is composed of three discrete components as illustrated in Fig. 2. The bottom part inserts into the dewar and contains two heaters, whose purpose is discussed in Section 2.3.

The midsection is primarily structural. The narrow section is only 25 mm wide, allowing for the spacing of the electromagnet poles to be minimized, increasing the magnetic flux density. The whole component is vacuum-walled and contains guides for the topmost part, which holds the sample.

The topmost part holds the sample board and all of the sensors and measurement wires. Its vertical position can be adjusted, so

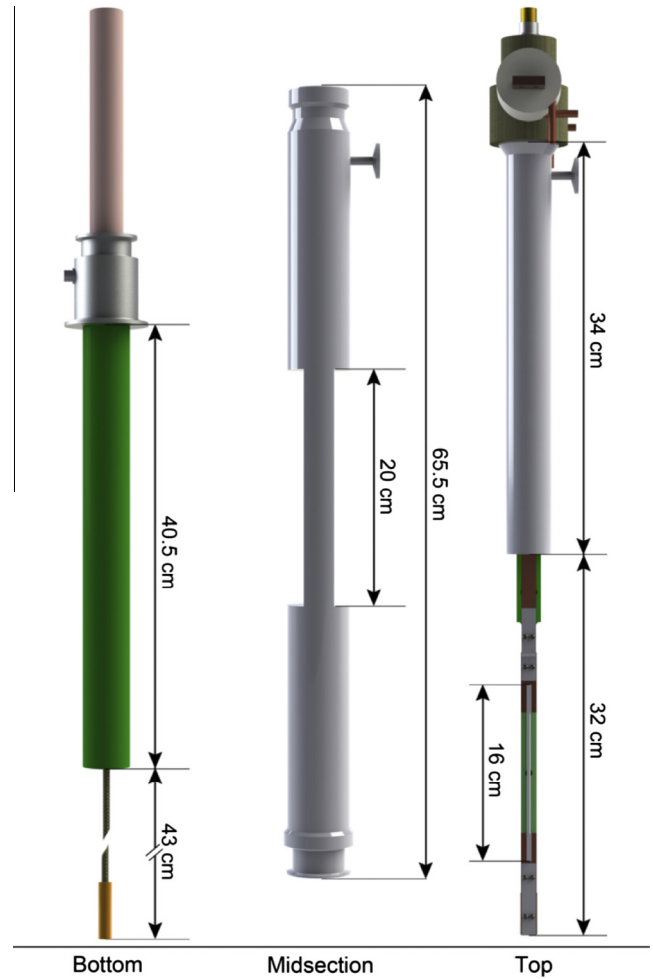


Fig. 2. The illustration shows the three separate parts forming the critical current testing facility, starting from the bottom-most part on the left, which inserts directly into a regular helium transfer dewar. The sample is placed on the topmost part, which slides into the midsection.

that the sample can be centered between the electromagnet poles. Furthermore, the topmost part can be rotated about its axis to orient the sample in the magnetic field. Hence, conductor anisotropy of I_c in magnetic field can be measured.

The low temperatures and high magnetic fields put constraints on the materials used in the system. Therefore, the vacuum walled parts were made out of non-magnetic stainless steel 316 with outer walls being thicker for structural rigidity (2 mm and 1 mm wall thickness for the midsection and topmost parts respectively), while the inner walls were made thinner to minimize thermal mass (less than 0.5 mm in each case). Care was taken to choose non-magnetic parts for all components near the magnetic field. The sample may experience significant forces during measurement, hence the central sample support in the topmost part was chosen to be glass-fiber/epoxy composite G10 from Tufnol due to its excellent stiffness, low thermal conductivity and excellent electrical insulation characteristics. Other parts that require non-conductive material like the cap on the topmost part were also manufactured out of Tufnol composite, but with cotton fibers due to easier machinability.

