STEP-BY-STEP: CRITICAL CURRENT MEASUREMENTS ON HIGH TEMPERATURE SUPERCONDUCTING TAPE

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HEALTH AND SAFETY

Please make sure to wear appropriate clothing for this workshop – standard shoes or trainers are fine (no sandals) and clothing that covers your arms and legs (no shorts). Do not wear ferroic jewellery.

In this workshop, we will be using high magnetic fields, very hot things (solder) and very cold things (cryogens).

Durham University is committed to make reasonable adjustments for all attendees. Could attendees with implants please contact Dr. Raine in advance and in confidence to help us with the use of magnetic fields?

In advance of the course, please ensure you understand all the technical terms in this document and in the document: 'Health and Safety for the CDT Icy Durham Practical Course' that you will be required to sign before on the first day.

Prof. Damian P. Hampshire, Durham University

December 2024.

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AIM

We intend to measure the critical current (I_c) of a high temperature superconductor (HTS) in the form of a (commercial) tape at 77 K in magnetic fields up to 0.5 T.

1. Check you have been given the equipment you need

Hardware

1. **I_c probe**: This is used to hold the sample in the cryogen, deliver electrical current to it, and measure the voltage of the sample throughout each measurement.
2. **Brass sample holder**: The HTS sample is first soldered to the sample holder, which is then mounted onto the end of the measurement probe.
3. **HTS sample tape**: 80 mm sample length.
4. **Hot plate**: Used to sufficiently heat the brass sample holder, prior to, and during, soldering.
5. **Soldering iron** (for applying heat during the soldering process) + **damp sponge** (for cleaning the soldering tip).
6. **Solder flux**: This is applied prior to soldering. It cleans metal surfaces and stops oxidation during soldering, allowing the solder to flow well, creating good, wetted, joints. It generally also makes soldering easier.
7. **Lead-tin solder**: You will use this to solder your sample to the sample holder, and to make all other electrical connections. The solder ensures that the sample is mechanically and electrically bonded to the sample holder.
8. **Polyimide insulated copper wire**: Used to make twisted pairs (voltage tap wires) that will be soldered to your sample and connected to the probe instrumentation wiring.
9. **A pair of connection pins**: To be soldered to the ends of your twisted pair wires for connection to the probe's instrumentation plug.
10. **Venier caliper gauge, measuring ruler and or tape rule**: For measuring voltage tap gauge-length and twisted pair length.

Safety Equipment

1. **Safety goggles** and **safety gloves** for use when transferring the cryogens or handling cold objects.

Any work involving a cryogen must be covered by a risk assessment that considers the physical risks such as burns, asphyxiation, manual handling, transport, air distillation and explosive atmospheres around cold pipework.

