CHARACTERIZATION OF THIN FILMS USING LOCAL MAGNEOMETER

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Abstract

SIS nanocomposite (Superconductor/Insulator /Superconductor) could improve the efficiency of bulk Nb accelerating cavities as proposed in 2006 by A. Gurevich [1]. The SRF multilayers concept takes advantage of the enhancement of H_{C1} of thin layers with thickness d~λ. The use of thin layers makes it easier to prevent avalanche penetration of vortices in case of local defects that could promote early penetration. The external field is not fully attenuated in such configuration, so several layers are necessary in order to screen the external field down to values below Nb H_{C1}, decoupled from each other with a dielectric layer. Many deposition techniques exist that can allow the deposition of such multilayers but a few of them are adapted for accelerating cavities shapes. Moreover we do not know yet how the predicted properties evolve in realistic deposition conditions. It seems reasonable to start the optimization of such structure on samples. Two parameters need to be measured to predict their behavior in conditions close to cavity operation: H_{C1} (or at least the penetration field for vortices) and surface resistance, in particular the residual resistance part value of which cannot be predicted by the current superconductivity models.

For that purpose a local magnetometer was developed at Saclay.

A local magnetometer allows measuring the vortices penetration on samples without the common orientation and edge effects encountered in classical SQUID magnetometers. Its operating conditions range from 2 to 40 K, with field up to 150 mT, and possible upgrade to higher field.

INTRODUCTION

In this paper, the working principle of local magnetometry, which is a tool of choice to characterize and optimize new superconductors dedicated to SRF application. In particular it allows measuring unambiguously the field at which vortices start to enter the superconducting samples, namely H_{C1}. Conventional Magnetometer (SQUID) give ambiguous results for very thin film samples because of demagnetization effects (field on the back and sides, alignment issues). With Squid measurement, the samples exhibit a strong transverse moment because, due to misalignment and very high demagnetization factor, the perpendicular field component is sufficient to let vortices enter the material. The local field B is then a combination of the uniform external applied field and the transverse moment. Hence even if the penetration field can be determined, the exact local field configuration is not known, it can be far from the cavities configuration. Moreover its physical meaning is difficult to establish. Indeed Squid magnetometry shows that thin layers/multilayers exhibit a strong enhancement of the penetration field H_P (which is expected to be close to H_{C1})[2-6]

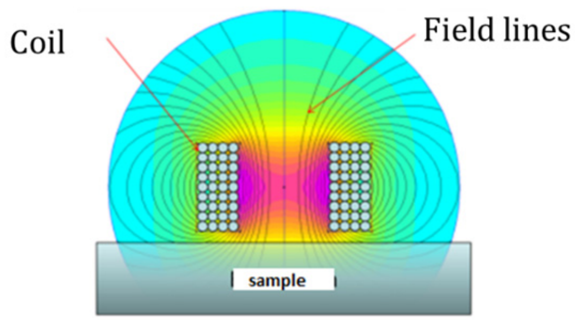
It is shown in [3,7] that the misalignment should be <0.005^0 to avoid demagnetization and edge effects. To overcome these issues there is a mandatory need to measure H_{C1} with another technique.

So, a local magnetometer specifically dedicated to measure thin films and multilayer samples is being developed at Saclay, the details of which will be described in this paper.

Local magnetometry is based on infinite slab approximation (Figure 1): when the field is generated in a coil which diameter is small compared to the size of the sample, the field becomes negligible about four to five diameter away from the center of the coil. So if one takes a sample with diameter larger than this dimension, it can be approximated like an infinite slab. This last condition makes negligible any demagnetization or edge effect.

Local measurement based ac third harmonic (V_3) analysis was proposed in ref. [8-10]. This technique depends on the hysteretic behavior of the magnetization in the critical state, which gives rise to none zero odd harmonics in the spectrum of the electrodynamic response of superconductors an AC magnetic field $b_0 \cos\omega t$ is applied to a zero field cooled sample, where b_o is the amplitude of the ac magnetic field and ω_o is the frequency. As b_o equals H_{C1}, vortices start to penetrate into the superconducting sample. Under these conditions, a nonlinear power law $J \propto E_n$ in the current-voltage curve applies, where the exponent n→∞ in the Bean limit, n>1 in the flux creep regime, n=1 in the flux flow linear regime. Thus odd harmonic components are produced in the spectrum of the sample response signal when it enters into a region of field and temperature in the magnetic phase diagram delimited by H_{C1} and the irreversibility field H_{irr}. Out of all the odd harmonics present, the 3rd harmonic is the most intense.

