

Loss of interplane correlation in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ single crystals

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By means of dc magnetization and the ac response of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ single crystals it is shown that at the dc irreversibility line the vortex system has no long-range order in the c direction. We find an energy dissipation peak at 7 Hz for interplane current that takes place at a temperature well below the irreversibility line. In this sense, the irreversibility line marks the temperature where quasi-two dimensional vortices are depinned. The experimental data clearly show the different nature of two dissipation peaks in the susceptibility: one related to the interplane currents and the other associated with the intraplane ones.

I. INTRODUCTION

The existence of a thermodynamically reversible¹ region in the H - T phase diagram is a remarkable characteristic of high-temperature superconductors. The origin of that region as well as the nature of the irreversibility line, separating the reversible from the nonreversible magnetic response, has been the subject of controversy and intensive theoretical and experimental investigation.

In a series of recent papers we have shown that the transition towards the reversibility region in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ (BSCCO) single crystals is made in two steps, as detected by mechanical oscillator experiments² and ac susceptibility³ and dc magnetization measurements.⁴ From those results we concluded^{3,4} that the irreversibility line should be associated with currents flowing in the Cu-O planes. Thus, while below that line the currents flowing in the Cu-O planes can be propagated essentially without dissipation, the superconducting currents in the perpendicular direction (c crystallographic direction) can only be established at lower fields and temperatures.

There are two well-determined characteristic temperatures: one, $T_M(H)$, determined by an ac dissipation peak related to currents flowing perpendicular to the Cu-O planes and the other, $T_I(H)$, at higher temperatures associated with the ac dissipation peak of currents flowing within them (see Fig. 1).³ These two lines in the H - T phase diagram are found to be frequency dependent. It was also pointed out⁴ that the dc zero-field-cooled (ZFC) flux exclusion measurements in these high-quality single crystals show two features that can be correlated to the ac $T_M(H)$ and $T_I(H)$.

A remarkable feature of the previous interpretation is that the relevant transition in the vortex system would take place at $T_M(H)$, where the long-range order in the c direction is established and the critical current is found⁴

to be strongly enhanced. If this suggestion is correct, the magnetic response at higher temperatures should be determined by the behavior of quasi-two-dimensional vortices, including the transition to the reversible state at $T_I(H)$.

The ac susceptibility experiments made by other researchers^{5,6} have been designed using an ac field configuration where the results are strongly determined by the currents flowing in the Cu-O planes. Up to $T_I(H)$ the shielding capability of those currents is strong enough to preclude the observation of the decoupling transition at $T_M(H)$.

Since the experimental results support the picture described before, we consider it important to study the almost unexplored region of the phase diagram between $T_M(H)$ and $T_I(H)$. We present data of the magnetic response to ac magnetic fields perpendicular to c , at frequencies as low as 7 Hz, showing that the dissipation peak at $T_M(H)$ is more than 10 K below the dc irreversibility line. The ac susceptibility data make evident that energy dissipation starts at a temperature well below the irreversibility line, at the point where dc measurements⁴ indicate that the critical current is strongly reduced. However, ac measurements which induce currents only in the Cu-O planes are found to be ineffective for detecting dissipation in the interesting temperature region below $T_I(H)$.

The ac susceptibility measurements for different dc fields,³ applied in the c direction, have been extended to a lower-field range. The results show that the nature of the peak at $T_I(H)$ does not change over the whole field range. On the other hand, the behavior of the ac response in the configuration $\mathbf{h}_{ac} \perp c$ is strongly modified for fields lower than 500 Oe, indicating a possible transition towards a three-dimensional vortex regime over the whole range of temperatures. The data show that in the low-field regime the dissipation of the ac currents flow-

ing in the c direction sets in at a frequency-independent temperature coincident with that of the dc irreversibility line. On the contrary, the dissipation of the currents flowing in the Cu-O planes is detected by a well-defined frequency-dependent peak at higher temperatures than that of the dc irreversibility line.

