Improvement of the superconducting transition lab experiment (LFM)

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Abstract: The object of this work was to improve the experimental procedure of the superconducting transition lab experiment in the "Modern Physics Laboratory" course. A new method of data acquisition has been implemented. In addition, a GPIB connector has been used to communicate the multimeters with a computer, using *Python* programming, allowing the direct computerized registering of data, instead of manual acquisition. A GUI application has been created in order to ease the control of the experiment and the performance of the student. New measurements have been made and a new method for data analysis is proposed. A comparison with the results obtained with the old acquisition setting is presented, showing an actual improvement of the experiment.

I. INTRODUCTION

The "Modern Physics Laboratory" course (LFM) consists in the study and realization of experiments in the fields of solid state, nuclear, atomic and quantum physics. The course has 14 laboratory sessions in each of which an experiment is done by students within 3 hours. High-temperature superconductivity is studied in one of the experiments, a phenomenon referred to superconductors with critical (or transition) temperatures above the boiling point of liquid nitrogen. The main goal of this lab experiment is to observe a superconducting transition and analyse the critical behaviour nearby. For that purpose, the resistance of a superconductor is measured as a function of temperature. A sample of YBaCuO is used, with a critical temperature around 90 K. The improvement of this lab experiment is the goal of this work and a new experimental procedure is implemented. In this introduction, the old procedure is exposed.

The sample is cooled from room temperature down to 80 K. For a matter of time, actual measurements are only taken between 180 and 80 K. A cryostat is used in order to achieve these low temperatures. A vacuum chamber isolates the internal components, including the sample, from the exterior. Isolation reduces the heat excess due to convection and conduction. It is important to remark that this heat excess limits the temperature at which the sample can be cooled, so good vacuum conditions are fundamental. Inside the cryostat, liquid nitrogen flows through a heat exchanger cooling the sample by radiation and convection. This flow is controlled by the student using a valve.

Two platinum resistances are used as thermometers. One of them is attached to the sample and the other to the heat exchanger. In general, these temperatures are not equal because the sample and the exchanger are not in thermal contact. Changing the opening of the valve and observing the difference between these temperatures gives the student a rough control of the cooling rate during the acquisition. The resistance-temperature dependence and the current injected in the Pt resistances (1 mA) are given in the experiment guide.

The other variable measured in the lab experiment is the voltage drop in the sample, and therefore, its resistance. It is measured using the 4-point probe method, to avoid including important electrical noise caused by the cables and the contacts. However, the metallic contacts in the sample add a non-desired voltage drop due to the thermoelectric effect. This can be subtracted making two voltage measurements, for each temperature measured, with reverse senses of the current injected to the sample. Since the thermoelectric voltage drop does not depend on the current sense, the voltage due to the resistance of the sample, V, can be then obtained as

$$V = \frac{V_+ - V_-}{2}, \qquad (1)$$

where V_+ is the positive-current voltage and V_- is the negative-current voltage. Then, the resistance can be obtained applying Ohm's law. The value of the current injected to the sample (10 mA) is found in the experimental guide.

A high-resolution multimeter (*Keithley 2000*) is used to measure the voltage drop in the sample, which is of the order of mV. On the other side, temperature is measured with a *Hewlett-Packard 3435A* multimeter, with a resolution of 0.1 mV, which corresponds to 0.25 K.

Two cables connect the electronics of the cryostat with a control box, where the multimeters are plugged to. This box acts as an intermediary and has all the elements to control the measurements. It also contains the DC power sources that inject the current to the sample and the platinum resistances. The main elements of this box that are of our interest are basically two switches. Both the temperature of the heat exchanger and that of the sample are measured with the same multimeter, and which of them is displayed is controlled by one of these switches. The other one reverses the sense of the current in the sample. The opening of the valve in the cryostat and the switches in the control box are the only manipulations done by hand by the students in order to perform the experiment.

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II. A NEW CONCEPT

There are different reasons that motivate seeking an improvement for this lab experiment. To begin with, temperature is not directly measured but actually the voltage drop in the platinum resistances. With the old acquisition setting, the conversion was done a posteriori, so the control of the temperature during the measurements was done quite blindly. In addition, the low resolution of the *Hewlett-Packard 3435A* multimeter limited the minimum step between measurements to 0.25 K. Therefore, the amount of data that could be taken was not enough for a high-significance analysis. An important lack is evident in the transition region (see crosses in *FIG.1*). The main analysis of data in the lab experiment is done within this region, so increasing data retrieval would ease the treatment and better justify the subsequent results obtained. Data analysis is discussed further in *Section IV*.

