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LOCAL MAGNETOMETER: FIRST CRITICAL FIELD MEASUREMENT OF MULTILAYER SUPERCONDUCTORS

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Abstract
S-I-S (Superconductor-Insulator-Superconductor) nanometric superconducting multilayers have been proposed by Gurevich [1] to increase the maximum accelerating field of Nb RF cavities. This enhancement of Hc_1 may be done by coating Nb with thin layers of thickness less than the penetration depth ($d \leq \lambda$). Therefore, it is necessary to find a particular tool, which allows us measuring Hc_1 directly. In fact, DC magnetometers (e.g. SQUID magnetometers) are largely used for magnetic measurements but these last are strongly influenced by orientation, edge and shape effects, especially in the case of superconductor thin films. For that reason, we developed at Saclay a specific local magnetic measurement of first critical field Hc_1.

INTRODUCTION

The first critical field Hc_1 is one of the key physical parameters characterizing a superconducting material. This parameter is usually measured by conventional magnetometers (SQUID). For thin superconducting films, these measurements are strongly influenced by orientation, edge and shape effects. These devices give ambiguous results for very thin samples because of demagnetization effects (field on the back and sides, alignment issues (Fig. 1a). Samples exhibit a strong transverse moment, due to misalignment, which is sufficient to let vortices entered the material.

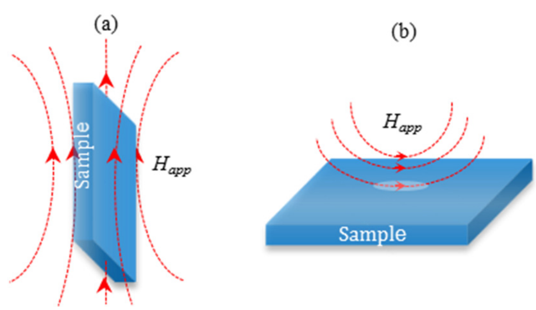

Figure 1: a) SQUID magnetometer principle. b) Local magnetometer principle.

Therefore, the development of a local magnetometer is necessary to measure directly the first critical field Hc_1 on superconductor sample without edge nor demagnetization effect.

When the coil is much smaller than the sample, the magnetic field decreases quickly away from the coil and the sample can be considered as an infinite plane for the field lines, with no edge and demagnetizing effect (Fig. 1b).

In this experimental set-up, the field configuration is similar to cavities, i.e. parallel to the surface and only on one side. In this paper, we will describe the evolution of the design of a local magnetometer specifically dedicated to measure thin films and multilayer samples, which is being developed at Saclay.

METHODOLOGY

The principle of our local magnetometer is based on the third-harmonic voltage method purposed by Claassen [2], it is non-destructive and contactless, but more importantly, without demagnetization effects The method of third harmonic analysis is currently used to study vortex behavior in superconducting (SC) samples (H_{irr} and/or J_C). Lamura showed in 2009 that it also can be used to measure Hc_1 [3].

By monitoring the intensity and/or the phase of the third harmonic signal (i. e. the higher harmonic) one can detect accurately the transition to the mixed state in a configuration that is close to the cavity operation conditions. In this configuration, if the samples were devoid of defects, we could in principle access to the superheating field. In the following we nevertheless will refer to it as Hc_1 as a precaution, meaning the transition of the composite sample to the mixed state.

The coil provides excitation as well as detection. It produces an AC magnetic field at the surface of the sample. As long as the sample keeps in the Meissner state, the sample acts as a perfect magnetic mirror: when the sample is in the Meissner state, the sample generates a magnetic moment opposed to the vertical component of H_{app} and the resulting induction is equals to twice the horizontal component of H_{app} : $2\mu_0\ H_{app}{}^{hor}$. The current (and voltage) in the coil keeps linear and shows the same sinusoidal curve as the reference signal at frequency ω. (Fig. 2).

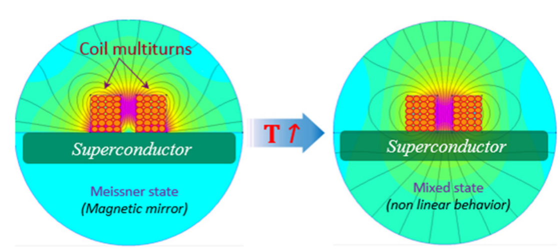

Figure 2: Repartition of the field lines in the Meissner state and in the mixed state.

Once vortices start to enter the sample (upon rising temperature or rising the current in the coil), they are pinned by defects, and the electrons from the coil experience a dragging force that gives rise to nonlinearity in the current/voltage inside the coil. In addition to the reference frequency ω, one start to observe higher odd harmonics. By monitoring the 3rd harmonic (most intense harmonic) one is able to determine the temperature of vortices penetration for a given field. The experiment is repeated for various

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current values, and the transition curve can be rebuilt by this way.

