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MAGNETIC FIELD PENETRATION OF NIOBIUM THIN FILMS PRODUCED BY THE ARIES COLLABORATION*

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Abstract

Superconducting (SC) thin film coatings on Cu substrates are already widely used as an alternative to bulk Nb SRF structures. Using Cu allows improved thermal stability compared to Nb due to having a greater thermal conductivity. Niobium thin film coatings also reduce the amount of Nb required to produce a cavity. The performance of thin film Nb cavities is not as good as bulk Nb cavities. The H2020 ARIES WP15 collaboration studied the impact of substrate polishing and the effect produced on Nb thin film depositions. Multiple samples were produced from Cu and polished with various techniques. The polished Cu substrates were then coated with a Nb film at partner institutions. These samples were characterised with surface characterisation techniques for film morphology and structure. The SC properties were studied with 2 DC techniques, a vibrating sample magnetometer (VSM) and a magnetic field penetration (MFP) facility. The results conclude that both chemical polishing and electropolishing produce the best DC properties in the MFP facility. A comparison between the VSM and the MFP facility can be made for 10 μm thick samples, but not for 3 μm thick samples.

INTRODUCTION

The current material used in superconducting radio frequency (SRF) cavities is bulk Nb which is reaching its theoretical limits. Superconducting (SC) thin film cavities are a good alternative to bulk Nb cavities. Copper is a preferable substrate to Nb for machining due to being more frequently used and being less brittle. Polishing Cu also uses less harmful chemistry than polishing Nb. Another advantage of Cu is its good thermal conductivity, providing better thermal stability and uniformity in comparison to Nb. Thin films reduce the amount of Nb used to make accelerating structures.

Systematic studies of SC thin films were performed by an international collaboration funded by H2020 ARIES project. These studies included the effect of substrate preparation[1] on the growth of Nb thin films. The SC properties of the Nb thin films were measured in RF conditions and in a DC magnetic field using a vibrating sample magnetometer (VSM) and a magnetic field penetration (MFP) facility.

Polishing of Cu Substrates

The condition of the substrate surface is critical for the quality of thin film growth, therefore attention must be paid to surface cleanliness and flatness prior to deposition. After the Cu substrates were cut, they were cleaned and polished with[1] [2]: Chemical polishing (SUBU5), Electropolishing (EP), EP + SUBU5, Tumbling. Once the Cu substrates had been polished and characterized, 15 substrates were equally distributed between INFN, Siegen and STFC for Nb deposition described in [1][2]. After deposition, the Nb thin films were characterized using AFM in non-contact mode. The SC properties of the Nb were then tested in a VSM, with the samples tested in both perpendicular and parallel orientation to the applied magnetic field (B_{app}). This paper is reporting the results of these samples obtained with a new MFP method.

It should be noted that the Nb deposited at STFC were 10 μm thick whilst the samples at Siegen and INFN are 3 μm. Before these samples were tested in the field penetration facility described in the next section, the samples from INFN were laser treated post Nb deposition described in [1].

FIELD PENETRATION FACILITY

Method

In an accelerating cavity the magnetic field induced by the RF wave is applied parallel to the surface of the cavity,

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where only one side of the superconductor interacts with B. In commercial magnetometry such as a VSM, a small sample is surrounded by a uniform DC magnetic field. As the sample is surrounded by B, flux enhancements can occur at edges or corners of the sample where B_{app} may enter the at a lower B_{app} than expected. In addition, it is difficult for the sample orientation to align perfectly with a parallel B_{app}, such that any normal component will enter the superconductor earlier than expected. The MFP method allows a DC magnetic field to be applied parallel to the surface, similar to the B_{app} in an RF cavity.

Facility

An original MFP method using cylindrical samples was suggested by A. Gurevich and implemented at Daresbury Laboratory [3]. Later the system was modified to use a C-shaped dipole magnet previously reported in [4]. The new facility uses a similar C shaped magnet to provide B_{app}, with the main difference between the 2 facilities is the cooling method.

The magnetic field is applied by a ferrite C shaped dipole magnet with a low temperature SC solenoid wrapped around the yoke shown in Fig. 1. The dipole has a small 2 mm gap which allows a strong field to be produced in the center, where the field is parallel to the sample surface whilst the sample is in the Meissner state. The magnet is housed in new facility which has been designed, built and commissioned at Daresbury Laboratory.