Rather than modifying the experimental setup, the new design affects the acquisition setting. However, the *HP 3435A* multimeter was changed to one with better resolution (*HP 34401A*), solving the limitation in the temperature step between subsequent measurement. The objective of the new acquisition method is to obtain more data in the same amount of time used in the old lab experiment, a 3-hour session. For that purpose, a computerized registering was implemented, improving the quickness at which measurements can be made. This also enables to convert automatically the voltages of the platinum resistances to temperature during the experiment, so that the value of the temperature can be displayed in the computer in real time. This actually increases the performance of the student, who can now control the experimental variables in a better way.

In addition to a computerized registering, a new method of acquisition was implemented, doubling the data that could be taken before. With the old method, for each temperature value, T_i, two measurements of the sample voltage were made, V_{i+} and V_{i-}, with opposite current senses. Two voltages were thus needed to have each data point. There is a problem with this method. In the time taken between the temperature measurement and that of the second voltage, the actual temperature could have changed, so this voltage was assigned to a temperature with which did not exactly correspond. The new acquisition setting solves this problem and increases simultaneously the amount of data points that can be obtained. The main idea behind is to measure a single temperature and voltage each time, (T_j, V_j). In other words, each time a voltage is measured, temperature is measured too. Besides, the computerized registering allows quasi-simultaneous temperature and voltage measurements. The new acquisition procedure consists in switching the current sense between subsequent measurements. Now each data point is constructed between two consecutive measurements. Instead of using equation (1) to obtain the resistive voltage drop, we assign the following temperature

$$T = \frac{T_j + T_{j+1}}{2} \qquad (2)$$

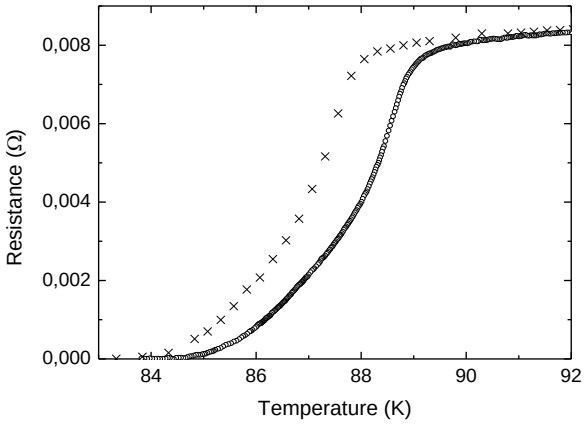

FIG. 1: Comparison between the data taken around the transition with the old acquisition setting (crosses) and the new one (circles).

to the resistive voltage drop

$$V = \begin{cases} \dfrac{V_j - V_{j+1}}{2} & ; \quad V_j > V_{j+1} \\ \dfrac{-V_j + V_{j+1}}{2} & ; \quad V_j < V_{j+1} \end{cases}, \qquad (3)$$

where (T_j, V_j) and (T_{j+1}, V_{j+1}) are two consecutive measures made with opposite current sense. Using this new concept, each voltage recorded gives rise to a data point, and this doubles the data points obtained with the old method. This way of acquiring the data leads to a better discretization.

It is worth noting that the improvement was made in the context of a laboratory experiment, with its constraints. All of what we thought either of physical interest or experimentally important for the student was kept unchanged. For instance, a lock-in or other AC measure techniques could have been implemented in order to avoid the thermoelectric contribution in the sample voltage, but that would have led us to an excessively automatized lab experiment. Moreover, we think that being aware of the physical existence of this contribution is of interest for the student.

III. METHODS

A. Maintenance of the hardware

Maintenance of the equipment was made in order to ameliorate the experimental conditions and to repair or substitute the components that presented imperfections. All upkeep was made in February, between academic semesters, when the experiment was not used. Everything that worked properly, such as the sample and the control box, was kept untouched. However, since the sample was left outside the cryostat during reparations, it was accidentally oxidised. This affects the critical temperature of YBaCuO, which depends, among other factors, on its oxygen content. Therefore, a change in this temperature would be expected between old and new measurements because of this oxidation. This difference is actually appreciated in *FIG.1*.

