# INVESTIGATION, USING Nb FOILS TO CHARACTERISE THE OPTIMAL DIMENSIONS OF SAMPLES MEASURED BY THE MAGNETIC FIELD PENETRATION FACILITY\*

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# Abstract

At Daresbury Laboratory, in depth Research and Development is attempting to push the maximum accelerating gradient  $(E_{acc})$  forward through its Thin Films SRF programme, by increasing the maximum magnetic field (B) that can be applied to an SRF cavity wall whilst maintaining the Meissner effect. Nb is the best performing single element as it has both the highest  $T_c$  and highest lower critical field  $(B_{c1})$  allowing the cavity to maintain the Meissner effect longer and improve efficiency. At Daresbury Lab, the Magnetic Field Penetration Facility (MFPF) tests flat samples by applying a DC magnetic field parallel to sample, replicating the conditions of the cavity wall. The field of full flux penetration  $(B_{fn})$  is determined by two Hall probes (HP1 and HP2), situated on either side of the sample. The facility can measure samples no larger than 50 mm diameter, but it is important to know how sample size can affect results due to field leakage around the sample. This paper shows results of a study that measured the effects of sample size (from 50 x  $50 - 10 \times 10 \text{ mm}^2$ ) and sample thickness in the range of 1 -100 µm to determine the ideal geometry for field penetration measurements using MFPF. Nb foil samples were used to allow easy alteration of size.

# **INTRODUCTION**

Charged particles are accelerated in particle accelerators by using radio frequency (RF) cavities. Superconducting Radio Frequency (SRF) cavities are used to enhance power and prevent energy loss through a resistive heat into the cavity walls, improving efficiency, whilst lowering operating costs. Superconducting materials are used to coat the cavities as they have unique properties, meaning they can remain in the Meissner state at higher acceleration field  $E_{acc}$ than other materials and therefore limit power loss [1]. In the Meissner state, DC resistance is equal to zero and all external magnetic fields are expelled, as long as the material remains below the critical temperature  $T_c$ . Therefore, it is important to develop new materials that can push the  $T_c$ higher and allow greater cavity performance. Nb is currently the leading, single element material used for cavity coating, as it has the highest critical temperature  $T_c = 9.25$  K and

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the largest, lower critical field  $(H_{C1})$  [2]. However, a new approach to sample structure using thin films which incorporate a thin Nb film, more than 1 micron on top of a Cu substrate to improve thermal conduction and multi-layers that use multi structures in the sample: Superconducting – insulation – superconducting (SIS) make it possible to push the boundaries of bulk Nb further, increasing  $T_c$  and  $H_{c1}$  [3, 4].

## **EXPERIMENTAL**

## Magnetic Field Penetration Facility

The Magnetic Field Penetration Facility (MFPF), built at Daresbury Laboratory, is described in details in Ref. [1]. It allows a practical and time efficient alternative to testing SRF cavity layers for their SC properties. Quicker turnaround times of sample testing, allows the optimal material composition to be identified without the need to coat the cavity beforehand thus, improving operational costs and time efficiency. The MFPF schematically shown in Fig. 1 operates by applying a DC magnetic field, through a 2 mm gap in a carbon based, steel, yoke magnet, parallel to the surface of a flat sample, to replicate the conditions met by the cavity wall.  $B_{fp}$  is defined using two Hall probes, HP1 and HP2. HP1 is situated between the yoke dipoles, directly above the sample, and measures the maximum applied magnetic field  $(B_1)$  [1], while HP2 sits in a carved out trench, directly below the sample. The MFPF operates within the temperature range of 2.6 and 30 K and can apply an optimal magnetic field of 600 mT. The facility operates by reducing the temperature below the  $T_c$  of the sample to induce the Meissner effect. Once the sample is in a SC state, a magnetic field is applied until the SC state is overcome and  $B_{fp}$  is defined as the point the field fully penetrates the sample. This operation is then repeated at different temperature set points to provide an overall picture of how the material is performing. With these applications in mind, it is sensible to determine a good understanding of the MFPF's limitations as a function of sample size and thickness in order to ensure the correct sample geometry is used and in turn provides the best results. An investigation was performed earlier [1], using a type one SC (Pb) to correctly identify the size limitations of a sample before leakage around the edges becomes an issue. Pb was used as it demonstrates a sharp transition and is malleable, making it simple to manipulate to different sizes. The MFPF

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focuses primarily, on type 2 SC, so this paper reports similar experiments using Nb.



Figure 1: Schematics of MFPF Experiment.