In other words, the coil provides excitation as well as detection; as long as the sample keeps in the Meissner state, the sample acts as a perfect magnetic mirror, the current (and voltage) in the coil keeps linear. Once vortices start to enter the sample (upon rising temperature and/or rising the current in the coil), the electrons experience a dragging force and this gives rise to non-linearity in the current/voltage inside the coil.



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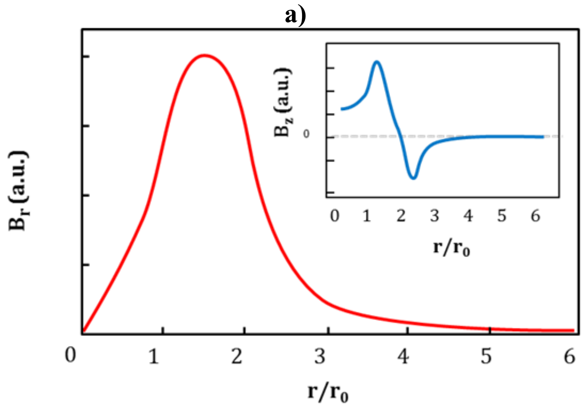

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Figure 1: a) Field repartition for a r_o diameter coil. b) Curve shows the radial repartition 200 µm away from the coils (approximate position of the sample is expected to be closer to the coil: ~60 µm). Inset shows the z component of field behavior.

The onset of non-linearity directly provides the couples (H_{C1}(T)), and allows to reconstruct the transition curve other a large set of fields and temperatures.

Specificities of the Saclay Magnetometer.

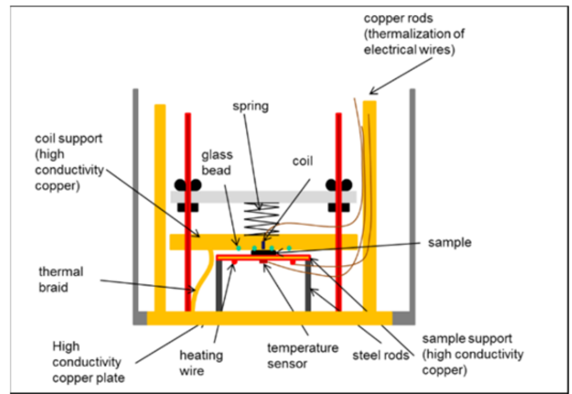
Similar set-ups exist worldwide, but most of them are dedicated to the study of HT_C superconductors in the mixed state close to T_C, hence with a moderate field (typically 10-20 mT). Most of these facilities are equipped for direct nitrogen or helium vapor cooling.

The design of the local magnetometer adapted at Saclay is based on cavity operating condition i.e. T= 2-4 K, B ~ 200 mT; i.e. at superfluid helium, and field 10 fold higher than usual.

To reach such stringent conditions, some improvement of the design had to be implemented. A coil had to be designed to go to higher fields. Modeling showed that putting a standard NbTi superconductor in AC would be more dissipative than copper, so we designed a 5 mm ∅ high conductivity copper coil (wire 100µ ∅, RRR 100).

One of the most important issue is thermal stabilization at high field because of high I in Cu coil. Efforts have been done to increase the thermal insulation between the sample and the coil:

- The experiment is under vacuum to prevent thermal contact between the sample and the coil.
- It is cooled by conduction, which allows pumping on the He bath and decreasing the temperature below 2 K when necessary.
- Glass beads provide point contact and fixed distance between sample and coil holder.
- Coil holder (copper, high thermal conductivity) is linked to a cold sink to evacuate dissipation. It is also designed to reduce Eddy current.
  (see Figure 2)



