ISBN: 978-3-95450-231-8 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2023-WEPA141

CHARACTERISATION FACILITIES FOR EVALUATING SUPERCONDUCTING THIN FILMS FOR SRF CAVITIES

D. Seal*[1,2], C. Benjamin[1,3], G. Burt[1,2], J. Conlon[1,3], N. Leicester[1,2], O.B. Malyshev[1,3], H. Marks[1,2], T. Sian[1,3], L. Smith[1,3], D. Turner[1,2], R. Valizadeh[1,3]
[1]Cockcroft Institute, UKRI/STFC Daresbury Laboratory, Daresbury, Warrington, UK
[2]Engineering Department, Lancaster University, Lancaster, UK
[3]ASTeC, UKRI/STFC Daresbury Laboratory, Daresbury, Warrington, UK

Abstract

Over recent years four dedicated facilities have been built at Daresbury Laboratory by a team working on thin film SRF cavities. Firstly, a conventional DC resistance facility allows measurements of critical temperature and residual resistance ratio. In addition, three other facilities were designed in house to address superconducting thin film (STF) characterisation specific to cavities. In a magnetic field penetration facility, a DC parallel magnetic field is applied locally from one side of the sample similar to the field within an RF cavity. The STF behaviour under RF conditions is tested with planar samples using a 7.8 GHz choke cavity with the main advantage of a quick turnaround. The final facility uses a novel idea of split single cell 6 GHz cavities. Such a cavity can be deposited with both planar and cylindrical magnetrons allowing for both deposition techniques to be tested in the same cavity. Also, the results can be compared to choke cavity measurements for planar samples. They can also be inspected easily both visually and with surface analysis instrumentation. All facilities are based on liquid helium free cryocoolers to simplify operation, safety and maintenance.

INTRODUCTION

The Cockcroft Institute (CI) team at Daresbury Laboratory are working on developing a technology for superconducting thin film radiofrequency (TF SRF) cavity production to enable the UK capability to coat TF SRF cavities. To meet this ambition, the team is systematically working on a few main directions:

- Surface preparation before deposition.
- Developing superconducting thin films (STF) and optimising deposition parameters and conditions.
- STF characterisation with various surface and thin film characterisation techniques.
- STF characterisation under DC conditions.
- STF characterisation under RF conditions.

This paper is devoted to latter two items.

For DC and RF characterisation of STF materials, four facilities have been built over recent years. Three of these facilities focus on measuring thin films on flat substrates. A fourth facility utilises a novel copper split cavity, allowing for RF, visual and surface analysis of thin films on a cavity-like geometry. All facilities utilise closed cycle refrigerators, eliminating the need for procuring and operating with liquid helium, thus making them simple and safe to operate.

* daniel.seal@cockcroft.ac.uk

FOUR FACILITIES

RRR and T_c

First of all, one needs to evaluate whether a deposited thin film is superconducting. To do this, the residual resistance ratio (RRR) and critical temperature (T_c) of the thin film are measured on a facility that utilises a four-point probe. This cryostat has two Vacuum Tubular Inserts (VTI) as described in Ref. [1]. An experimental insert is loaded with a sample (max. 5 mm × 5 mm) as shown in Fig. 1, which can be used with either VTI. Whilst the cryostat is at base temperature, the sample can be changed without stopping the cryocooler and fully warming up the cryostat, allowing for a rapid sample changeover and up to 20 sample tests per week.

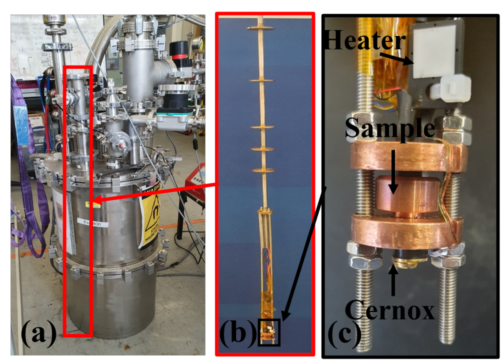

Figure 1: The RRR facility. (a) The cryostat showing the position of one of the VTIs, (b) an insert, (c) sample holder.