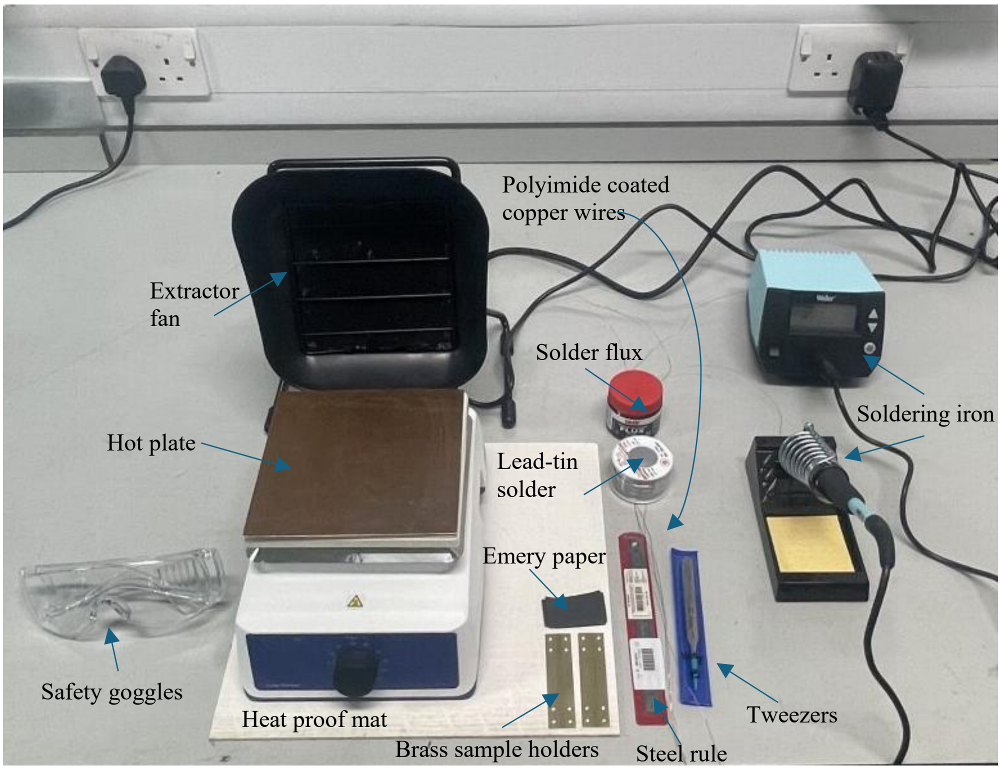


Fig. 1: The equipment that makes up the soldering station.

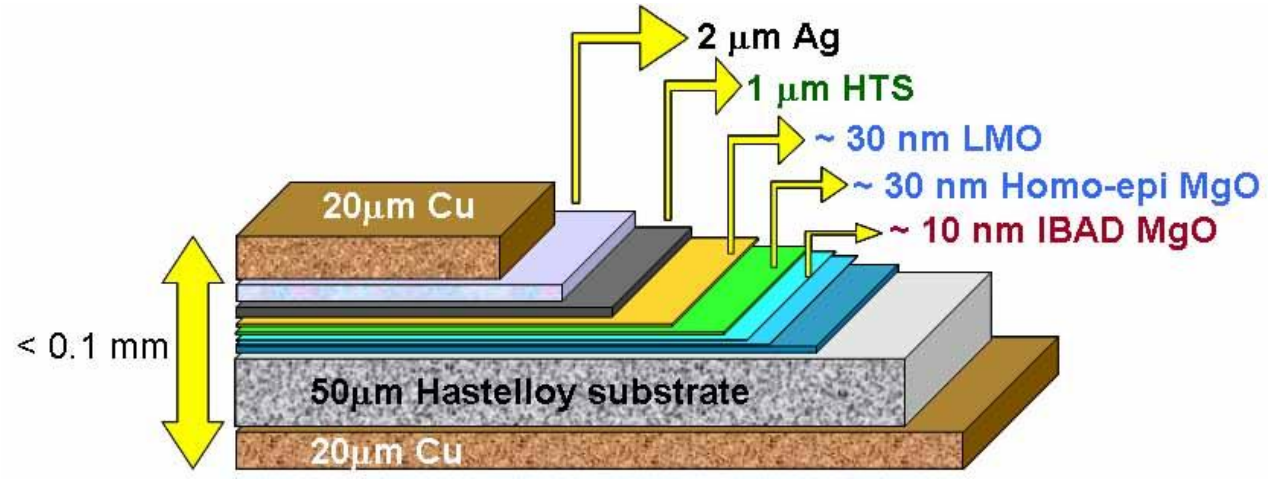
2. Soldering the HTS tape to the sample holder

Aim 1: Attach the HTS sample to the sample holder with lead-tin solder. The whole area of the sample should be bonded flat to the sample holder. The sample holder is designed so that the sample should sit inside the groove in the top surface.

Solder: We use standard Pb-Sn solder that plumbers use in your house. Nevertheless, the Pb-Sn materials must be disposed of safely/professionally (see below).

Important – Disposal of Waste

Any cotton buds, paper towel or similar item contaminated with hazardous materials (e.g. Solder flux) should be disposed of in the designated hazardous waste bin. A demonstrator will make it clear to you where this is. All disposable gloves must also be thrown away here, whether they are contaminated or not