2.3. Cooling system and thermal insulation

The temperature is actively controlled via two heaters, made of coiled resistive Kanthal wire. Heater 1, referred to as the evapora-

tor, controls the flow rate of helium gas in the system and thus the minimum achievable temperature. Fig. 3. illustrates the temperatures achieved for given helium consumption. The temperature is asymptotic to 10 K, which is set by the thermal insulation of the cryostat and the heat leak through the current leads to the sample. The helium consumption is relatively small for temperatures above 15 K, considering that a typical critical current measurement takes less than 2 min, and the temperature can be stabilized in less than 5 min if the temperature set-point is changed by less than 20 K. Given the increasing helium price [5] it is important to keep the helium usage as low as possible.

In most cases it is convenient to decouple the helium gas flow rate and sample temperature, to obtain variable cooling power at a constant temperature. Therefore, the gas heater, located in the neck of the dewar, is used to adjust the temperature of the helium gas to the required value. The gas heater is in a PID loop with sample temperature as the input, which helps to eliminate temperature drift as the system cools or during a current ramp.

Current to the sample is supplied via superconducting (RE)BCO coated conductor leads, however they are coupled to the outside by large copper terminals, required due to currents of up to 900 A. These terminals present a thermal leak into the system. Moreover, the junction between the superconducting current leads and the copper terminals must be kept below the critical temperature of the current leads by an acceptable margin. For these reasons, the copper current terminals are pre-cooled by liquid nitrogen. Fig. 5. shows the copper tubing coiled around and soldered to the current terminals. The copper tubing is encased in polystyrene insulation, to further minimize the heat leak. Liquid nitrogen is passed through the copper tubing. The tubing on the two copper terminals can be connected in series via a non-conducting link. The precooling helps to dissipate the heat generated in the copper during a current ramp and anchor the temperature of the top of the superconductive current leads close to ~77 K.

One of the key features of the cryostat is the narrow section where the sample is contained, allowing the poles of the electromagnet to be separated by only 25 mm. The sample space is 16 mm wide leaving a thermal gradient of up to 280 K over 4.5 mm. For this reason, the thermal insulation of the cryostat is of paramount importance. The cryostat is composed of two cylindrical parts labeled as the midsection and topmost parts in Fig. 2, each of which are vacuum walled. The two parts slide into each other and are linked by an Ultra-Torr compression fitting from Swagelok®. This allows the vertical sample position as well as orientation to be changed, while overlapping vacuum insulated

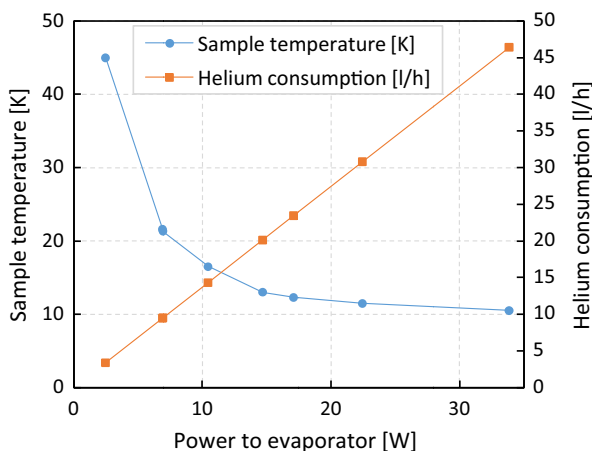


Fig. 3. The minimum sample temperature and helium consumption rate for a given power supplied to the evaporation heater.

