

MAGNETIC ENERGY ABSORPTION IN SINTERED  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  SAMPLES

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A.c. magnetic susceptibility measurements of eight microscopically characterized sintered  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  samples are reported. The samples show losses  $0.2 > 4\pi\chi'' > 0.002$ .  $\chi''$  vs  $\chi'$  plots derived for all samples agree with models and results where flux pinning plays an important role.

1. INTRODUCTION

Low field a.c. magnetic susceptibility,  $\chi = \chi' - i\chi''$  is a good probe to study granular superconductivity. Reported  $\chi(T)$  data for high  $T_C$  superconductors in sintered and powdered samples show special features clearly related with the microstructure (1). To investigate the influence of preparation and sintering techniques on the diamagnetic response of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ , systematic measurements of a series of sintered samples obtained by different methods have been performed.

2. EXPERIMENTAL

Preparation method and other relevant parameters are presented in Table 1.  $\chi(T)$  measurements were performed in an automated susceptometer. Samples were cut into bars and oriented parallel to the applied field,  $1 \text{ mOe} < h_0 < 11 \text{ Oe}$ ,  $\nu = 120 \text{ Hz}$ .

3. RESULTS AND DISCUSSION

For samples I to VII, the onset of diamagnetism,  $h_0$  independent, starts at  $T_0$ , where the resistive transition,  $T_0(\rho)$ , does too. The strong decrease in  $\chi'$  takes place at  $T_1 = T_C(\rho=0)$ . At low temperature and low applied field the apparent volume susceptibility corrected from demagnetizing effects reaches an asymptotic value within 10% of perfect flux exclusion. At  $T_1$  a strongly field amplitude dependent peak in  $\chi''$  accompanies the transition (2). Sample VII shows a second peak in  $\chi''(T)$  at  $T_0$ .  $\chi'(T)$  and  $\chi''(T)$  curves for sample I and  $h_0 = 110 \text{ mOe}$  are given in figure 1. From inspection of table 1 the trend of increasing hysteresis losses for smaller grain size is apparent.

For sample VIII no anomaly is observed in  $\chi'$  at  $T_C = 91.8 \text{ K}$ . The lowest value of  $-4\pi\chi'$  yields only 45% of the ideal one, and it is  $h_0$  independent.  $\chi''$  values for  $h_0 = 110 \text{ mOe}$  are negligible but a sharp peak appears at  $T_0$  for  $h_0 = 11 \text{ Oe}$  (see inset of figure 1).

Since for ceramic superconductors no frequency dependence exists (3), results will be discussed assuming that magnetic energy absorption is due to hysteresis in the M-H curve of a type II superconductor. Collected results are presented in a  $\chi''$  vs  $\chi'$  diagram (figure 2).  
 3.1. The weak-links model

The sample is considered as an array of superconducting grains connected by weak-links. It can be approximated by an equivalent superconducting loop with a well defined transition temperature,  $T_{C1} = T_1$ , and a critical current  $J_{c1}$  which shows a temperature behaviour depending on the type of weak-links considered (3). All experimental diagrams lay below the theoretical

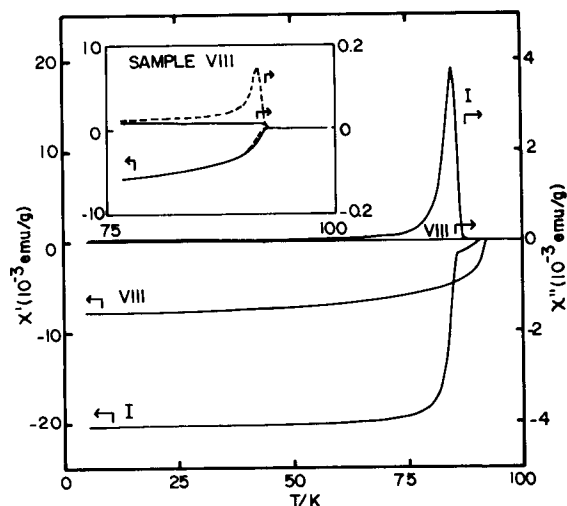


FIGURE 1  
 $\chi'$  and  $\chi''$  curves for samples I and VIII,  $\nu = 120 \text{ Hz}$ ,  $h_0 = 110 \text{ mOe}$  (full line);  $11 \text{ Oe}$  (dashed line). The inset shows enlarged area for sample VIII.