To perform a sample changeover, the VTI is filled with continuous He gas flow and the experimental insert is quickly removed. The sample is then loaded and pressed against the four-point probe. The insert is then placed back into the VTI and pumped with scroll and turbo-molecular pumps. To perform the measurements, the VTI is filled with gaseous He to a pressure in the range 0.01 mbar ≤ P ≤ 10 mbar. The higher pressure is required to reach the lowest sample temperatures. A heater and Cernox thermometer connected to the sample stage allows one to control the sample temperature in the range 3.5 K ≤ T_s ≤ 80 K. Whilst applying a

14th International Particle Accelerator Conference,Venice, Italy JACoW Publishing
ISBN: 978-3-95450-231-8 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2023-WEPA141

constant current across the four-point probe, the voltage drop as a function of T_s is measured, allowing one to determine both the DC resistance (R_{DC}) and T_c of the sample.

T_c is defined at the 50 % transition between $R_{DC} = 0$ and R_{DC} just before superconducting transition begins: i.e. $R(10\ K)$ for Nb. The temperatures corresponding to 10 % and 90 % define the width of transition. For example, RRR for Nb is given by: $RRR = R_{DC}(300\ K) / R_{DC}(10\ K)$. A demonstration of resistance measurements for a V_3Si sample near T_c are shown in Fig. 2. In this example, $T_c = 12.4 \pm 0.3$ K measured with $I = 3$ mA.

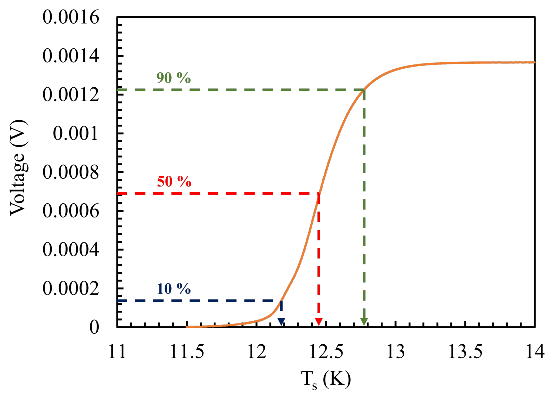

Figure 2: Resistance measurements for a V_3Si on sapphire sample indicating the 10, 50 and 90 % transition points.

Magnetic Field Penetration

Development of STFs and optimising their deposition parameters requires a quick test for small planar samples in conditions similar to those found in an RF cavity. In an RF cavity, the field is applied (a) only to the internal surface and (b) parallel to the surface. In commercially available facilities for evaluating superconductivity, a small sample is usually placed inside a large coil, hence the magnetic field is completely around the relatively small sample, and alignment parallel to the magnetic field is a challenging task. I.e. measurement conditions are very different from an RF cavity. Thus, one needs a characterisation facility that would reproduce the conditions in an RF cavity. The original idea of a magnetic field penetration (MFP) facility, where a DC magnetic field is applied from one side and parallel to the sample [2, 3], has developed into an in-vacuum facility described in detail in Ref. [4, 5]. In these measurements, the magnetic field on one side (B_1) is steadily increased to detect its value when the magnetic field penetrates through the STF and is detected on the other side (B_2).

In this facility, a magnetic field can be produced in a 2-mm gap between the poles of a C-shaped superconducting dipole magnet in the range $0 \le B_1 \le 500$ mT. The magnetic field is measured with Hall probes on both sides of the sample. A sample is placed on a copper plate mounted between Stage 2 of the coldhead and the magnet, so the magnetic field is applied parallel to the sample surface. A loaded sample in the facility is shown in Fig. 3. Two 12-Ω heaters and a

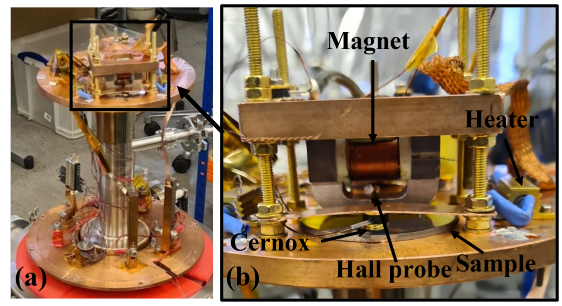

Figure 3: The MFP facility: (a) cryostat, (b) sample stage.