Inside the cryostat, the superinsulator surrounding the liquid nitrogen reservoir was found wet with oil. This was a consequence of a malfunction in the pump used to make the vacuum in the insulation chamber. The superinsulator is used to isolate its internal components from the external thermal radiation. Although superinsulation can be omitted in liquid nitrogen cryostats [1], a low-emissivity aluminised film was added in the vacuum chamber as replacement for the previous superinsulator.

All the maintenance that was done is listed below:

- The cables connecting the cryostat to the control box were changed and contacts were soldered again.
- Vacuum leakage detection in the cryostat was performed using a helium mass spectrometer. Small leakages in the sample chamber were covered with varnish and important ones were solved using vacuum solder.
- The vacuum pump was sent to repair.
- The superinsulator in the cryostat vacuum chamber was replaced with a new one.

B. GPIB communication

In order to implement the computerized register, we needed a way to send data from the multimeters to the computer. To this end, a General-Purpose Interface Bus (GPIB) controller was used. GPIB Instrument Control is a communication channel which can be used to transfer data between scientific instruments and data-registering devices. A *Prologix GPIB-USB Controller* [2] was used in this work. This specific connector is provided with internal commands that facilitate its usage.

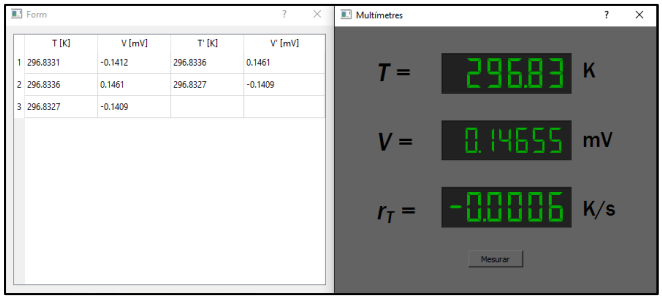

FIG. 2: Screenshot of the data acquisition graphic application developed for the lab experiment

A program for acquiring data from the multimeters was developed. Any programming environment with access to serial ports could have been used to develop custom applications involving the *Prologix GPIB-USB Controller*. However, for this work *Python* [3] was the preferred programming language. For accessing serial ports, *Python*'s *pyserial* package [4] was used. Data from one multimeter is obtained with the following process:

(1) The computer's communication serial port ('COM' port) is opened using *pyserial* commands.
(2) The desired multimeter is addressed using the GPIB controller's commands. Each multimeter has an assigned GPIB address that can be changed using the menus from their front panel.
(3) Multimeters are configured and measurements are made by sending specific multimeters commands, found in the corresponding manuals [5, 6].

C. Creation of a software

Keeping in mind that this lab experiment has to be done by students, a graphic application was created in order to ease the performance. This application is a graphical extension of the program for data acquisition. There are several packages available for Graphical User Interface (GUI) development using *Python*. Although *Tkinter* is the preset package provided, another package (*PySide*) was used [7, 8]. *PySide* is better-looking than *Tkinter* and also comes with a GUI builder tool to design applications, *Qt Designer*.

The application consists of three windows. When it is started, a *Measurement Parameters* dialog (or window) is opened. In this dialog, the student introduces the values of the temperature-resistance conversion parameters and the currents injected both to the sample and the platinum resistances. These parameters are needed for the background calculations done by the program. Having introduced all the parameters, the *Data Table* and *Multimeters* windows appear.

The *Multimeters* window contains the displays of three virtual multimeters and a *Measure* button. The upper display (T) gives the temperature (expressed in K), calculated in real time with the data of the real multimeter and the parameters introduced before. The middle one (V) displays the voltage drop in the sample (expressed in mV), measured directly with the *Keithley* digital multimeter. The last display (r_T) gives the cooling rate (expressed in K/s), calculated as the difference of two current consecutive temperature values divided by the time step between them. A measurement is made every 1 second. The *Measure* button is used to register the last pair displayed, (T_j, V_j), to a '.txt' data file. Finally, the *Data Table* window contains a table with all the registered measurements. The table is set in a way in which each row corresponds to a data point. Therefore, during the analysis of data, the student has to apply equations (2) and (3) to each row in order to obtain a data point. Each resulting voltage is converted to resistance using Ohm's law. This window is useful to control the voltage sign of the last register, and therefore set the correct current sense for the next registered measurement.

