Transport J_c in Bulk Superconductors: A Practical Approach?

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***Abstract*— The characterisation of the critical current density of bulk high temperature superconductors is typically performed using magnetometry, which involves numerous assumptions including, significantly, that J_c within the sample is uniform. Unfortunately, magnetometry is particularly challenging to apply where a local measurement of J_c across a feature, such as a grain boundary, is desired. Although transport measurements appear to be an attractive alternative to magnetization, it is extremely challenging to reduce the cross-sectional area of a bulk sample sufficiently to achieve a sufficiently low critical current that can be generated by a practical current source.**

In the work described here, we present a technique that enables transport measurements to be performed on sections of bulk superconductors. Metallographic techniques and resin reinforcement were used to create an I-shaped sample of bulk superconductor from a section of Gd-Ba-Cu-O containing 15 wt % Ag_2O. The resulting superconducting track had a cross-sectional area of 0.44 mm^2. The sample was found to support a critical current of 110 A using a field criterion in the narrowed track region of 1 $\mu V\ cm^{-1}$. We conclude, therefore, that it is possible to measure critical current densities in excess of 2.5 x 10^8 A m^{-2} in sections of a bulk superconductor.

***Index Terms*— Critical current density, current transport measurements, high temperature superconductors, rare-earth barium copper oxide**

I. Introduction

The critical current density, J_c, denotes how much current a superconductor can carry before it starts to become resistive. As such, it is essential to be able to quantify this parameter accurately for different superconducting systems and materials. In the case of bulk rare earth [(RE)BCO (where RE is Y, Sm, Gd or Nd)] superconductors J_c is usually estimated from the magnetic moment (or magnetization) of a sample of well-defined geometry measured in an applied magnetic field. It is usually assumed in this calculation that the sample is uniform throughout its volume and that its behaviour is described by Bean's critical state model [1]–[3].

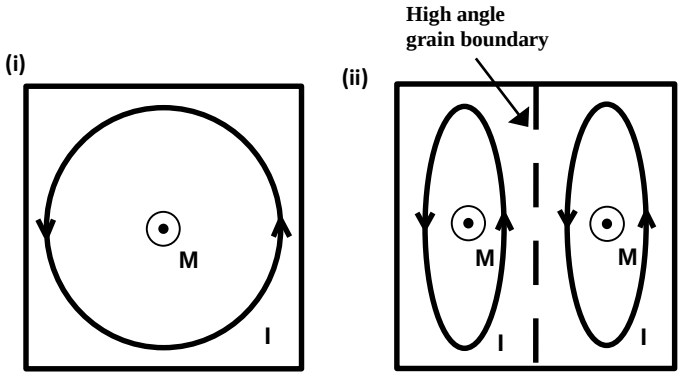

Figure I. Typical magnetization loops for (i) a homogenous material and (ii) a material with an inhomogeneity (e.g. a grain boundary). Note that it would not be possible to determine the value of the critical current at the grain boundary simply from the magnitude of the magnetization alone.

However, when J_c across a sample is not uniform, the conversion between magnetization and J_c becomes non-trivial. J_c decays exponentially with grain misalignment angle in tape superconductors [4]–[6] and figure I demonstrates how such a region cannot be distinguished the basis of a simple magnetization measurement alone. Such issues are also relevant at growth sector boundaries, where the J_c may be significantly higher than in the rest of the bulk by up to a factor of four [7].

It is possible using SQUID magnetometry to apply minor hysteresis loop cycles to a granular, or polycrystalline, coated conductor sample in order to estimate the J_c of the grain and grain boundaries independently, in addition to estimating the grain size. However, this method does not yield information about individual features of the sample microstructure and, therefore, so is of limited use for investigating J_c as a function of field and angle [8]. This point is best illustrated by a quotation from [9]: "It is difficult to correlate local J_c properties with trapped field behaviour on the bulk superconductor."

J_c can also be investigated across a sample using an array of hall probes [10], [11] or magneto-optical methods [12], [13], in which a magnetically active material is placed on top of the bulk to directly observe the flux lines. These methods also require certain assumptions in order to numerically calculate J_c via the Biot Savart law whereas transport measurements only require the choice of a field criterion.

