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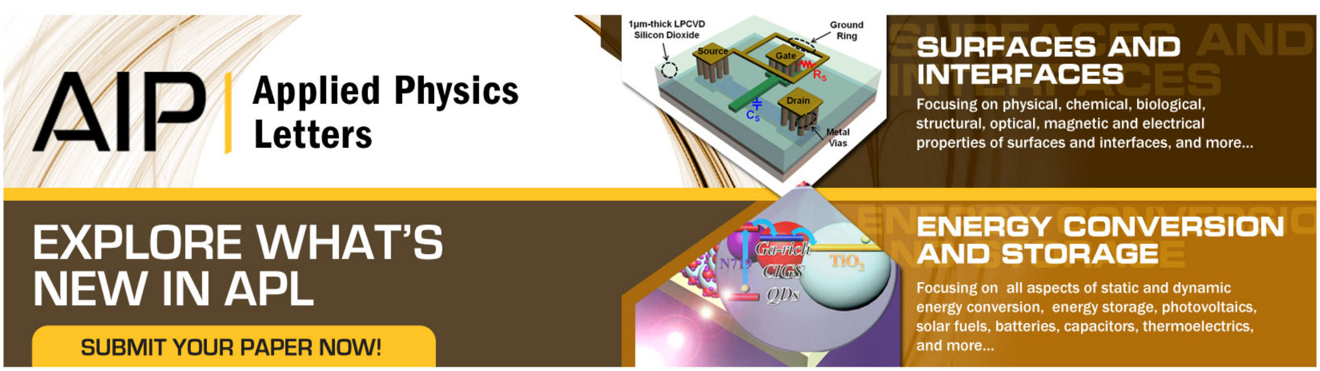

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Investigation of harmonic generation in the alternating magnetic response of superconducting Y-Ba-Cu-O

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The magnetic behavior of a sintered Y-Ba-Cu-O superconductor has been investigated by monitoring the harmonic components of its magnetic response to an alternating field. As the steady bias field is incrementally raised, a transition from a linear, reversible behavior of the magnetization to a nonlinear, irreversible behavior is indicated by the appearance of magnetic losses and odd harmonic components in the response. These harmonics disappear as the bias field or the temperature is increased above a certain point, indicating a linear behavior. The disappearance of the odd harmonics, while the magnetic losses persist, is interpreted as signifying a transition to a state of dissipative flux motion without pinning. These measurements demonstrate a new technique for determination of the lower critical field and the "irreversibility" field below which irreversibility in the magnetization sets in as a result of flux pinning.

The magnetic behavior of the new Y-Ba-Cu-O superconductors has been widely investigated using both dc and ac techniques.[1-9] Nevertheless, the values of fundamental characteristics, such as the lower and upper critical fields H_{c1} and H_{c2}, are still controversial.[4,7] Accurate measurement of these fields is required for determination of key parameters such as coherence length and penetration depth, which are vital for understanding the superconducting mechanism. The lower critical field is usually determined from measurements of the superconducting magnetization curves. The onset of nonlinear behavior in the magnetization versus field curve is identified as H_{c1}. The accuracy of this method is limited because of the difficulty in detecting relatively small deviations from linearity just above H_{c1}. Estimations of the upper critical field commonly rely upon measurements of the transition exhibited by the electrical resistivity or the magnetic susceptibility at a given field. The drawback of this technique is that the transitions broaden with increasing field and the methods for extracting the transition temperature are somewhat arbitrary. In a recent paper, Malozemoff *et al.*[7] argue that the transitions previously used to indicate H_{c2} in Y-Ba-Cu-O actually represent an "irreversibility" field, H_{irr}, i.e., a field below which irreversibility in the magnetization sets in as a result of flux pinning. Above this field, thermal activation permits unpinning of flux lines within the time scale of the experiment. They further infer that the line representing H_{c2} in the field-temperature plane lies above the irreversibility line and may be better determined by the onset of the resistivity drop.

In this letter we propose a new approach for characterizing the magnetic behavior of Y-Ba-Cu-O by which the lower critical field and the irreversibility line can be determined more accurately. This approach is based on monitoring odd harmonic components of the ac magnetic response of the material in steady bias fields. A theory of odd harmonics in the magnetic response of type II superconductors was first developed by Bean.[10] Although this model forms the basis for an understanding of the harmonic generation, it is not directly applicable to critical-field measurements since it assumes that the lower and upper critical fields are zero and infinity, respectively. A model predicting odd harmonics in the ac magnetic response of superconductors was also developed by Ishida and Mazaki.[11] According to this model the third-harmonic component of the response is proportional to the out-of-phase susceptibility. Campbell *et al.*[12] developed methods for measuring H_{c1} and H_{c2} using the transient response to the superposition of a sinusoidal and ramped fields. They also reported the appearance of a second-harmonic component in this response when the applied field was swept between H_{c1} and H_{c2}. However, no second-harmonic signal was observed when the dc field was held constant during the measurement. Harmonics above the second were not examined.

