

# RF CHARACTERISATION OF BULK NIOBIUM AND THIN FILM COATED PLANAR SAMPLES AT 7.8 GHZ \*

D. Seal<sup>1,2</sup> †, O.B. Malyshev<sup>2,3</sup>, R. Valizadeh<sup>2,3</sup>, T. Sian<sup>1,2</sup>, H. Marks<sup>1,2</sup>, O. Hryhorenko<sup>4</sup>,  
D. Longuevergne<sup>4</sup>, C. Pira<sup>5</sup>, E. Chyhyrynets<sup>5</sup>, E. Marshall<sup>3</sup>, G. Burt<sup>1,2</sup>

<sup>1</sup>Engineering Department, Lancaster University, Lancaster, UK

<sup>2</sup>Cockcroft Institute, UKRI/STFC Daresbury Laboratory, Daresbury, Warrington, UK

<sup>3</sup>ASTeC, UKRI/STFC Daresbury Laboratory, Daresbury, Warrington, UK

<sup>4</sup>Université Paris-Saclay, CNRS/IN2P3, IJCLab, Orsay, France

<sup>5</sup>Legnaro National Laboratories INFN, Legnaro, Italy

## Abstract

Research is ongoing into the use of superconducting thin films to replace bulk niobium for future radio frequency (RF) cavities. A key part of this research requires measuring the RF properties of candidate films. However, coating and testing thin films on full-sized cavities is both costly and time-consuming. Instead, films are typically deposited on small, flat samples and characterised using a test cavity. A cost-effective facility for testing such samples has recently been built and commissioned at Daresbury Laboratory. The facility allows for low power surface resistance measurements at a resonant frequency of 7.8 GHz, temperatures down to 4 K and sample surface magnetic fields up to 1 mT. A brief overview of this facility as well as recent results from measurements of both bulk Nb and thin film coated samples will be presented.

## INTRODUCTION

For over 50 years, the overwhelming majority of superconducting radio frequency (SRF) cavities used in particle accelerators have been manufactured from bulk niobium. This material's extremely low surface resistance,  $R_S$ , means that cavities reach very high intrinsic quality factors ( $Q_0 \approx 10^{10}$ - $10^{11}$ ) at liquid helium temperatures. This also allows for continuous wave operation in order to create very stable beams with low energy spread.

However, Nb cavities are now pushing the theoretical limits in accelerating gradient,  $E_{acc} \approx 55$  MV/m [1]. For example, 1.3 GHz TESLA shaped Nb cavities have recently reached  $E_{acc} \geq 50$  MV/m [2]. As a result, there is a push to develop cavities using alternative superconducting materials to reach higher  $Q_0$  and  $E_{acc}$ . These materials are typically deposited onto copper cavities as thin films tens of nanometers to a few micrometers thick. One of the main incentives for using bulk Cu instead of Nb as the substrate is the significant reduction in material and production costs. In addition, the much higher thermal conductivity of Cu allows for increased thermal stability of cavities during operation.

Ultimately, for these reasons, thin film cavities could lead to shorter, more sustainable accelerator structures.

The development of thin film SRF cavities requires 5 main areas of research: (1) substrate surface preparation, (2) thin film deposition, (3) film characterisation, (4) superconducting DC properties measurements, (5) superconducting RF evaluation. So far, the focus has been on studying the properties of thin films on small planar substrates mainly due to cost savings and ease of deposition and analysis. Results from all 5 areas of research will be used to determine which samples are most likely to perform well on a cavity-shaped geometry for further testing.

A facility for the superconducting RF evaluation has recently been commissioned [3]. The addition of this facility now allows for research and development in all 5 areas of the thin film development programme at Daresbury laboratory.

## FACILITY OVERVIEW

The facility for RF testing of planar samples 90-130 mm in diameter is now in full operation [3, 4]. A diagram of the facility is shown in Fig. 1.