Data from current transport measurements, where current is forced through the sample by a voltage, are therefore much

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easier to interpret. In this case J_c is defined simply as the current divided by the cross-sectional area of the sample when the electric field generated across a well-defined section, such as between two voltage tapes, reaches a critical value [14] (1 μVcm^{-1} is commonly used e.g. in [14]–[16]). By patterning the sample in the correct position, such as has been done for coated conductors in [17], [18], it is possible to select particular microstructural features of interest, including grain and growth sector boundaries.

Whereas magnetization methods are relatively quick and easy to perform, several assumptions are made about the path of the current. Crucially in transport measurements, the current is forced to travel across a specific feature and in a particular direction. This allows relatively easy characterisation of the behavior of J_c as a function of applied field, field angle and temperature.

Practical transport measurements are difficult to perform in bulk (RE)BCO superconductors and, as a result, relatively little work has been performed in this area recently. As the J_c of bulk superconductors continues to rise, higher currents or, conversely, lower cross sectional areas are required to see the transition from superconducting to normal at around J_c. Experimentally, DC transport measurements have become more difficult to perform and as such work has largely been abandoned in the last twenty years [6], [19]–[23]. In [19], there is a discrepancy of an order of magnitude between the J_c obtained via magnetization and transport techniques, which is mostly unexplained.

Preparation of the bulk sample for transport current measurements must be carried out very carefully to avoid introducing cracks and holes into the sample microstructure. In addition, J_c varies strongly with temperature [24], [25], so the contact resistance must be low to avoid local heating during the measurement. Therefore, the sample must be loaded carefully with sufficient pressure to keep the contact resistance low, but not so much that the sample cracks at the weaker thin, notched section. A method for testing bulk samples has been developed in which a notched, dog-bone shaped section is prepared. Such a method could be extended easily to characterise the behaviour of interesting microstructural features of the samples, such as growth sector boundaries.

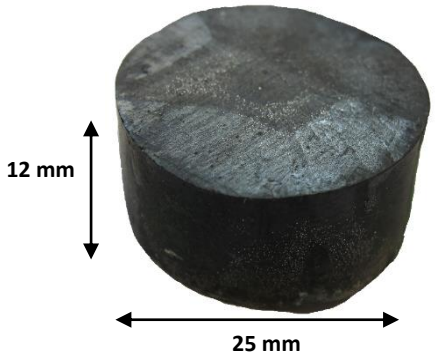

Figure II. A silver loaded, single grain GdBCO bulk superconductor used in this experiment. Each bulk typically yields 20 dog-bone sections suitable for transport current measurements.

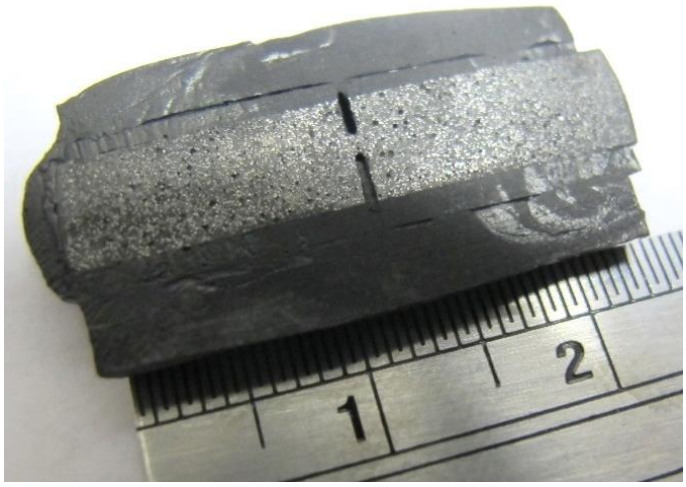

Figure III. A notched sample reinforced with Stycast. The cross-sectional area of the bridge is 0.44 ± 0.03 mm^2. The sample is approximately 5 mm wide, 25 mm long and 0.70 ± 0.02 mm thick, with a bridged width of 0.65 ± 0.02 mm.