The experimental procedure of this study is similar to the conventional ac susceptibility technique.[13] Small oscillations in the magnetization are induced by a small sinusoidal field that is superimposed on a colinear steady field. The sinusoidal field is produced by a primary coil coaxial with a pair of balanced coils, one containing the sample. The off-balance voltage induced in the coil pair is monitored using a spectrum analyzer (HP 3585A) and a two-phase lock-in amplifier (Ortholock-SC 9505). In order to probe H_{c1} and H_{irr} at a given temperature, the bias field is slowly swept over a wide range. On raising the field above H_{c1}, the material transforms into a mixed state and its magnetic response becomes nonlinear. The magnetization induced by the sinusoidal field traverses minor hysteresis loops as a result of flux pinning. Thus a nonsinusoidal voltage is induced across the sample coil and components of the voltage at harmonics of the applied frequency are generated.[10] Hysteresis loops that are symmetrical with respect to the origin cause the generation of odd harmonics.[10] The transition to the mixed state at H_{c1} is also accompanied by the onset of hysteresis losses. The disappearance of the harmonic components, on raising the bias field above a certain point, indicates linear behavior of the system and hence flux motion without pinning. Thus H_{c1} is indicated by the appearance of odd harmonics and hysteresis losses, whereas H_{irr} is indicated by the disappearance of the odd harmonics. The losses may persist further up beyond H_{irr} because of the flux flow resistance effect.[7] The

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experiments described in this note were made on sintered Y-Ba-Cu-O material. Although the above analysis may have to be modified to account for the granular nature of this material, the experimental results clearly demonstrate the phenomena of appearance and disappearance of the odd harmonics.

The Y-Ba-Cu-O material was prepared by ball milling stoichiometric amounts of yttria, barium carbonate, and copper oxide powders. Calcination of the powders was performed at 900 °C in flowing oxygen (500 cc/min) for 16 h followed by a 10 h ramped cooling to room temperature. The calcined powders were ground and then pressed at 200 MPa into pellets and fired at 950 °C for 16 h in flowing oxygen (500 cc/min). An x-ray diffractogram of this material agreed with that of the superconducting "1-2-3-7" standard material.

We measured the amplitude of the third-harmonic component V_3 and the out-of-phase ac susceptibility χ'' in a slab of Y-Ba-Cu-O ($1\times5\times7.4$ mm) with the long dimension parallel to the bias field. An ac field of amplitude 0.04 Oe and frequency 20 kHz was applied in the same direction. Figure 1 shows the variation of V_3 and χ'' as a function of the bias field at a constant temperature of 77 K. The third-harmonic signal V_3 and the out-of-phase susceptibility χ'' show similar behavior. Both remain below the background noise level as the bias field is increased from 0 up to about 60 Oe, and rise abruptly thereafter. This indicates a transition from a linear, reversible behavior to a nonlinear, irreversible behavior of the magnetization. Interpreting it as a transition from the superconducting state to the mixed state of the material, the lower critical field H_{c1} can be determined by extrapolating the roughly linear rise of $\chi''(H)$ and $V_3(H)$ downward to where they intersect the noise level. This gives the value of 63 ± 5 Oe for H_{c1} in this sample at 77 K. However, because of the granular nature of the sample, this value of H_{c1} may represent the decoupling of grains rather than the intrinsic H_{c1} of the material.

Figures 2 and 3 show the temperature dependence of the third-harmonic signal and χ'' as the sample was cooled in zero field and then slowly heated through the transition under a steady bias field. It is seen that the third-harmonic signal drops sharply to the noise level at a well-defined temperature. This temperature shifts down as the bias field increases. In contrast, χ'' shows a gradual approach to zero,

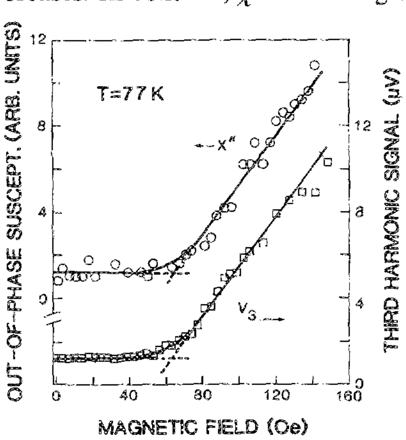

FIG. 1. Third-harmonic signal (V_3) and out-of-phase susceptibility (χ'') measured in Y-Ba-Cu-O ceramic as function of bias field at constant temperature of 77 K. The solid lines are a guide for the eye.

