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Superconducting Properties of Polycrystalline YBCO Prepared by a Pyrolytic Process (*).

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Summary. — Polycrystalline YBCO was prepared by a pyrolytic process starting from citrate and tartrate precursors. The effect of the precursor on the superconducting properties was investigated by means of magnetic measurements using a SQUID magnetometer, a Vibrating-Sample Magnetometer and an a.c. susceptometer. The critical temperature is not affected by the type of precursor (T_c = 85 K for both). On the other hand, the precursor plays an important role on the critical current density, which is found to be an order of magnitude higher in the sample obtained from citrates (J_c(77 K, H = 0) = $2.3 \cdot 10^4$ A/cm^2 and 10^3 A/cm^2 for the from-citrate and from-tartrate sample, respectively).

PACS 74.70 – Superconducting materials.
PACS 74.60.Mj – Material effects on T_c, κ, critical currents (including impurities, ion implantation etc.).
PACS 82.30.Lp – Decomposition reactions (pyrolisis dissociation and group ejection).
PACS 01.30.Cc – Conference proceedings.

1. – Introduction.

Among the different methods to prepare bulk high-T_c superconducting materials (ceramic, melt textured, sol-gel, etc.) the use of suitable precursors has received a great attention in the last few years [1-4]. In particular, the pyrolysis of organic as well as inorganic precursors has been developed succesfully for YBCO and BSCCO [5, 6], giving rise to ultrafine and high-reactive powders. Pyrolysis powders can be calcined and, after pressing, sintered in a controlled atmosphere. The resulting material is a granular bulk HTSC pellet.

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In this work we have developed the pyrolysis method to produce YBCO bulk materials. Three different materials were prepared starting from citrates and tartrates as precursors. The superconducting properties are compared and the role of the precursor is discussed.

2. – Results and discussion.

2·1. *Sample preparation.* – Basically the preparation process is the following: nitrates were formed by adding nitric acid to a CuO, Y_2O_3 and $BaCO_3$ mixture (purity of 99.99%). The addition of citric acid or tartaric acid to the solution leads to citrates (sample *A*) or tartrates (sample *B*). The *p*H value is adjusted (~ 6.8) by adding 25% ammonia. By heating the solution at about 250 °C the boiling mud shows a spontaneous-combustion reaction. The final result of the combustion process was a very black powder with granulometry of the order of 100 nm. Pyrolyzed powders were calcined at $T = 900$ °C for 10 hours and after crushed, sieved and then pressed in cylindrical shape (pellets) at about 200 kg/cm^2. Finally, the pellets were sintered in an ozone-enriched atmosphere at $T = 900$ °C for 10 hours. A third sample (*C*) was prepared by adding citric acid to commercial nitrate powders and then following the previous procedure. XRD shows that the materials consist of YBCO single phase.

2·2. *Magnetic characterization.* – The superconducting properties were investigated using a commercial SQUID magnetometer ($2 \leqslant T \leqslant 400$ K; $H_{max} = 5.5$ T), a vibrating-sample magnetometer ($77 \leqslant T \leqslant 300$ K; $H_{max} = 1.4$ T), and an a.c. susceptometer ($77 \leqslant T \leqslant 300$ K; $\nu = 200$ Hz, $H_{a.c.} = 1$ Oe). The critical temperature was taken as the onset of the diamagnetism in the a.c. susceptibility measurements (fig. 1.). The intragrain critical current density was extracted from the hysteresis cycles (fig. 2) by using the Bean critical-state formula, ($J_c = 15\,\Delta M/d$, where ΔM is the width of the hysteresis cycle and d the pellet dimension perpendicular to the applied field).

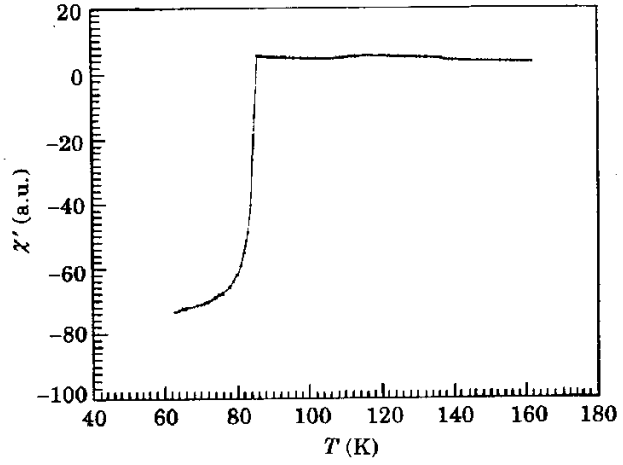

Fig. 1. – A.c. susceptibility as a function of temperature for the sample *C*.