II. Methodology

A. Sample Preparation

Single grain, GdBCO bulk superconductors doped with 15 wt % AgO_2, corresponding to the composition that achieved a record trapped field in 2014 [26], were used in this study. HTS bulks often contain many internal cracks and voids generated during the melt process and, therefore, the preparation of notched, dog-bone sections requires careful application of metallographic techniques to ensure that the sample retains its structural integrity during preparation.

Each bulk sample was cut initially into circular discs using a diamond cutting wheel. Each disc had a thickness of between 0.3 and 0.8 mm, which allows typically for 10 circular sections to be cut from each bulk sample. At this stage, the slices could be thinned further using P800 SiC paper and their surfaces buffed using P2400. These discs were then cut into three rectangular bars of width approximately 5 mm. The thickness of the bars was found to an accuracy of ± 0.02 mm using Vernier calipers.

The surfaces of the samples were cleaned thoroughly using acetone prior to the application of silver paint to their ends to form two current pads of approximate dimensions 5 mm x 5 mm. The silver was then annealed into the sample in an oxygen furnace at 400 ºC for one hour in order to reduce the contact resistance, as reported previously [27]. The rectangular bars are subsequently backed using the two part resin, Stycast 2850FT. The bars are notched from one side using the diamond cutting wheel and the notch resin-reinforced. The second notch is carefully aligned with the first and a bridge of between 0.4 and 1.0 mm is created. The notch width is investigated using an optical microscope with an expected accuracy also of ± 0.02 mm. Finally, Stycast is added to the final notch and the sample preparation is complete, with the sample used in this investigation seen in figure III.

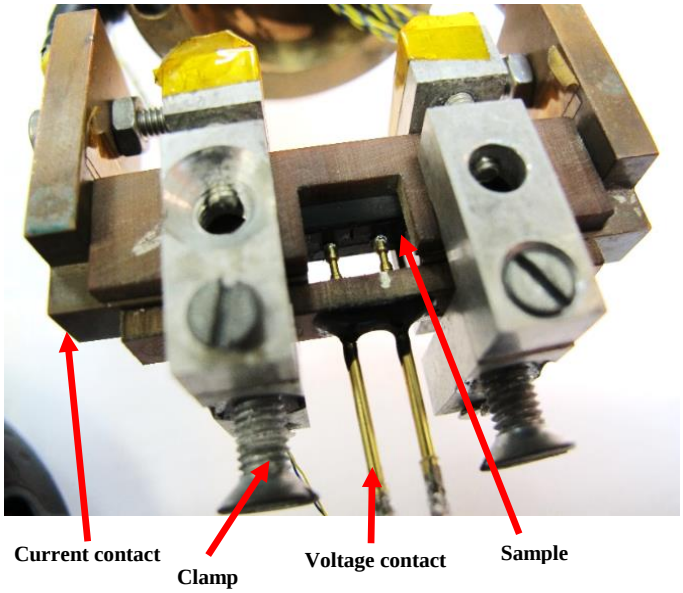

Figure IV. The sample for transport measurement clamped firmly against a Tufnol support. Indium is placed between the sample and the copper blocks with sufficient clamping pressure to ensure good electrical contact.

B. *Sample Mounting*

The sample is mounted by firmly clamping it against a Tufnol block using indium foil beneath a copper block, as shown in figure IV. It is important that enough pressure is applied to the contacts that good surface connectivity is achieved, but not so much that the fragile bridged section cracks. It was found that a torque of ~ 0.3 N m on a 5 mm screw was optimum. Using this technique, a resistance of 50μΩ cm^2 was found across the two pads (each approximately 0.3 cm^2 in area).

