Persistence of the first-order transition lines in mesoscopic Bi$_2$Sr$_2$CaCu$_2$O$_8$ vortex matter with less than hundred vortices

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The persistence of the first-order transition line in the phase diagram of mesoscopic Bi$_2$Sr$_2$CaCu$_2$O$_8$ vortex matter is detected down to a system size of less than hundred vortices. Precise and highly-sensitive to bulk currents AC magnetization techniques proved to be mandatory in order to obtain this information. The location of the vortex matter first-order transition lines are not altered by decreasing the sample size down to 20 µm. Nevertheless, the onset of irreversible magnetization is affected by increasing the sample surface-to-volume ratio producing a noticeable enlargement of the irreversible vortex region above the second-peak transition.

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I. INTRODUCTION

The vortex matter phase diagram in macroscopic samples of clean layered high-temperature superconductors presents a first-order transition (FOT) line between a solid phase at low fields and temperatures and a liquid or decoupled gas of pancake vortices with reduced shear viscosity. At low temperatures the solid phase presents irreversible magnetic behavior ascribed to bulk pinning and surface barriers, each of them dominating at different temperature and measuring-time ranges. Direct imaging of the low-field phase in pristine samples of the extremely anisotropic Bi$_2$Sr$_2$CaCu$_2$O$_8$ compound reveals a vortex solid with quasi long-range positional order. Heavy-ion-irradiated Bi$_2$Sr$_2$CaCu$_2$O$_8$ samples also present a FOT at low fields though in this case the vortex solid is polycrystalline. These findings indicate that the positional order of the solid phase is therefore not relevant for the order of the transition. Josephson-plasma-resonance measurements reveal the FOT corresponds to a single-vortex decoupling process between stacks of pancake vortices in adjacent CuO planes. Therefore, there is evidence that the first-order transition is actually a single-vortex transition that depends, at best, on the density of the surrounding vortex matter. This can be further tested by decreasing the sample size down to a few micrometers such that, at low fields, the system size is reduced to only a few vortices.

The FOT of vortex matter in Bi$_2$Sr$_2$CaCu$_2$O$_8$ can be detected at high temperatures, 0.66 $T_c$ < $T$ < $T_c$, as a jump in the local flux density, or, in some cases, in the reversible magnetization, or, alternatively, by a frequency-independent paramagnetic peak in the in-phase component of the first-harmonic of the AC screening signal. Both features develop at the same field, identified with the first-order transition field $H_{\text{FOT}}$. The irreversible magnetic response of the system in this high-temperature regime is dominated by a surface barrier. Depending on the sample geometry, this barrier can be of the geometrical or Bean-Livingston type, while the bulk of the sample remains magnetically reversible. In the intermediate temperature region 0.39 $T_c$ < $T$ < 0.66 $T_c$, bulk pinning increases its relevance on cooling and the paramagnetic peak is masked by bulk shielding currents, entailing a sudden decrease of the AC signal. Finally, in the low-temperature regime $T$ < 0.39 $T_c$ = 35 K, bulk pinning plays a dominant role and the so-called second-peak effect or order-disorder transition is detected. This transition is manifested as a local increase of the width of DC hysteresis loops and as a minimum in AC magnetization loops with the field-location of the feature, $H_{\text{SP}}$, being frequency-independent.

The persistence of these transition lines when decreasing the sample size down to few vortices is still open to discussion. Whether the thermodynamic $H_{\text{FOT}}$ transition remains in mesoscopic vortex matter consisting of only a few vortices has, to our knowledge, not yet been investigated. Regarding the $H_{\text{SP}}$ transition, two works of the same authors have reported on its disappearance on decreasing the sample system size down to 30 µm. The authors attributed this phenomenon to the sample size becoming smaller than the temperature-dependent Larkin-Ovchinikov correlation length down which the vortex structure is insensitive to the pinning potential. This interpretation is contradictory with their own data since they invoke bulk current arguments for a system that clearly presents a surface-barrier-dominated physics as deduced from the two-quadrant locus of their DC magnetization loops. Other work reports on a sample-size-dependent $H_{\text{SP}}$, even when not extremely varying the millimeter-range sample size, attributed to a distribution of metastable disordered vortex states with different lifetimes.