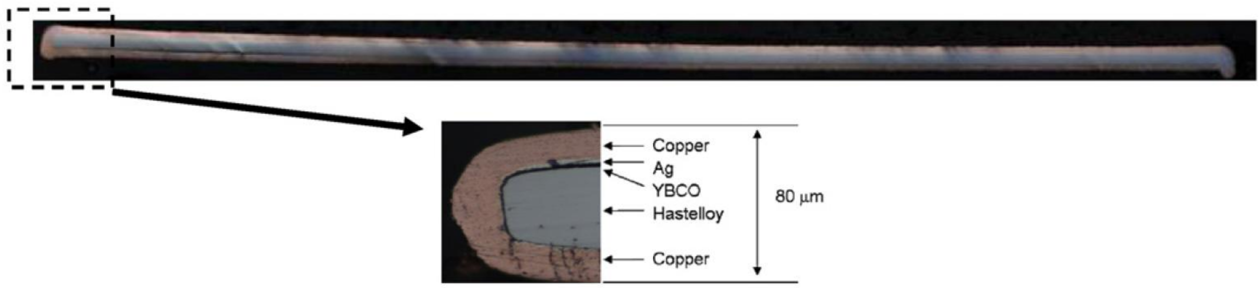


Fig. 2: HTS coated conductors - Kilometre long (quasi-) single crystals REBCO (RE: Rare-Earth) – eg $GdBa_2Cu_3O_7$. Note that the tape is flexible and the superconductor is brittle.

- DO NOT throw HTS tape or copper wire into this bin. Place any waste samples or solder-contaminated wire into the designated plastic bag that is labelled clearly. A demonstrator will ensure this is disposed of properly at the end of the course.

Terminology: Soldering involves two processes: *pre-tinning* the surfaces of the components (sample holder and the sample) with a thin coating of good quality solder, followed by *sweating* the soldered components together. (There can also be a third process for instrumentation leads involving mechanically arranging (if necessary) the components so they are sufficiently close together that there is no stress on the solder.)

Pre-tin the sample

The sample is easier to pre-tin but, it is *brittle and delicate*. It will need to be mounted with the superconducting side down, so you should pre-tin this side. The tape will have a natural curvature – the superconducting layer is on the convex (outside) side.

a) Set the soldering iron temperature to around 250 deg C and melt some solder on the tip.
b) Apply solder flux to the surface to be tinned using a wooden stick (other end of cotton bud).
c) Clean the tip of the soldering iron if needed by wiping it on the wet sponge. Re-apply solder to the iron tip – not too much!
d) Hold the soldering iron tip on the sample for a few seconds, until you see the solder start to flow and start to coat the sample. Then sweep the soldering iron SLOWLY along the length of the sample. You should now have a thin shiny silver layer of solder on the sample - see fig. 3. Ensure the whole area of the sample is covered – go back over areas if needed, applying more flux if needed. Don't forget to be careful when handling the sample… it is fragile !
e) Leave to cool and clean off flux residue using a cotton bud soaked in isopropanol.

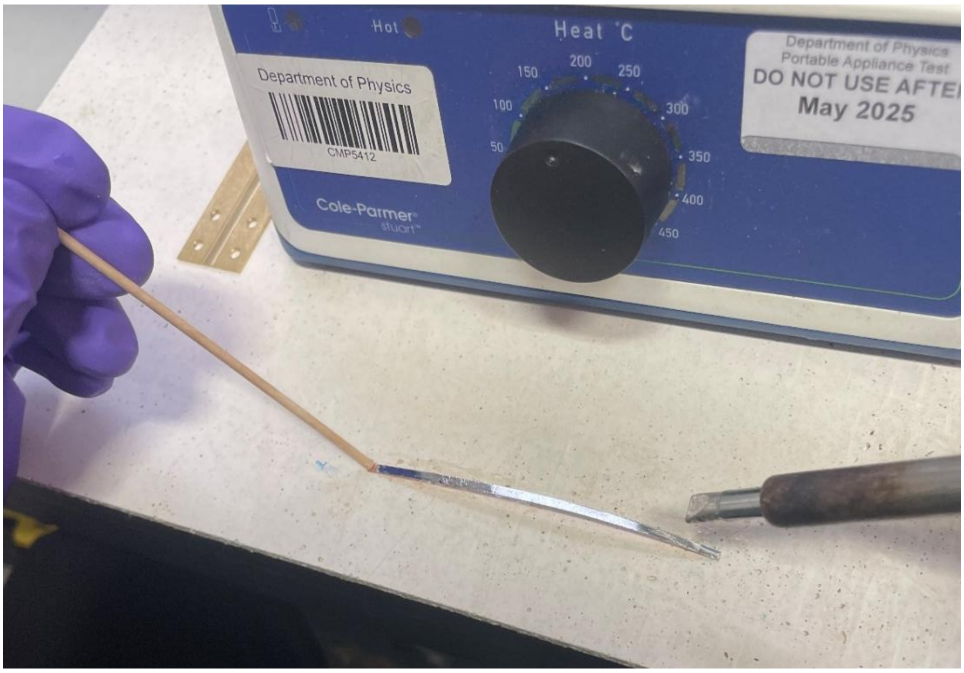


Fig. 3: A well tinned sample.