DESIGN

After initial measurements at Napoli with a standard magnetometer [4] we had to develop a design specific to our operating conditions: 1.8 – 40 K and over than 200 mT on the sample. Moreover, we need to use a copper coil even at high field, because most of the available superconducting wires at our disposal would be very dissipative at our operating frequency (1 kHz). So, the main challenge in our facility is the thermal design. Moreover even if used a wire adapted to high frequency, as it would be in the mixed state, it might produce its own non linearities.

Structure of the Set-up

Figure 3 describes the general features of the set-up. The set-up is placed under vacuum tank to prevent thermal contact between the sample and the coil. A high-quality copper plate is brazed to the bottom of this cell and provides a cold sink. The vacuum tank is immersed in the He bath (liquid or superfluid).

The sample is placed on a copper plate which will ensure a good uniformity of temperature. The sample holder is related to the cold sink with steel supports, having a thermal conductivity sufficient to cool the sample rather quickly, but not too much to be able to heat it anyway. A heater and a temperature sensor placed under this support will allow regulating the temperature.

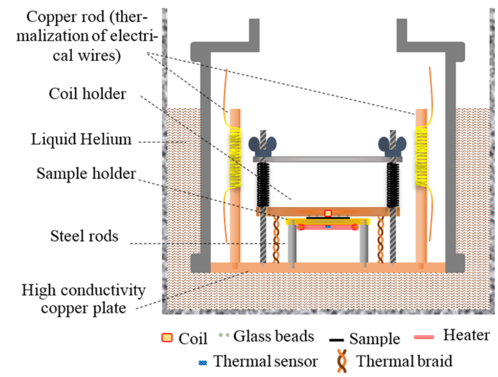

Figure 3: Scheme of the experimental set-up.

The coil creating the field is made of high conductivity copper (outer diameter of 5 mm, wire 100µm, RRR = 114). It is fixed on a copper plate, which can adapt to the thickness of the sample and is kept in place with springs. The coil is cooled by thermal conduction, by means of a copper braid connecting the plate and the cold point. In order to avoid the heat transfer between the coil and the sample while keeping them at a known and constant distance, we use glass beads as spacers (punctual contacts). Copper rods fixed on the cold plate are used to thermalize the connection wires.

Both sample holder and coil holder had to be modified several times to face thermal instabilities and transient state thermal calculation had to be conducted to assert the actual origin of the losses (see below).

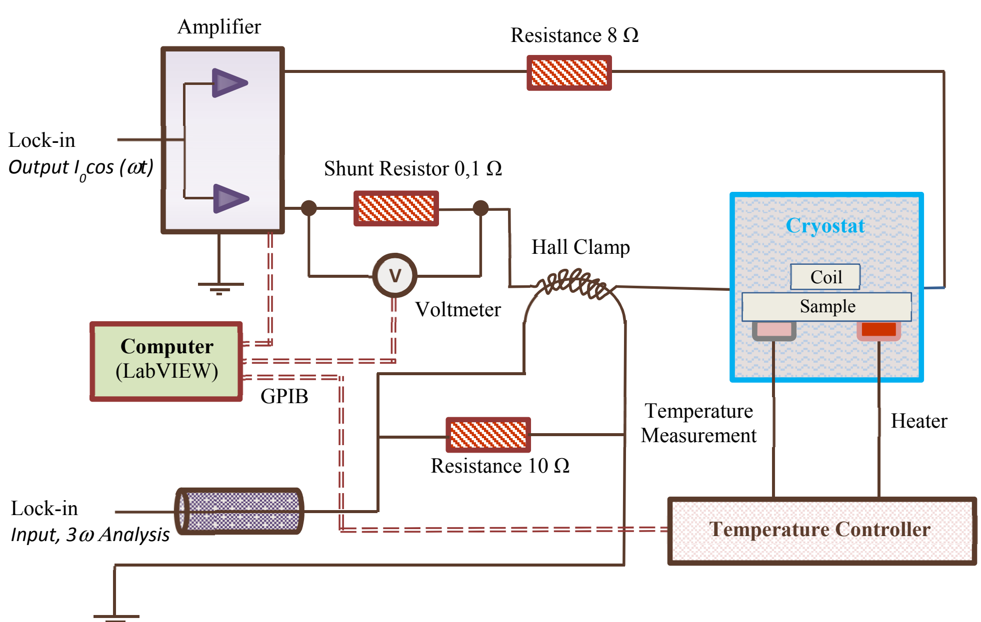

Figure 4: The block diagram of an electronic system for measuring Hc_1 by using the 3^{rd} harmonic method.