In this work we report on the phase-location of the $H_{\text{FOT}}$, $H_{\text{SP}}$, and irreversibility, $H_{\text{irr}}$, lines for macro-
scoplic and micron-sized Bi$_2$Sr$_2$CaCu$_2$O$_8$ single crystals, with the field applied parallel to the sample c-axis. We show that the $H_{\text{FOT}}$ and $H_{\text{SP}}$ transition fields do persist down to a system size of the order of hundred vortices. In addition, we reveal that these features present the same signature and location in the phase diagram, independently of decreasing the sample size down to 21 µm.

II. EXPERIMENTAL

Single-crystals of optimally-doped Bi$_2$Sr$_2$CaCu$_2$O$_8$ with $T_c = 90$ K were grown by means of the traveling-solvent floating zone technique. We selected two high-quality crystals from the same batch. The first one was taken as the reference 220 × 220 × 30 µm$^3$ macroscopic crystal and the second one was used to engineer micron-sized disks. The latter are obtained by means of optical lithography of the sample surface and subsequent physical ion-milling in the disks’ negative. The resulting micron-sized towers are removed by cleaving the milled surface with a resist-wetted silicon substrate. The resist is then chemically eliminated. The resulting circular samples have typical thicknesses of 1 µm and diameters ranging from 10 to 50 µm. The disks are then mounted onto Hall-sensor chips with micron-precision manipulators, and glued with Apiezon N grease.

The local magnetization or, more precisely, the stray field, of the disks was measured with microfabricated 2D electron gas Hall sensors with 6 × 6 µm$^2$ active surface embedded with an excitation coil on a single chip. Figure 1 shows a photograph of one of the studied disks mounted on one of two adjacent sensors. The remaining empty sensor is used as a local reference of the applied magnetic field when performing DC experiments. The macroscopic sample was measured by means of similar chips supporting several Hall sensors with active areas ranging from 6 × 6 to 40 × 40 µm$^2$. The Hall array is always placed over the center of the sample. The on-chip embedded coil generates an AC field parallel to the applied DC field $H$. In all the experiments presented here this ripple field has an amplitude of $H_{\text{AC}} = 0.9$ Oe rms, while its frequency ranges from 1 to 1000 Hz. The current applied to the sensors is in the range of 25 to 50 µA. A digital-signal-processing lock-in technique is used to simultaneously measure the in- and out-of-phase components of the fundamental and the third-harmonic frequencies of the Hall voltage.

In this way, several DC and AC local magnetic measurements were performed using the same set-up, as a function of temperature, magnetic field, and frequency. The DC magnetic hysterisis loops are obtained by measuring the magnetization, $H_S = B - H$, while its frequency ranges from 1 to 1000 Hz. The current applied to the sensors is in the range of 25 to 50 µA. A digital-signal-processing lock-in technique is used to simultaneously measure the in- and out-of-phase components of the fundamental and the third-harmonic frequencies of the Hall voltage.

Figure 2: High-temperature DC and AC magnetic hysteresis loops of the reference Bi$_2$Sr$_2$CaCu$_2$O$_8$ macroscopic crystal measured at 75 K. Top: ascending and descending branches of the DC hysteresis loop. Insert: zoom-in of the DC loop at the vicinity of the first-order transition entailing a $B$-jump (see black arrow). Red arrows indicate the ascending and descending branches. Bottom: AC transmittivity loops with paramagnetic peaks fingerprinting the first-order transition field $H_{\text{FOT}}$. The loops were measured with ripple fields of 0.9 Oe rms and frequencies of 7.5 and 437 Hz. Black arrows indicate the ascending and descending branches.
induction $B$. The modulus of the third harmonic signal $|T_{h3}| = |B_{h3}^\text{AC}| / [B'(T > T_c) - B'(T \ll T_c)]$ becomes non negligible at the onset of irreversible magnetic properties (see Fig. 3). This onset is then used to track $H_{\text{irr30}}$.