Pre-tin the sample holder

This prepares a solder layer on the sample holder for the pre-tinned surface of the sample to bond to. This is more difficult than tinning the sample, as there is a greater mass of material to maintain above the solder melting temperature.

a) Place the sample holder onto a hot plate and set it to ~ 170 deg C and leave for a few minutes to heat up. (An LED indicator around the temperature dial will indicate the hot plate's current temperature.)
b) Make sure the small square sponge at the bottom of the black soldering iron holder is wet (use water bottle if necessary).
c) Apply solder flux to the area you need to pre-tin – which in this case is the inside of the groove – using the wooden end of a cotton bud.
d) Apply solder to the soldering iron tip.
e) Hold the soldering iron tip in place on the sample holder, in the groove, until solder begins to flow. If you don't observe this happening after 10 seconds, you will need to increase the temperature of the soldering iron, hot plate or both!
f) When solder begins to flow, sweep the soldering iron SLOWLY along the length of the sample holder. Ensure an area larger than the sample is thinly but completely covered and go back over areas as needed, applying more flux if necessary. See fig.4.
g) Leave to cool and clean off excess flux residue using a cotton bud soaked in isopropanol.
h) If the solder surface is visibly dirty, you will need to remove it from the hot plate, leave to cool and clean. Otherwise leave on the hot plate

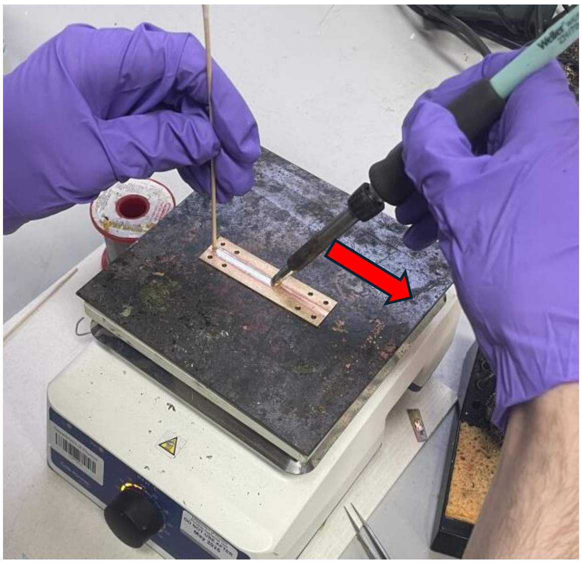


Fig. 4: Pre-tinning the sample holder.

Sweat the sample to the sample holder

a) Apply more flux to the pre-tinned surfaces of the sample and sample holder.

b) Ensure the sample holder is on the hot plate at a temperature around 200 °C (adjust this if needed).

c) Place the sample onto the sample holder – ensure the pre-tinned surface is facing down!

d) Hold one end of the sample in place with the wooden end of a cotton bud. Apply a soldering iron on top of the sample near the cotton bud stick and wait for the solder between the sample to flow out the sides – this indicates a good bond. Then slowly drag the soldering iron along the length of the sample, holding it in place with the stick, while applying moderate pressure, ensuring good solder flow as you go – see fig.5. Go back over any areas that aren't properly bonded.

e) Remove from hot plate, leave to cool and clean off flux residue.