Signal Acquisition

The harmonics are detected by means of a Lock-in amplifier with synchronous detection (SR830). This device sends the reference signal at a specific frequency (1 kHz in our case) and compares it with a pick-up signal. An internal filter allows to select only the 3 signal (see block diagram in Fig. 4. Experiment is performed by zero-field cooling the sample below the transition temperature TC. Then we apply a constant current $I_0 \cos(\omega t)$ to the coil

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(field $= b_0 \cos(\omega t)$ on sample). The temperature of the sample was then slowly increased (0.1-0.5 K/s). When the temperature reached the transition temperature for that applied field, the vortices started to enter the sample and odd harmonic signal appears. We made series of measurements of different b_0 corresponding to series of transitions Bc1(T), thus reconstructing the Bc1 curve. But this approach had thermal stabilization issues at higher fields (when high current is 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).

CALIBRATION

The calibration of our measurement system was done with bulk Nb monocrystalline which has RRR = 300 and T_C = 8.9 K. The main problem is the thermal stabilization and occasional shift or instabilities in the temperature measurements (Fig. 5), superimposed with EMC troubles. Once the EMC trouble solved [5], we discovered that our first thermal calculations, done supposing that our slow temperature rise allowed the system to reach equilibrium, led us to an error in the design of the sample holder. Transient state calculation allowed us to better understand the origin of the trouble.

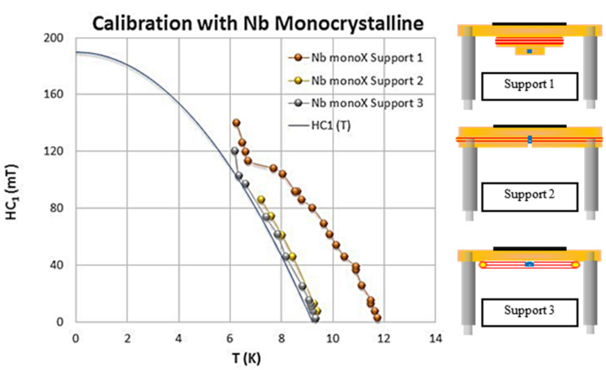

Figure 5: Several improvements for sample holder to achieve good calibration with Nb RRR =300.

Thermal Calculation

In fact, the temperature shift problem was due to the design of the sample holder and the temperature sensor type (Cernox AA) while the temperature instability was due to the coil support cooling.

Sample holder: To confirm the temperature shift hypothesis, a thermal simulation in transient state has been performed with Solidworks. Materials properties, such as thermal conduction and specific heat, were implemented in the solver. The copper sample holder with a RRR=30 and the supports are in 304L stainless steel.

To reproduce the conditions of the experiment, the initial temperature of the assembly is 4.2 K and the basis of the 304L supports temperature is fixed at 4.2 K because they are connected to the liquid helium bath. We assume that the contacts between the different pieces are perfect and there is no convection since everything is placed in a vacuum box. We can see that after 180 s of 0.2 W constant heating; there is a 3 K temperature difference between the top of the sample holder and the temperature sensor emplacement (Fig. 6). Furthermore, the temperature sensor type, Cernox AA, have a relatively high response time due to its mass (about 390 mg) so the temperature measurement of the sample was not accurate enough.

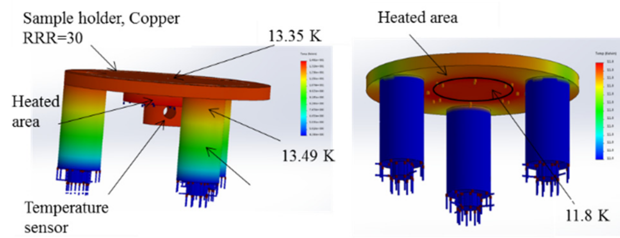

Figure 6: heat repartition for two different sample holder designs at 0.05 W, 180 s, transient state calculation.

In order to resolve the temperature shift problem, a new sample holder has been constructed. It's a simple circular copper plate screwed to the 304 L stainless steel supports. A new simulation in transient state has been performed with the same conditions. Since the heater is directly placed below the sample holder, the temperature is more uniform. Furthermore, the temperature sensor is replaced by a Cernox-SD, which is much smaller than a Cernox-AA, and have a better response time.

A further model is under study to accommodate larger samples. Indeed, at very high field the "infinite plane" approximation does not apply anymore and one start to observe two transition signal (the new one probably originating from the edges of the sample). One way to overcome this problem is to use larger samples and/or reduce the coil size (see also below).

Coil support: On the other hand, the coil support is a 3 mm thick copper plate cooled with a cooper braid (150 mm long and 20 mm² and a RRR about 80) tightened with a brass countersunk head screw(Fig. 7). If the contacts support/braid and braid/copper cold point, were perfects, this braid could evacuate 80 mW (approximately the coil dissipation for a 1 A supply) with a temperature difference of 1.2 K at 4.2 K but since it's tightened with a countersunk head screw, the contact is really bad and the coil overheated and created temperature instability in the whole set-up.