The results presented in this paper are particularly relevant since most explanations^{7,8} of the magnetization of BSCCO are based on the influence of thermal fluctuations in the response of three-dimensional vortices. In particular, the theoretical results of Bulaevskii, Leivij, and Kogan⁸ predict that the three-dimensional vortex lattice in thermodynamic equilibrium loses the long-range coherence in the Cu-O planes at much lower temperatures than that where the vortices make the transition to a two-dimensional behavior. This theoretical result has been used to interpret the magnetization at temperatures and fields where, according to our interpretation of the experimental data,³ the vortex system is quasi-two-dimensional.⁹ Although the dynamic response of the vortex system is not treated in that theoretical work, our experimental results seem to contradict the picture provided by that model.

II. EXPERIMENT

The ac susceptibility data presented in this paper were obtained by means of two complementary techniques. The measurements at frequencies greater than 30 Hz were made using a conventional mutual inductance technique. In these measurements two different ac field configurations were employed: $\mathbf{h}_{ac} \perp c$ and $\mathbf{h}_{ac} \parallel c$. Further experimental details can be found in Ref. 3. For frequencies lower than 30 Hz the measurements were carried out using a superconducting quantum interference device (SQUID) magnetometer as a preamplifier. In these experiments the sample was thermally connected to the cold finger of a custom-built cryostat by means of a sapphire pad.¹⁰ The ac excitation field was induced by a primary copper coil of 0.5 cm diameter and 2 cm length. The secondary coils were made of Nb wire wound on top of the primary in a gradiometer configuration. The sample was placed in one of the secondaries with its c axis perpendicular to the ac field. The superconducting secondary coils are part of the superconducting transformer of the SQUID magnetometer. In order to keep the detection system at constant temperature the coils were mounted on a stainless-steel vacuum jacket immersed in liquid He. The excitation field was taken from the reference signal of a two-phase lock-in amplifier (PAR 5301). The change of the sample susceptibility as a function of field and temperature induces a current in the secondary that is detected by the SQUID. The in- and out-of-phase components of this current were measured by the lock-in amplifier, connected at the output of the SQUID electronics. The proper phase of the lock-in amplifier was set measuring the sharp superconducting transition of an indium sample. The dc magnetic field was applied perpendicular to the ac field (parallel to the c axis of the crystal) by means of a homemade superconducting magnet operated in permanent mode. Superconducting and

magnetic shieldings reduced the ambient magnetic field in the cryostat down to 10 mOe and screened spurious ac external fields. Further details on the cryostat can be found in Ref. 10. All the ac susceptibility measurements have been taken in field-cooling (FC) experiments. In the context of this paper the susceptibility is expressed using SI units.

The single crystals used in these experiments were obtained and characterized as described in Ref. 11.

III. RESULTS

Figure 1(a) shows the change of magnetic flux in the BSCCO single crystal as a function of temperature in ZFC and FC experiments, as detected by the SQUID magnetometer. The external magnetic field was applied perpendicular to the Cu-O planes. The measurements were made at constant field, sweeping temperature at a rate of 1° per minute. Changes of the sweeping rate by