The B_{app} is measured with a Hall probe sensor (HP1) placed inside the gap between the poles of the dipole, and the penetrated field (B_{pen}) can be found from the magnetic field measurements (B_{HP2}) with a second Hall probe sensor (HP2) placed directly under the sample shown in Fig 1. The maximum field that has currently been tested is B_{app} = 612 mT at an applied current 8 A.

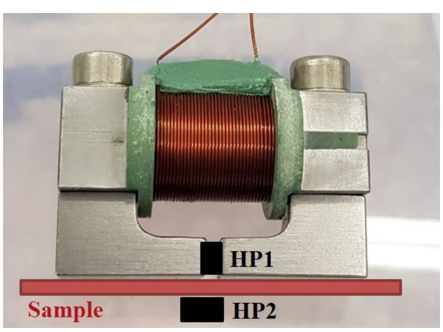

Figure 1: The C shaped dipole magnet with a sketch of the sample and Hall probe sensor set up.

The MFP facility is mounted directly onto a cryo-cooler which allows fast and reliable testing of samples. The testing stage is shown in Fig 2. The center of the sample is placed under the center of the dipole to reduce the amount of field that can leak around the sample. A thermometer is compressed onto the sample using a brass strip, to determine the temperature of the sample. Two resistors either side of the sample are used to control the sample temperature, such as to heat the sample above the critical temperature (T_c) and release any pinning. The resistors are also used to control the temperature of the sample for temperature sweeps. Twisted wires are used for the resistors as to not form a loop, to ensure there is no extra field being applied near the sample that could affect results. Two thermal radiation shields are used to reduce the heat load of the system, and to ensure no higher energy photons hit the sample during testing. The minimum temperature the sample can reach is 2.6 K.

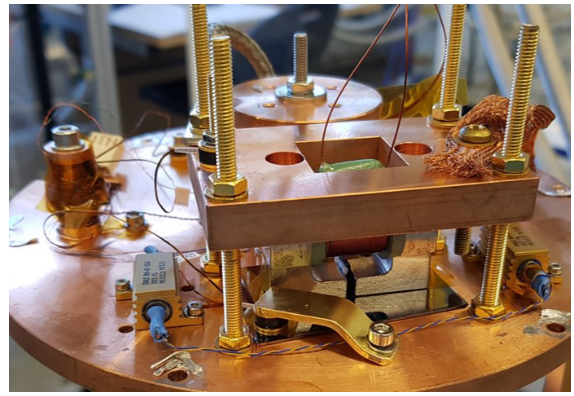

Figure 2: The testing stage for the field penetration facility.

Operation

The sample is cooled in absence of an B_{app}, known as a zero field cooldown (ZFC). Initially the heaters are turned off to reach a minimum sample temperature. The current applied to the magnet is increased in steps defined by the operator, resulting in B_{app} measured by HP1 to increase in steps. The sample temperature, applied current and both B_{app} and B_{pen} are recorded with in-house software. After reaching a B_{app} defined by the operator, the applied current is set to 0 A. To ensure the magnet does not become magnetised it is degaussed after each run. The degaussing procedure is performed by ramping the magnet upto the maximum current applied from the previous test in the opposite polarity, followed by reducing the applied current to 90% of the maximum value and reversing the polarity. This is repeated until the applied current is reduced to the minimum step the power supply can apply (ΔI=1.8 mA which is equivalent to $\approx$ 0.8 mT). The sample is then heated above T_c to release any possible trapped flux, before undergoing ZFC to the next set temperature. The system stabilises the temperature and ensures the temperature is within 0.05 K of the set temperature for 20 minutes before the next step, to ensure the sample has thermalised. The following runs repeat the same procedure at temperatures between 3 K and the T_c. The temperature is set by a temperature controller, which applies a current to the two resistors described in previous the subsection.