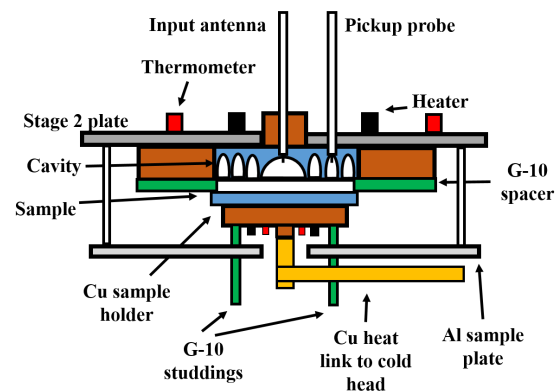


Figure 1: A schematic of the cavity and sample mounted to the stage 2 plate of the cryostat, previously reported at [3].

This facility utilises a bulk Nb half-cell cavity, first reported in [5], operating at a resonant frequency,  $f_0 = 7.8$  GHz. The cavity itself is surrounded by three quarter wavelength chokes designed to contain the fundamental mode frequency within the cavity and minimise leakage. This means that no physical welding is required between the cavity and sample,

\* 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.

† daniel.seal@cockcroft.ac.uk

allowing for an easy sample changeover. The cavity and sample are mounted in a liquid helium free cryostat detailed in [6].

The cavity recently received a light buffer chemical polishing (BCP) treatment at INFN followed by a 120 °C vacuum bake for 48 hours at Daresbury Laboratory. This was carried out to reduce RF power dissipated on the cavity and maximise power on the sample. Recent tests have demonstrated a minimum stage 2 temperature of 3.1 K. Overall, the facility is used to make measurements of  $R_S$  at sample temperatures,  $T_S \geq 4$  K and sample peak magnetic fields,  $B_{S,pk} \leq 1$  mT. Crucially, with the lack of sample-cavity welding, a very high sample throughput rate of 2-3 per week can be achieved.

### Sample-Cavity Gap Optimisation

During the final stages of commissioning, an investigation into the optimum sample-cavity gap size was carried out in CST [7] with the aim of maximising the RF power dissipated on the sample. The results from these simulations are shown in Fig. 2.

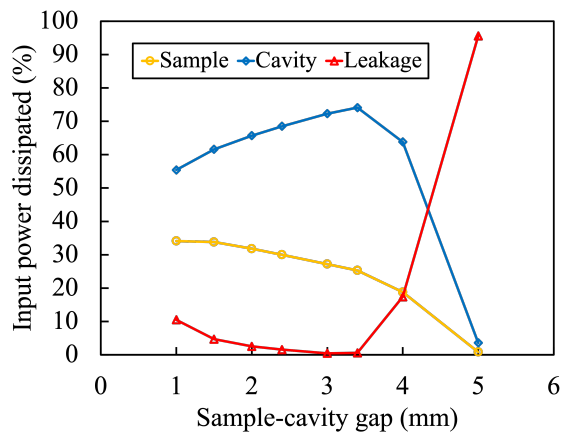


Figure 2: Results from sample-cavity gap simulations.

The method for calculating the  $R_S$  of samples relies on a calorimetric method detailed in [4]. Therefore, it is desirable to maximise the percentage of input power dissipated on the sample. Assuming a critically coupled antenna, the results of modelling demonstrate that this occurs with a gap size of 1 mm, as shown in Fig. 2. Above 3.5 mm, the leakage through the gap rapidly increases due to significant de-tuning of the chokes. Therefore, a gap of 1 mm used and maintained with G-10 spacers between the cavity and the sample.

## SAMPLE CHARACTERISATION

### Bulk Nb Sample

In order to provide a baseline for future sample measurements, a bulk Nb sample disk was tested ( $RRR = 400$ ). Previous measurements on a different bulk Nb sample highlighted the sensitivity of the  $R_S$  to surface treatments [3], however a polished sample had yet to be tested. Polishing is an important step in the preparation of all bulk Nb cavities in order

to remove any defects from manufacturing and produce a smoother surface. This is to reduce  $R_S$  as well as the effect of field emission at higher fields.

The Nb sample being evaluated was treated using a recently developed metallographic polishing method at IJCLab [8, 9]. This technique has the potential for cost-savings and higher levels of repeatability compared to standard BCP and electropolishing (EP) used for Nb cavities. Images of the sample pre- and post-polishing are shown in Fig. 3.

