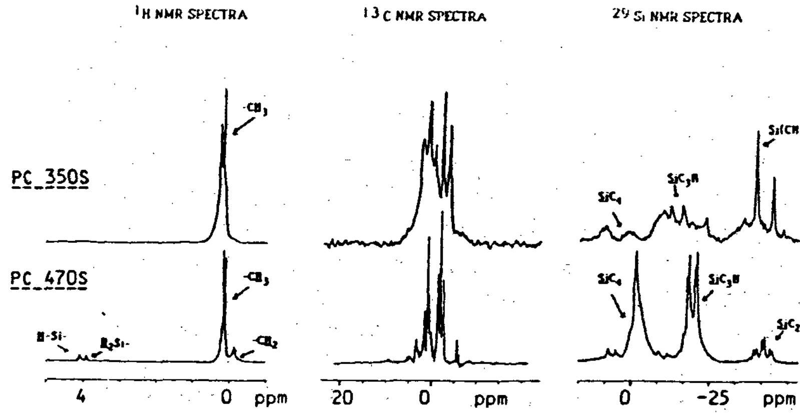

Figure 1a Figure 1b Figure 1c

PC 470S

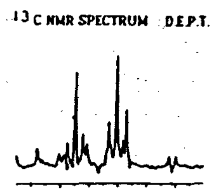

Figure 2a

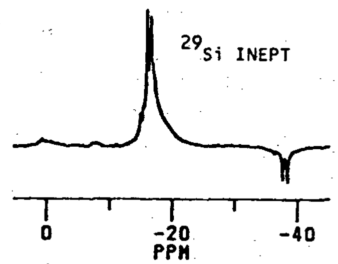

Figure 2b

^{1}H-^{13}C heteronuclear shift correlation

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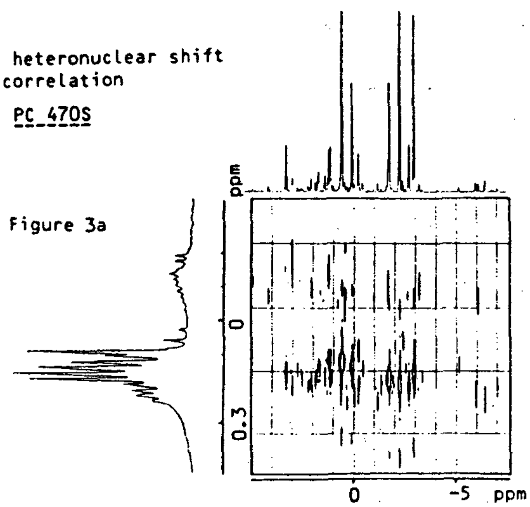

Figure 3a

^{1}H-^{29}Si heteronuclear shift correlation

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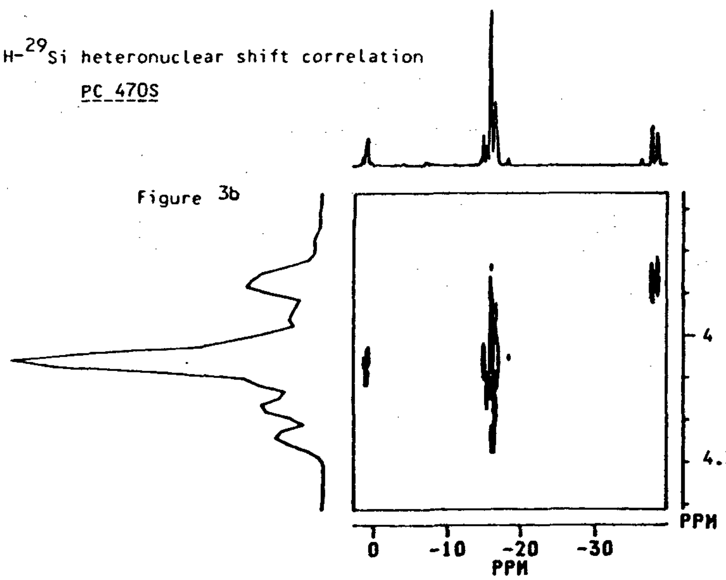