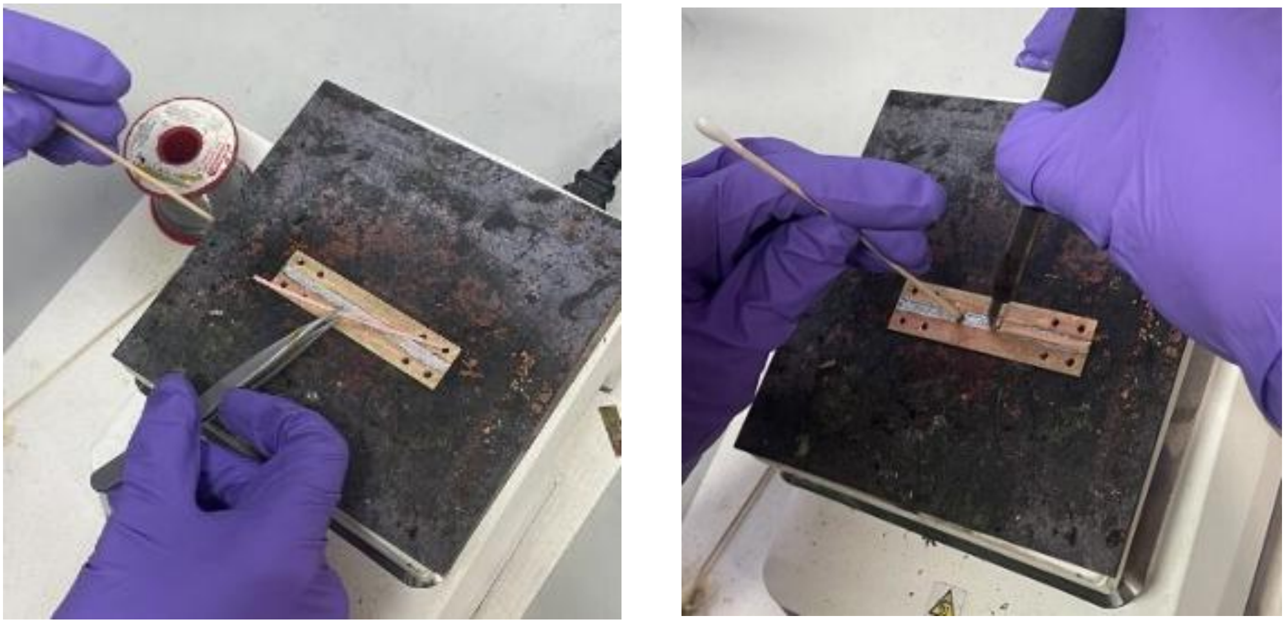


Fig. 5: (Left) Positioning the pre-tinned sample (tinned side *down*) with tweezers. (Right) Sweating the tape to the sample holder.

2. Soldering the voltage taps

We need to attach two wires to the top surface of the sample. These are used to measure the voltage across a region of the sample – called the gauge length. You should use two polyimide insulated copper wires (ask the demonstrator what length to use), forming a twisted pair (you may need to twist them together yourself). The two ends at the bottom of the probe should be soldered onto the sample approximately 1 cm apart. You should solder the other ends to the small connector pins you have been given.

1) Pre-tin the wires:
    a) Remove the polyimide insulation from a short length at the end of each wire with a sharp blade or Emery paper.
    b) Apply flux to the ends of the wires
    c) Apply solder to the tip of a soldering iron. Touch this onto the fluxed wire ends. A shiny, silver layer of solder should coat the ends of the wires.
    d) Clean off flux residue
2)
    a) Pre-tin areas of the sample and attach wires – make two small patches of fresh solder on the tape for the wires to bond to as easily as possible.
    b) Apply flux to the areas to be tinned.
    c) Heat the sample to ~ 170 deg C on the hot plate. Set a soldering iron with a fine tip to ~ 250 deg C.
    d) Tin these areas with a similar technique to steps 2d and 2e in phase 1. If the solder is not flowing easily, you will need to increase the temperature.
    e) Apply more flux to the tinned wire tips. Apply more solder to the soldering iron. One by one, hold the voltage tap wires in place where they are to be soldered using tweezers. Apply heat with the soldering iron to bond the wires to the sample – see figure 6. Gently pull the wires and find out if the come off easily. If they do the solder bond is not strong enough so have another go – this is probably the trickiest part of the process so you may need a few tries! Note – try to make sure sections of the wire do not touch the hot plate as this may burn off the insulation, potentially leading to unwanted short circuits. When you have finished, your soldered connections should have shiny surfaces. If they are a dull grey, consult a demonstrator to check their quality – poorly wetted solder joints can come off at cryogenic temperatures!

Fig. 6: Soldering the voltage taps.

f) Remove the sample from the hot plate and place on a heat proof mat as soon as good bonds are made. Ensure you use the heat proof gloves to avoid burning yourself.
g) Leave to cool and clean off flux residue
h) Measure the separation of your voltage taps – this will allow you to convert a voltage to an electric field later.

3) Solder the other ends of your wires to the connecting pins. This should be easier!