Results

The purpose of this study is to measure the field of full flux penetration (B_{fp}). For an infinitely large sample, B_{fp} is the minimum applied magnetic field corresponding to

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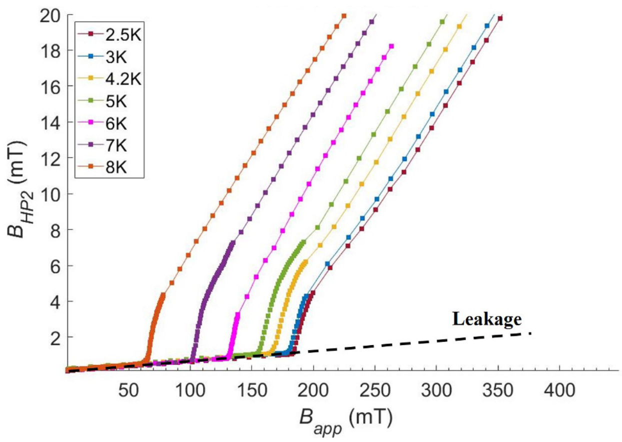

Figure 3: The raw data for sample L19 with a 10-μm thick Nb film deposited at STFC on SUBU5 polished Cu at INFN.

when the magnetic field on the other side of the sample is no longer zero.

It was expected that no magnetic field would be measured using HP2 until the magnetic field had penetrated through the film at B=B_{fp}. However, it was found the sample size does not allow full screening of B_{app}, and a fraction of B_{app} leaks around the sample. An example of the raw data from one of the polished samples is shown in Fig. 3. The magnetic field leaking around the sample can be seen in the low B_{app} region. As the leakage is linear to B_{app} it can be subtracted from the results. It can be seen in Fig. 3, as B_{app} is increased further, there is a sharp increase in B_{HP2} when the field penetrates all the way through the sample, which defines the field of full flux penetration, B_{fp}.

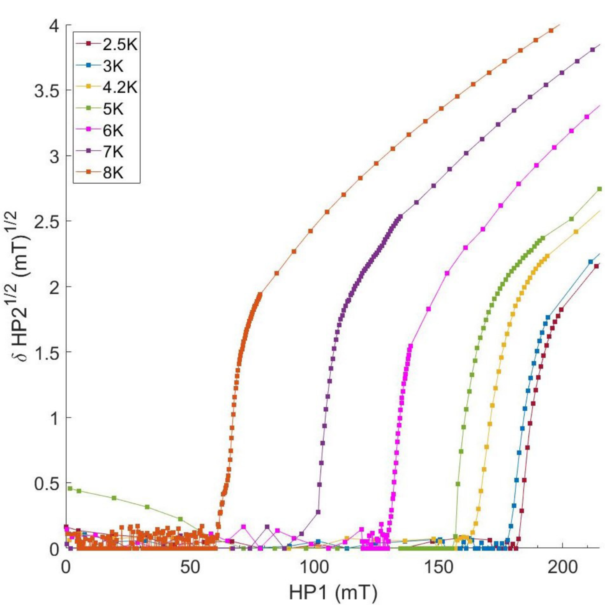

Figure 4: The $\delta_{HP2}{}^{\frac{1}{2}}$ as a function of B_{app} for L19.

To accurately determine B_{fp}, the square root of the standard deviation of B_{HP2} from the linear dependence of the leakage field ($\delta_{HP2}{}^{\frac{1}{2}}$) is used. At B_{fp} there is a sharp increase in $\delta_{HP2}{}^{\frac{1}{2}}$, as shown in Fig. 4. In the following results in B_{fp} correspond to the B_{app} when $\delta_{HP2}{}^{\frac{1}{2}} = 0.2\,\mathrm{mT}^{\frac{1}{2}}$.

The B_{fp} as a function of T can then be plotted to determine the temperature dependence of the superconductor, shown in Fig. 5. This data can be fitted well using the T^2 dependence shown in Eq. 1[5].

$$H_c(T) = H_c(0K)\left(1 - \left(\frac{T}{T_c}\right)^2\right) \tag{1}$$

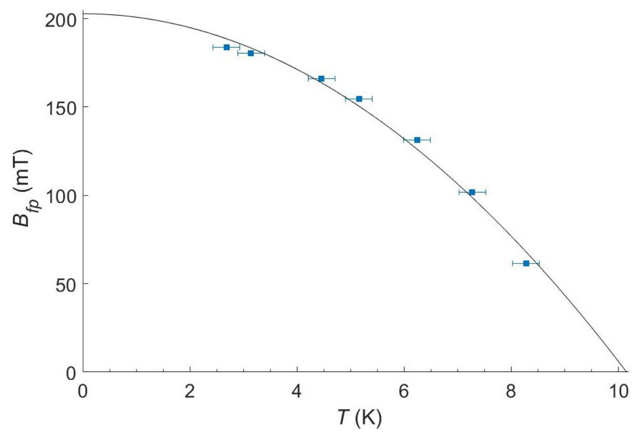

Figure 5: The B_{fp} as a function of temperature, plotted with a T^2 fit.