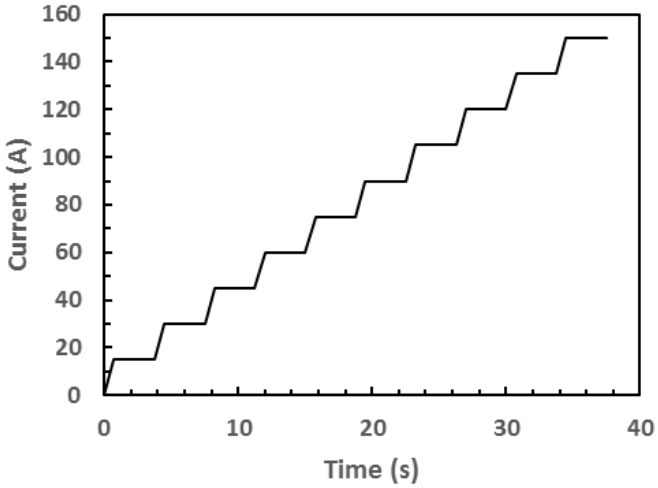

Figure V. A typical current sweep for a ramp rate of 4.2 A s^{-1}. The current was allowed to stabilise for some time before the voltage is measured, thus removing any frequency dependent components.

Voltage contacts were made using pogo pins that were 5 mm apart with indium pressed against the sample. By having independent voltage contacts as in a typical four-point measurement, the resistance of the circuit outside of the pins was ignored.

C. *Measurement Procedure*

The sample was left to cool in liquid nitrogen vapour for an hour before being completely immersed, thus minimising thermal shock. The current was then ramped up in well-defined steps with average rates of between 1.7 and 22 A s^{-1}, as illustrated in figure V for an average ramp rate of 4.2 A s^{-1}. If the supposed increase in E was due to heating, it is expected that the sample would go normal earlier at lower rates. This was