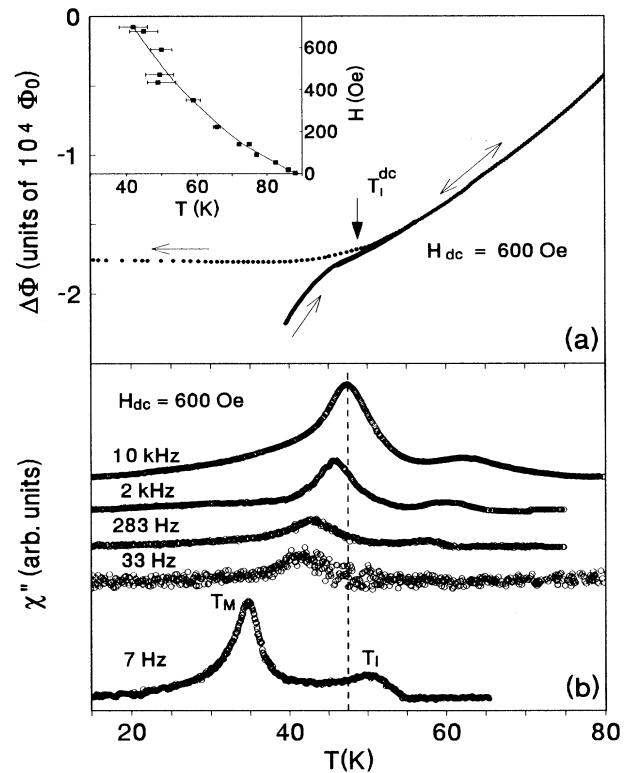


FIG. 1. (a) Magnetic flux in the sample measured by the SQUID magnetometer as a function of temperature, in the ZFC and FC experiments, for an applied field of 600 Oe. The thin arrows indicate the thermal sweep direction. The solid arrow indicates the temperature where irreversibility is first detected. In the inset the irreversibility line from dc measurements is plotted (Ref. 4). (b) In-phase voltage in the lock-in amplifier, proportional to χ'' as a function of temperature for different frequencies in the configuration $\mathbf{h}_{ac} \perp c$. In order to allow the comparison the curves have been offset. The 7 Hz curve has been taken using the SQUID as a current detector. The dashed line indicates $T_I^{dc}(600 \text{ Oe})$.

factors of 2 did not introduce significant variation in the data.

The results of Fig. 1(a) show the temperature where the irreversibility is detected. Similar data of the same single crystal were used to determine the irreversibility line reported in Ref. 4. In order to distinguish the irreversibility line as detected by dc SQUID magnetization it is denoted as $T_I^{\text{dc}}(H)$.

Figure 1(b) shows the imaginary component of the ac susceptibility χ'' in a configuration where the dc magnetic field is parallel to the c axis and the ac field perpendicular to it. In this case the dc field is 600 Oe and the ac amplitude 1 Oe for the data taken at frequencies higher than 30 Hz and 30 mOe for those at 7 Hz. Both, $T_I(H)$ and $T_M(H)$ were found independent of the ac field for amplitudes up to 1 Oe. The results of Fig. 1(b) show the shift of the frequency-dependent dissipation peaks, in the range of frequencies between 7 Hz and 10 kHz. The peak at the higher temperature defines³ the corresponding frequency-dependent $T_I(H)$, and the one at lower temperatures determines the frequency-dependent $T_M(H)$.

The temperature $T_I(H)$ coincides with that obtained from the single dissipation peak observed³ in the configuration where the ac currents are induced only in the Cu-O planes. As a consequence, the peak at $T_I(H)$ shown in Fig. 1(b) is believed to be due to some misalignment between the sample and the ac field. This assumption is supported by the experimental result showing that the intensity of the peak at $T_I(H)$ changes from experiment to experiment, probably due to small changes in the relative position of the sample within the mutual inductance. The dashed line in the figure marks $T_I^{\text{dc}}(600 \text{ Oe})$.

The data at 33 Hz show that the peak at $T_M(H)$ has been shifted to a temperature lower than $T_I^{\text{dc}}(H)$. On the other hand, the reduction of the signal-to-noise ratio at that frequency does not allow the determination of the corresponding $T_I(H)$. The use of the SQUID electronics as an ac current detector in the mutual inductance measurements proved to be a quite powerful technique in the low-frequency range. The results using this technique at 7 Hz are plotted also in Fig. 1(b). It is evident from the data that the dissipation peak at $T_M(H)$ is well below $T_I^{\text{dc}}(H)$.

Figures 2(a) and 2(b) display the behavior of χ'' at 10 kHz as a function of temperature for the two ac configurations $\mathbf{h}_{\text{ac}} \perp c$ and $\mathbf{h}_{\text{ac}} \parallel c$, respectively and dc fields always perpendicular to the c axis.