The Nb on Cu substrates deposited as part of the ARIES program were the first samples to be tested in the MFP facility. Figure 6 summarizes all the results obtained in this study. The B_{fp} as a function of T for each polishing technique is shown in Fig. 6 a, c and e, split into the institutes that deposited the Nb thin film. The B_{fp} is compared to earlier reported results in the from the VSM in Fig. 6 b, d and f, again split into each individual institution. Each institutions set of samples had a variation, such that the effect of substrate polishing could only be analysed within each set. It was observed across each institution shown in Fig. 6 that EP followed by SUBU5 had lowest B_{fp}. It was also observed that both SUBU and EP had a larger B_{fp} individually than both treatments combined. The reduction due to the EP + SUBU polishing technique is more pronounced in the samples from INFN and Siegen. The effect of the other polishing techniques is not as pronounced, but there is still a trend between each institution shown in Table 1. The B_{fp} is also compared to the field of first flux penetration, B_{vp} in a VSM at 4.2 K in Fig. 6 b, d and f. It can be seen that for all institutions the B_{vp} is smaller than B_{fp}. For the STFC samples it is observed that the greater the B_{vp}, the greater B_{fp}. However, this is not the case for Siegen and INFN as shown in Fig. 6 b and d. For example, sample C1 has a greater B_{vp} than L1, however L1 has a greater B_{fp} than C1. It must be noted that if B_{vp} is similar in both the perpendicular and parallel orientation then there must be a normal component of B_{app} in the parallel test, causing early flux penetration. This portrays the difficulty in aligning a sample to be parallel inside a VSM.

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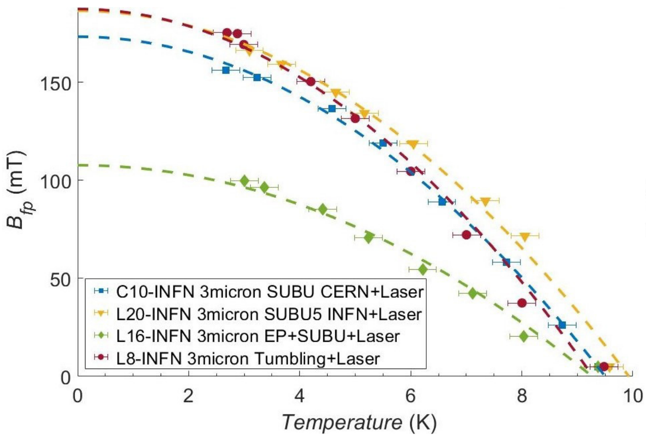
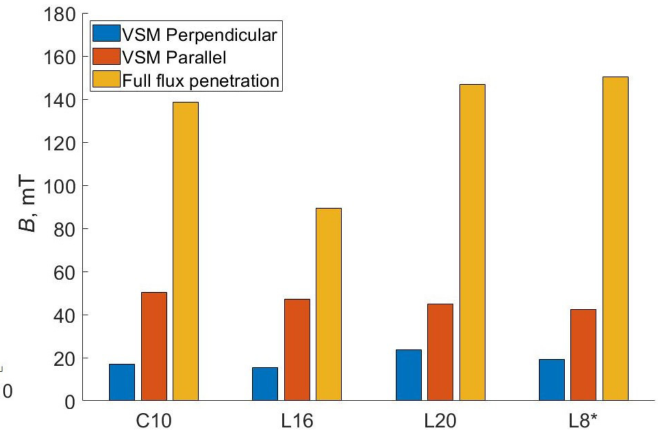
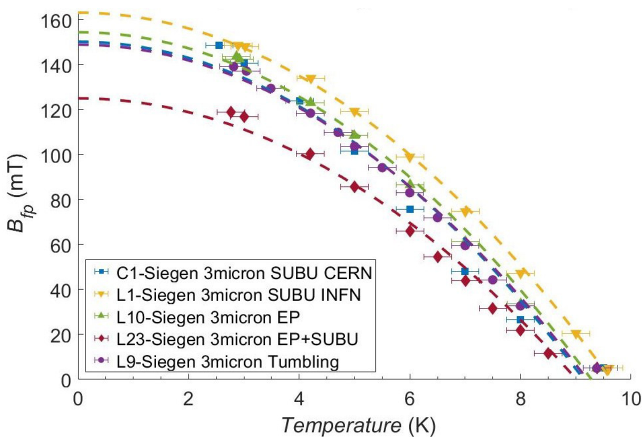