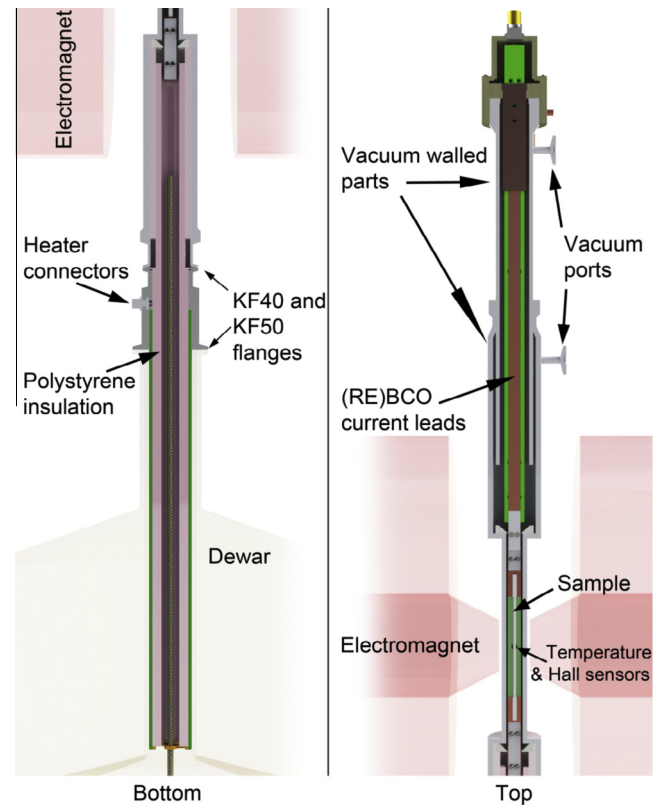


Fig. 4. Illustration of various parts of the cryostat. The image is split into two parts, the left image showing the bottom half and the right showing the top part of the cryostat.

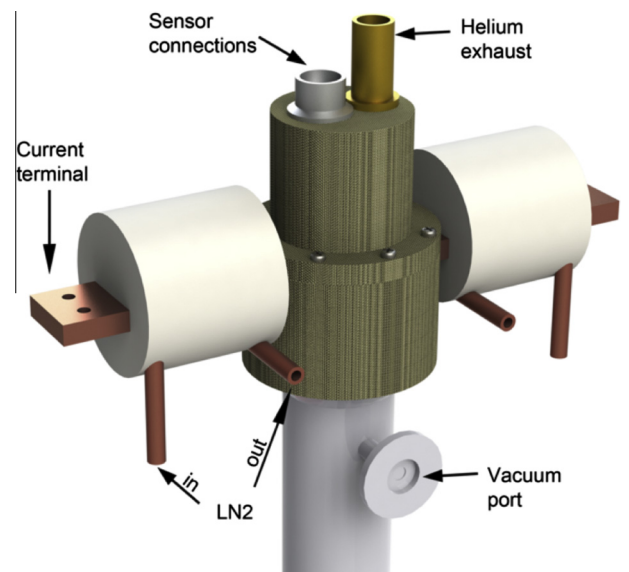


Fig. 5. Illustration showing the copper terminals supplying the current to the sample. The terminals are pre-cooled via liquid nitrogen passed through copper tubing soldered to the current terminals. The copper tubing is encased in polystyrene insulation.

sections maintain good thermal insulation. The vacuum walls are evacuated prior to each experiment and sealed by a vacuum valve. Even better performance is expected if the cryostat is pumped continuously during the experiment. The link between the cryostat and the dewar is insulated by overlapping sections of expanded polystyrene as illustrated in Fig. 4.

2.4. Estimation of thermal losses in the system

To estimate the thermal losses and identify possible ways to reduce helium consumption, four cryogenic temperature sensors were placed in different parts of the system, allowing heat losses to be estimated in different segments of the system from the thermal gradients. The measurements were made once the system reached thermal equilibrium for estimates of static heat loss. It is worth noting that temperature gradients across the system are much lower during actual sample testing, as a higher helium flow rate is used than the minimum needed to reach the target temperature. Instead, the second (gas) heater is used to maintain the desired temperature. Also segment 4 in Fig. 6 does not contribute to the sample temperature as it is upstream of the sample, and a high temperature gradient is expected as the top of segment 4 is weakly clamped to 77 K by the LN₂ cooled copper terminals.