TABLE 1: Relevant parameters of the eight sintered samples

| SAMPLE | PREPARATION METHOD         | X-RAY IMPURITIES                              | GRAIN SIZE ( $\mu\text{m}$ ) | $T_0(x')$ | $T_1(x')$ | $4\pi\chi''_{\text{max}}$ |
|--------|----------------------------|---|------------------------------|-----------|-----------|---------------------------|
| I      | Liquid mix (7)             | $\text{Y}_2\text{BaCuO}_5$ , $\text{BaCuO}_2$ | 3-5                          | 90,5 K    | 85,7 K    | 0.190                     |
| II     | Solid state reaction (2)   | $\text{Y}_2\text{BaCuO}_5$                    | Inhomogeneous                | 88,0      | 75,0      | 0.150                     |
| III    | Solid state reaction       | $\text{Y}_2\text{BaCuO}_5$                    | 3-10                         | 88,5      | 88,5      | 0.110                     |
| IV     | Metallorganic precursor(8) |   | 3                            | 87,7      | 84,2      | 0.110                     |
| V      | Citrate route (9)          |   | 10-100                       | 91,5      | 85,0      | 0.060                     |
| VI     | Solid state reaction       |   | 3-10                         | 88,4      | 84,1      | 0.060                     |
| VII    | Citrate route (9)          | $\text{Y}_2\text{BaCuO}_5$                    | 10-100                       | 91,9      | 85,0      | 0.060                     |
| VIII   | Solid state reaction (10)  |   | 0.5 and Crystallites         | 92,5      | --        | 0.002                     |

curve a in figure 2, probably due to rounding effects in the magnetic hysteresis loop. These could be due to the existence of many weak loops with a random distribution of transition temperatures and critical currents centered at  $T_{c1}$  and  $J_{c1}$ . The existence of two peaks in  $\chi''$  of sample VII could be explained as caused by random distributions centered at  $(T_{c1}, J_{c1})$  and  $(T_{c2}, J_{c2})$  respectively.

The zero offset on the experimental  $\chi''$ - $\chi'$  diagrams reflects that for samples I to VII  $T_1 < T_0$ . This may be understood if a transition from a paraohereant state of superconducting grains, with transition temperature  $T_0$ , to a coherent state of weakly connected grains occurs at  $T_1 = T_c$  (4).

The negligible field dependence and the absence of anomaly in  $\chi'(T)$  for sample VIII yields the conclusion that electrical connectivity has not weak-link nature, the

shielding effect being not so efficient as in the other samples. The very small grains of average size  $0.5 \mu\text{m}$  and crystallites of  $10 \mu\text{m} \times 5 \mu\text{m}$  may be responsible of the observed behaviour. Following the model proposed in (3) we conclude that specimen I is a good example of weakly coupled grains, while specimen VIII shows mainly bulk effects.

### 3.2. The critical field model

In a type II superconductor hysteresis losses can be due to flux penetration subject to pinning forces, when  $h_0$  exceeds the lower critical field. Curve b, figure 2, corresponds to a prediction for the bulk pinning case (5). Flux pinning at low fields in the ceramic superconductors has been evidenced from magnetic hysteresis loop measurements (4).

$\chi''$  vs  $\chi'$  curves for samples I to III agree qualitatively with the model. If pinning is considered weaker in the surface layer than in the bulk core, lower hysteresis losses are expected (5), in agreement with IV-VIII curves. Finally, the existence of some of the samples of two peaks in  $\chi''(T)$  has been already explained as caused by two superconducting components with different critical fields (6).

### ACKNOWLEDGEMENTS

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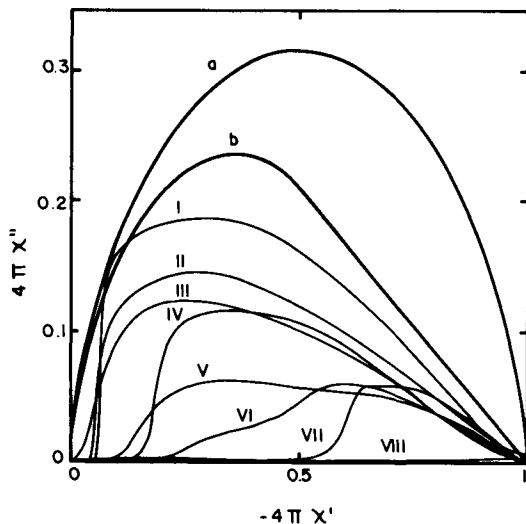


FIGURE 2

$\chi''$  vs  $\chi'$  diagrams for samples I to VIII. Curve a: Weak-links single loop model. Curve b: simplest bulk pinning case of the critical field model.