We perform two types of measurements, temperature evolution of $T'$ and $|T_{h3}|$ on field-cooling in several fields, and isothermal DC and AC hysteresis loops as, for instance, shown in Fig. 2. By means of the AC technique, the first-order transition field and the onset of magnetic irreversibility are detected with better resolution than by DC loop measurements. In the high-temperature regime, the FOT is manifest in the AC transmittivity as a prominent paramagnetic peak that develops at the same $H$ as the jump in local induction detected in DC hysteresis loops.

III. RESULTS

A. Reference macroscopic sample

The magnetic properties of the reference macroscopic Bi$_2$Sr$_2$CaCu$_2$O$_8$ sample present three characteristic temperature regimes. Figure 2 depicts the magnetic hysteretic response of the reference macroscopic sample at
independent, and is therefore considered as 

The frequency dependence of 

In addition, the 

decreases on increasing the applied field, see Fig. 3 (b). 

This feature is the fingerprint of the first-order transi-

For frequencies smaller than 7.5 Hz a sudden drop of 

A low-

temperature AC loop at 35 K is shown in Fig. 4 (b) depicting local minima in both, the ascending and de-

Figure 5 presents the evolution of the nor-

We therefore use the AC hysteresis loop technique in order to track the first-order transition up to higher temperatures. The AC transmittivity reflects the dimensionless normalized sustainable-current density, 

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The crossover temperature for the detection of the 

The high-temperature, 

The top panel shows a two-quadrant DC loop such as typically obtained in this temperature regime. Closer inspection of the DC data in the vicinity of 80 Oe reveals a 

On increasing frequency, the system enhances its shielding capability manifested as a 

Figure 2 shows that, in AC loops, this transition is detected with improved resolution: paramagnetic peaks emerge at the flanks of the central 

developing at the flanks of the central 

Figure 3 shows a set of AC magnetic data for low applied fields ranging from 10 to 200 Oe. The paramagnetic peak in 

The second-peak transition 

The crossover temperature for the detection of the 

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Finally, Fig. 6 presents the vortex matter phase di-

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temperatures, the $H_{\text{irr}}$ line deviates towards higher fields and is strongly frequency-dependent. The higher the frequency, the larger is the deviation from the FOT line. The onset of $|T_{h3}|$ indicates the electric field $E = E(H_{\text{AC}}, f, J)$ becomes non-linear below a working point given by the experimental resolution. As temperature decreases, the linear response limit shrinks to smaller current-density values. Therefore, the onset of third harmonic response shifts to lower temperatures for smaller frequencies (smaller currents).

### B. Micron-sized samples

As discussed in the previous section, performing AC measurements is necessary in order to properly track the FOT line, particularly in the case of samples of highly-reduced size in which surface barriers for vortex flux entry/exit completely dominate the electromagnetic response. The insert to Fig. 6 shows the transmittivity data for the 21 $\mu$m diameter disk at the smallest measurement field of 5 Oe. The paramagnetic peak fingerprinting the $H_{\text{FOT}}$ is clearly visible for this system consisting of only 80 vortices. Tracking the location in the temperature-field plane of this peak yields the first-order transition line in the vortex phase diagram shown in the main panel of Fig. 7. An important finding of this figure is the persistence of the $H_{\text{FOT}}$ transition line for micron-sized vortex matter from $T_c$ down to 75 K = 0.83 $T_c$. The first-order transition line is also detected down to similar temperatures in larger disks. This indicates that, even for samples with a large surface-to-volume ratio, the $H_{\text{FOT}}$ transition measured at high-temperatures remains robust. For $T < 75$ K no paramagnetic peak is detected in the transmittivity data, presumably due to a masking effect produced by enhanced surface barriers in small samples. The $H_{\text{FOT}}$ line for the disks merges that of the macroscopic reference sample, indicating that the nature of this transition does not change even for a system with just 80 vortices, for the smallest applied field of 5 Oe in the 21 $\mu$m sample (see Fig. 7).

