phys. stat. sol. (a) 120, K (1990)
Subject classification: 74.70: S10.15

Laboratory of Superconductivity, University of Havana[1])

Hysteretic Critical Currents in Y-Ba-Cu-O Superconductors: a Microstructural Approach

By
E. ALTSHULER, S. GARCÍA, and A. AGUILAR

Critical current measurements have been extensively performed for the superconducting system Y-Ba-Cu-O as a standard part of its transport characterization /1 to 4/, but the hysteretic behaviour of the J_c versus B curves has been much less studied. Here we study this behaviour in relation to metallographic pictures of the samples with the aim of interpreting some features of those curves in terms of sample microstructure.

Samples were prepared by standard techniques using high purity Y_2O_3, $BaCO_3$, and CuO powders. Sample 1: three pre-firing treatments at 950 ^oC, extra-firing of compact disks at 800 ^oC for 2 h, sintering at 950 ^oC, and slow cooling (1 K/min) to room temperature in oxygen flow, including 6 h annealing at 650 ^oC. Sample 2: same procedure, save extra-firing before sintering.

I_c values measured at liquid nitrogen temperature were taken as those corresponding to an electric field of approximately 10^{-6} V/cm in the standard four-probe measurement method. Firstly, a J_c versus B curve was obtained for each sample by increasing the magnetic field from B = 0 to a certain B_{max}, in which the sample was kept for a few seconds; then a second J_c versus B curve was obtained by decreasing the magnetic field from B_{max} to B = 0, after which the sample was taken to room temperature. The whole process was performed three times for each sample, corresponding to three different values of B_{max} (3, 6, and 9 mT).

To take the required microstructural photographs of the samples, these were previously ground, polished with diamond paste and absolute ethanol, and gently etched in a HCl solution. Both samples showed rectangular grains (defined as regions containing sets of parallel twin planes) with a great variety of areas ranging from 10 to 1000 $1 \mu m^2$ in good agreement with /3/. Pores both inter- and intragrain were also observed.

Fig. 1 shows the J_c versus B experimental curves, where the values of J_c for B = 0 are substantially different for the two samples (≈ 300 A/cm^2 for sample 1 and ≈ 160 A/cm^2 for sample 2). At 800 ^oC, the first steps of a homogeneous sintering process take place with a higher equilibrium oxygen concentration than for 950 ^oC. Thus, it may well be that a more convenient microstructure is

[1]) IMRE, S. Lázaro y L, 10400 Habana, Cuba.

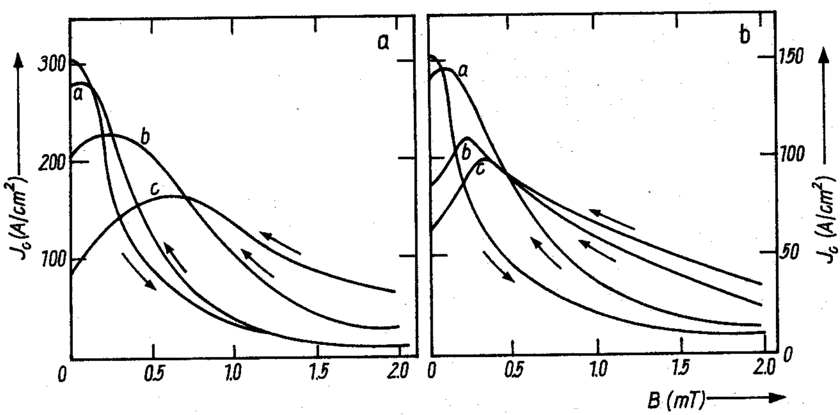

Fig. 1. J_c curves for samples a) 1 and b) 2. Arrows indicate increasing or decreasing B. Curves a, b, and c correspond to decreasing B curves with B_{max} = 3, 6, and 9 mT, respectively

stabilized in sample 1 as compared with the one resulting from direct sintering of sample 2 at 950 ^{o}C, for which a more intense intercalation process of oxygen through cooling will occur, possibly generating a higher density of crystalline defects, twinning planes, microcracks, or intergrain links of lower quality.

The general shape of the J_c versus B curves for a given sample shows the following features: 1) For increasing B the curves first decrease abruptly from B = 0 to B ≈ 0.5 mT, and then slowly approach J_c = 0 for higher values of B. 2) Every curve obtained for decreasing B shows a relative maximum which is smaller in value and more shifted to the right the larger B_{max} is. These features agree with those described in Evetts and Glowacki's extensive work /5/, and can be explained, as they do, in terms of the effect produced by the flux expelled or trapped by the superconducting grains at the weak links between the grains, thus controlling their capability of carrying transport currents.

