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Synchrotron study of oxygen depletion in a Bi-2212 whisker annealed at 363 K

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Direct evidence is reported of structural and electronic effects induced on a single $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) whisker during a progressive annealing process. The crystal was investigated by micro X-ray diffraction (μ-XRD), micro X-ray fluorescence and electrical characterization at the European Synchrotron Radiation Facility, during a series of three *in situ* thermal processes at 363 K. Each step increased the sample resistivity and decreased its critical temperature, up to a semiconducting behaviour. These data correlate with μ-XRD analysis, which shows an increase of the *c*-axis parameter from 30.56 Å to 30.75 Å, indicating an oxygen depletion mechanism. Mild temperature annealing could be an effective process to modulate the intrinsic Josephson junctions' characteristics in Bi-2212 whiskers.

Keywords: superconductor; microfocused X-ray beam; Bi-2212; whisker; THz; oxygen doping; Kirkpatrick–Baez mirrors; micro-XRF; micro-XRD.

1. Introduction

Electromagnetic emissions in the THz range have extensive applications for information and communication technologies, biology and medical sciences, non-destructive evaluation, global environmental monitoring and ultrafast computing (Tonouchi, 2007, and references therein). Materials for THz radiation solid-state sources are presently the subject of intensive research efforts. Several studies have been published describing the possibility of producing or sensing coherent THz radiation from layered high-temperature superconductors owing to their intrinsic Josephson junctions (IJJs) properties (Wang *et al.*, 2002; Ozyuzer *et al.*, 2007; Kadowaki *et al.*, 2008). Among the possible crystalline forms, Bi-2212 whiskers, owing to their microscopic size, are promising candidates for the fabrication of miniaturized devices (Kim *et al.*, 2003; Inomata, Kawae, Kim *et al.*, 2003; Badica *et al.*, 2006). Furthermore, some authors have reported on the possibility of controlling whisker IJJ parameters, *i.e.* the critical current density (J_c) and the junction resistance (R_N), by modifying the oxygen content, which is well known to be strongly related to the carrier density (Inomata, Kawae, Nakajima *et al.*, 2003; Kawae *et al.*, 2005). In previous publications we noticed that electrical characteristics modification of Bi-2212 whiskers, owing to oxygen content variation, was accompanied by structural modifications, indirectly measured by atomic force microscopy (AFM) (Truccato *et al.*, 2005; Cagliero *et al.*, 2007, 2009). In a previous paper, where we performed a structural study, only a single severely underdoped sample was measured, so that a correlation between measured *c*-axis and electrical properties was not possible (Truccato *et al.*, 2005).

Most of the diffraction studies present in the literature refer to data acquired on clusters of whiskers (Rahman Khan *et al.*, 2009; Cagliero *et al.*, 2009). This approach increases the intensity of the diffraction peaks but has severe drawbacks owing to averaging. Moreover, many measurements are reported in the literature showing double superconductive transitions in a single whisker, usually explained by the presence of intergrowths of different phases in the Bi–Sr–Ca–Cu–O system, which are not visible by structural analysis because of the low relative volumes (Inomata, Kawae, Nakajima *et al.*, 2003; Hatano *et al.*, 2001; Matsubara *et al.*, 1989). Moreover, such types of whiskers have been found to undergo modifications of the relative amount of the two constituent phases even at low temperatures (Truccato, Cagliero *et al.*, 2006). In order to avoid misinterpretation of the experimental results, in the present work we chose a single Bi-2212 whisker, previously electrically characterized, in order