Automatic system for the direct and continuous measurement of the irreversibility line of high T_c superconductors

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A system for the direct and continuous determination of the irreversibility line in high T_c superconductors is described. The system, based on a standard ac susceptometer, operates by applying a controlled dc field to the sample that maximizes the imaginary (out-of-phase) component of the ac susceptibility. This control is achieved using a proportional-integral-derivative loop with the reference set to zero and with the magnetic field derivative of the imaginary component of the ac susceptibility taken as the feedback sample (error signal). To obtain a dc voltage proportional to this derivative a double lock-in detection scheme is used. This apparatus is able to carry out a continuous measurement of this line by sweeping the temperature in the range of interest while the sample is maintained, by the controller, at the irreversibility magnetic field at each temperature. Hence, the whole process is accomplished in the same time as a typical resistivity-versus-temperature measurement, which is faster than other methods. © *1998 American Institute of Physics.* [S0034-6748(98)02408-3]

I. INTRODUCTION

In the H-T plane of a type II superconductor three main regions can be found: Meissner, mixed, and normal,[1,2] delimited by the lower (H_{c1}) and upper (H_{c2}) critical field lines, as the field increase at any fixed temperature. The Meissner state is characterized by a perfect diamagnetic behavior, while in the mixed state, normal and superconductive regions coexist.[3]

From a practical point of view one important parameter in superconductors is the critical current density (J_c), the highest current that can be applied to the sample without dissipation. The mixed state can also be separated into two regions by a line called ''irreversibility line''[4,5] below which J_c has a finite value and it turns to zero by crossing this boundary.[6] The existence of this line reduces the range of application of high temperature superconductors. This justifies the experimental determination of this line and the necessity of its physical understanding.

In order to obtain the irreversibility line in superconductors several techniques have been used,[7–12] a common approach among them is the study of the magnetic properties of the material under the external excitation of a magnetic field either constant or periodic in time. The studies that use a constant (dc) field can be further divided into two types: (1) The first one is based on the determination of the temperature at which the magnetization-versus-temperature curve of a sample, cooled under zero magnetic field, splits from the same curve obtained when the sample is cooled under nonzero field.[7] This procedure has to be repeated at different field values and the curve constructed point by point. (2) The second dc technique requires the measurement of the field at which the hysteresis loop closes at different temperatures.[8]

Another set of techniques widely used to obtain the irreversibility line are based on ac susceptibility measurements. A large constant magnetic field is modulated by a relatively low level periodic field. One criterion is to find the value of the magnetic field that maximizes the imaginary component of the susceptibility (χ'')[9–11] at different temperatures. Another criterion which has been used is the onset of the third harmonic in the response of the superconductor to the ac signal.[12]

Thermal activation plays an important role in the magnetic properties of high T_c superconductors;[13] hence, the critical current and the irreversibility line depend on the characteristic time scale of the measurement technique. For instance, the position of the dissipation peak in the ac susceptibility is modified by the measurement frequency.[14] Therefore, irreversibility lines obtained by using different techniques show measurable differences.

Although these methods differ in several ways, they all have one thine in common: they take a long time and several thermal and magnetic cycles to obtain an irreversibility curve with few points. In this work we present a technique that allows, through the use of a proportional-integral-derivative (PID) controller,[15] the determination of the irreversibility

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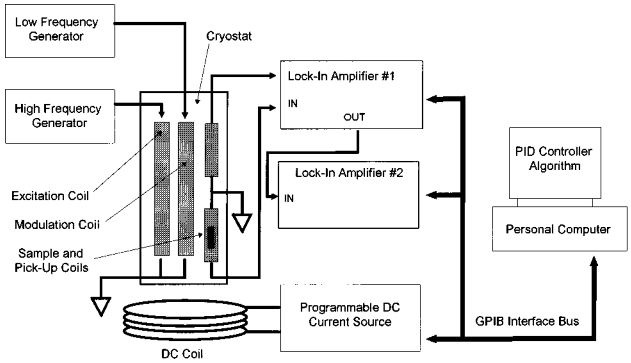

FIG. 1. Schematic diagram of the experimental setup. The excitation, modulation, and pick-up coils are wounded coaxially inside the cryostat. The dc coil is immersed in liquid nitrogen surrounding the cryostat.

line continuously and fast. Our method needs only one thermal cycle, is with an automatic adjustment of the dc field.

