



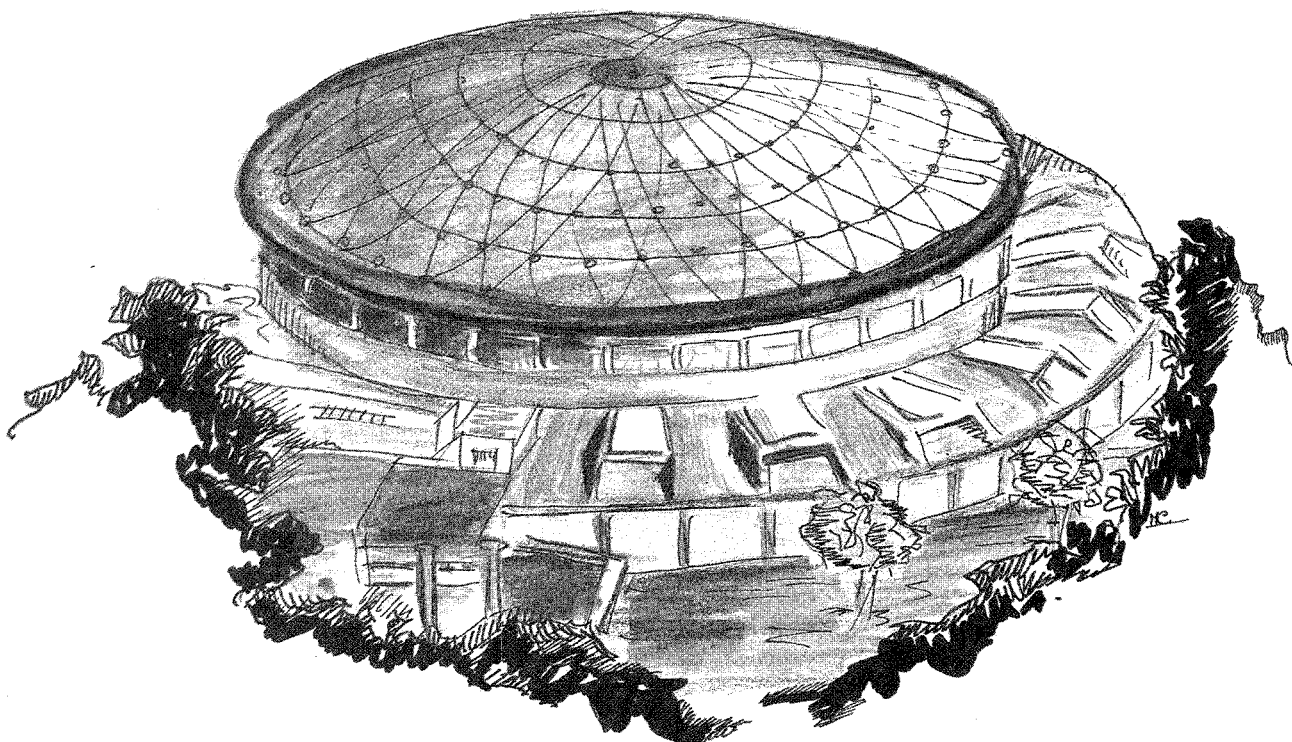
Laboratori Nazionali di Frascati

Submitted to Physica C

LNF- 89/092(P)
22 Dicembre 1989

F. Celani, A. Saggese, V. Calzona, M. Putti, L. Liberatori, S. Pace, B. Polichetti,
R. Scafuro:

**IMPROVEMENTS OF SINTERED YBCO SAMPLES BY CITRATE
PYROLYSIS AND OZONE ANNEALING**



Servizio Documentazione
dei Laboratori Nazionali di Frascati
P.O. Box, 13 - 00044 Frascati (Italy)

**IMPROVEMENTS OF SINTERED YBCO SAMPLES BY CITRATE
PYROLYSIS AND OZONE ANNEALING**

F. Celani, A. Saggese
INFN Laboratori Nazionali di Frascati, P.O. Box 13, 00044 Frascati (Italy)

V. Calzona, M. Putti
Dept. of Physics Univ. of Genova, Via Dodecaneso 33, 16146 Genova (Italy)

L. Liberatori, S. Pace, B. Polichetti and R. Scafuro
Dept. of Physics Univ. of Salerno, Loc. Lancusi, 84100 Salerno (Italy)