3. Securing the sample holder/sample to the I_c probe.

The probe will be pre-assembled for you – but you do need to attach the sample holder to the probe yourself. Make sure you have the following parts:

- Sample holder with sample soldered into the grove
- 8 x M2.5 screws and bolts

1) Decide which sample from your group will be measured.
2) Fit the sample holder to the probe (check with the demonstrator if you are in doubt), ensuring the sample is facing up and the holes line up.
3) Connect the samples voltage tap twisted pair connector to the probe connector.

4) Secure any loose wires or excess length with Kapton tape so it can't move around during the measurement.
5) Ask a demonstrator to check that your probe is ready for cryogenic measurements.

4. Set up the apparatus for the Ic experiment.

Set up apparatus for experiment.

1) Position the probe in the sample space for your measurement (inside liquid nitrogen Dewar). This job will need at least two people as the probes are heavy – make sure you ask for help!
2) Identify the correct polarity for the current leads (Flemings left-hand rule or (better) Maxwell's equation and the right hand-rule for the curl of a vector field).
3) You will need to attach the current leads from the 120 A power supply to the copper current contacts at the top end of your probe. This is done by using nuts and bolts – ensure these are sufficiently tightened to ensure a good electrical connection.
4) Plug the instrumentation plug into the socket at the top of your probe. This connects your voltage taps to the data acquisition instrumentation. Refer to figure 7.

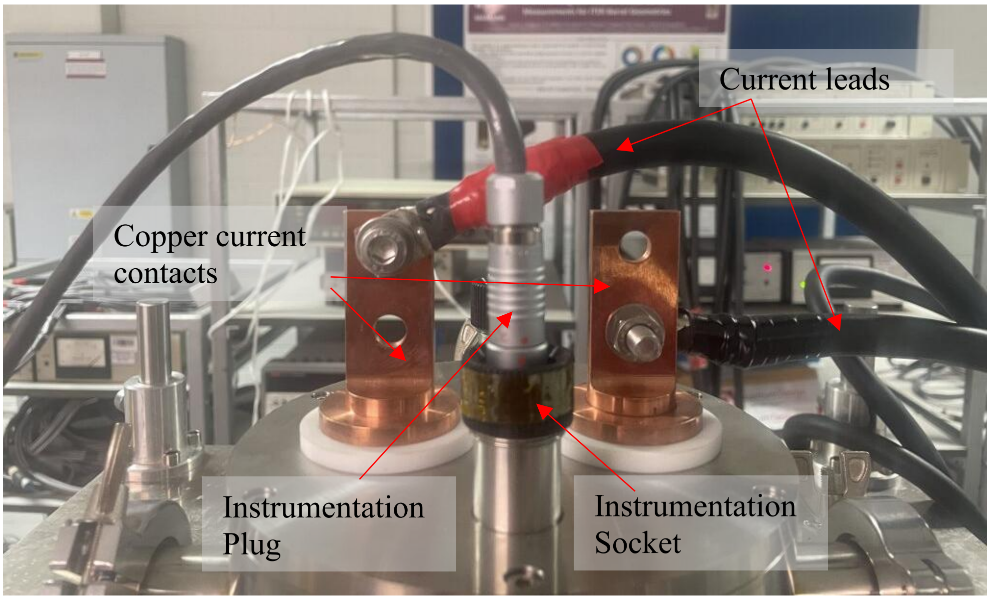


Fig. 7: The components of the top end of the probe.

5. Make the I_c measurement 😊

1) When you are ready to start the measurement fill the Dewar with liquid nitrogen. Ensure you use all the correct protective equipment as per the training.
2) The measurement will be controlled using a Labview program – the power supply and voltage measurements are all controlled and read automatically by the computer. You can (and should!) also monitor the measurement data in real time. Refer to figure 8 for an illustration of the program you will be using, and follow the steps outlined.