Initial tests showed substantial heat losses in segment 2, exceeding 180 W at a He flow rate of 1.6 g/s. The losses were successfully reduced to 17 W by placing the gas heater lower down in segment 1, and installing overlapping polyurethane foam insulation on the inside, extending across the joint connecting the bottom and midsection parts of the system (see Fig. 4).

At the lowest temperatures, heat losses are dominated by segment 3, which are due to radiation losses across the vacuum double wall (up to 10 W by approximating the segment as concentric cylinders and assuming an emissivity of 0.6 for unpolished stainless steel), radiation from the top of the system and conduction across the sample rod and current leads (<1 W), and residual pressure in the vacuum walled cryostat as it is not pumped during the experiment. Localized frost points were observed at low temperatures in segment 3, and contact bridges between vacuum walls were suspected due to imperfections in the manufactured part and the small separation of the walls, with the vacuum gap of less than 2 mm. These contact bridges are expected to contribute significantly to losses at low temperatures.

2.5. Sample mounting and current leads

One of the major considerations when designing the system was achieving fast sample exchange, whilst retaining the possibility of remeasuring samples at a later date without re-soldering. The samples are soldered to a sample board only once, and the sample

is kept on the sample board permanently. The sample board is affixed to the measuring probe via flexible silver joints as shown in Fig. 7. It is expected that the largest heat source in the system will be the sample contacts, hence to further decrease the contact resistance a strip of indium foil is inserted between the silver tabs and the sample. Samples of up to 16 cm in length can be measured, ensuring that at the measuring point all of the current is in the superconductor even if the superconducting wire/tape has a high resistivity sheath material [6,7]. The voltage drop, temperature and magnetic field are measured in the center of the sample board. The sample board has a hole for a carbon ceramic temperature sensor which is attached to the test probe as illustrated in Fig. 7. Apiezon N thermal grease is applied between the sample and the temperature sensor to ensure good thermal contact. Two perpendicular GaAs based Hall effect sensors HG-116C from Asahi Kasei are placed next to the temperature sensor, to measure the magnetic field strength and its orientation. The voltage drop across the superconductor is measured across 1 cm above the temperature sensor.

Current to the sample is supplied by superconducting current leads. Each current lead is composed of three stacked (RE)BCO tapes supplied by SuperPower: details are given in Section 3.1. The operating temperature of the current leads is kept below 77 K. The temperature of the current leads is monitored near the terminations with a carbon ceramic temperature sensor and the current is cut automatically if for some reason it exceeds 77 K to prevent damage. Also, in case of a short term overcurrent, a 100 μm thick silver shunt is attached parallel to both current leads.

Superconducting current leads help to save space and reduce heat leak into the system, however, they also limit the upper operating temperature of the system. The current leads must be maintained superconducting during operation, unless very small currents are used. The silver shunt can carry a current of 10 s of Amps without significant heating. This makes the system usable for measurements near T_c of high temperature superconductors (HTS) such as rare earth barium copper oxide, (RE)BCO, as well.

2.6. Control and measurement system

A simplified block diagram of the control and measurement system is depicted in Fig. 8. The temperature is controlled via a PID loop implemented in software. Sample temperature (process vari-