The $H_{\text{irr}}$ line for micron-sized samples is located at higher fields than for the macroscopic sample. For example, Fig. 7 shows that at low temperatures $H_{\text{irr}}$ is $\sim 35\%$ larger for the 21 $\mu$m disk than for the macroscopic sample at a fixed frequency (177 Hz in this case). This result might have origin in the different aspect ratio of the macroscopic and disk samples, and in their probably dissimilar surface roughness originating from the different preparation methods for both specimens. Ascertaining the origin of this discrepancy is beyond the aim of this paper. Figure 7 also shows that for smaller frequencies $H_{\text{irr}}$ approaches the first-order transition line. Finally, in the high-temperature range the irreversibility lines for the macroscopic and micron-sized samples merge into a single bunch of data with the $H_{\text{FOT}}$ line.

We also study the effect of decreasing the sample size down to microns on the $H_{\text{SP}}$ transition. Previous works using DC magnetic techniques reported that this tran-
Figure 8: Low-temperature magnetic hysteresis loops for Bi$_2$Sr$_2$CaCu$_2$O$_{8+}$ micron-sized disks for DC (top red curves) and AC (bottom blue curves) measurements: (a) smallest 21 µm diameter measured disk at 30 K and (b) 42 µm diameter disk at 35 K. The ripple field is of 0.9 Oe rms and 194 Hz for the smaller disk and 17 Hz for the largest one. The transition field $H_{SP}$ considered as the mid-point between the onset and the full development of the minimum in the ascending branch is indicated with an arrow.

Figure 8: Low-temperature magnetic hysteresis loops for Bi$_2$Sr$_2$CaCu$_2$O$_{8+}$ micron-sized disks for DC (top red curves) and AC (bottom blue curves) measurements: (a) smallest 21 µm diameter measured disk at 30 K and (b) 42 µm diameter disk at 35 K. The ripple field is of 0.9 Oe rms and 194 Hz for the smaller disk and 17 Hz for the largest one. The transition field $H_{SP}$ considered as the mid-point between the onset and the full development of the minimum in the ascending branch is indicated with an arrow.

The three characteristic temperature regimes of the first-order phase transition of macroscopic Bi$_2$Sr$_2$CaCu$_2$O$_8$ vortex matter are also found for micron-sized samples. In the latter case, the identification of the first-order transition is rendered more difficult by the enhancement of the surface to bulk-currents ratio. The nearly-vanishing remanent magnetization and the two-quadrant locus of the DC loops in micron-sized samples indicate the preeminence of surface barriers for vortex flux entry/exit. Therefore applying AC magnetic techniques is imperative in order to have access to the faster decaying bulk currents emerging from the surface-barrier background.

In the intermediate- and low-temperature regimes $T'$ and $|T_{13}|$ present particular features that are related to the bulk-current contribution. For $0.39T_c < T < 0.66T_c$ we detect, at a field $H_{step}$, a discontinuous decrease of the bulk shielding-currents associated with the first-order transition. On cooling below $T = 0.39T_c = 35$ K, the opposite effect of an increase of the shielding currents is observed at almost the same field indicating the $H_{SP}$ transition. In the vicinity of this reversal of current behavior, varying the frequency of the AC ripple field tunes a decrease (low-frequencies) or an increase (high-frequencies) of the shielding currents. The latter case is equivalent to probing magnetic relaxation on a shorter time-scale, in analogy to DC magnetization experiments, or to choosing a higher electric field in an transport $I(V)$ measurement. Since the $I(V)$ curves just below and above the FOT cross — with the electric field in the high-field phase being larger than that in the low-field phase for the low-current density limit, and vice-versa for the high-current density limit, varying the working point (by tuning the frequency) leads to either a step-like-behavior of the screening current (at low electric fields) or a peak-like curve (at high electric fields). The energy barriers for flux creep have $U(J)$ and $E(J)$ curves with different functionalities for fields larger or smaller than the transition one. On varying field close to the transition these curves cross, and the phase transition produces a discontinuous change on the electrodynamics of vortex matter. Detecting the transition with a high electric field (short measurement times) leads to an enhancement of shielding currents with increasing field, whereas with a low electric field is not detected for samples with diameters smaller than 30 µm.