Cernox thermometer control the sample temperature in the range 2.6 K $\le T_s \le$ 30 K. This facility is currently able to test samples up to a maximum size of 70 × 50 mm^2 at a rate of 2-3 samples per week.

The results obtained with the MFP facility can be represented with a parameter $R = 1 - B_2/B_1$. Thus, $R = 1$ corresponds to full screening of the applied magnetic field by the STF, whilst $R < 1$ means a partial screening. The field of full flux penetration (B_{fp}) can be defined using a few different methods, for example at $R = 0.99$ (or 1% MFP). An example of $R(B_1)$ measurements are shown in Fig. 4.

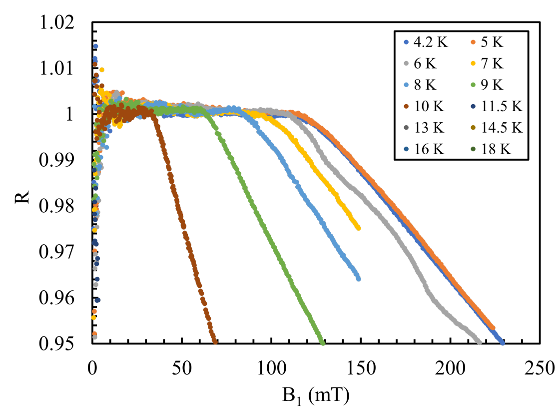

Figure 4: An example of $R(B_1)$ measurements with the MFP facility for a V_3Si on Nb on Cu multilayer sample.

Planar Samples at RF

Varying and optimising deposition parameters are much easier with a planar deposition facility. Planar samples are also lower cost and easier to inspect and characterise. However, no DC characterisation of superconducting properties provides good prediction of STF behaviour under RF conditions. Therefore, in order to complete the evaluation of planar STF samples, these films also need to be characterised under RF conditions. This facility, described in Refs. [6, 7], uses a test cavity consisting of two parts: a choke cavity - a half-cell elliptical bulk Nb cavity surrounded by quarter-wavelength RF chokes, and a planar sample disk - 90 to 130 mm diameter made from either bulk Nb or STF coated Cu. The test cavity operates at 7.8 GHz in the TM_{010} mode.

ISBN: 978-3-95450-231-8 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2023-WEPA141

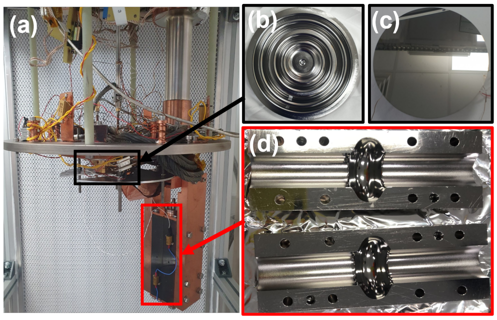

Figure 5: The shared RF facility for planar samples and split cavities. (a) the cryostat, (b) choke cavity, (c) Nb coated planar sample, (d) both halves of a Nb coated split cavity.

The RF facility with the two-part test cavity are shown in Fig. 5. The sample temperature, T_s, is measured with two Cernox thermometers and controlled with two 10 Ω resistors in the range 3.5 K ≤ T_s ≤ 30 K. The use of RF chokes means that the choke cavity and planar disk are physically and thermally isolated. This allows for a simple measurement of the average sample surface resistance (R_s) using an RF-DC compensation method (described in [8]) at peak magnetic fields B_{pk} ≤ 1.2 mT. R_s can be measured at fixed B_{pk}, i.e. $R_s(T_s)$, and at fixed T_s, i.e. $R_s(B_{pk})$. As the bandwidth of the cavity is large at 7.8 GHz the RF measurements can be made with a VNA and amplifier rather than requiring a phase-locked loop or self-excited loop. In addition, a low power sweep with the VNA allows one to track the shift in resonant frequency, Δf, at temperatures close to T_c. Given the simple cavity design and mounting procedure, up to 3 samples can be fully characterised per week. Crucially, this facility allow for direct comparison the R_s of different films.