The data in Fig. 2(a) do not show the peak at $T_I(H)$ due to a better alignment between the ac field and the sample, in this particular experiment. The peak at $T_M(H)$ for $H = 600 \text{ Oe}$ coincides with the corresponding one shown in Fig. 1(b). The χ'' behavior of the high-field regime⁴ is well represented by the 600-Oe curve, while the low-field regime is characterized by the results for $H < 300 \text{ Oe}$. The high-field data show a continuous dissipation when increasing temperature, starting near 20 K, then a peak, and, at higher temperatures, a shoulder related to the dissipation of current loops flowing in the Cu-O planes. As a consequence, the low-field behavior in this case is essentially determined by the currents flow-

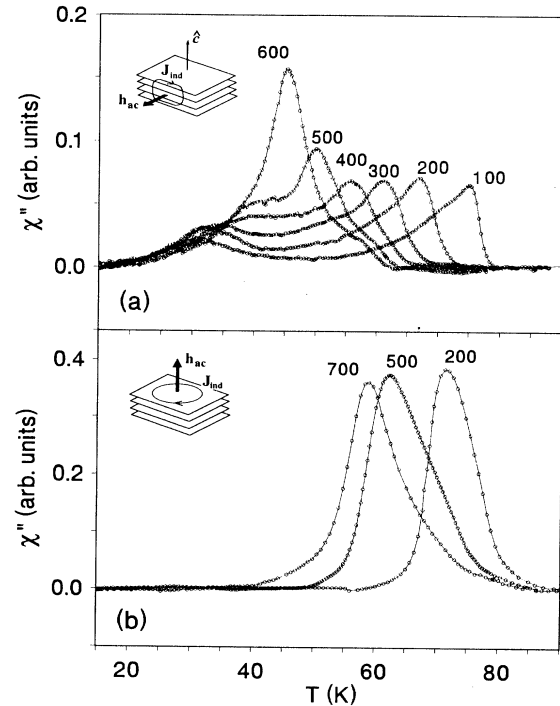


FIG. 2. Dissipative component χ'' of the ac susceptibility as a function of temperature for different applied dc fields in the two configurations of \mathbf{h}_{ac} used: (a) $\mathbf{h}_{\text{ac}} \perp c$ and (b) $\mathbf{h}_{\text{ac}} \parallel c$. The numbers indicate the applied field in Oe. All the curves have been taken at $\nu = 10 \text{ kHz}$ and $\mathbf{h}_{\text{ac}} \simeq 0.1 \text{ Oe}$.

ing between the Cu-O planes. It is interesting to see that in the low-field region the temperature where the maximum dissipation takes place rapidly increases as the field is decreased. The shift of this temperature allows the observation of a dissipation maximum at a nearly field-independent temperature. This peak is only seen in the low-field range. It is not observed at zero field, can be detected for fields greater than 10 Oe,¹² and reaches its maximum intensity at $H_{\text{dc}} \simeq 300 \text{ Oe}$. Its origin is unknown and will be the subject of further investigations.

The data of Fig. 2(b) corresponding to the configuration where $\mathbf{h}_{\text{ac}} \parallel c$ show the shift of $T_I(H)$ with field. It should be noticed that in this configuration no losses are detected in the low-temperature range. The whole peak shifts with field without major changes in its shape and intensity.

Figure 3 shows χ'' as a function of temperature for three different fields and two frequencies, in the configuration $\mathbf{h}_{\text{ac}} \perp c$. The low-temperature dissipation peak mentioned before is clearly seen in this figure. The data shows that while the dissipation at low temperatures is frequency dependent, the position of the dissipation peak at high temperatures is frequency independent for changes in frequency of four orders of magnitude. This last result marks a notorious difference between the ac response of the low- and high-field ranges, where the whole dissipation curve shifts in temperature when varying the measuring frequency, as shown in Fig. 1(b).