Figure 3b

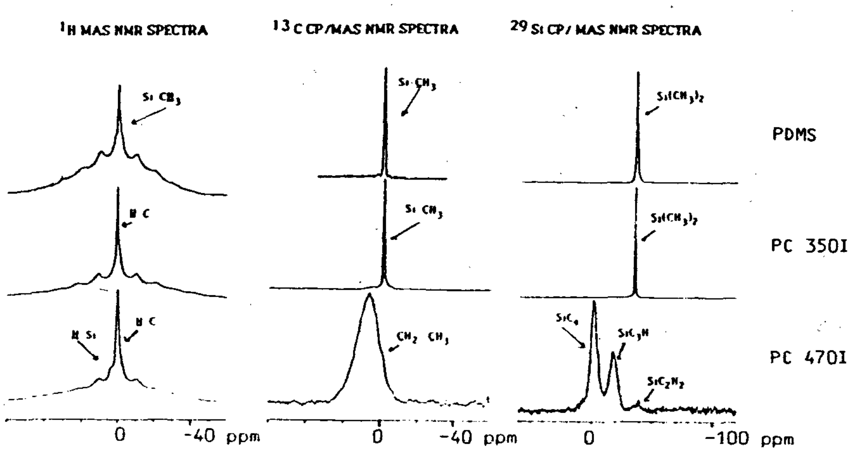

Figure 4a Figure 4b Figure 4c

Microwave Hysteresis in ESR Measurements in Copper-Oxide Superconductors:Y-Ba-Cu-O

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I. Introduction

The observation of so-called "related phenomena" in magnetic resonance experiments sometimes allows one to obtain additional information about the subject of inquiry. With high-T_c superconductors it becomes clear at present that all ESR signals attributed to Cu^{2+} ions are connected with structural irregularities or different phase impurities. There is still no remarkable progress in the magnetic ion substitution experiments (1,2). So information about the superconducting state of novel compounds may come from non-resonant absorption phenomena.

We distinguish two accompanied effects which cause microwave absorption in ESR experiments at $T \leq T_c$. The low magnetic field (m.f.) signal (LFS) at $H \leq 50$ G, as shown in recent papers, could be successfully attributed to the absorption of Josephson's network based on weak intergranular links (3) or twins (4). The opposite phase of the LFS indicates that the LFS has a non-resonant origin. The high-field ($H \geq 100$ G) hysteresis results from the pinning-depinning processes taking place when the m.f. sweep direction is reversed (5). The determination of the δ(T,H) value involves relative measurements of the first derivative of the absorbed mw-power dP/dH at different m.f. sweep directions within the H_o-environment: $\delta(T,H_o)=(dP/dH^-)_{H_o}-(dP/dH^+)_{H_o}$, where "+" and "-" correspond to the linear growth and reduction of m.f., respectively. The general and partial hysteresis loops are shown in Fig. 1. Note that the $dP/dH^- \Longleftrightarrow dP/dH^+$ transition width was narrow enough ($\Delta H \approx 10^{-2}$G) to allow $\delta(H_o)$ to be measured within sufficient accuracy. Details of the behavior of other parameters are presented elsewhere (5).

II. Discussion

The significant feature to discuss is the quasi-universal temperature behavior of the hysteresis amplitude δ (Fig. 1) observed in the series of traditional superconductors (Nb_3Ge, V_3Si, $PbMo_6S_8$ etc.) $\delta(T)=\delta(0\ K)(1-\beta T/T_c)^2)$ where $\beta \approx 1$. Such behavior, together with a peculiar field dependence $\delta(H) \sim H^{-1}$, could be interpreted in terms of critical-current-density ruled processes (6). However, the stoichiometric ceramics $YBa_2Cu_3O_{6.9}$ have shown an unexpected knee at $T \approx 60$K, which is reported in (5). The question of the origin of that knee arise.

X-Ray experiments within 2% accuracy show the absence of phase compositions with different $T_{c2} < T_c$. To clarify the origin of the anomaly, we measure δ(T) dependences in ceramic $YBa_2Cu_3O_{7-x}$ with different oxygen concentration: x=0, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.9 (all the quantitites presented are chemical data). The samples with x>0.5 did not