We performed AC measurements in order to study the persistence or absence of the $H_{SP}$ transition. Fig. 8 shows the comparison of DC and AC loops data: while the DC loops do not show evidence of the $H_{SP}$ feature, the local minima in the ascending and descending branches of the AC loops are evident. Even measuring at intermediate (194 Hz) and low (17 Hz) frequencies, the application of the AC technique enables the observation of the emergence of the bulk current contribution, therefore allowing the detection of $H_{SP}$. Figure 8 depicts a remarkably well-developed $T'$ local minimum for the 21 and 42 µm diameter disks, giving a $H_{SP}$ field that is within 10% of the value observed in macroscopic samples (roughly 330 Oe).
field (long times) a sudden decrease is observed. The lack of sensitivity of DC magnetization loops to the FOT in the intermediate-temperature regime can be understood as the consequence of a distribution of Bean-Livingston surface barriers.\textsuperscript{32} The transit of individual pancake vortices over these barriers is not affected by the nature of the vortex phase inside the sample.

The irreversibility line obtained from the onset of $|T_{h3}|$ merges with the first-order transition line $H_{FOT}$ in the high-temperature regime. At lower temperatures or higher applied fields, the shielding of the AC field starts at higher temperatures than the occurrence of the first-order transition, namely, the onset of $|T_{h3}|$ develops before the paramagnetic peak on cooling. This indicates the existence of a non-linear vortex region spanning at higher temperatures than the first-order transition line.\textsuperscript{22,23} This phenomenon might have origin from a residual effect of pinning,\textsuperscript{33} or Bean-Livingston barriers,\textsuperscript{34} on the high-temperature liquid phase. This phenomenology is observed in macroscopic as well as micron-sized Bi$_2$Sr$_2$CaCu$_2$O$_8$ samples.

The most important finding of this work is that the first-order transition persists even for a very small vortex system. In the high-temperature regime, the clear detection of the paramagnetic peak at 5 Oe in the 21 µm disk is shown in the insert to Fig. 7. This result indicates that, independently of its nature, the first-order transition is robust and its thermodynamic nature remains unaltered even for a reduced system size of less than hundred vortices. In the low-temperature regime, the detection of the $H_{SP}$ transition as a sudden increase of shielding currents (decrease of $T^*$) persists in micron-sized samples. The observation of this feature was made possible by the use of an AC magnetic measurement technique. The possibility of working at shorter time-scales (larger frequencies) reveals currents flowing in the sample volume with improved sensitivity than DC measurements. Therefore, our results indicate that reducing the sample size down to few dozens of microns does not produce a disappearance nor a dramatic decrease of $H_{SP}$ due to size effects as previously claimed.\textsuperscript{22,23,24}

V. CONCLUSIONS

In the high-temperature phase region, the first-order transition of Bi$_2$Sr$_2$CaCu$_2$O$_8$ vortex matter remains robust and persists at the same $H - T$ location even when decreasing the system size down to less than hundred vortices. We found that the second-peak transition detected in the low- and intermediate-temperature regions also persists on decreasing the system size down to roughly 20 µm, in contrast with previous reports.\textsuperscript{22,23} The identification of these transitions increases in difficulty when decreasing the sample size, due to the predominance of surface-barrier-related currents. The application of AC magnetic techniques, with better sensitivity to bulk currents, allowed us to detect the transition-related features.

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