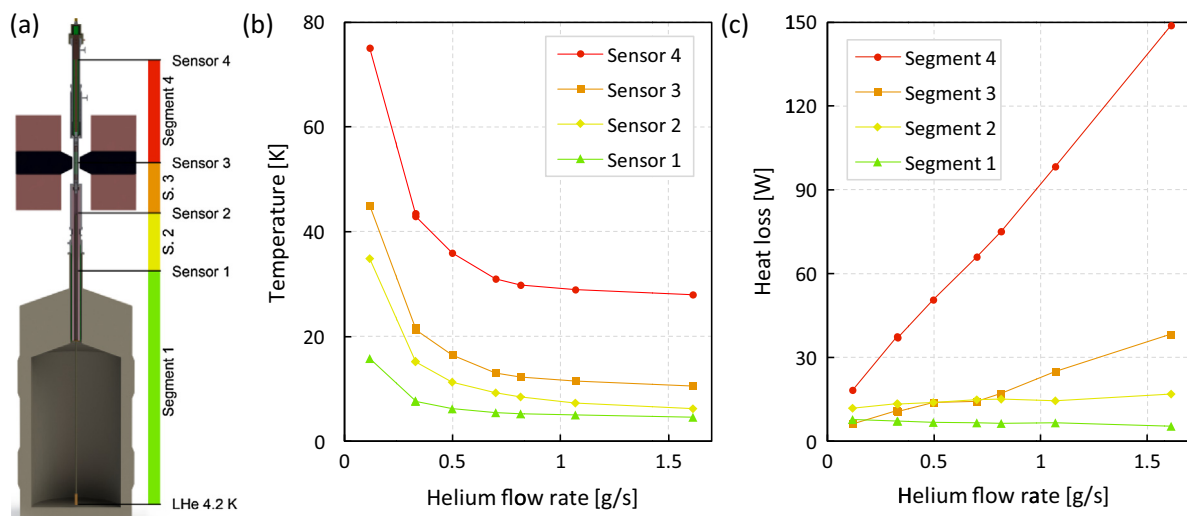


Fig. 6. The temperatures (b) at different positions (a) along the system as a function of helium flow rate, as well as estimates of the heat loss in the segments between the measured temperature sensors (c).

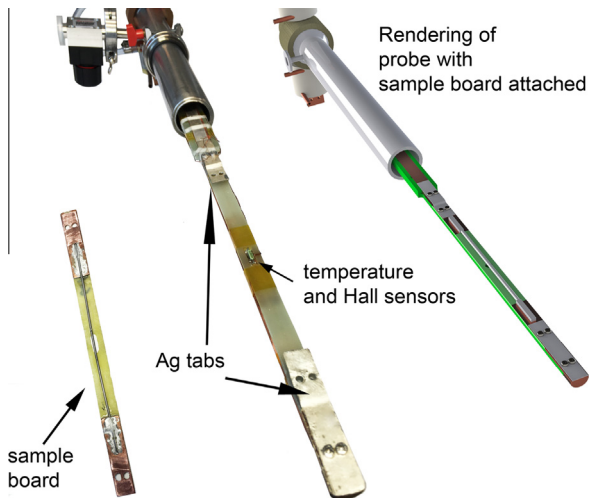


Fig. 7. Photographs of the sample board with a MgB_2 sample illustrating how it is attached to the measurement probe. The location of the temperature and Hall sensors is also indicated.

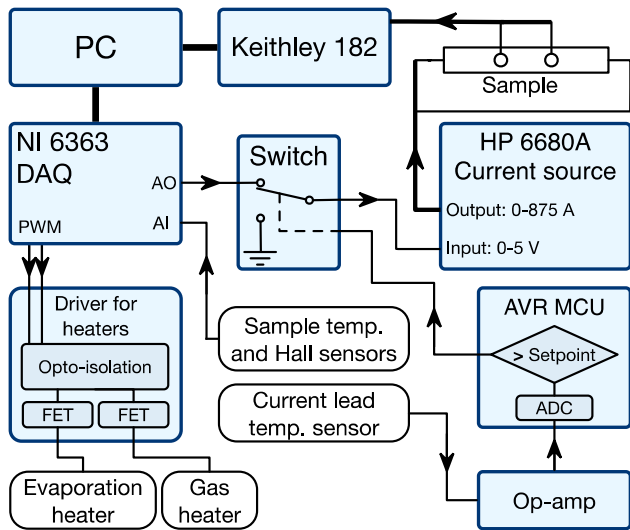


Fig. 8. Simplified block diagram of the measurement and control equipment used. Acronyms used: AI/AO – analog input/output; PWM – pulse width modulated signal; FET – field effect transistor driving the heaters; AVR MCU – microcontroller with ADC (analog-to-digital converter) outputting a high signal when temperature of current leads exceeds a set point.

able) is measured via a National Instruments (NI) 6363 digital acquisition system (DAQ). The gas heater power (control variable) is controlled by a pulse width modulated (PWM) signal generated by the DAQ which is fed into an opto-isolated driver circuit. The power of the evaporation heater is set independently.

