Dependence of vortex phase transitions in mesoscopic Bi$_2$Sr$_2$CaCuO$_8$ superconductor at tilted magnetic fields

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Abstract. A micron sized single crystal of the superconductor Bi$_2$Sr$_2$CaCuO$_8$ was studied using silicon mechanical micro-oscillators at various tilt angles of the dc magnetic field with respect to the c axis of the sample. Different phases of the vortex matter were detected by measuring changes in the value and sign of the oscillator resonant frequency variation with temperature. We could explain the change in the sign of this variation at high temperatures as the transition from the 2D liquid of decoupled pancakes to a reversible 3D vortex lattice. The data indicates that this transition only depends on the magnetic field perpendicular to the superconducting layers while the dissipation involved in this process depends on the component parallel to them.

1. Introduction
The vortex matter structure in high $T_c$ superconductors is determined by the competition between three important energy scales: thermal, repulsive interaction between vortices and attractive interactions between them and materials defects (pinning). The Bi$_2$Sr$_2$CaCuO$_8$ (BSCCO) is a strongly anisotropic type-II superconductor and because of all these factors it presents an ample, complex and rich phase diagram with numerous phase transitions and crossovers. A magnetic field, $H$, perpendicular to the superconducting planes ($H_c$ parallel to c axis) penetrates the sample as 2D pancakes vortices, PV’s, [1] while a transverse field ($H_{ab}$ parallel to ab planes) penetrates as Josephson vortices, JV’s. The JV’s couple PV’s from different superconducting planes and this connection disappears at high temperatures where the lattice of PV’s melts into a liquid. Under tilted fields this transition in macroscopic samples has been studied intensively [2]. In this paper we show that in a micron sized sample of BSCCO this transition only depends on the magnetic field perpendicular to the superconducting layers ($H_c$) although the dissipation involved in this process depends on the component parallel to them ($H_{ab}$).

In order to detect the small signals produced by microsized samples, new instruments designed for the microscale are needed. Our approach for studying the magnetic properties of a Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ microscopic disk is to use silicon micro-oscillators (following the work of [3] and [4]) which have a torsional mode with a resonant frequency $\nu_r \approx 45$ kHz and a quality factor $Q > 10^4$ at low temperatures. This instrument integrates high sensitivity and reduced size with a small signal loss.
2. Experimental
The poly-silicon oscillators were fabricated in the MEMSCAP [5] foundry using its Multiuser process (MUMPS). The oscillator consists of a $50 \times 100 \ \mu m^2$ released plate anchored to the substrate by two serpentine springs. A more detailed description of the experimental detection setup can be found elsewhere [4, 6]. Single crystals of BSCCO were grown using the self flux technic [7] and by means of optical lithography and ion milling we fabricated disks of 13.5 microns in diameter and 1 micron in high. A disk was mounted on the micro-oscillator's plate with the Cu-O layers ($ab$ planes) parallel to it (figure 1a). The measurements were taken in vacuum inside a closed-cycle cryogenerator where the temperature can be varied between 14 and 300 K. The $dc$ magnetic field was provided by a split electromagnet that can be rotated in the plane perpendicular to the axis of rotation of the oscillators with an accuracy of $1^\circ$. Therefore, the direction of $H$ can be varied from the $c$ axis of the sample ($H = H_c$) to an axis perpendicular to it ($H = H_{ab}$).

![Image](image_url)

Figure 1. a) Scanning electron micrograph of a high-Q mechanical silicon oscillator with a BSCCO disk with a diameter of 13.5µm and a thickness of 1µm. b) Change in the resonant frequency as a function of temperature under a tilted external magnetic field.

The natural resonant frequency ($\nu_0$) of an oscillator in the torsional mode is given by:

$$2\pi \nu_0 = \sqrt{\frac{k_e}{I}}$$

where $k_e$ is the elastic restorative constant of the serpentine springs and $I$ is the plate moment of inertia. In our system (oscillator+sample) this mode has a resonant frequency close to 40 kHz and a quality factor $Q$ greater than $2.6 \times 10^3$. When a magnetic sample is attached to the oscillator and $H$ is applied the resonant frequency $\nu_r$ changes to:

$$2\pi \nu_r = \sqrt{\frac{k_e + k_M}{I}}$$

where $k_M$ is the variation in the effective elastic constant originated by the magnetic interaction between the sample and the magnetic field. It can be expressed as:

$$k_M \simeq 8\pi^2 I \nu_0 \Delta \nu,$$

where $\Delta \nu = \nu_0 - \nu_r$ is the change in the resonant frequency when $H$ is applied. This $k_M$ is associated with the sample magnetization, $M$, and can be calculated by differentiating two times
the magnetic energy, as in [6]. The experiment consist in measuring the oscillator's torsional mode resonant frequency as a function of temperature to different values and directions of $H$.