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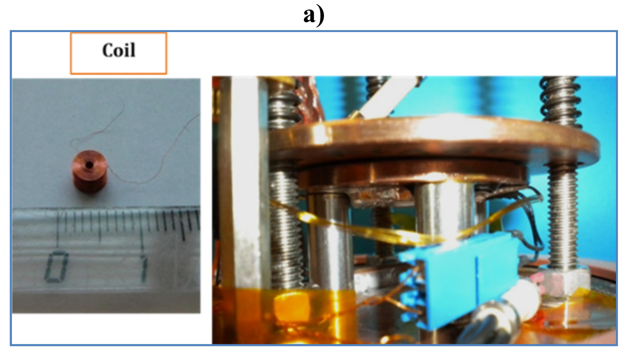

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Figure 2: a) Schematic of the local magnetometry facility at Saclay, b) Details of the coil and sample holder.

EXPERIMENTAL DEVELOPMENTS

Experiment is performed using two methods. Initially the setup was zero-field cooled down below the transition temperature of sample. Than we applied a constant current I_0cos (ωt) to the coil (field = $b_0$cos (ωt) on sample). The temperature of the sample was then slowly increased (0.1 K/s). When the temperature reached the transition temperature for that applied field, the vortices started to enter the sample and odd harmonic signal appeared. We made series of measurements of different b_0 corresponding to series of transitions B_{C1}(T), thus reconstructing the B_{C1} curve. But this approach had thermal stabilization issue at higher fields (when high current was applied, temperature rises for a short while because of the inertia of the thermal regulation and the transition can be reached before the measurement can start). So a new approach was developed for the measurement of the signal. In this method, after zero field cooling, the experiment is kept at a fixed temperature T,

ISBN 978-3-95450-178-6

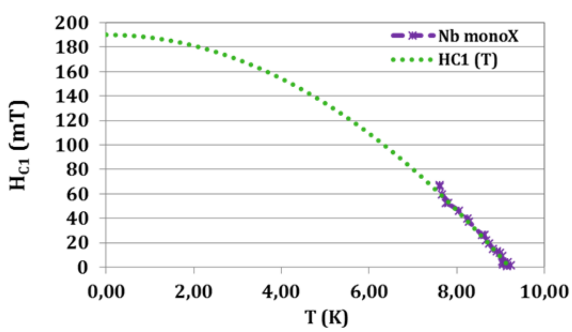
then a current $I_0\cos(\omega t)$ (field = $b_0\cos(\omega t)$ is increased slowly in the coil until the apparition of 3rd harmonics. Series of experiments are done at different T and corresponding applied current is measured, allowing to reconstruct the $B_{C1}(T)$ curve (Fig. 2). We have checked this method manually and temperature was stable within 0.1 K range at high fields without any unwanted upsurge.

This experiment had moreover to be fully refurbished because it was very sensitive to external parasitic signal. New grounding and new shielded cables were installed and filtered power supply was provided.

Present Performance

The measurement using initial method was done for Nb monocrystalline and Multilayers. The curve in Figure 3 shows the results. From the curve it can be seen that for monocrystalline Nb the measured data curve follows the standard curve up to 180 mT. Thanks to recent improvements described here above, we can operate at fields ~150 mT.

At field higher than 80 mT, we noticed that the transition was following the initial part of the curve, but was sitting on the top of another signal. We think that at high field, the infinite slab approximation is not valid anymore for our 2x2 cm^2 samples. And that somehow the field has gone around the sample (as in Squid magnetometry).



a)

The use of larger samples should easily overcome this problem. It is somehow comforting that the experimental points seem to follow the initial trend.

CONCLUSION AND PERSPECTIVES

This set-up has proven to be very useful to measure the penetration field of samples in field configuration close to cavity operation conditions. Right now it was able to assess unambiguously the screening effect of very thin layers [2] and the field enhancement effect of multilayer structures [2, 3, 11] on model samples deposited on thick sputtered Nb sublayers.