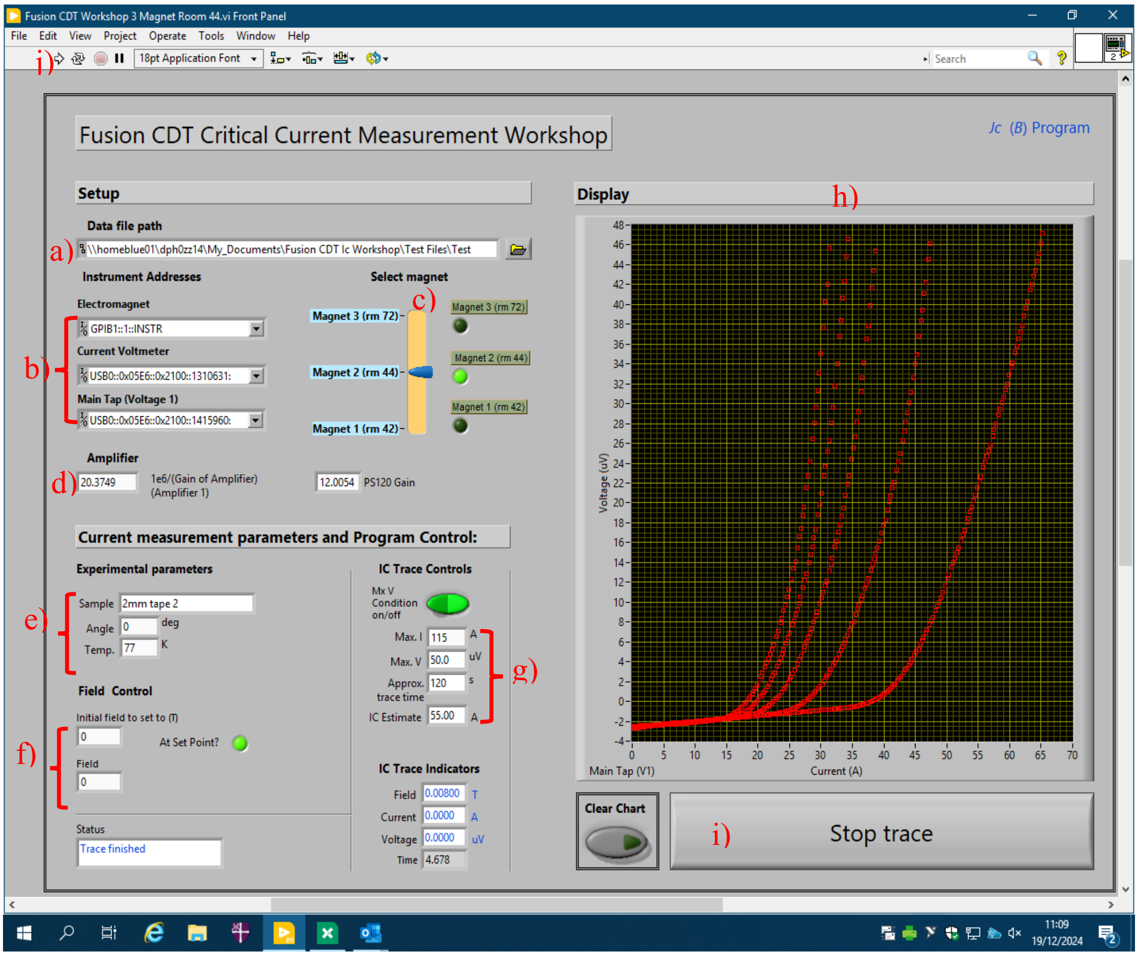


Fig. 8: An annotated screenshot of the Labview program used for the measurements. The annotated letters are explained below.

a. Create a folder for your group's data in the 'Student data files' directory. Choose this new folder and enter an appropriate file name where your data will be saved.

b. Ensure your program communicates with the correct instruments to record the data for your measurement by selecting the correct instrument addresses from the drop down menus. *Refer to table 1 to check the correct addresses for your Lab. Rm.*
c. Important: Ensure you have selected the correct magnet for your particular lab you are in, using the slider.
d. We use a nanovolt amplifier to enable us to measure the low voltages required through the noise – the program accounts for the amplification automatically but you need to enter the correct correction factor for the amplifier you are using. Refer to table 1 for the correct number for your lab.
e. You can enter appropriate values here which will be included in the metadata of your data file, to help you identify the sample being measured, field angle and temperature of the measurement.
f. Set the applied magnetic field for your measurement. It is good practice to initially set the magnetic field slightly higher, then reduce it to the target field for your measurement. This will happen automatically, but you will need to enter values in the indicated boxes. Discuss with your demonstrator some appropriate magnetic fields to try.
g. These fields control various parameters of the measurement. Max I and Max V set the current and voltage at which the measurement trace will be cut off automatically. Approx trace time and Ic estimate will control the current ramp rate for the measurement. Discuss appropriate values for these with your demonstrator.
h. Your I-V trace will be plotted here in real time. You can choose whether to clear the chart at the start of each measurement or plot subsequent traces together using the ‘clear chart’ switch.
i. If you need to stop your trace for any reason, press the ‘Stop trace’ button.
j. When you are ready to start your measurement, click this white arrow.