IV. MEASUREMENTS AND NEW ANALYSIS

All measurements were made cooling down the sample within 3 hours. If the experiment was performed warming up the sample from the lowest temperature attained by the cryostat (liquid nitrogen), temperature changed too fast to take high-resolution measurements. The low precision in the opening of the valve made difficult to control the slowness of the cooling rate during the transition. Therefore, we had to be careful not to slow down excessively the measurements to

avoid undesired warming of the sample and, hence, the appearance of thermal hysteresis.

Data obtained with the new setting are shown in *FIG.3*. The improvement does not only affect data retrieval in the transition but now measurements can begin at room temperature, without time loss (measurements with the old setting started at 180 K to avoid excessive time consumption).

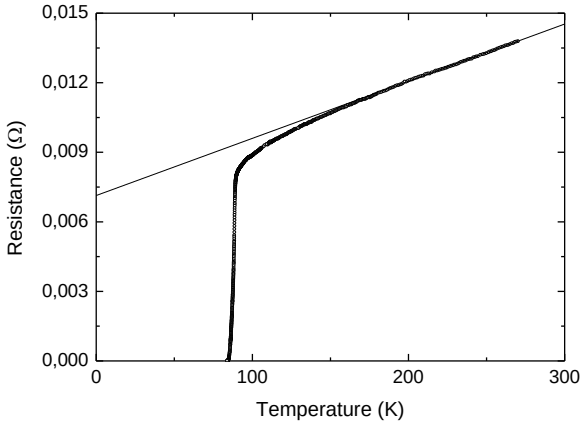

FIG. 3: Resistance of the YBaCuO sample as a function of temperature, obtained with the new acquisition setting.

Further in the lab experiment, with the results obtained in the laboratory session, the student has to determine the value of the critical temperature, T_C, and analyse the critical behaviour near the transition. The inflection point of the resistance profile $R(T)$ can be used as a first estimate of the critical temperature. However, a better evaluation is found from the critical analysis. A model for high-temperature superconducting transitions is needed in order to define a critical temperature. Depending on the model used, a different value for this temperature is obtained. In this lab experiment, a dynamic scaling model based in Aslamazov-Larkin (AL) theory is used [9, 10, 11]. This analysis is based on the fluctuations of the excess conductivity, $\Delta\sigma$, which acts as the order parameter of the phase transition. According to AL theory, the critical behaviour of the excess conductivity near the transition can be written as

$$\Delta\sigma = At^{-\varepsilon} , \qquad (4)$$

where A is a temperature independent constant, ε is the critical exponent and $t = (T - T_C)/T_C$ is the reduced temperature. The excess conductivity can be obtained using the data from the measurements, as will be explained latter. Then, the critical temperature can be determined by imposing the value of the critical exponent.

The Ginzburg-Landau (GL) theory of phase transitions predicts a mean-field exponent $\varepsilon = 1/2$ far from the transition. However, GL theory does not account for the order parameter fluctuations. Including these fluctuations, the mean-field exponent is lowered and, in addition, two critical regions appear closer to T_C, as predicted by Lobb [12]. A full critical dynamic region, with $\varepsilon = 1/3$, is found for reduced temperatures $t < 10^{-5}$. A crossover region, with $\varepsilon = 2/3$, separates the mean-field and the full critical region.

In practice, the full critical region is difficult to be accessed and, in addition, corrections may be made to account for other contributions in this region. Moreover, anisotropy and inhomogeneities in the sample may affect the mean-field region and fluctuations can lower its critical exponent, diverging from $\varepsilon = 1/2$. Therefore, the crossover region is used instead to determine T_C [9, 10, 11].

Returning to the analysis, first a linear fitting of the so-called background resistance has to be made in the metallic region, where the sample behaves like a normal conductor. A proper estimate of the background resistance is needed, since it affects the subsequent analysis. With the new acquisition setting the linear fit can be made for temperatures $T \geq 2T_C$, where the linear behaviour is better accomplished. The normalised excess conductivity can be then obtained as

$$\frac{\Delta\sigma}{\sigma_0} = R_0\left(\frac{1}{R} - \frac{1}{R_B}\right). \qquad (5)$$

Here R is the measured resistance and R_B is the background resistance the sample would have if it would stay metallic at any temperature and is calculated by extrapolating the linear fit performed in the metallic region. R_0 and σ_0 are, respectively, the resistance and conductivity at 300 K calculated with the background fit.