The system is based on a standard ac susceptometer,[16] and works by controlling the applied dc field to adjust it to the value that provokes a maximum in the out-of-phase component of the ac susceptibility. This is done by superimposing a low frequency field on the ac field commonly used in the ac susceptibility measurements, which modulates the ac susceptibility signal and allows (using a double lock-in detection scheme) the measurement of the magnetic field derivative of the out-of-phase component of the susceptibility as a dc voltage. This voltage is used as an error signal fed into a PID controller, whose output drives a programmable current source used to apply the dc magnetic field. As the controller tends to null the error signal at its input, the loop will lock in a state where the applied field matches the irreversibility one, i.e., the field for which the out-of-phase component of the susceptibility is a maximum (and its derivative, the error signal, equals zero). This system allows the measurement of the irreversibility line by sweeping only the temperature in the range of interest, while the controller adjusts the irreversibility field at each temperature.

II. SYSTEM DESCRIPTION

Figure 1 shows a general block diagram of the system described in this work; it is based on a standard three-coil ac susceptometer setup, where a fourth coil is added. The four coils are coaxially placed inside a cryostat, immersed in liquid nitrogen, with temperature regulation provided by a Lake Shore 330 temperature controller. The pick-up coils are would in opposite directions to null the signal induced when no sample is placed inside them. One of the outer coils is used to generate the ac excitation field (about 0.1 mT at 1 kHz) from which the ac susceptibility is obtained. The other one is used to generate a low frequency field (about 0.2 mT at 10 Hz) for modulation purposes, as explained below. Another coil placed outside the cryostat is used to generate dc fields up to 0.1 T (1000 Oe). The signal of the pick-up coils is differentially injected to the first lock-in amplifier with the phase adjusted for the measurement of the out-of-phase component of the susceptibility.

Although the low frequency field applied to the sample generates an ac susceptibility signal at its frequency, it also

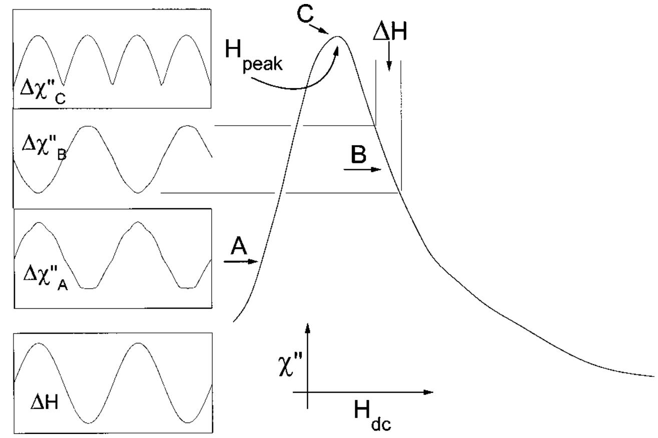

FIG. 2. Illustration of the working principle for the determination of the peak position (H_{peak}), through the double lock-in detection technique. The curve is a typical measurement of χ'' vs applied magnetic field. Three different regions (A, B, and C), and the modulation produced by the low frequency ac field (ΔH) in each one of them, are observed in the graphs vs time.

can be understood in terms of a small modulation (ΔH) of the dc applied field (H_{dc}). Figure 2 shows the effect of such modulation in a χ'' versus H_{dc} curve; where three regions are observed: $H_{\mathrm{dc}}<H_{\mathrm{peak}}$, $H_{\mathrm{dc}}=H_{\mathrm{peak}}$, and $H_{\mathrm{dc}}>H_{\mathrm{peak}}$. In each case the ac low frequency field provokes some modulation of the χ'' signal. For $H_{\mathrm{dc}}<H_{\mathrm{peak}}$ (case A) the χ'' modulation is in phase with the field oscillation, for $H_{\mathrm{dc}}>H_{\mathrm{peak}}$ (case B) the χ'' modulation is dephased 180°, and when $H_{\mathrm{dc}}=H_{\mathrm{peak}}$ (case C) the signal at the modulation frequency disappears, and a signal at twice this frequency is obtained.