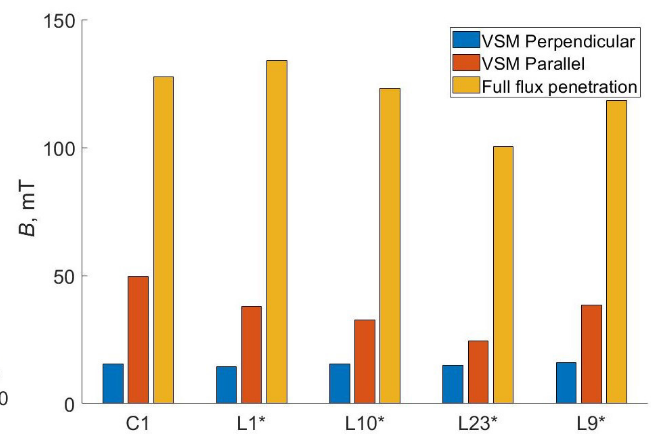
(a) INFN - B_{fp} as a function of temperature



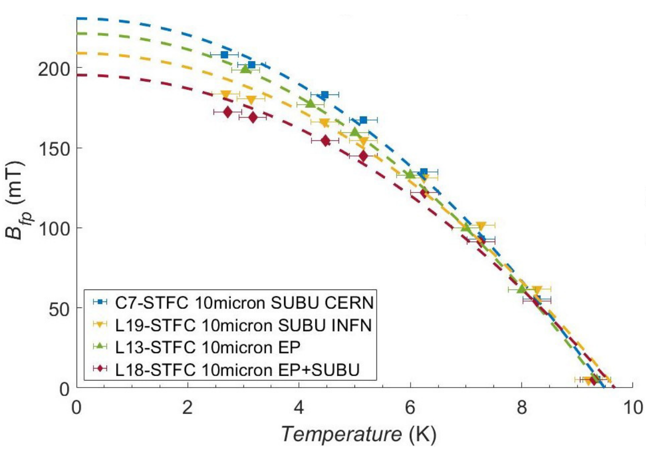
(b) INFN - A comparison between B_{vp} from a VSM and B_{fp} at 4.2 K



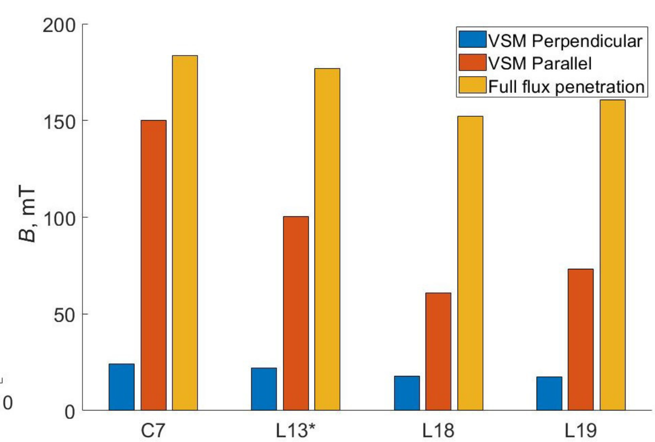
(c) Siegen - B_{fp} as a function of temperature



(d) Siegen - A comparison between B_{vp} from a VSM and B_{fp} at 4.2 K



(e) STFC - B_{fp} as a function of temperature



(f) STFC - A comparison between B_{vp} from a VSM and B_{fp} at 4.2 K

Figure 6: The effect of polishing on B_{fp} for each institution as a function of temperature, and the comparison of B_{fp} compared to B_{vp} in a VSM at 4.2 K for each institution.