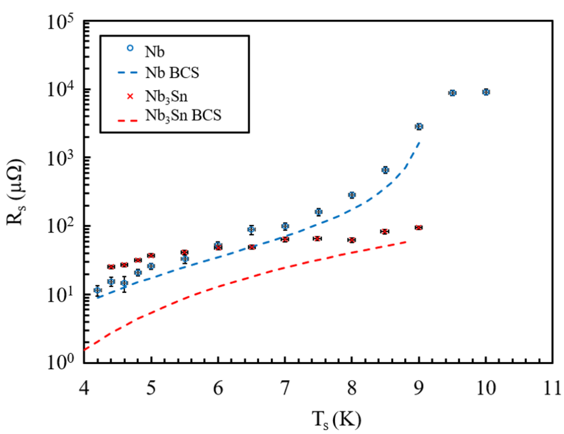

Figure 6: An example of $R_s(T_s)$ measurements for Nb and Nb_3Sn TF planar samples with the choke cavity.

An example of $R_s(T_s)$ measurements for both a Nb sample and Nb_3Sn sample with this facility and are shown in Fig. 6. The theoretical BCS resistance is calculated using the SRIMP code based on the widely used Halbritter code [9].

Split Cavity

Having fully characterised a film on a planar substrate, the next step is to test a deposited film on a cavity-like geometry. In order to do this, a novel longitudinally split single cell 6 GHz cavity was designed at Lancaster University and manufactured at Daresbury Laboratory, as shown in Fig. 5. The idea of using this cavity allows for depositions with both planar and cylindrical magnetrons, with the former allowing for direct comparison with planar samples measured with the choke cavity. Also, a split cavity allows for easy visual inspection and surface analysis of the film on a curved surface. For cryogenic testing, the cavity is mounted directly to the stage 2 cold head of the cryocooler in the same system used to test planar samples under RF conditions. Four 10-Ω resistors allow for R_s measurements in the range 3.5 K ≤ T_{cav} ≤ 30 K. As the whole cavity is coated, $R_s(T_{cav})$ can be calculated from direct measurement of the ohmic Q factor of the cavity. As the cavity is made from a solid block, microphonics are very low, again allowing measurements to be performed with a VNA. Details of the design and preliminary tests of this cavity are detailed in Refs. [10, 11].

DISCUSSION

Having four fully operational facilities enables completion of all five stages of the full characterisation cycle of a STF detailed in the introduction. A flat sample starts with polishing and cleaning procedure followed by STF deposition and characterisation, examples of which are shown in [12–15]. Then, the DC and RF superconducting properties are measured using: RRR, MFP and choke cavity facilities. This workflow allows for a sample to be fully characterised in two weeks, enabling quick optimisation of STF parameters to produce the best performing films. Then, the optimised polishing and deposition procedure can be applied to the split cavity to gain an understanding of whether a good flat sample performance is transferable to a cavity-like geometry.

CONCLUSIONS

Four facilities for evaluating superconducting samples and cavities have been designed, built and put in operation. This enables the TF SRF programme running at CI to optimise TF deposition parameters initially on planar samples and ultimately on closed RF cavities. This complete cycle now enables an accelerated R&D programme for TF SRF technology and production in the UK.

ACKNOWLEDGEMENTS

The authors wish to acknowledge support received by members of ASTeC, namely A. Vick, S. Hitchen, A. Blackett-May, A. Palmer and A. Wootten. We also wish to acknowledge work carried out by staff in the Engineering Technology Centre at Daresbury Laboratory. D. Seal would like to thank UKRI for his PhD funding. This work has been supported by: the IFAST collaboration which has received funding from the European Union's Horizon 2020 Research and Innovation programme under Grant Agreement No 101004730.