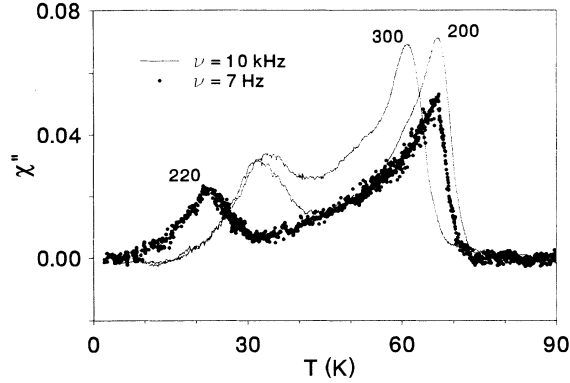


FIG. 3. Plot of χ'' as a function of temperature for three applied dc fields and two frequencies. The numbers indicate the applied field in Oe.

We measured χ'' as a function of temperature for different dc fields up to 400 Oe in the frequency range from 7 Hz to 10 kHz. The temperature where the maximum dissipation takes place for different fields is plotted in Fig. 4. The data show that this temperature is frequency independent within experimental error. In the same figure we have plotted the irreversibility line $T_I^{\text{dc}}(H)$ obtained⁴ from dc SQUID measurements; see Fig. 1(a). At higher fields than those shown in Fig. 4 the data become frequency dependent, giving origin to the already discussed $T_I(H)$. We show that the use of the SQUID in the ac susceptibility measurements permits the detection, in the low-field range, of the two main features observed by the dc measurements and to elucidate the origin of the dissipation as measured by χ'' : the collapse of the critical current and the irreversibility line.

We have investigated the \mathbf{h}_{ac} amplitude dependence of the resistive response. It is found that for low enough ac field amplitudes the response is linear within our experimental sensitivity. This is shown in Fig. 5 where χ'' at 10 kHz is plotted as a function of temperature for ac field amplitudes as small as 5 mOe and up to 1 Oe.

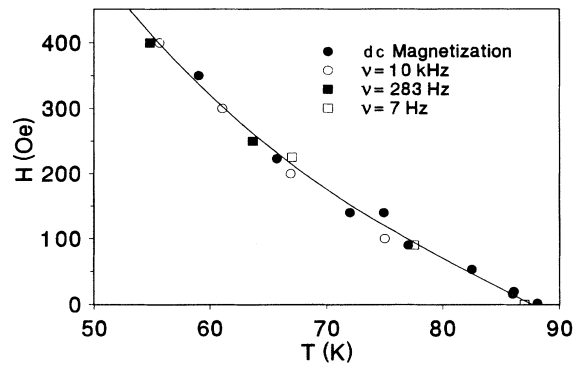


FIG. 4. Irreversibility line as measured at different frequencies. The symbols of the ac measurements correspond to the maximum of $\chi''(T)$. The solid line is a spline fit of the dc data.

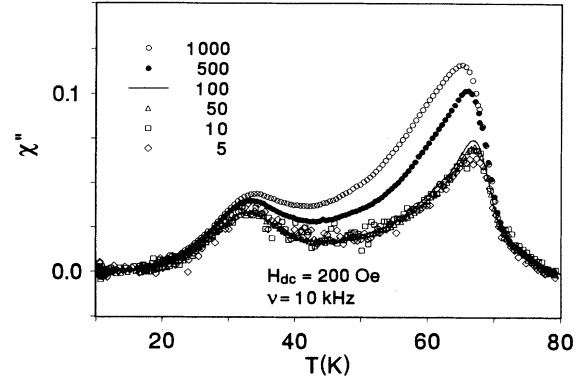


FIG. 5. Plot of $\chi''(T)$ for different amplitudes of $\mathbf{h}_{\text{ac}} \perp c$. The applied dc field was 200 Oe and the data were taken at $\nu = 10$ kHz. The numbers indicate the \mathbf{h}_{ac} amplitude in mOe. For $\mathbf{h}_{\text{ac}} \geq 100$ mOe the response becomes nonlinear in almost the whole range of temperatures.

