

Superconducting properties of doped and off-stoichiometric $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ single crystals

G. Balakrishnan, D.M^cK. Paul and M.R. Lees

Department of Physics, University of Warwick, Coventry CV4 7AL, UK

High quality single crystals and grain aligned rods of off-stoichiometric Bi-2212 superconductor have been grown by the 'floating zone' method using an infra-red image furnace. Growths starting from off-stoichiometric compositions have been performed to optimize the size of the crystals and their superconducting properties. Aligned rods and single crystals of the Y doped compound, $\text{Bi}_{2.2}\text{Sr}_{1.64}\text{Ca}_{0.96}\text{Y}_{0.2}\text{Cu}_2\text{O}_8$, have also been grown successfully and characterized. The effects of annealing and oxygen stoichiometry on the superconducting properties of these crystals are presented.

1. INTRODUCTION

Single crystal specimens are essential for any experiment which attempts to probe the character of the superconducting or the "normal" state in high temperature superconductors and their magnetic analogues. The extreme anisotropy of such materials also requires that at least heavily textured or aligned material is necessary for many of the possible applications. We have used an Infra-Red Image furnace to successfully produce large aligned rods and high quality single crystals of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ (BSCCO) using a "floating zone" method [1]. This technique is particularly well adapted for the growth of oxide materials [2-4] and has advantages over other methods since there is no contamination from supporting crucibles, as none are used, and the crystals produced are easily separated from the bulk and have large, flux-free surfaces. This paper describes the optimization of the crystal growth of some off-stoichiometric and Y doped BSCCO single crystals by the floating zone method and their superconducting properties.

2. EXPERIMENT

The single crystals were grown by the "floating zone" method using a double ellipsoi-

dal infra-red image furnace. A description of the furnace and the crystal growth procedures can be found in an earlier paper [1]. Several starting compositions were tried to optimize the conditions required to obtain large crystals. Starting compositions where the compositions were varied from 2:2:1:2, by changing the Sr/Ca ratio as in 2.2:1.64:1.16:2 were tried. As this method is well suited to produce homogeneous impurity contents in crystals, we have tried to grow crystals of the Y doped compound, $\text{Bi}_{2.2}\text{Sr}_{1.64}\text{Ca}_{1.16-x}\text{Y}_x\text{Cu}_2\text{O}_8$, for $x=0.2$. All the crystal growths were carried out at the rate of 0.5 mm/h, in one atmosphere of oxygen gas, with the feed and the seed rods counter rotated at 10 rpm. Growth rates of 1 to 1.5 mm/h were sufficient to produce aligned rods in all cases.

3. RESULTS AND DISCUSSION

The boule processed by the floating zone method is a well aligned rod, with the growth axis (rod axis) being along the 'a' axis. The alignment is well defined to within 10-14 degrees, determined by measuring a neutron rocking curve about the axis. The grown boule may be cleaved longitudinally to produce a large number of crystals. The largest BSCCO crystals were obtained for the off-stoichiometric starting composition, 2.2:1.64:1.16:2. Crystals with edge lengths of up to 15 mm could be separated out. Of

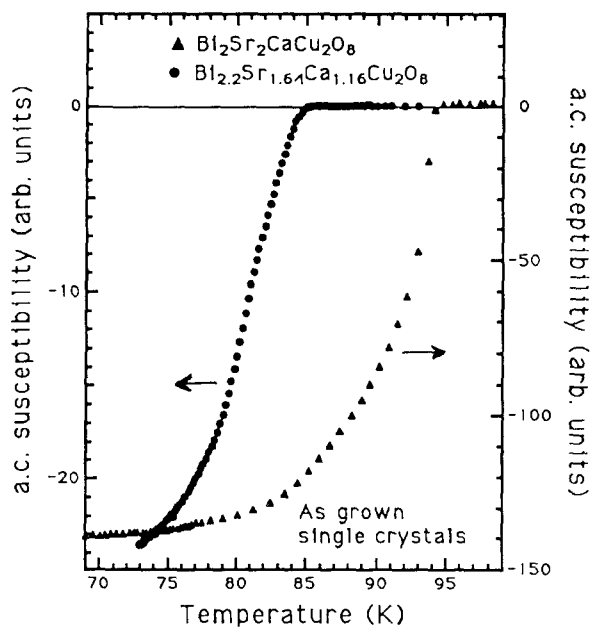


Fig. 1 Superconducting transitions of single crystals of two different stoichiometries measured by ac susceptibility

the Y doped material, the crystals obtained were smaller, the average size being $2 \times 3 \times 0.1 \text{ mm}^3$.

The superconducting transition temperatures of the crystals were determined by AC susceptibility and resistance measurements. The pure BSCCO 2:2:1:2 crystals exhibit sharp transitions with onsets at 93K. The T_c s may be altered by controlling the oxygen content in the crystals. The crystals of the 2.2:1.64:1.16:2 composition show comparatively broad transitions, with onsets at 85K. Here again the T_c depends on the oxygen content, slightly oxygen deficient crystals show sharper transitions, at 87K. Fig.1 shows a comparison of T_c s of BSCCO crystals of the two different starting compositions. The Y doped crystals are superconducting for Y=0.2, the as grown crystals show relatively broad transitions. This is probably due to the disorder in the system due to the doping. The superconducting transitions of the Y doped crystals meas-

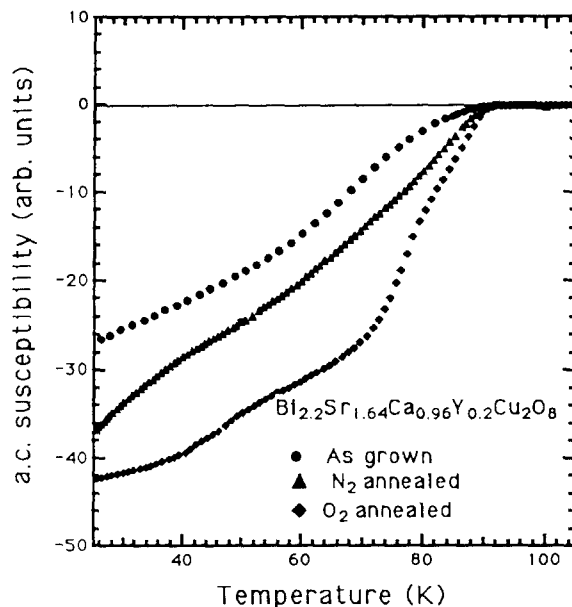


Fig 2. Superconducting transitions of Y-doped single crystals, after various annealing treatments, measured by ac susceptibility.

ured by a.c.susceptibility on the as grown and annealed crystals are shown in Fig.2. The transitions could be made sharper by annealing in oxygen; nitrogen annealing does not raise the T_c as much as it does in the pure BSCCO crystals [1].

REFERENCES

1. G. Balakrishnan, D. M^cK. Paul, M.R. Lees and A.T. Boothroyd, *Physica C* 206 (1993) 148
2. I. Tanaka and H. Kojima, *Nature* 337 (1989) 21
3. I. Tanaka, K. Yamane and H. Kojima, *J. Cryst. Growth* 96 (1989) 711
4. I. Shigaki, K. Kitahama, K. Shibutani, S. Hayashi, R. Ogawa, Y. Kawate, T. Kawai, M. Matsumoto and J. Shirafuji, *Jpn. J. Appl. Phys.* 29 (1990) L2013