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## Electrical study of an unusual phase transformation in a $Bi_2Sr_2Ca_2Cu_3O_{10+x}$ whisker at room temperature

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## Abstract

We report the observation of a phase transformation which can occur in a microscopic double phase BSCCO whisker at room temperature. We performed electrical resistivity measurements by thermally cycling the whisker in the range 78–300 K in a helium atmosphere and observed a decrease of the Bi-2223 phase amount in favour of the Bi-2212 phase, accompanied by an increase in the  $T_c$  of the Bi-2212 phase. A longer ageing of the whisker increased its resistance by about a factor of four at room temperature and caused a semiconducting behaviour at low temperature. We demonstrate that a simple electrical model can account for the experimental data and disentangle the contributions of the two phases.

## 1. Introduction

The bismuth-based family of high- $T_c$  superconductors has been extensively studied in the past 20 years, both from the point of view of its fundamental physical properties and from that of the thermodynamical stability of its different components. In fact, it is well known that the BSCCO material consists of at least three phases: Bi<sub>2</sub>Sr<sub>2</sub>CuO<sub>6+ $\gamma$ </sub> (Bi-2201), Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8+ $\delta$ </sub> (Bi-2212) and Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\epsilon$ </sub> (Bi-2223). The thermodynamical stability limits between them have been investigated in polycrystalline samples of macroscopic sizes [1, 2] and this information has been exploited in order to design the most suitable processes for the synthesis of pure samples in the different phases.

As far as bulk (i.e. with sizes greater than about  $1000 \times 200 \times 10 \ \mu \text{m}^3$ ) single crystals of the Bi-2212 phase are concerned, the effect of several thermal treatments and atmospheres on the doping and transport properties has already been studied [3–9]. The vast majority of these studies reported that temperatures higher than about 500 °C are required to induce significant changes in the structure and in the electronic features of the material, which is consistent with the results from the phase stability limit studies.

On the other hand, a few more recent papers have shown that the same modifications can also be induced in these crystals at much lower temperatures [10–12]. The reason for this discrepancy is still not clear. The necessity of longer treatment times in the latter case seems to indicate that not only the thermodynamic equilibrium conditions but also the kinetic processes play an important role. This means that some important factors might have been overlooked in the past. Among them, the crystal sizes could directly affect the experimental results if the kinetics of the phase transformation process is a limiting experimental factor.

In this framework, Bi-2212 whisker-like crystals are expected to give a significant contribution to clarifying the problem both because of their micrometric sizes along the b- and c-axis directions and because some transformations in their crystal structure have already been observed at room temperature [13]. Actually, these particular crystals have recently attracted a lot of attention. Besides having been used for the realization of submicrometric SQUIDs [14, 15], the effect of the details of the atmosphere and annealing temperature on their carrier density and on their critical current densities has been studied by many authors [16–18]. Nevertheless, the interplay between the Bi-2223 and the