These results disagree with previous mechanical oscillator experiments² where nonlinearity was always evident. Although this subject deserves further investigation, the origin of the discrepancy may be found in the lower current densities induced in the mechanical oscillator measurements.

In order to study the different behavior of the ac response seen in the high- and low-field ranges it is also useful and complementary to analyze the real component of the susceptibility χ' , which represents the shielding capability of the currents in the sample. In Fig. 6 we present the experimental results for the two components of the susceptibility, as a function of temperature, at 7 Hz and in a range of dc fields from 0 Oe up to 600 Oe. The low frequency shielding response shown in Fig. 6(a) is similar to that reported³ previously at 10 kHz and resembles the dc results as measured by the SQUID, as was discussed in Ref. 4. In the case of the data of Fig. 6(a) the similarity is even closer due to the low frequency used in the experiments.

It is clear from Fig. 6(a) that the shielding capability is strongly depressed for fields higher than 500 Oe at temperatures well below $T_I^{\text{dc}}(H)$. On the other hand, the low-field range χ' curves (220 Oe and 100 Oe) show that the overall shielding capability is increased at temperatures close to $T_I^{\text{dc}}(H)$. This transition towards the low-field region is characterized by a decrease of the intensity of the peak in χ'' at $T_M(H)$, which diminishes and eventually disappears, as seen for $H_{\text{dc}} = 220$ Oe in Fig. 6(b) [see also Fig. 2(a)].

It is important to remark that in the low-field range, the dissipation peak at high temperatures is frequency independent in the range investigated (7 Hz–10 kHz) while the curves for $H_{\text{dc}} > 500$ shift more than 10 K.

Despite the overall increase in the shielding capability in the low-field range, the appearance of the dissipation peak at low temperatures followed by a small jump in χ' is remarkable (see 100-Oe and 200-Oe curves). For $H_{\text{dc}} = 220$ Oe the shielding is found to be reduced, at 20

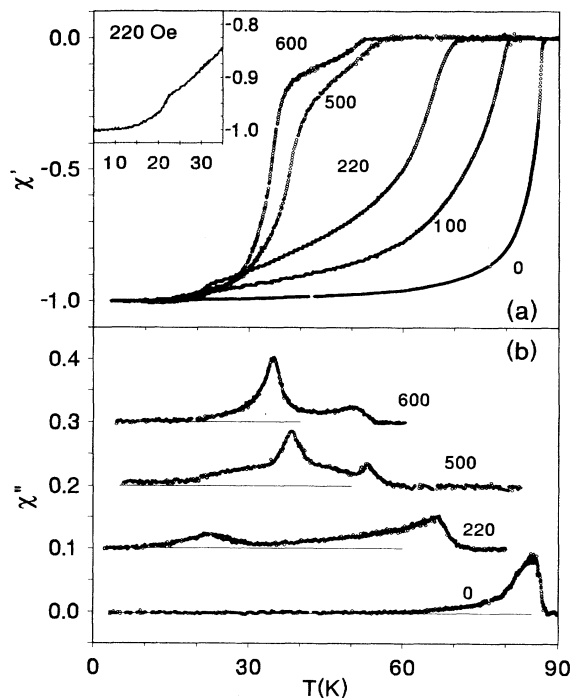


FIG. 6. ac susceptibility data as a function of temperature for different applied dc fields in the configuration $\mathbf{h}_{ac} \perp c$. The numbers indicate the applied dc field in Oe. The data were taken at 7 Hz using the SQUID. (a) χ' . The inset shows an expanded view of the low-temperature region of the 220-Oe curve. (b) χ'' . For the sake of clarity the curves have been offset, and the zero-field curve has been multiplied by 4.

K, by 8%; see inset of Fig. 6(a). This is also detected by a fairly sharp peak in χ'' as discussed before in Fig. 3.