3) If you are one of that special band of people who can overcome the blood, sweat and tears required to get great (computational or experimental) data, fill your boots! It is one of the few times you should show-off by securing a cornucopia of data – measure $J_C(B, \theta, 77K)$.
4) After you have finished your measurements;
   a. Download your data onto a USB or your own OneDrive.
   b. Please take 5 minutes to annotate a paper copy of this step-by-step with all your corrections and suggested improvements and give it to the lab demonstrator/Prof. Hampshire.

	Lab. Room 42	**Lab. Room 44**	**Lab. Room 72**
Amplifier gain	49.57 x 10^3 (10^6 / gain = 20.173)	49.64 x 10^3 (10^6 / gain = 20.145)	49.15 x 10^3 (10^6 / gain = 20.345)
Electromagnet address	TDK magnet power supply	GPIB1	GPIB6
Current voltmeter address	USB 1415930	USB 1310631	USB 1310629
Main tap (voltage 1) address	USB 1270177	USB 1415960	USB 1269999

Table 1: Amplifier gain parameters and instrument addresses for the apparatus in each of the 3 labs.

Appendices

A1. Good conduct

Durham University adheres to the University Policy on Good Conduct in Research, which is accessible at:
https://www.durham.ac.uk/research/ethics--governance/research-integrity-policy-and-code-of-good-practice/

A2. Soldering

Overview of soldering - Soldering - Wikipedia
Very basic step-by-step introduction to soldering How to Solder : 8 Steps (with Pictures) - Instructables

A3. Solder flux

In standard I_c measurements, such as those here, we use plumbers' flux or rosin flux. For more advanced measurements that include say multiple 10 pin plugs or high precision thermometry, we recommend the use of acid flux to eliminate 'tracking'.
Good overview of solder fluxes - "Choosing the Correct Flux – Types and Their Advantages/Disadvantages?" https://www.pillarhouse.co.uk/wp-content/uploads/301-Choosing-a-Flux-11-2016.pdf

A4. Cryogenics safety literature

The most common way to learn cryogenics is from other experts in the field. There is useful reading material from BOC "Care with Cryogenics": https://mriquestions.com/uploads/3/4/5/7/34572113/care-with-cryogenics410_39400.pdf .

A5. Academic literature for I_c

A5.1 Core reading materials

M J Raine, S A Keys and D. P. Hampshire **Characterisation of the Transport Critical Current Density for Conductor Applications** – **Handbook of Superconductivity**. Publisher: Taylor and Francis (2021)

P. Sunwong J. S. Higgins and D. P. Hampshire **Angular, Temperature and Strain Dependencies of the Critical Current of DI-BSCCO Tapes IEEE Trans Magn. 21 No3 p2840 (2011)**

R. G. Hutson, M. J. Raine, M. Kiuchi, T. Matsushita and D. P. Hampshire, "**HTS Cable International Round Robin: 4.4 kA Critical Current Measurements**," in IEEE Transactions on Applied Superconductivity, doi: 10.1109/TASC.2023.3263143.

A5.1 Post-measurement I_c reading materials

C.W.W. Haddon, A.I. Blair, F. Schoofs, and D.P. Hampshire **Computational Simulations using Time-Dependent Ginzburg–Landau Theory for Nb–Ti-like Microstructures** IEEE Transactions on Applied Superconductivity, Article vol. 32, pg. 5, Jun 2022, Art no. 8800105, doi: 10.1109/tasc.2022.3156916.Associated movie: **https://www.youtube.com/watch?v=7KmiTSx_Rok**

A. I. Blair and D. P. Hampshire **Critical current density of superconducting-normal-superconducting Josephson junctions and polycrystalline superconductors in high magnetic fields**, Phys. Rev. Research **4**, 023123, 16 May 2022

P.O. Branch, Y. Tsui, K. Osamura and D. P. Hampshire. **Weakly-Emergent Strain-Dependent Properties of High Field Superconductors. Nature Scientific Reports 9:13998 (2019).**

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