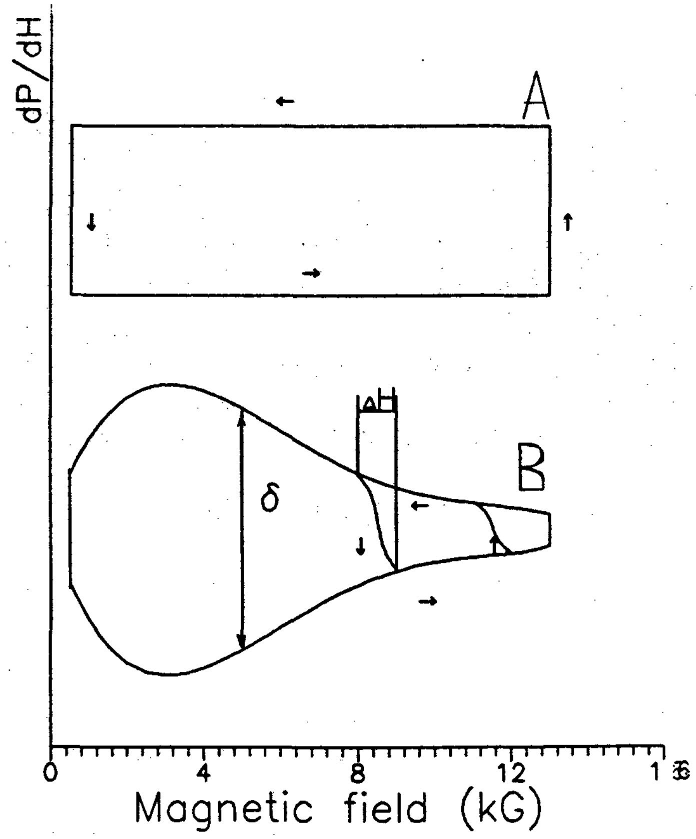

Figure 1: Field dependences of the mw-absorption dP/dH for Y-Ba-Cu-O samples in superconduction state (80K): (a) - single crystals at H||(ab) orientation and ceramics. (b) - single crystals at H⊥(ab) orientation. Partial hysteresis loop is shown schematically ($\Delta H \sim 0.01 + 5$ G depends on orientation).

show a mw hystersis, a fact which is consistent with the absence of superconductivity measured by resistivity technique.

The relative $\delta(T)$ dependences shown in Fig. 2 demonstrate a gradual degradation of the knee-like anomaly until it vanishes at x=0.5 (T_c=43K).

To describe the data presented a few possible mechanisms should be taken into account. The oxygen distribution near the granular surface may create a low-T_{c2} layer and in spite of the extremely small relative volume of the layer, could cause effective coating processes at $T< T_{c2}$. This supposition does not look realistic at present because a lot of experiments fail to find near-surface layers with low oxygen concentration in Y-Ba-Cu-O ceramics (7). A quite different approach may be considered, if the effective pinning strength shows a temperature-dependent behavior. It is resonable to assume that such a dependence is present within the high temperature flux creep approach discussed by Yeshurun and Malosemoff (8) and Iye (9).

In an attempt to investigate this question more properly, we have measured the temperature behavior of the hysteresis amplitude $\delta(T)$ in single crystals of $YBa_2Cu_3O_{6.8\pm0.1}$ at different m.f. orientations (Fig. 3). A non-monotonous field dependence $\delta(H)$ at 100 G<H<10 kG (Fig. 1) was observed for the perpendicular orientation H⊥(ab). A similar picture was observed in deformed Nb single crystals for both m.f. orientations (H_{max}(4 K)≈1.5 kG). In this case the maximum in the $\delta(H)$ dependence was shifted toward higher fields and spread as the sample was deformed (5). A comparison of magnetization data (10) and the behavior of the hysteresis amplitude maximum allows us to ascribe that maximum with the extreme of pinning force. As for H||(ab) orientation in $YBa_2Cu_3O_{7-x}$ crystals, a dependence typical of ceramics (Fig. 2) was observed. Comparing Fig. 1 and Fig. 2 data, one can notice that the appearance of maxima in $\delta(T)$ dependence (T_{max}=70 K for H_o=7.5 kG and T_{max}=55 K for H_o=3.0 kG) correlates strongly with the motion of $\delta(H)$ maximum along the m.f. axis. The field independent behavior of the $\delta(T)$ curve for Y-Ba-Cu-O ceramics is probably due to the spreading of the above mentioned $\delta(H)$ maximum over the