YBCO sintered samples have been fabricated by the citrate pyrolysis method. The annealing, with the usual thermal processes, but using a mixture of oxygen and ozone, generates large microcrystals. Samples fabricated with this process have been characterized by measuring the a.c. susceptibility. Low field measures show a very narrow transition width, while high field measures performed both at 50 and 100 Gauss denote the higher strength of the intergranular junctions in the ozone made samples.

1. INTRODUCTION

It is a common opinion that sintered YBCO have a complex behaviour caused by the presence of grains and weak-link between them. The weakness of the weak-link in the sintered YBCO pellets is a serious handicap for all applications that require high current densities. In this paper, together with a.c. susceptibility measurements a process able to improve the sample performances is presented.

2. FABRICATION AND MEASUREMENTS

Our fabrication procedure consists in the realization of a YBCO powder, by using the pyrolysis followed by a thermal treatment with an oxygen atmosphere enriched with a low percentage of ozone⁽¹⁾. The procedure generates more uniform mixture and thinner powder with granulometry from 50 to 100 nm. Moreover the higher reactivity of the ozone in the thermal treatments ensures a stronger oxygenation. Pellets were fabricated by using the previously annealed powder and an hydraulic press. On these pellets a final thermal treatment, as previously described, is executed. The ozone treatments produce the following effects: increase the pellet density achieving values up to 5.9 gr/cm^3 , cut down the spurious phases. Moreover sometimes an enlargement of the grain dimensions (Fig. 1) and a texture is obtained⁽²⁾. Low field a.c. susceptibility measurements were performed by using a two coil system. The sample was measured with an a.c. magnetic field of 0.2 Gauss and 59 Hz frequency. The observation in Fig. 2 of the real part of the susceptibility gives a transition width of only 1K and the complete shielding just 2K below the critical temperature T_c . The presence, show in Fig. 3, of a single sharp peak in the imaginary part denotes the inseparability, at this low field, between grain and junction transitions.

FIG. 1 - Microscope picture of YBCO crystals observed in polarized light (—— = 40 μm).

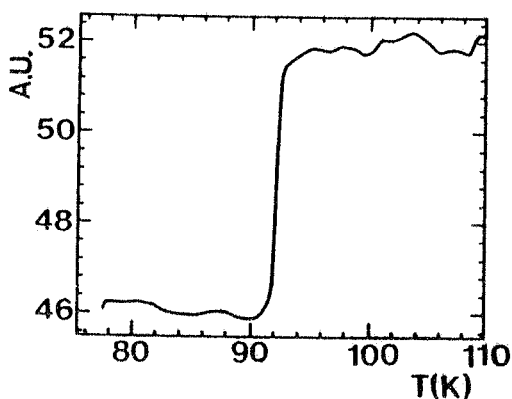


FIG. 2 - Real part of the low field susceptibility. $H = 0.2 \text{ Gauss}$, $f = 59 \text{ Hz}$.

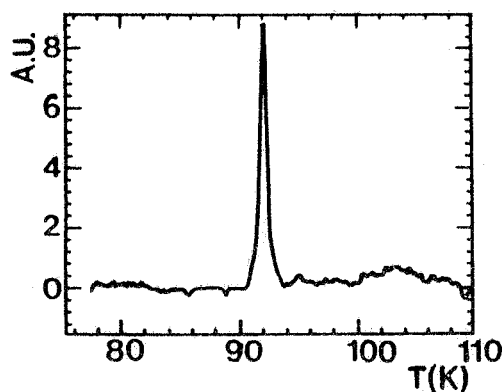


FIG. 3 - Imaginary part of the low field susceptibility. $H = 0.2 \text{ Gauss}$, $f = 59 \text{ Hz}$.