As Küpfer et al. /6/ have shown similar effects occur not only in grains and intergrain links but also in intragrain decoupled regions and intragrain junctions. However, since intragrain J_c's are of the order of 10^6 to 10^7 A/cm^2 for our materials, hysteretic intragrain effects will not be considered here.

Normalized J_c versus B curves for samples 1 and 2 corresponding to two values of B_{max} are shown in Fig. 2 where in the first place it is seen that for B_{max} = 9 mT, as compared to sample 2, the maximum for sample 1 occurs at a higher value of the external field. This could be explained by assuming that sample 1 is capable of trapping more flux than sample 2 (which is more visible at a higher B_{max}),

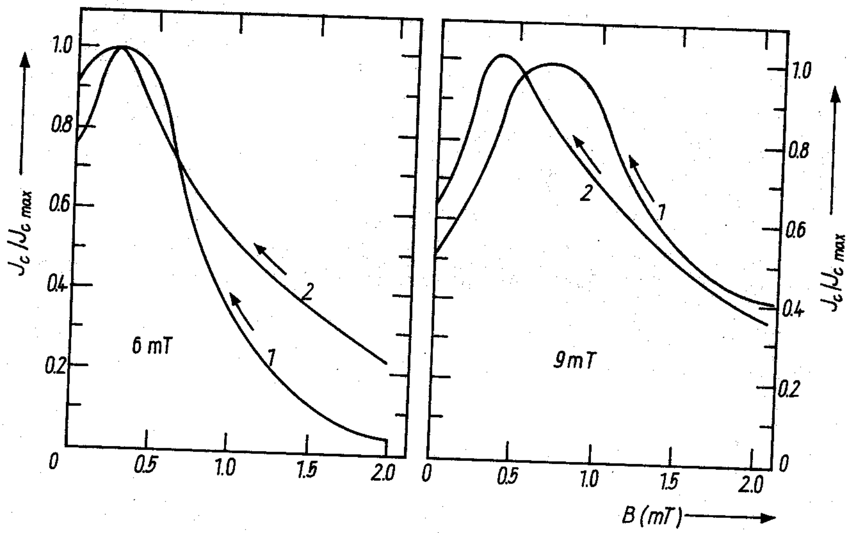

Fig. 2. Comparison of normalized J_c versus B behaviour between samples 1 and 2 for decreasing B with different values of B_{max}

since a curve peak corresponds to that value of the external field which produces maximum cancellation of the flux trapped by the grains at the weak links, a conclusion which is coherent with our previous assumption that the grains of sample 1 are less defective than those of sample 2.

As to the different widths of maxima shown by the curves, the larger widths may well be due to broader statistical distributions of "cancellation configurations" between the external field and the flux trapped at the links. The character of those distributions could be geometrical, intrinsic, or both. Geometrically, the existence of a great variety of grain link sizes and shapes would offer a broader distribution of "cancellation configurations" including the effect of many different demagnetization factors for the grains, all of which might give rise to a wider maximum. On the other hand, a broader distribution of J_c's for the individual weak links and for the grains could produce a similar effect.

Since from our microstructural photographs we were not able to detect any significant difference in the statistical geometrical distribution of grains and links between samples 1 and 2, we conclude that pure geometrical features are of little consequence to the widening of the maximum of our curves, so that the intrinsic superconducting properties of grains and links must be much more significant for the explanation of the J_c versus B differences shown by our samples.

REFERENCES

/1/ H. KÜPFER, I. APFELSTEDT, W. SCHAUER, R. FLÜKIGER, R. MEIER-HIRMER, and H. WÜHL, Z. Phys. B 69, 159 (1987).

/2/ J.W. EKIN, A.I. BRAGINSKY, A.J. PANSON, M.A. JANOCKO, D.W. CAPONE II, N.J. ZALUCEK, B. FLANDERMEYER, O.F. DE LIMA, M. HONG, J. KWO, and S.H. LIOU, J. appl. Phys. 62, 4821 (1987).

/3/ R.L. PETERSON and J.W. EKIN, Phys. Rev. B 37, 9848 (1988).

/4/ J. HALBRITTER, Internat. J. mod. Phys. 3, 719 (1989).

/5/ J.E. EVETTS and B.A. GLOWACKI, Cryogenics 28, 641 (1988).

/6/ H. KÜPFER, I. APFELSTEDT, R. FLÜKIGER, C. KELLER, R. MEIER-HIRMER, B. RUNTSH, A. TUROWSKI, U. WIECH, and T. WOLF, Cryogenics 29, 268 (1989).

(Received March 6, 1990)