Current to the sample is supplied by a Hewlett Packard (HP) 6680A current source controlled via an analog input by the National Instruments DAQ. The actual current supplied to the sample is read back directly from the power supply via GPIB interface. The current can be cut quickly if the temperature of the current leads becomes too high. This is done by monitoring the temperature sensor voltage is first amplified by an instrumentation amplifier then read by a microprocessor. If the temperature exceeds a set point of ~ 77 K, the microprocessor outputs a high signal to an analog switch IC, which grounds the input of the current source. Implementing the protection in separate hardware adds redundancy

and avoids communication delays that are present between the DAQ and PC.

The magnetic field is applied using a Walker Scientific HV-4H electromagnet with 2-inch diameter field poles. The magnetic field and its direction is measured with Hall probes on the sample probe using the NI 6363 DAQ. The voltage drop across the superconducting sample is measured using a Keithley 182 sensitive digital voltmeter. The experiment was controlled using bespoke software.

3. I_c measurements of commercial (RE)BCO coated conductor and a MgB_2 wire samples

For a system test run, the critical current of a 2G HTS (RE)BCO tape from SuperPower was tested, with the same specification as the one used for the current leads in the system. The sample was tested at a magnetic flux density of 1 T with magnetic field parallel and perpendicular to the tape surface in a temperature range from 40 K to 60 K. A MgB_2 wire was also tested at 1 T in a temperature range from 20 to 40 K.

3.1. Samples

The (RE)BCO sample measured was produced by SuperPower with specification SCS12050-AP. The tape has a width of 12 mm, 50 μm Hastelloy substrate, 2 μm silver over-layer and 20 μm copper stabilizer. At 77 K and self-field the tape has a minimum I_c of 309 A, while the average is 340 A, as stated by the manufacturer. Given the large critical currents expected at a field of 1 T (I_c for the full width sample exceeds 1000 A at 45 K), the tape width was reduced to 4 mm for testing. The critical current was also measured at 77 K in liquid nitrogen.

The MgB_2 sample was produced by Columbus Superconductors, specification 700RM13609. The wire is 1.52 mm in diameter and has a Monel sheath. The conductor contains 37 MgB_2 filaments in a Ni matrix. Reference data from the manufacturer for a wire of this specification are shown in Fig. 9.

3.2. Experimental setup

The system described in Section 2, together with a liquid helium dewar, was used to measure the critical current. The sample voltage was measured across 1 cm of the superconductor and a criterion of $1 \mu\text{V}/\text{cm}$ was used to determine the critical current. The current ramp rate of 8–10 A/s was used for critical currents larger

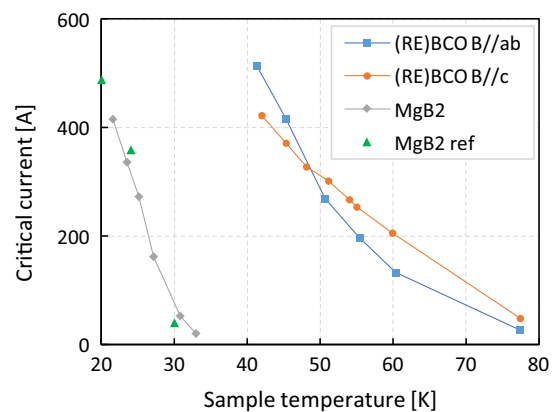


Fig. 9. Critical current data measured with the system described in this work. 4 mm wide sample of (RE)BCO tape was tested at a field of 1 T parallel and perpendicular to the width of the tape (near a-b lattice plane). Measurements at 77 K were made in liquid nitrogen. MgB_2 sample was also tested at a field of 1 T and displayed in the graph together with reference data obtained from manufacturer (MgB_2 ref series).

than 100 A, and as low as 5 A/s for lower currents. Sample voltage and current measurements were taken at a rate of 4 samples/second.