The zero dc field curve is particularly relevant. It indicates perfect shielding at low temperatures up to a temperature somewhat above 60 K where dissipation is made evident. This is an interesting result, because careful dc magnetization measurements¹³ show that in these single crystals a full Meissner state is detected only up to 60 K where the low critical field $H_{C1}(T)$ collapses to zero,¹⁰ indicating the presence of magnetic excitations even for $H_{dc} = 0$.

The data reported in this paper were obtained on the same single crystal and have been reproduced by measurements in several crystals of the same batch.

IV. DISCUSSION

The comparative analysis of the ac and dc data show the coexistence of a dc irreversibility line $T_I^{dc}(H)$ and energy dissipation at temperatures well below it, when ac currents flow perpendicular to the Cu-O planes at low enough frequencies. The data make evident that the generally accepted assumption of an electrical resistivity tending to zero at $T_I^{dc}(H)$ is incorrect for BSCCO 2:2:1:2, at least for fields greater than 400 Oe. The results indicate that the phase correlation length of the

order parameter in the c direction is strongly reduced well below $T_I^{dc}(H)$, at the temperature where the critical currents are seen⁴ to collapse to small values. The shielding capability of the currents flowing only in the Cu-O planes indicates that the phase coherence in the planes is stronger than that in the c direction, at least up to $T_I(H)$.

The experimental results definitely show that the two dissipation peaks, initially observed in other experiments,^{2,3} correspond to two different dissipation processes. The existence of simultaneous current dissipation and magnetic flux metastability are evident. The dc irreversibility line $T_I^{dc}(H)$ separates the reversible magnetic flux region from one having metastable pinned flux at lower temperatures and fields. As was mentioned in Sec. III the characteristic experimental time in these dc experiments is at least of the order of 1 minute. On the other hand, the 7-Hz data for the $\mathbf{h}_{ac} \perp c$ configuration show energy dissipation well below the irreversibility line. This is possible only if two different currents in different regions of the sample are responsible for the magnetic response of the material. These are one associated with two-dimensional Abrikosov vortices, nucleated in the Cu-O planes, and the other with Josephson currents determined by the phase difference of the order parameter nucleated in the Cu-O planes. As was suggested previously³ the results are consistent with a picture in which energy dissipation is induced by the currents flowing between the Cu-O planes in a force-free vortex configuration, while the currents in the planes remain essentially superconducting up to $T_I(H)$ where the quasi-two-dimensional vortices become completely depinned.

The results show the different nature of the superconducting transition where inter- and intraplane currents are involved. The dissipation of the current loops in the planes induce a peak with an intensity of the order of 40% of the total shielding change, as measured by the variation of χ' when the sample goes from complete superconducting to the normal state. This result could be consistent with a matching of a skin depth with some dimension of the sample, in agreement with previous results.⁵ On the other hand, the interplane current induces a dissipation that never exceeds 20% of the change in χ' . In particular, in the low-field region the maximum of the dissipation is always less than 10% of the total change of the real part of the susceptibility.

The use of the SQUID as a current detector in the ac measurements is shown to be a very sensitive technique that allowed to extend the measurements to very low frequencies, making clear that the dissipation related to the loss of long-range correlation length in the c direction takes place well below the dc irreversibility line.

The results of Fig. 3 are a demonstration of the different nature of the dissipation processes taking place at low temperatures and those at high temperatures. It is remarkable that in the low-field regime and for low ac amplitudes the response is linear and frequency independent. This indicates that the low-field irreversibility line represents a very sharp change in the conduction properties of the superconducting state, suggesting a possible phase transition of the vortex structure at that

temperature.¹⁴

The comparative study of the two components of the susceptibility shows the change in the superconducting response when decreasing the applied field. In principle, the larger shielding capability at low fields tends to indicate a transition towards a three-dimensional regime.

The experimental results presented in this paper strongly support that in the high-field range the vortex correlation length in the c direction is strongly reduced¹⁵ by thermal-induced disorder, at a temperature well below that of the irreversibility line.

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