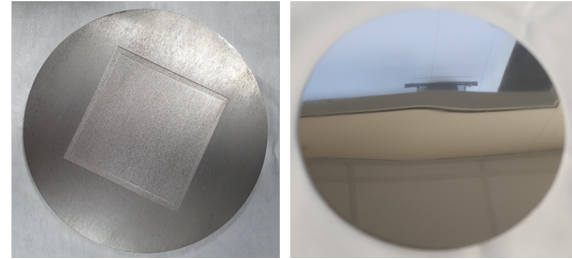


Figure 3: The bulk Nb sample pre- and post-metallographic polishing shown from left to right.

Measurements of  $R_S$  as a function  $T_S$  for this sample before and after polishing are shown in Fig. 4. These measurements, and subsequent results shown, were taken at constant  $B_{S,pk} < 1$  mT. Previous measurements showed  $R_S$  to be constant as a function of low  $B_{S,pk}$  at constant  $T_S$  [3].

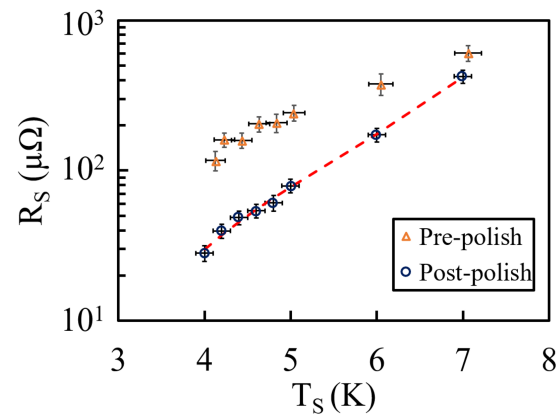


Figure 4:  $R_S$  as a function of  $T_S$  for the bulk Nb sample before and after metallographic polishing. Dashed line shows the BCS fit to post-polished data with  $R_{res} = 5.70 \mu\Omega$ .

These results show a significant improvement in  $R_S$  after polishing of  $\approx 120 \mu\Omega$  at 4.2 K. Visually, the sample surface appears much smoother and more reflective as shown in Fig. 3, so those results are as expected. The theoretical BCS resistance for the polished bulk Nb sample at 7.8 GHz has been fitted using a multi parameter least squares fit [10] based on the widely used Halbritter code [11]. Initial parameters:  $T_c = 9.25$ K,  $\Delta/kT_c = 1.85$ , London penetration depth = 32 nm, coherence length = 39 nm were used. The fit calculated a residual resistance,  $R_{res} = 5.70 \mu\Omega$ . This can likely only be reduced with further surface treatments.

The sample will now be used as a substrate to test the RF performance of thin film multilayers.

### Nb Thin Film Coated Cu Samples

In addition to the bulk Nb sample, three Nb on Cu thin film samples were also tested. Samples 1 and 2 were deposited using pulsed DC magnetron sputtering onto 100 mm diameter Cu disks. Sample 3 was deposited by high impulse magnetron sputtering (HiPIMS) onto a 130 mm Cu disk. Details of these deposition techniques can be found in [12, 13]. Sample substrates were mechanically polished before deposition: Sample 1 and 2 with a diamond abrasive and sample 3 with diamond turning. Based on previous measurements, the thin film samples measured in this paper are  $\approx 1\text{-}3 \mu\Omega$ . Future surface analysis will be carried out to measure thickness accurately. The results of measurements of  $R_S$  as a function of  $T_S$  for three samples are compared in Fig. 5.

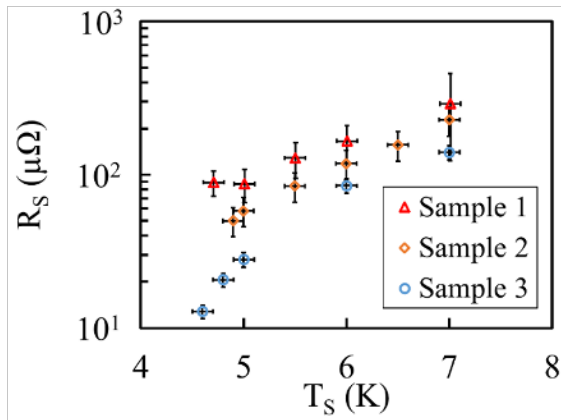


Figure 5: Nb on Cu samples 1-3 shown from left to right.