Figure 2:  $B_{fp}$  as a function of thickness between 100 and 1300 µm in comparison to the data from ODU [5].

#### RESULTS

# Samples

For study of the effect of sample thickness, *d*, Nb Foils (99.99% purity) were procured from Kurt J. Lesker Ltd. All Nb foil samples are 50 x 50 mm<sup>2</sup>, with a thickness ranges of 1 to 100  $\mu$ m. In addition, a bulk Nb sample with *RRR* = 400 with a diameter of 50 mm and thickness of 1.3 mm, was manufactured by Niowave inc. (USA).

## Experimental procedure

Two sets of measurements were performed in this work:

- Studying the effect of sample thickness,
- Studying the effect of sample size.

The 1.3-mm thick sample was placed between the sample plate and the magnet. Temperature is uniform between the magnet and sample plate, while the magnet sits on top of the sample and provides sufficient pressure to ensure adequate thermal contact between the sample and the sample plate. Thin foil samples are tested with an underlined 0.16-mm thick brass plate beneath to ensure the sample flatness.

The samples were tested at a fixed temperature  $T_s$  in the range of 2.6 to 10 K by increasing magnetic field  $B_1$  from 0 to 280 mT, followed by a demagnetisation procedure with  $B_1 = 0$  and  $T_s = 30$  K in each run. Temperature  $T_s$  was measured by a Cernox sensor using a 335 temperature controller (LakeShore, USA), the temperature sensor was positioned on top of the sample. Magnetic field is defined using two Hall probes, HP1 for applied magnetic field and HP2 for penetration.

For studying the effect of sample size, Nb Foils are used (10  $\mu$ m thickness), supplied be Kurt J Lesker (99.99% purity). A 50 x 50 mm<sup>2</sup> sample was tested first, before being cut down to lower sizes in 5 mm<sup>2</sup> increments ending at 10 x 10 mm<sup>2</sup>.

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Figure 2 displays  $B_{fp}$  for a 100 µm thick foil and a 1.3 mm thick bulk Nb samples measured at  $T_s = 4.2$  K. These results can be compared to the results presented bu Old Dominion University (ODU) team, obtained from a separate MFPF at a slightly higher temperature of 4.35 K [5]. The ODU results show a linear dependence of  $B_{fp}$  for bulk Nb samples with a thickness between 160 – 1000 µm, extrapolated to zero thickness to obtain  $B_{fp}(0T) = 132.5$  mT which is close to  $B_{c1} = 135.1$  mT [5]. These ODU results are largly comparable to DL results displayed on the same figure.



Figure 3:  $B_{fp}$  as a function of thickness between 1 and 100 µm.

Figure 3 displays the results for the all foil samples with thickness from 1 to 100  $\mu$ m, to determine correlations and trends. An increase of  $B_{fp}$  is observed as thickness increases up to 50  $\mu$ m, which is sensible. Conversely, 10 and 50  $\mu$ m thick samples shows a higher  $B_{fp}$  than 100  $\mu$ m thick one, which is unexpected and contrary to the expected trend.

Figure 4 uses the raw data to plot  $B_2$  as a function of  $B_1$  to display size ranges from 50 x 45 to 40 x 40 mm<sup>2</sup> for 10-µm thick Nb foil.

Smaller dimensions were also tested, however, once below  $40 \times 40 \text{ mm}^2$ , their results became even less sensible to



Figure 4:  $B_2$  as a function of  $B_1$  for samples of varying size.

the presence of sample, again, demonstrating how smaller samples show a limiting factor of results due to field leakage around the sample. Analysis of figure two shows that as  $B_2$ increases on 50 x 45 mm<sup>2</sup> sample a sharp  $B_{fp} = 250$  mT can be observed, 45 x 45 mm<sup>2</sup> has a slight drop off and the same trend continues through the remaining size ranges before size becomes a limiting factor for further measurements.

## DISCUSSION

As a magnetic field is applied to a type 2 SC, one would expect that the thicker a sample the higher magnetic field should be applied to penetrate. Figure 2 is clearly demonstrating this trend in the range from 100 to 1300  $\mu$ m.

Also, there is a clear correlation between the results obtained at ODU and DL for Nb thickness in this range observed in Fig. 2. It shows that samples measured above 100 µm follow a similar linear path, were the samples with a greater thickness display higher  $B_{fp}$ . The DL data points are slightly higher than the linear fit displayed for ODU data that could be attributed two factors: a difference between the facilities and a difference in the samples.