The results reported in Fig. 6 do not allow us to conclude if there is a good correlation between MFP measurements and VSM. The same data has been plotted differently, shown in Fig. 7. One can see that there is a clear correlation for the 10 μm thick Nb samples from STFC, however there is no visible correlation for the thinner 3 μm thick Nb samples from INFN or Siegen. A possible explanation for this is due to thinner samples being more sensitive to alignment in VSM. This needs to be validated by testing more samples. Another explanation could be that the nature of the results are different. The VSM measures the magnetic response which is proportional to the total SC volume. The MFP

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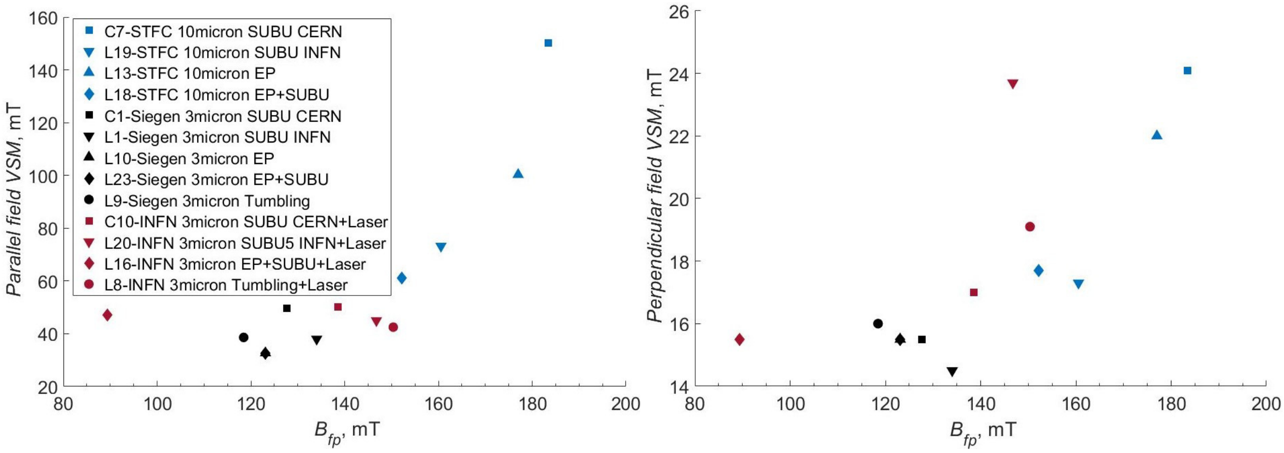

(a) (b)

Figure 7: A comparison of B_{fp} in the MFP facility to B_{vp} in (a) parallel and (b) perpendicular magnetic field in the VSM.

Table 1: The polishing technique that produced the largest B_{fp} (top) to lowest B_{fp} (bottom) for each institutions Nb thin film deposition

STFC	Siegen	INFN
SUBU5 CERN	SUBU5 INFN	SUBU5 INFN
EP	EP	
SUBU5 INFN	SUBU5 CERN	Tumbling
	Tumbling	SUBU5 CERN
EP + SUBU5	EP + SUBU5	EP + SUBU5

facility could be sensitive to any imperfections in the SC thin film. The film is grown and contains many interstitial grain boundaries which may have dislocations such as lattice miss match allowing the field to penetrate through the sample earlier.

For thicker films, the imperfections may allow the magnetic field to enter the sample early, however not allow B to penetrate all the way through the sample such that B_{fp} is observed.

CONCLUSION

A new local magnetometer has been introduced and we have demonstrated that it is a powerful instrument in SC thin film characterization. The facility is built directly onto a cold head and operates in a cryogen free environment, with the sample being able to reach temperatures as low as 2.6 K. A local parallel DC magnetic field is applied from one side of the sample to the other to measure B_{fp}, and has been tested up to a field of 612 mT at 8 A. The field penetration facility can apply a parallel field to planar samples which is both easier to align and less sensitive to misalignment than other methods, which allows us to shed light onto the SC properties of thin films.

The effect of polishing Cu substrates on the SC properties of sputtered Nb films has been investigated using the new facility. It has been shown that EP and SUBU5 are preferred as a polishing technique based on providing a larger B_{fp}. This conclusion matches our earlier results based on visual microscopy and SEM. These results have also been compared to the results produced in a VSM in both parallel and perpendicular orientation, showing some correlation for 10 μm thick films, and no correlation for 3 μm thick films.

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