ISBN: 978-3-95450-231-8 ISSN: 2673-5490 doi: 10.18429/JACoW-IPAC2023-WEPA141

REFERENCES

[1] O. B. Malyshev *et al.*, "Design, Assembly and Commissioning of a New Cryogenic Facility for Complex Superconducting Thin Film Testing," in *Proc. IPAC'18*, Vancouver, Canada, Apr.-May 2018, pp. 3859–3861. doi:10.18429/JACoW-IPAC2018-THPAL089

[2] O. B. Malyshev *et al.*, "A Facility for Magnetic Field Penetration Measurements on Multilayer S-I-S Structures," in *Proc. SRF'15*, Whistler, Canada, Sep. 2015, pp. 716–719. https://jacow.org/SRF2015/papers/TUPB059.pdf

[3] O. B. Malyshev *et al.*, "First Results of Magnetic Field Penetration Measurements on Multilayer S-I-S Structures," in *Proc. IPAC'16*, Busan, Korea, May 2016, pp. 2245–2247. doi:10.18429/JACoW-IPAC2016-WEPMB057

[4] D. A. Turner *et al.*, "A facility for the characterisation of planar multilayer structures with preliminary niobium results," *Superconductor Science and Technology*, vol. 35, no. 9, p. 095 004, 2022. doi:10.1088/1361-6668/ac7fbf

[5] D. A. Turner *et al.*, "Investigating the superconducting properties and surface morphology of sputtered nb films on cu due to laser treatment," *IEEE Transactions on Applied Superconductivity*, vol. 33, no. 4, pp. 1–12, 2023. doi:10.1109/TASC.2023.3243459

[6] O. B. Malyshev, G. Burt, J. A. Conlon, P. Goudket, N. Pattalwar, and S. M. Pattalwar, "The SRF Thin Film Test Facility in LHe-Free Cryostat," in *Proc. SRF'19*, Dresden, Germany, Jun.-Jul. 2019, pp. 610–612. doi:10.18429/JACoW-SRF2019-TUP070

[7] D. J. Seal *et al.*, "RF Characterisation of Bulk Niobium and Thin Film Coated Planar Samples at 7.8 GHz," in *Proc. LINAC'22*, Liverpool, UK, 2022, pp. 818–821. doi:10.18429/JACoW-LINAC2022-THPOGE10

[8] D. J. Seal *et al.*, "A Low Power Test Facility for SRF Thin Film Testing with High Sample Throughput Rate," in *Proc. SRF'21*, East Lansing, MI, USA, 2022, p. 100. doi:10.18429/JACoW-SRF2021-SUPFDV016

[9] J. Halbritter, "Fortran-program for the computation of the surface impedance of superconductors.," 1970. https://www.osti.gov/biblio/4102575

[10] B. S. Sian, G. Burt, O. B. Malyshev, H. S. Marks, D. J. Seal, and R. Valizadeh, "Split Thin Film SRF 6 GHz Cavities," in *Proc. LINAC'22*, Liverpool, UK, 2022, pp. 814–817. doi:10.18429/JACoW-LINAC2022-THPOGE09

[11] B.-T. Sian *et al.*, "Split 6GHz SRF thin film cavities," presented at IPAC'23, Venice, Italy, May 2023, paper WEPA185, this conference.

[12] R. Valizadeh *et al.*, "PVD Depostion of Nb3Sn Thin Film on Copper Substrate from an Alloy Nb3Sn Target," in *Proc. IPAC'19*, Melbourne, Australia, May 2019, pp. 2818–2821. doi:10.18429/JACoW-IPAC2019-WEPRB011

[13] C. Pira *et al.*, "Impact of the Cu Substrate Surface Preparation on the Morphological, Superconductive and RF Properties of the Nb Superconductive Coatings," in *Proc. SRF'19*, Dresden, Germany, Jun.-Jul. 2019, pp. 935–940. doi:10.18429/JACoW-SRF2019-THP041

[14] R. Valizadeh *et al.*, "A First 6 GHz Cavity Deposition with B1 Superconducting Thin Film at ASTeC," in *Proc. IPAC'22*, Bangkok, Thailand, 2022, pp. 1279–1282. doi:10.18429/JACoW-IPAC2022-TUPOTK031

[15] R. Valizadeh, C. Benjamin, and J. Conlon, "Using HiPIMS to Deposit V3Si Super Conducting Thin Films of Single Target Deposition," presented at IPAC'23, Venice, Italy, May 2023, paper MOPL146, this conference.