Finally, new copper braids have been installed on the coil support. Their total cross section is about 42 mm² for 10 cm long and they are tightened with stainless steel CHC screws on three different points of the coil support. Since the thermal contact is much better and the braid can evacuate 250 mW, with a temperature difference of 1.2 K at 4.2 K, the temperature instability is solved as long as the current in the coil does not exceed 1 A corresponding to a 100 mT field.

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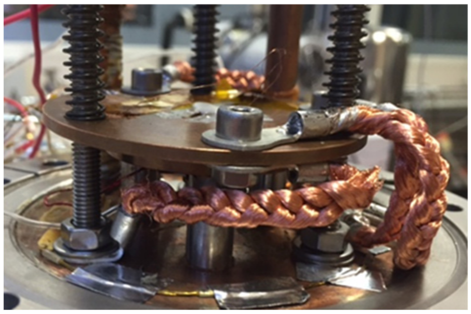


Figure 7: Copper braids used to increase the heat transfer between the coil holder and the cold point.

As a matter of fact, the contacts braids/support and braids/copper cold point are still not good enough to reach the desired magnetic field (about 150-200 mT). A new version of this measurement facility is under construction. The coil cooling is enhanced by increasing the total cross section of the copper braids, by improving their contacts with the cold point and the coil support and finally improving the cooling power spread.

Magnetic head: In order to get higher field with less dissipation we designed an inductor that allows to concentrate the field line in a more restrained area (Fig. 8) . This way we can not only reach higher field for the same level of dissipation, but we reduce the surface exposed to magnetic field and thus do not need to use too large samples.

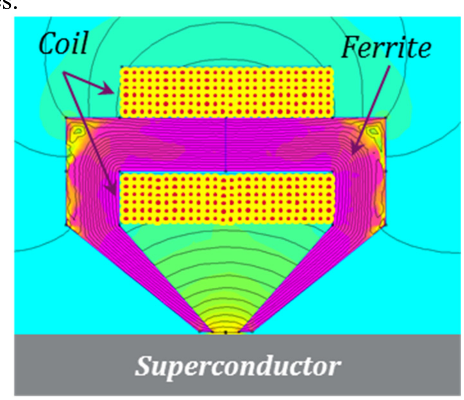
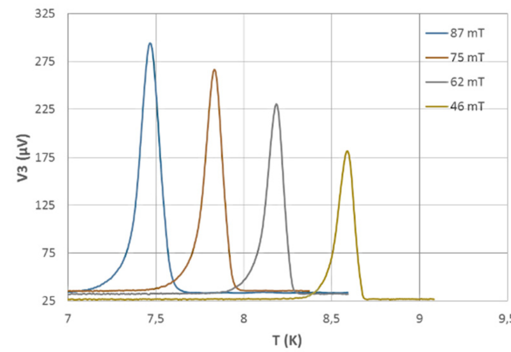


Figure 8: With a gapped core inductor made from ferrite, the field can be enhanced for the same intensity of current.

We are currently testing ferrites for cryogenic use and will implement the inductor along with the new thermic design in the new system under construction.

With our actual set-up, we are able now to measure our samples correctly and have acceptable results with very clear transitions (Fig.9).



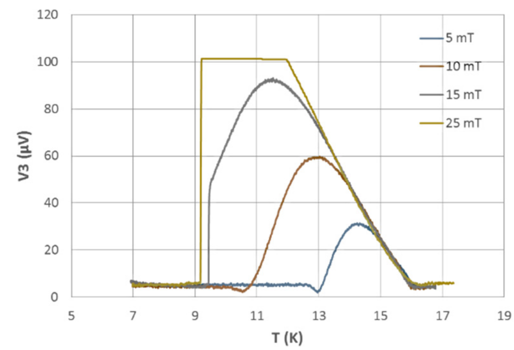


Figure 9: example of series of measured transitions; top: monocrystalline Nb after a light mechanical chemical polish (same results as the bulk Nb after etching), down: multilayer Nb/MgO/NbN (NbN ~ 200 nm). See reference [6] for complete experimental results on Nb/MgO/NbN tri-layers.

CONCLUSION

The new local magnetometer facility was built at Saclay. This set-up has proven to be very useful to measure the penetration field of samples in field configuration close to cavity operation conditions. We are able to measure our samples correctly and have acceptable results with very clear transitions. Present design successful to reach fields higher than 150 mT, but thermal instabilities starting at ~100 mT.

This local magnetometer is appropriate for thin films as well as bulk samples. In the future, we hope to increase the applied magnetic field on the sample up to 250 mT by replacement the coil by a ferrite core inductor (Figure 8) and working on a novel thermal design of the experimental setup, in order to be able to measure more realistic samples, deposited onto bulk Nb.

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