It is obvious that if we synchronously rectify this signal with the second lock-in amplifier (using as a reference a voltage proportional to the low frequency ac field) at this lock-in output there will be a dc voltage: positive for $H_{\mathrm{dc}}<H_{\mathrm{peak}}$, negative for $H_{\mathrm{dc}}>H_{\mathrm{peak}}$, and zero in the peak position, and, in general (for a fixed amplitude of the modulation signal), proportional to the magnetic field derivative of χ''. If we use this voltage as an error signal in a PID controller that adjust the current applied to the dc solenoid, and with the proper settings of the PID and circuit parameters, we obtain a control loop that will try to adjust the dc field to guarantee that this error signal equals zero.

In our system, a C++ program running in a personal computer (PC) performs the PID control action. The output of the second lock-in amplifier is read by the computer through the general purpose interface bus (GPIB). The first lock-in amplifier, the current source, and the temperature controller are also connected in this bus. The computer handles all the measurement processes: it adjusts the phase settings of both lock-in amplifiers, sweeps the temperature in the range of interest, and controls the dc magnetic field through the PID controller program. The algorithm implemented dynamically adjusts the PID coefficients to improve the controller response,[15] but maintaining it in an overdamped mode to avoid field oscillations around the peak.

The whole system works as follows: the temperature is stabilized for a while at the highest value desired, then the PID controller starts to adjust the dc field until the peak

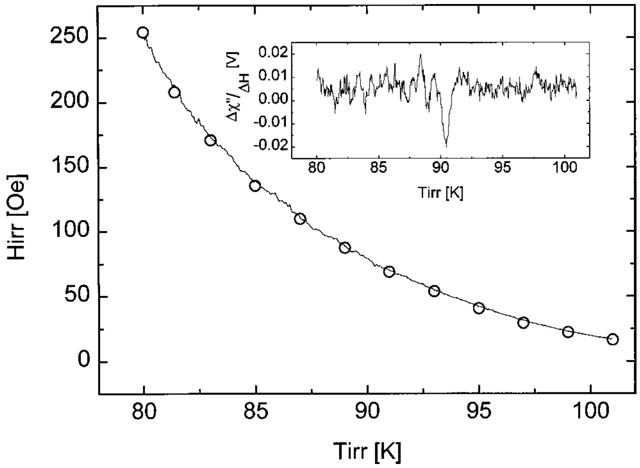

FIG. 3. Intergranular irreversibility line for a BSCCO ceramic sample measured using the system described in this work (solid line), and determined from the processing of several χ'' vs applied field curves (circles) in a point-by-point method.

position at that temperature is reached. At this point the temperature is swept down, at a fixed rate, while the controller follows the variations of the peak position. This process ensures that the magnetic field will be continuously increasing, avoiding hysteretic phenomena in the measurement. As a result we will obtain a curve $H_{irr}(T)$, for the criterion used.

III. RESULTS AND DISCUSSION

In order to test our system, intergranular irreversibility lines of several BSCCO ceramic samples were measured. The result for one sample (Fig. 3, line) shows excellent agreement with the curve measured point-by-point (circles), i.e., the one obtained by measuring several χ'' (H_{dc}) curves at different temperatures. In the inset of the figure we can see the voltage at the second lock-in output, i.e., the error signal at the PID input. This voltage gives a criterion of the controller performance. Its value must be as close to zero as possible and, more importantly, it must remain as constant as possible during the measurement, i.e., it cannot drift away from its initial value. The stability of this voltage is good and its deviation from zero is small and constant enough to ensure that the peak position was maintained during the whole measurement. One important advantage is the fact that the obtained curve has a continuous appearance due to the amount of experimental points composing it (typically 500). This facilitates mathematical processing of the experimental data.

This method could be useful not only for these polycrystalline materials, but for the determination of irreversibility lines of samples that exhibit loss peaks in the out-of-phase component of the susceptibility, such as single crystals or thin films. Also this technique could be easily extended to other criteria for the determination of the irreversibility regime, such the onset of the peak, onset of the third harmonic, etc.

These measurements were made at temperature sweeping rates up to 3 K/min, and no major differences were found between the curves obtained (we only show the one for 3 K/min). It is important to stress that this limit is imposed by the cryogenic system and not by the controller. For the fastest temperature sweeps attainable, the controller performance was excellent.

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