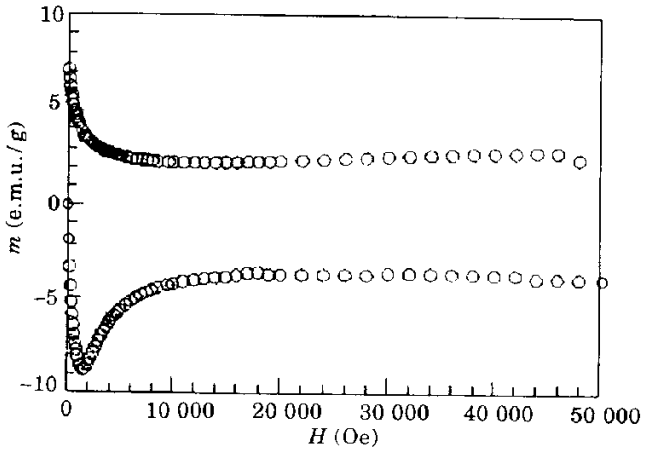

Fig. 2. – Hysteresis cycle at $T = 20$ K for the sample A.

The results show that the critical temperature is not affected by the precursor ($T_c = 85$ K for the three samples). The low-T_c values could be due to unsufficient oxygenation in the samples. Differences are observed, however, in the diamagnetic signal at 77 K, which is much lower for the sample obtained starting from tartrates, revealing a lower flux exclusion.

Hysteresis cycles measurements were performed at low temperature by the SQUID magnetometer (see, for example, the cycle at 20 K in fig. 2) and at 77 K by the vibrating sample magnetometer (fig. 3, inset). The field dependence of the critical current density at 77 K is reported in fig. 3. The critical current density is an order of magnitude higher in the sample obtained from citrates and from oxides ($J_c \approx 2.3 \cdot 10^4$ A/cm^2 in zero field). Such differences should not be related to a macroscopic difference in oxygen content between the samples, the T_c values being identical in the

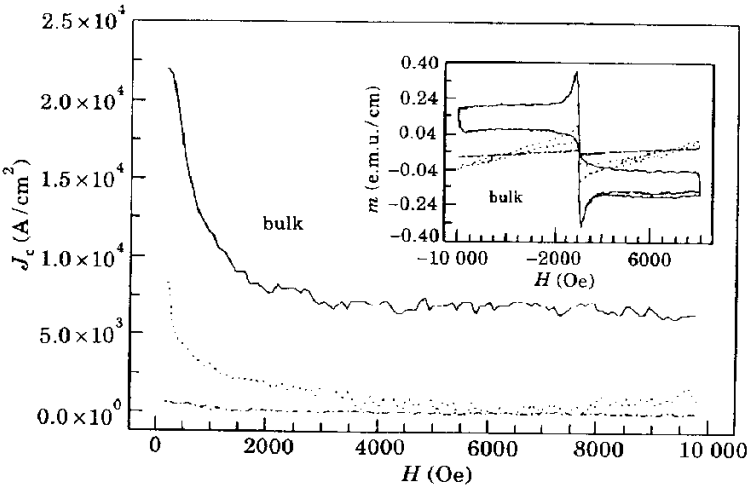

Fig. 3. – Field dependence of the critical current density at $T = 77$ K. Inset: hysteresis cycles at $T = 77$ K. —— A, ····· C, -·-·- B.

three samples. The X-rays diffraction patterns do not show significant differences between the three samples. In principle a different density of twin planes, acting as pinning centres, could be responsible for different J_c values. However, in the absence of microstructural investigations it is difficult to draw conclusions about the reasons of the J_c differences.

3. – Conclusions.

We have investigated the role of the precursor in the preparation of the polycrystalline YBCO by pyrolitic process. The results indicate that while the critical temperature is not affected by the precursor, the critical current density is an order of magnitude higher in the sample obtained using citrates as precursors and starting from the oxides.

REFERENCES

[1] S. JIN, T. H. SHERWOOD, M. E. DAVIS, R. B. VAN DOVER G. W. KAMMLOTT, R. A. FASTNACHT and H. D. KEITH: *Appl. Phys. Lett.*, 53, 2074 (1988).
[2] K. SALAMA, V. SELVAMANICKAM, L. GAO and K. SUN: *Appl. Phys. Lett.*, 54, 2352 (1989).
[3] M. MURAKAMI, M. MORTA, K. DOI and K. MIGYAMOIN: *Jpn. Appl. Phys. Lett.*, 28, 1189 (1989).
[4] Y. FENG, L. ZHOU, P. ZANG, P. JI, X. WU, CH. LOU and X. XIN: *J. Supercond.*, 2, 95 (1992).
[5] J. FLOKSTRA, G. J. GERRITSMA, D. H. A. BLANCK and E. KEIM: *Proceedings of the European Workshop on High T_c Superconductors and Potential Applications, Genova 1-3 1987*. (Academic Press, London, 1987), p. 429.
[6] F. CELANI, L. FRUCHTER, C. GIOVANNELLA, R. MESSI, S. PACE, A. SAGGESE and N. SPARVIERI: *IEEE Trans. Magn.*, 25, 2348 (1989).