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LETTER TO THE EDITOR

Thin-film Bi–Sr–Ca–Cu–O superconductor preparation using a single resistive evaporation source

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Abstract. Thin films of the high-T_c superconducting family Bi–Sr–Ca–Cu were prepared by the use of a conventional vacuum system housing a single resistively heated source. A mixture of CaF_2, SrF_2, Cu and Bi pulverised and weighed to yield a stoichiometric $Bi_{2.3}Sr_{1.5}Ca_1Cu_{3.9}$ film was evaporated from a single tungsten boat onto well polished $SrTiO_3$ and ZrO_2 substrates held at room temperature. The films thus obtained were annealed in a preheated furnace in two consecutive stages. The first one was at 725 °C for 15 minutes in flowing O_2 bubbled through water and the second one was at 850 °C for a shorter time. The films grown on ZrO_2 substrates exhibited better electrical characteristics compared with the ones grown on $SrTiO_3$.

Because of their application in microelectronics and many other fields, extensive efforts have been made to prepare high-T_c superconducting thin films. Since the recent discovery of the new family of Bi–Sr–Ca–Cu oxide superconductors [1] thin films with many different compositions have been grown by a variety of methods such as three-gun sputtering [2], sequential electron-beam evaporation [3], RF sputtering [4], ion-beam deposition [5], spin-on coating [6], co-evaporation [7] and ArF excimer-laser ablation [8]. However, these techniques require many elaborate peripheral accessories such as thickness and stoichiometry monitoring systems along with control equipment. A simple evaporation system without any electron-beam or control system will reduce film production costs and will make it even more practical and applicable. In this Letter a study of the preparation of superconducting Bi–Sr–Ca–Cu oxide thin films using a simple conventional vacuum system equipped with a single resistive evaporation source is presented.

The films were made in a conventional vacuum system housing only one resistively heated tungsten boat [9]. The starting materials, Bi, CaF_2, SrF_2 and Cu powder, were weighed in atomic proportions to yield a stoichiometric $Bi_{2.3}Sr_{1.5}Ca_1Cu_{3.9}$ film. The pulverised mixture, after being well ground in a pestle and mortar, was placed in the boat at a distance 6 cm below the substrate holder. The substrates used in this experiment were well polished and clean $SrTiO_3$ and ZrO_2 held at room temperature during evaporation. No thickness monitor or any control system were used other than evaporation to completion. The vacuum chamber was flushed with oxygen, then pumped down to a typical pressure of 10^{-5} Torr and kept at this pressure during the evaporation time which lasted for 30 minutes altogether. The films thus obtained were inserted into a preheated

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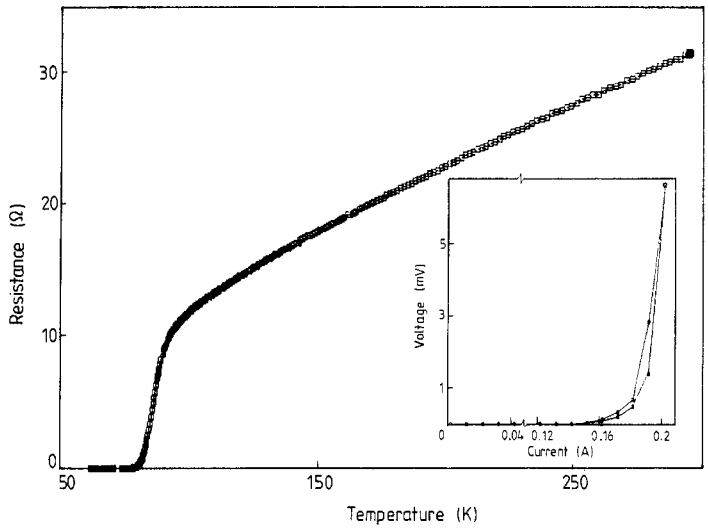

Figure 1. Temperature dependence of resistance at 1.6 mA for Bi–Sr–Ca–Cu oxide thin film grown on a ZrO_2 substrate. Inset: voltage–current characteristic at the liquid-He temperature taken with current increasing and decreasing. Thus the double voltage values reflect some instability in temperature.

tube furnace with flowing oxygen bubbled through water (to remove the fluorine [10]) for about 15 minutes at 725 °C, followed by 5 minutes at about 840 °C in an atmosphere of flowing dry oxygen for crystallisation and then quenched by removing the sample from the quartz-tube furnace.

A standard four-point DC method was used for resistance–temperature and current–voltage characterisations. Voltage values were averaged for both direct and reverse current direction to eliminate any possible thermal EMF effects. Electrical contacts were made by pressing indium onto the film. A typical film thickness was measured to be around 1.5 μm. The temperature dependence of the resistance at a current density of 20 A cm^{-2} is shown in figure 1. Zero resistance was first detected at 78 K. The inset in this figure shows the *I–V* curve of the same sample at the liquid-He temperature from which a critical current density of 2000 A cm^{-2} was deduced using 1 μV voltage onset as its measure.

It should be noted here that films grown on ZrO_2 substrates exhibited superior properties to those grown on $SrTiO_3$ substrates, as was found when two such films of the same run and post-annealing treatment were compared. The zero-resistance onset of the film on ZrO_2 is 10 K higher and the critical current density at the liquid-He temperature is almost a factor of 2 greater than the corresponding parameters of the film grown on $SrTiO_3$, thus supporting the findings reported in [11]. The reverse has been observed in Y–Ba–Cu–O superconducting thin films [9], reflecting the difference in structure between the two families of superconductors.

The surface structures of the films on ZrO_2 substrates were examined with a scanning electron microscope; a picture of this is shown in figure 2. It exhibits soft contours of size about 25–30 μm on top of which is a fine texture formed by randomly oriented elongated grains, similar to the picture reported in [12], suggesting a multiphase structure for these films.

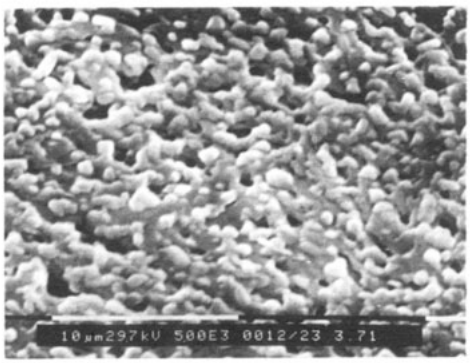

Figure 2. A typical scanning electron microscope picture of a Bi–Sr–Ca–Cu oxide film grown on ZrO_2.

In conclusion, a simple method of preparing high-T_c superconducting thin films by use of a single resistively heated source has been presented. This new method has been applied successfully to Y–Ba–Cu oxides [9] as well as to Bi–Sr–Ba–Cu oxides. Films of the latter system grown on ZrO_2 substrates displayed superior electrical properties to those grown on $SrTiO_3$ substrates.

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