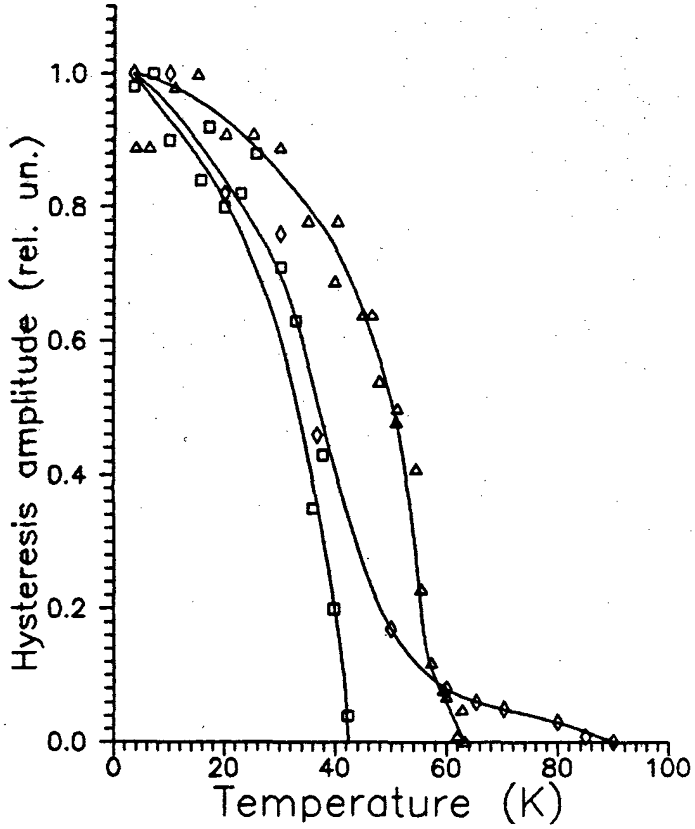

Figure 2: Temperature dependences of hysteresis normalize amplitude $\delta(T)/\delta(4\ K)$ of <u>ceramics</u> $YBa_2Cu_3O_{7-x}$: ($\diamond$) - x = 0.0, (Δ) - = 0.2, ($\square$) - x = 0.5 (H$\sim$3 kG).

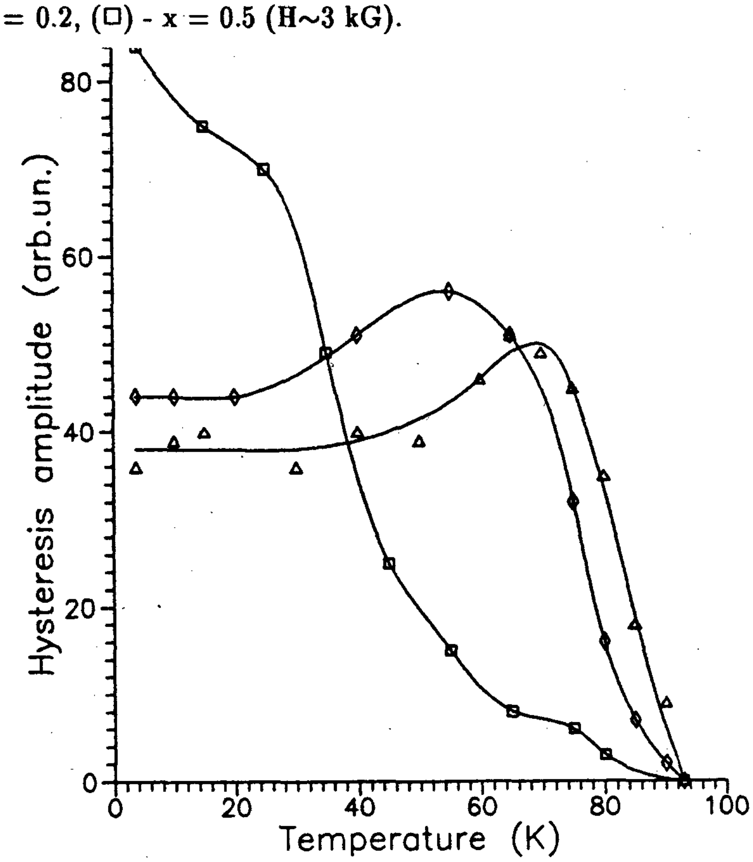

Figure 3: Thermal behavior of $\delta(T,H_o)/\delta(4\ K)$ in single crystals $YBa_2Cu_3O_{6.8}$: (Δ)-H$\perp$(ab), H_o=3 kG, ($\diamond$)-H$\perp$(ab), H_o=7.5 kG, ($\square$)- H$\|$(ab), H_o=3 kG.

broad (H>10 kG) m.f. region. This assumption appears reasonable for single crystals as well (orientation H||(ab)), because of the highly orientated twins network that is typical for Y-Ba-Cu-O compounds. Under such conditions, the δ(T) curve should not be influenced by the additional contribution of the δ(H)-maximum and a ceramic-like behavior setting in.

The high anisotropy of the superconducting parameters (ξ_c/ξ_{ab}=2/13) allows us to consider a different pinning strength in both orientations (8,9), so while strong pinning determines the δ(T) behavior for the perpendicular orientation (H||c), the same mechanisms could be suppressed for the parallel m.f. orientation (H||(ab)) because the value of ξ_c is extremely small. Recent torque anisotropy measurements made by Ashimov (11) show a sharp decrease of pinning force above T=40 K in stoichiometric ceramics. Such behavior was interpreted in terms of the ineffectiveness of a certain type of pinning centers as the temperature is varied. These assumptions may be successfully applied to weak hysteresis observed at H<100 G, if Sonin's hyper-vortexes (12) are taken into consideration. The pinning processes in this case are complicated, but a hysteresis appearance is liable to arise.

III. Concluding Remarks

On the strength of the foregoing considerations we conclude that the measurements of the mw absorption hysteresis provide an additional sensitive rapid technique for analyzing hard superconductors, especially high-T_c superconductors. This method allows us to detect a pinning anomaly in stoichiometric samples of Y-Ba-Cu-O above T≈50 K. However, further experiments and a detailed theoretical description are needed for this technique to be used for quantitative estimates.

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