These results show that sample 3 has the lowest  $R_S$  whilst sample 1 has the highest  $R_S$ . Though surface analysis has yet to be carried out on these samples, these results might be as expected due to the smoother surface produced from diamond turning on sample 3 compared with using a diamond abrasive on the other two samples. This means that sample 3 is likely to have fewer defects and less field emission causing an increase in  $R_S$ . Also, sample 2 experienced some de-lamination which could contribute to an higher  $R_S$ . Sample 1 appears to have larger grain size which could lead to increased losses at the grain boundaries.

### NbTiN Thin Film Coated Cu Sample

For the first time, an alternative superconductor has been characterised with this facility. The sample characterised was a NbTiN thin film deposited onto a 100 mm Cu substrate disk (polished with diamond abrasive) by pulsed DC magnetron sputtering. With a  $T_c = 11.6 - 17.5$  K [14], NbTiN would be a good alternative to Nb. It is also a suitable candidate for multilayer films which has the potential to increase the lower critical field,  $H_{c1}$ , and  $Q_0$  of the cavity and hence increase  $E_{acc}$ .

The measurements of  $R_S$  as a function of  $T_S$  are shown in Fig. 6. These results show a low temperature  $R_S$  to be lower

than both the Nb films and bulk Nb. For example, at 5 K,  $R_S$  is  $\approx 70 \mu\Omega$  lower than the metallographically bulk Nb and  $\approx 19 \mu\Omega$  lower than thin film Nb. Given these results, NbTiN is a promising candidate film that should be analysed further in addition to the Nb samples.

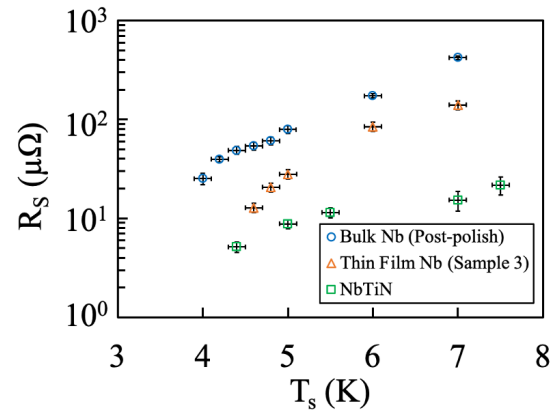


Figure 6:  $R_S$  as a function of  $T_S$  for the NbTiN sample compared with the polished bulk Nb and Nb thin film sample 3.

The next step will characterise the films in a magnetic field penetration facility [15]. This would allow investigation of the effect of a DC magnetic field applied parallel to the sample surface, in similar conditions to the field induced inside a cavity. Surface characterisation of all samples shown will also be carried out to further understand the results. With full results for each sample, measurements on cavity depositions can be considered.

## CONCLUSIONS

A facility at Daresbury Laboratory capable of measuring  $R_S$  of planar thin film samples at  $T_S \geq 4$  K is now fully operational. Crucially, this facility allows for a very high throughput rate of 2-3 samples per week, enabling the quick RF characterisation of samples to keep up with the high rate of sample production. In addition to the samples already tested, more single layers, including  $Nb_3Sn$ ,  $Nb_3Al$ ,  $NbN$ ,  $MgB_2$ ,  $V_3Si$ , as well as multilayers, will be characterised with this facility and followed up with DC magnetic field penetration measurements and surface characterisation.

Daresbury Laboratory now has facilities fully operational in all 5 areas of our thin film development programme. As a result, the programme can move at a faster rate to find candidate samples for future cavity depositions and accelerate the progress of thin film SRF research.

## ACKNOWLEDGEMENTS

The authors wish to acknowledge support received by members of ASTeC, namely J. Conlon, S. Hitchen, A. May, K. Dumbell, D. Turner, A. Hannah and L. Smith. 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.

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