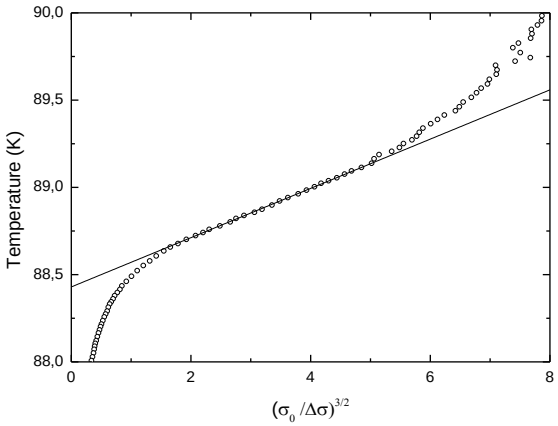

FIG. 4: Plot used to obtain the critical temperature, by linear fitting the data in the crossover region. Critical temperature is the ordinate-intersect of this fit, found in this case to be $T_C = 88.429 \pm 0.004$ K.

With the old analytic procedure, the critical temperature was defined as the T_C value for which the plot of $\ln(\Delta\sigma)$ versus $\ln(t)$ had the best linear behaviour near the transition. This method does not account for the different critical regions, only the mean-field one, and induced to error if further regions were actually accessed. A new method is proposed, using the crossover critical exponent. From equation (4), imposing $\varepsilon = 2/3$, we can obtain

$$T = (T_C \cdot A'^{3/2})\left(\frac{\sigma_0}{\Delta\sigma}\right)^{3/2} + T_C , \qquad (6)$$

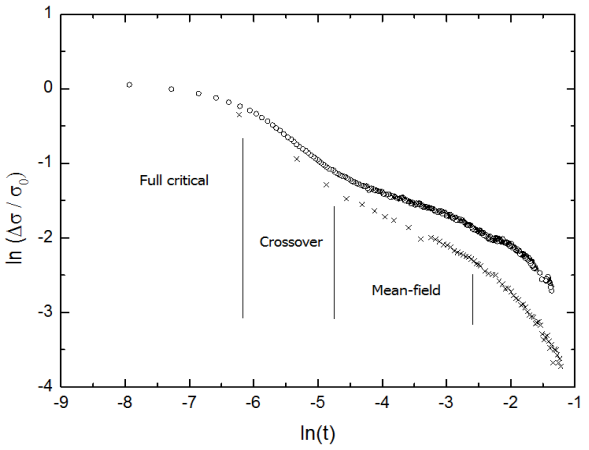

FIG. 5: Log-log plot of the normalised excess conductivity as a function of the reduced temperature. Circles and crosses correspond, respectively, to data obtained with the new and the old setting. The critical temperature value obtained with the new analysis method for each data set has been respectively used. The different critical regions are shown.

where we have replaced $\Delta\sigma$ with the normalised excess conductivity. Therefore, the critical temperature can be obtained by fitting equation (6) in a T versus $(\sigma_0/\Delta\sigma)^{3/2}$ plot. This method is shown in *FIG.4* using the data from the new measurements. The adjustment *R*-square of the linear fit in this figure is 0.9989, and the critical temperature was found to be $T_C = 88.429 \pm 0.004$ K. Finally, we can make a plot of $\ln(\Delta\sigma)$ against $\ln(t)$ using the crossover critical temperature found in *FIG.4* in order to see qualitatively the different critical regions explained before (*FIG.5*). For comparison, the results found with the old setting are also plotted in this figure, using the respective critical temperature as obtained with the new analysis method. As it can be seen, with the old setting we could only measure four data points in the crossover region. With the new acquisition setting, even a few points entering the full critical region can be accessed.

V. CONCLUSIONS

We have improved the superconducting transition lab experiment of the LFM course with the introduction of a new acquisition method that allows obtaining a higher amount of data than it could be achieved with the old one. A GPIB controller has been used in order to implement a computerized data registering via *Python* programming. Since the lab experiment is performed by students, a graphic application has been created using *Python* for the purpose of easing the control of the experiment and the data acquisition. All the maintenance has also improved the experimental conditions, as for example the state of the vacuum pump, the quality of the vacuum in the cryostat and the whole wiring. Moreover, the new analysis proposed enables a better way to determine the critical temperature with precision and the new setting allows accessing all the critical regions in the temperature dependence of the excess conductivity. The new acquisition method developed in this work is ready to be implemented in the next semester.

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