In the case of the ODU, the samples were measured at a slightly higher temperature (4.35 K instead of 4.2 K at DL). A different cool down method was used, the experimental set-up was submerged in liquid helium (LHe) bath, contained within a cryogenic Dewar [4]. While the DL facility cooling method is different, using a cold head compressor to deliver cryogenic temperatures to the stage two, sample plate, which creates good thermal conduction between the plate and sample [1]

Another difference between the measurements at DL and ODU is the samples and the preparation before experiment. At DL the sample were used "as-received", while the bulk samples at ODU were etched with BCP.

This can be observed in Fig. 2 as the thicker sample (bulk Nb) shows the highest  $B_{fp}$  while ODU shows a linear fit as  $B_{fp}$  lowers as a function of thickness decrease from 160 - 1000 µm and DL Nb foil sample (100) displays the lowest

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 $B_{fp}$ . Figure 2, therefore, is an example of the relationship between sample thickness and penetration of the magnetic field as  $B_{fp}$  increases with thickness.

Figure 3 provides a further examples of this relationship, were only foils measured at DL are displayed. The results obtained at T = 4.2 K have been plotted for the foil thicknesses ranged of 1 - 100 µm. The trend shows that  $B_{fp}$  increases with thickness but the  $B_{fp}$  for 100-µm thick samples is lower than ones for samples with thickness of 10 and 50 µm. Samples 50 and 100 µm have been plotted separately as they had a different manufacturing process, were, annealing was carried out. Further investigation will be performed to address this: foils should be characterised for structure, morphology, chemical composition, etc.

An example of how sample size has a direct impact on results of  $B_{fp}$  is shown in Fig. 4, where there is a plot of the raw data, with  $B_2$ , field penetration as a function of  $B_1$ , the applied magnetic field, for three samples with difference size. One can see that the results for samples with sizes of  $50 \times 45 \text{ mm}^2$  and  $45 \times 45 \text{ mm}^2$  provide practically the same value for  $B_{fp}$ , 170 and 175 mT respectively, while for the 40 x 40 mm<sup>2</sup> sample  $B_{fp} \approx 100$  mT. this can be explained as the following. Magnetic field has the highest strength in the 2-mm gap between the poles, however, there is a similar magnetic field outside the gap. As sample size decreases the magnetic field can begin to reach (and be detected by) the Hall probe on the under side of the sample, HP2. in other words, a small sample is less efficient to screen HP2 from the applied magnetic field  $B_1$ , and for sample size less or equal to  $40 \times 40 \text{ mm}^2$ , it becomes impossible to separates magnetic field penetrating through the sample from the magnetic field travelling around the sample.

As  $B_1$  is applied to the sample, the flux propagates parallel to the sample, however, as the magnet has a two mm gap between the dipoles it produces a field large enough measure a sample diameter of 50 x 50 mm<sup>2</sup>. This means, as the sample size decreases the field becomes larger than the sample and *B* loss around the sample starts to occur. Eventually magnetic field leakage around the sample becomes so great that results become invalid. Figure 4 shows an example of this effect, it can be observed that 50 x 45 mm<sup>2</sup> sample follows  $B_1 \cdot K_2$ line in a SC state before an obvious transition is noted at approximately 180 mT. The 45 x 45 mm<sup>2</sup> sample is just as clear to identify a transition, however, for samples under 40 x 40 mm<sup>2</sup> it is observed that the point of transition becomes much harder to define.

## CONCLUSIONS

This investigation has identified how sample size and thickness impact measurements and results using the MFPF. Nb foils where used to measure different thickness and size variations, and analysis was performed to identify the optimum dimensions to achieve the best results. Results of this investigation show that sample sizes 40 x 40 mm<sup>2</sup> and less should not be used as results become invalid due to magnetic field leakage around a sample. For thickness, trends show

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that samples with a greater thickness provide a higher  $B_{fp}$  from 1 - 50 µm showed a trend of  $B_{fp}$  increase with thickness, although the 100 µm sample did not show the same linearity, it did provide a good comparisons between results taken from a previous Nb Bulk (1.3 mm) measurement, conducted at DL and a further Nb (160 - 1000 µm) measurement conducted at ODU. Further investigations should be carried out on this in the future as, previously stated, irregularities between 10 and 100 µm were noted. The MFPF provides a more efficient way to test samples and replicate the conditions met on the cavity walls, without the need to test the cavity itself. This allows a higher turn around of measurements and now further progress to push the efficiency of the facility can be made as a results of this investigation.

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