3.3. Results

The critical current of the (RE)BCO coated conductor was measured at 1 T with the direction of the magnetic field parallel and perpendicular to the plane of the tape, and perpendicular to the current direction. The results are shown in Fig. 9. Critical currents in the temperature range of 40–60 K were measured using the system described in Section 2, whereas the measurements at 77 K were performed in liquid nitrogen. Measurements from the MgB₂ sample are also included, along with reference data from the manufacturer for wire of the same specification. The measurements match the reference data well. Some deviations from the reference data are expected as the supplied reference data are not for the same batch.

Due to the low thermal mass of the system, the time required to stabilize at a new temperature was below 1 min for temperature steps as large as 10 K. The overall cooling time to 60 K from room temperature was about 8 min once the probe was attached to the helium dewar. Cooling in a gaseous medium provides some flexibility of sample geometry, however it also presents some challenges. During the superconducting-normal transition, heat is generated in the sample, and due to the steep *I*-*V* curve, the sample temperature may suddenly change, which is hard to adjust for quickly. This is partially helped by increasing the helium gas flow (but maintaining the same temperature) which effectively increases the cooling power in case of a sample temperature change, however this also increases the helium consumption. Due to these effects it may be difficult to determine accurate *n*-values, but the *I*_c value is more accurate due to the *n*-value being large in most cases. Possible system improvements to allow *n*-value estimation are outlined in Section 4. Nevertheless, temperature stability better than 0.2 K is maintained during the current ramp before the onset of the transition.

4. Future improvements

Further planned improvements to the system include faster sample voltage acquisition, allowing *n*-value measurements in the range of 0.1–1 μV/cm where sample heating is negligible, as in [8]. Currently the acquisition rate is limited to 4 samples/second: readings are transferred from the Keithley 182 in real time over GPIB, to allow the current ramp to be terminated under software control when a threshold voltage is reached. This rate can be increased to 15 samples/second by buffering samples before readout, triggering the end of the current ramp using the pre-amplified analogue output of the Keithley 182. This would also allow the ramp to be ended more quickly at the pre-set threshold voltage, reducing over-heating of the sample, which is particularly useful for measuring poorly-stabilized samples.

There is scope to further reduce heat losses by continuously pumping the vacuum spaces. Some helium is also lost during insertion of the probe into the dewar that could in principle be used for pre-cooling the system. Nevertheless, full characterization of a sin-

gle sample can be achieved with 5–10 l of liquid helium, depending on the temperature range and number of data points gathered. For example, characterization of the MgB₂ sample at 1 T used 5–6 l of liquid helium, with a significant fraction of this expended during the initial insertion of the probe into the dewar.

Currently the sample orientation with respect to the magnetic field is changed manually, but this could be automated using an approach similar to the one described in [9].

5. Conclusions

The design of a low-cost critical current measurement system for superconductor characterization was presented. Temperatures down to 10 K can be achieved and the design allows for variable cooling power (helium flow rate) at a constant temperature. The superconducting current leads allow for a very compact design with low thermal mass, minimizing the helium consumption while cooling down and maintaining a stable temperature. Custom computer software enables rapid measurements with minimal time between experiments, further reducing helium usage. The system was tested in operation with HTS (RE)BCO and MgB₂ wire samples. The system performed as expected, and critical currents measured in the range from ~20 to 60 K were in good agreement with reference values.

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