3. HIGH FIELD RESULTS

After the low field characterization the superconducting shielding at 77K was measured. For d.c. fields as high as 200 Gauss, as shown in Fig. 4, the screening does not reduce significantly. Later this sample was packed, without any care, into a plastic box. After three months, the previously measured sample was cut in several blocks with dimensions 11 x 4 x 1 mm and measured at high field by using a high sensitivity mutual inductance susceptometer⁽³⁾. The sample was measured with a.c. magnetic fields of 20, 50 and 100 Gauss. The results are shown on Fig. 5 where the imaginary part shows a first peak related to the grain transitions and, at lower temperature, a second peak associated to the weak link and then related to the macroscopic currents. In the same temperature range the behaviour of the real part of the susceptibility between 50 and 100 Gauss does not change appreciably. Further considerations on the 20 Gauss measurement, shown in Fig. 5, allow to estimate the mean critical current density⁽³⁾. At 82K this value is about 400 A/cm².

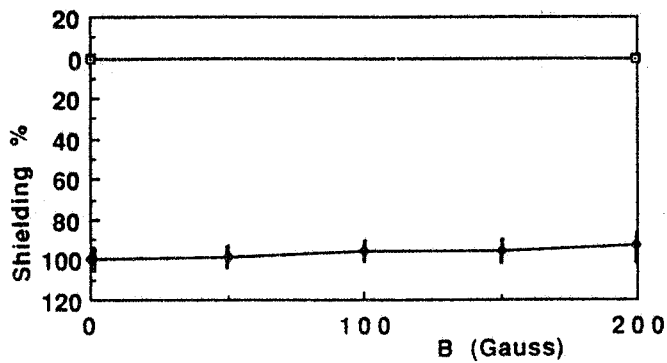


FIG. 4 - Superconducting shielding at 77K as function of the d.c. magnetic field.

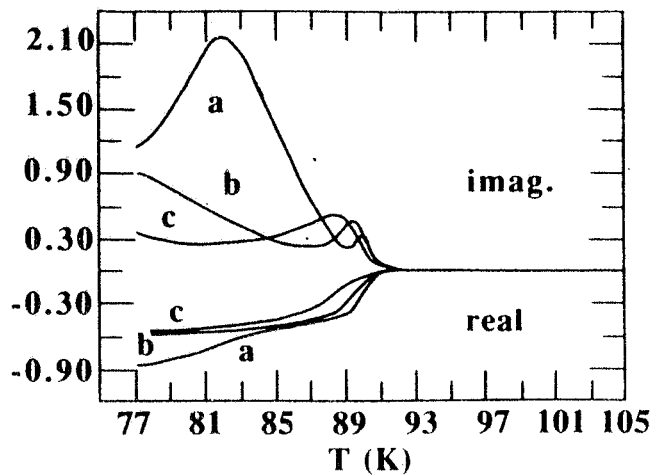


FIG. 5 - High field susceptibility measurement. Top: imaginary part magnified by a factor 10. Bottom: real part (- 1 is the full shielding). $f = 20$ Hz, $H = 20$ (a), 50 (b), 100 (c) Gauss.

4. CONCLUSIONS

In conclusion the different behaviour of measures on Fig. 4 and Fig. 5 is not clear but, between them, the sample was cut and badly stored. In spite of that, high field measures proved that links between grains in samples made with pyrolysis and ozone are very strong.

ACKNOWLEDGMENTS

The authors acknowledge prof. A.S. Siri for kindly permitting the use of his apparatus. We thank also D. Riondino for his quick and skilful drawings.

REFERENCES

- (1) F. Celani, R. Messi, S. Pace and N. Sparvieri, *Il Nuovo Saggiatore* **4**, 7 (1988).
- (2) F. Celani, L. Fruchter, C. Giovannella, R. Messi, S. Pace, A. Saggese and N. Sparvieri, *Proc. of the Applied Superconductivity Conference ASC88* in print on *IEEE Trans. on Mag.* 25 (1988).
- (3) V. Calzona, M.R. Cimberle, C. Ferdeghini, M. Putti and A.S. Siri, *Physica* **C157**, 425 (1989).