3. Results and Discussions

In the two limit cases where $H = H_c$ and $H = H_{ab}$ we showed [4] that $\Delta \nu_r$ is always negative and positive, respectively. In the first case this is due to the non-restorative torque between $H_c$ and the magnetic moment generated by Meissner currents. In the second case the restorative torque is generated by the interaction between $H_{ab}$ and the superconducting currents that shield the alternating perpendicular magnetic field $h_c = H_{ab} \times \sin \alpha$, where $\alpha$ is the angle of the oscillator with respect to its equilibrium position. On the other hand, the response of the system to tilted fields $H = H_\theta$, where $\theta$ is the angle between the field and $c$ axis of the sample, can be positive or negative depending on the vortex system phase. Figure 1b shows ZFC (zero field cooling) and FC (field cooling) measurements for $H$ at $\theta = 30^\circ$. The presence of $ac$ magnetic fields generated by the tilt of the micro-oscillator, causes that $\Delta \nu_r$ is related with the $dc$ magnetization, $M$, and the $ac$ response, $\chi$, of the sample. It is known that the melting transition imply features in $M$ and $\chi$ [8]. The only peculiar or characteristic behavior in our measurements at high temperatures is the change of sign of $\Delta \nu_r$, and in this way we can relate it with the melting transition from 2D liquid of decoupled pancakes to a reversible 3D vortex lattice. On the other hand, from low temperatures at the irreversible area the non-restorative torque in ZFC measurements is basically due to the Meissner currents and for FC measurements the response $\Delta \nu_r > 0$ is due to the pinning's vortices.

![Figure 2](image_url)

**Figure 2.** a) $\Delta \nu_r$ vs reduced temperature applying a constant magnetic field perpendicular to the superconducting planes for different values of $H_{ab}$ as indicated in the figure. b) Dissipated energy of the sample vs reduced temperature. Inset: Integral of the dissipation at the melting transition vs $H_{ab}$.

The measured $\Delta \nu_r$ vs. $T$ data for different angles and values of $H$, keeping constant $H_c = 136$ Oe is showed in figure 2a. We observe that for microscopic BSCCO samples the
melting transition occurs at the same reduced temperature $t \sim 0.815$ and does not depends on the applied $H_{ab}$. This result agrees with the conclusions obtained in ref [9] who proposed that in BSCCO, the phase transition from the vortex liquid phase to the vortex solid state depends only on $H_c$.

Due the Lorentzian fit of the amplitude vs. frequency curve from which we obtain $\nu_r$, we also can obtain $Q$. In this way we can find the energy, $\epsilon$, dissipated for the sample by cycle in each temperature (figure 2b):

$$\epsilon \propto Q_0/Q_H - 1,$$

(2)

where $Q_H$ and $Q_0$ are the quality factors with and without $H$. The integral of $\epsilon$ over the range of temperature where the transition is present, is a quadratic function of $H_{ab}$ (inset figure 2b). While the melting temperature no depends on $H_{ab}$, the jump in $\Delta \nu_r$ does it. The term of $\Delta \nu_r$ (or of energy) related with the ac currents shielding $h_c$ is a quadratic function of $H_{ab}$. This means that the dissipation of the system is controlled only by ac currents generate in the superconducting planes.

4. Conclusions

In conclusion, we have presented measurements of a micron sized single crystal of the superconductor $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ using silicon mechanical micro-oscillators. The change in the resonant frequency can be positive or negative, depending on how the currents and magnetic moments are generated in the sample. The melting transition can be detected as the change of sign in $\Delta \nu_r$ at high temperatures and it is independent of the value of the magnetic field parallel to the superconducting planes and only depends on the magnetic field perpendicular to them, in agreement with [9]. However, at the transition the dissipated energy is a quadratic function of $H_{ab}$, indicating that the ac currents in the superconducting planes are the most important source of dissipation.

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