Design and operation improvement should now open the route toward the higher field, in order to be able to measure more realistic samples, deposited onto bulk Nb,

- We need to find a way to increase the field without increasing the power sent in the set-up. For that we have explored 3 different routes:
- As mentioned, we had tried to design a superconducting coil, but the simulation with standard SC DC wire (NbTi) showed 10 times more dissipation than copper. Specific cables exist for AC application, but we were not able to get it in a single wire shape.
- We also have tried using R/W heads as presented initially by S. Anlage (see e.g. [12]) but this requires microscopic bonding machine, and up to now we were not successful in getting good connections.
- Last we also tried to implement an Iron "nail" inside the same coil as the one presently used in the experimental set-up.

Figure 4 shows the simulation carried with and without iron core inside the coil. It can be seen with the use of iron core the field can be enhanced from 90 mT up to 400 mT on the sample for the same intensity of current in the coil, which would be high enough for the foreseen applications. This solution will be soon tested in the facility.

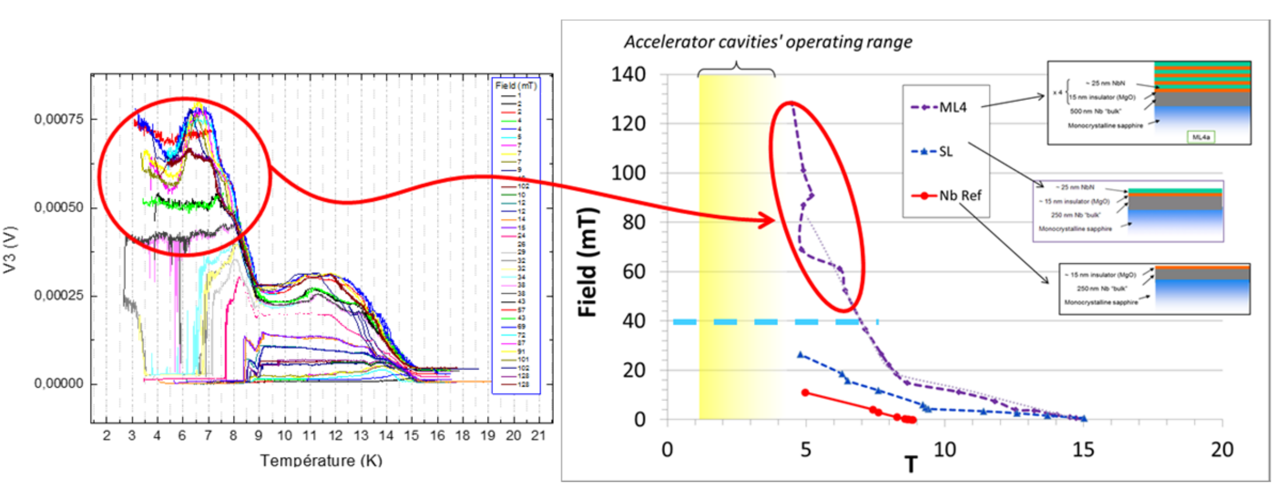


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Figure 3: Example of preliminary results for a) Nb. b) multilayer samples.

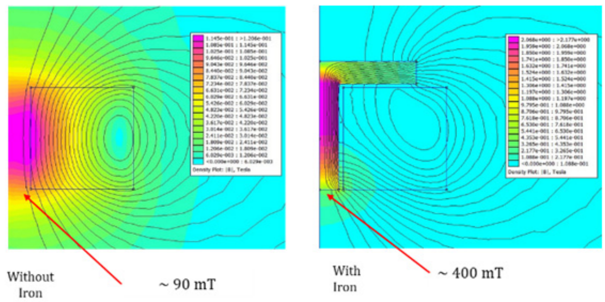

Figure 4: Field lines before and after the insertion of iron core inside the coil to enhance the magnetic field ~ a factor 4 on the sample.

The drawback of having Iron in the system is that iron may bring some additional non linearity at high field. Since they are not expected to be temperature dependent, one can in principle easily differentiate them from the vortex contribution. The local magnetometer has proven to be effective and is currently undergoing upgrade for higher field operation. A series of ML samples with varying thickness will be measured in near future.

ACKNOWLEDGMENT

We sincerely thank our technical staff and cryogenic staff for helping us carrying out the experiment. The work is part of EuCARD-2, partly funded by the European Commission, GA 312453.

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ISBN 978-3-95450-178-6