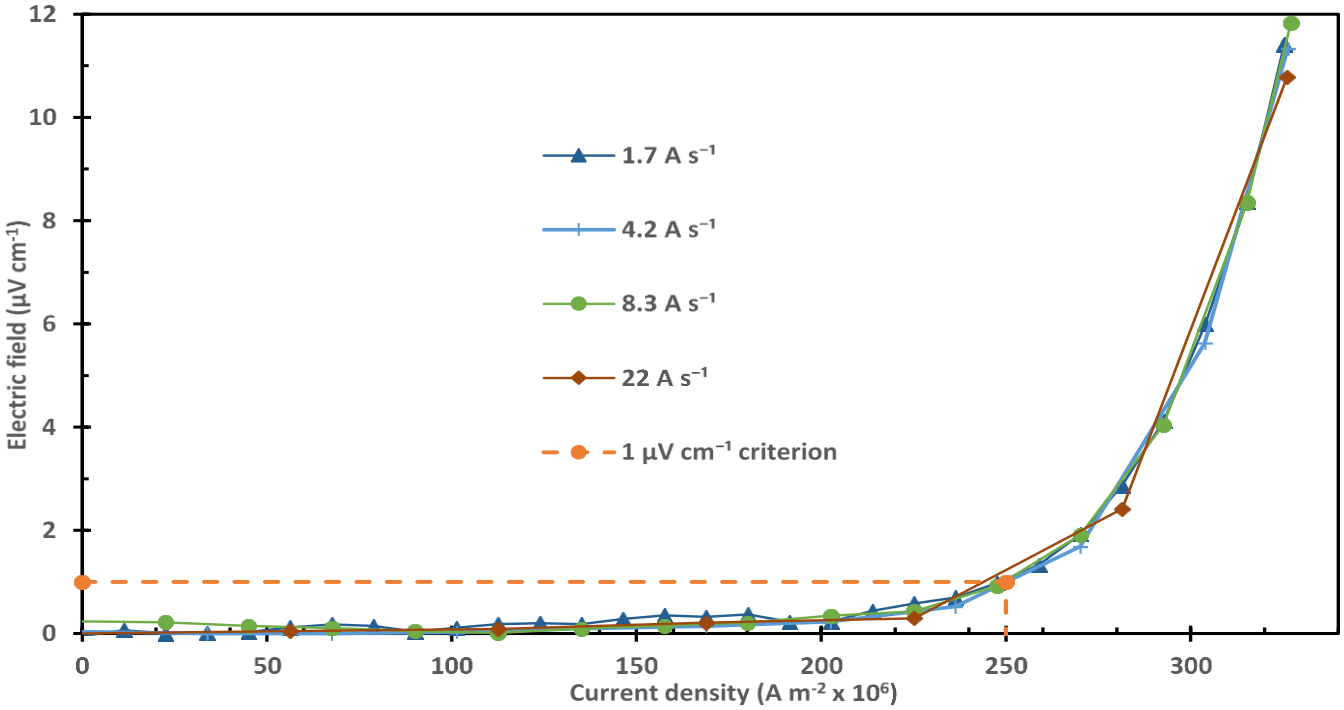

Figure VI. Four E-J curves measured at 77 K using different ramp rates, each of which indicate a transition to the normal state at J_c = 2.5 x 10^8 Am^{-2}. The shape of the curve is independent of ramp rate, which indicates that the effects of heating during the measurement are insignificant. These data are comparable to the values of J_c obtained from SQUID magnetometry, which are typically 5 ± 1 x 10^8 A m^{-2} [28].

verified directly from the measured *E-J* curves, which were independent of average ramp rate. Noise in the measurement was minimised by allowing the current to settle (thus avoiding any time-transient effects from increasing the current which allows a true DC measurement to be taken) before the voltage was measured using shielded, twisted wire pairs and a Keithley 2128A nanovoltmeter, which was set to average the signal over three power line cycles [NPLC = 3].

This ramping is distinct from work such as [22], in which the current is pulsed through the sample over a short period of time which makes it difficult to obtain an accurate result due to the time related effects involved in vortex motion.

III. Results and Discussion

It was found that a sample with cross-sectional area 0.44 mm^2 exceeded the 1 $\mu V\ cm^{-1}$ field criterion at a current of 110 A, corresponding to a critical current density of 2.5 x 10^8 A m^{-2}. The reproducibility of the E-J curves for the different ramp rates shown in figure VI indicates that sample heating effects are negligible, since consistent values of J_c are observed (shape of the curves is independent of the rate at which the current was stepped). This is a strong indication that the sample was not heating significantly during the period of the test and that the measured J_c is a true value at 77 K.

The value of J_c derived from the transport measurements is comparable to those obtained from SQUID magnetization measurements on melt processed GdBCO-Ag samples grown in the same batch, which typically yield critical current densities of (5 ± 1) x 10^8 Am^{-2} at 77 K [28]. There are many assumptions inherent to converting magnetization into critical current density using SQUID data, as outlined in the introduction. It is expected, therefore, that some level of discrepancy will exist between the two sets of data, although there are other explanations for these differences.

It is possible that the sample underwent partial cracking either during preparation or during loading. The notches present in the dog-bone shaped section create a stress concentration during loading and therefore act as a site for cracks to propagate through the sample. Any microscopic crack can act as a region of poor connectivity and inhibit the flow of transport current across that region.

Typical bulk samples contain a large amount of inherent inhomogeneity [29] and, therefore, it is entirely possible that an area with low J_c was selected for measurement. As a result, some variation in J_c is to be expected, with the magnitude of the fluctuation increasing as the size of the region being investigated decreases.

Finally, there is some uncertainty in the exact value of J_c introduced by the uncertainty in the value of the cross-sectional area (~ 6 %). However, this uncertainty is also present in typical SQUID measurements in which a small cube of material is cut and the J_c calculated using Bean's formula [1], with J_c inversely proportional to the fourth power of it's length.

IV. Conclusion

There are compelling arguments that transport J_c measurements of bulk superconducting samples are easier to interpret than J_c measurements performed by traditional magnetometry. Transport measurements enable samples in which a two-dimensional irregularity, such as a growth sector or grain boundary, exists to be studied systematically. Field-angle transport measurements across grain boundaries have been conducted on coated conductors and tapes, but relatively little work has been carried out on bulk superconductors.

We have shown that it is possible to create a notched section of bulk, single grain GdBCO-Ag capable of supporting a DC current of 110 A using metallographic techniques and resin reinforcement to give a resultant J_c of 2.5 x 10^8 A m^{-2}, which is comparable to the results of SQUID magnetometry measurements.

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