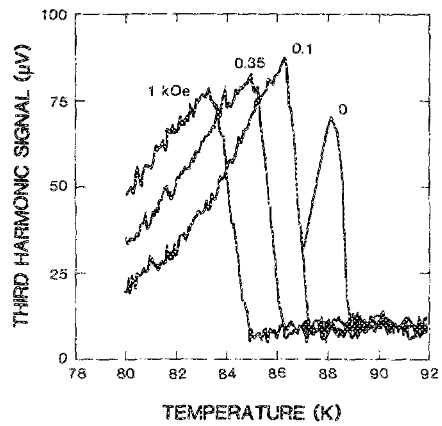

FIG. 2. Temperature dependence of the third-harmonic signal for Y-Ba-Cu-O ceramic in various bias fields.

trailing up to higher temperatures. As mentioned previously, a plausible interpretation of these data is that the disappearance of the third-harmonic signal, while the magnetic losses persist, signifies a transition to a state where flux motion is retarded only by viscous damping. Following this interpretation, we obtain the irreversibility line in a close vicinity of T_c, as shown in Fig. 4. The curvature of the irreversibility line can be fit to a relation $T_{\rm irr}(0)-T_{\rm irr}(H)\propto H^q$ with $q=0.4$ as shown by the dashed curve in Fig. 4. This is in variance with the theoretical prediction $q=2/3$ of the spin-glass model (the "de Almeida–Thouless" line), which was found in high-field dc measurements on powder samples of Ba-La-Cu-O[14] and some Y-Ba-Cu-O crystals.[9] Deviations from an $H^{2/3}$ dependence in low-field measurements on Y-Ba-Cu-O ceramics has been reported by Yeshurun *et al.*[15] and by Cronemeyer *et al.*[16]

It should be noted that the line determined from the peaks in χ'' (shown in Fig. 4 as squares) cannot indicate the irreversibility line, as sometimes proposed,[7] since the positions of these peaks depend on the sample dimensions (in our case thickness of the slab) and the amplitude of the ac field. This dependence is predicted by the critical-state model[17] and was confirmed in our experiments on Y-Ba-Cu-O ceramics.

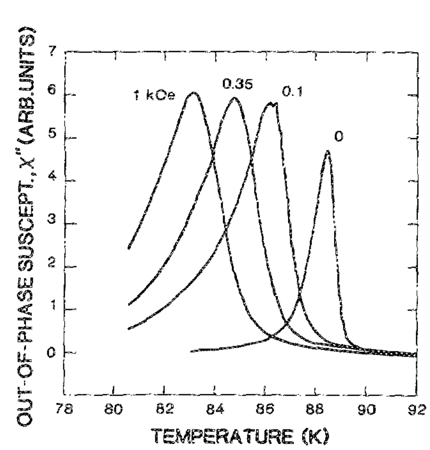

FIG. 3. Temperature dependence of the out-of-phase ac susceptibility for Y-Ba-Cu-O ceramic in various bias fields.

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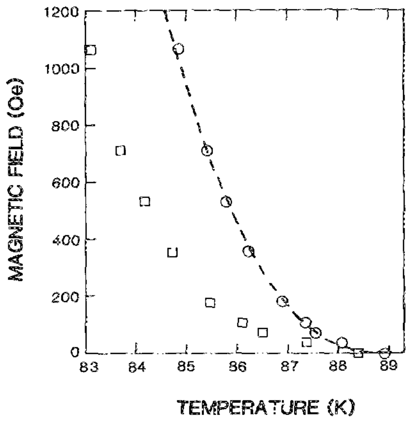

FIG. 4. Irreversibility line (circles) in Y-Ba-Cu-O ceramic as determined from the transitions exhibited by the third-harmonic signal. The dashed line is a fit to a relation $T_{irr}(0) - T_{irr}(H) \propto H^{0.4}$. The field-temperature relation obtained from the positions of the peaks in χ'' is indicated by squares.

In conclusion, measurements of higher harmonics in the ac magnetic response offer an effective tool for investigating the magnetic behavior of high T_c superconductors. Of particular interest is the sharp transition which is exhibited by the third-harmonic component (Fig. 2). We have interpreted it as signifying a transition from a state of flux pinning to a state of dissipative flux motion without pinning. This interpretation is supported by our recent observation that this transition is strongly frequency dependent.[18] As expected, the transition temperature shifts up as the frequency increases. Measurements of the harmonic components also enable more accurate determination of the lower critical field H_{c1}. The measurements reported here on a polycrystalline material are affected by the anisotropy of the randomly oriented crystal grains and the grain boundaries. Similar measurements should be done on single crystals of Y-Ba-Cu-O to reveal the bulk properties. Measurements of the frequency dependence of the transition exhibited by the third-harmonic component will be published elsewhere.

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