ESRF	Experiment title: Low temperature ionic mobility in micrometric BSCCO whiskers investigated by micro-XRF and Cu-K edge micro-XANES	**Experiment number**: **CH-2654**
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The Bi-Sr-Ca-Cu-O (BSCCO) system is a multilayered perovskitic structure with CuO superconducting plains intercalated by other cationic insulating plains. The $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212 phase, T_c~80K) whiskers, owing to their microscopic size and their intrinsic Josephson junctions (IJJs) properties, are promising candidates for the fabrication of miniaturized devices able to produce or sense coherent THz radiation. In previous publications we noticed that electrical characteristics modifications of Bi-2212 whiskers, owing to oxygen content variation, was accompanied by structural modifications, indirectly measured by atomic force microscopy (AFM) [1,2]. In a previous experiment at the ID22 beamline, we performed a structural study only on a single severely underdoped sample and therefore a correlation between measured *c*-axis and electrical properties was not possible [1].

Now we report direct evidence of structural and electronic effects induced on a single Bi-2212 whisker during a three-step annealing process at 363 K aimed at progressively reducing the oxygen content. The process was monitored by following the evolution of both the *c*-axis parameter (μ-XRD, Table 1 and Fig. 3) and the electrical properties (R versus T, see Fig.1) of the Bi-2212 whisker. This multi-technical investigation allowed us to directly correlate structural properties and electrical behaviour of a single monophasic whisker during the superconducting to semiconducting transformation induced by thermal annealing.

Fig. 2 shows the μ-XRF maps collected to check the chemical homogeneity of the as-grown crystal: variations of about 4% noticeable along the crystal length (which is aligned along its *a*-axis) are of the same order of magnitude of the background noise. Consequently, the crystal, at this stage, was considered to have a uniform elemental distribution. The significant variation in the ratios observed in the bottom part of each map (Y <35 mm) is due to the higher efficiency of Ag contacts in re-absorbing the lower energy fluorescence lines. μ-XRF maps, repeated after each annealing step, did not exhibit any significant difference with respect to those shown in Fig. 2 and are consequently not reported.

For each step of the annealing process we performed μ-XRD measurement, obtaining single-crystal (00l) diffraction peaks, as shown in Fig. 3. Powder rings owing to the Ag/Au contacts were also visible, which served as internal standards for adjusting both the sample-to-detector distance and the tilt angle. Two main (00l) reflection series, owing to two crystalline domains misaligned by 14°, are visible and indexed as shown in Fig. 3. Such crystalline domains are probably coupled by a small-angle grain boundary positioned along the *ac*-plane, which in principle can arise from a series of edge dislocations stacked along the *c*-axis, similar to those observed in previous crystallographic studies on Bi-2212. We decided to evaluate the *c*-axis value by averaging the parameters obtained from the two series, which are in any case compatible within the error bars. This procedure is further justified by comparison between XRD and electrical measurements (see Table 1), as the latter involved the whole crystal volume between the voltage contacts.

TABLE 1. Summary of the electrical and structural characteristics of the Bi-2212 whisker for the three annealing steps

Annealing time (h)	T_c (K)	ρ_{ab} at 275 K (Ω μm)	*c*-axis (Å)
0	79.9	3.08	30.56
6	79.7	3.50	30.68
36	-	6.19	30.75

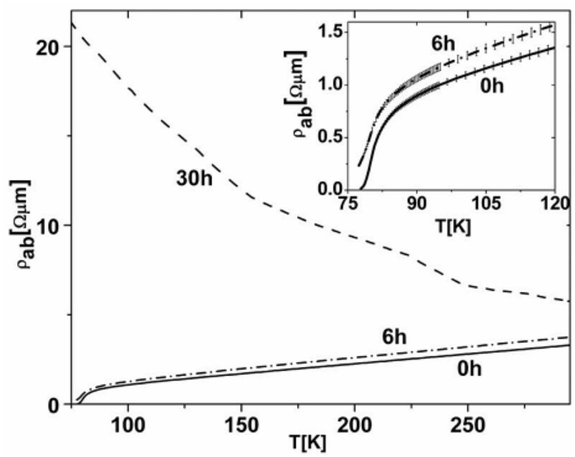

FIG. 1. Summary of the three resistivity versus T measurements obtained on the Bi-2212 whisker during the three-step annealing process: solid curve, as-grown; dot-dashed curve, after 6 h at 363 K; dashed curve, after 30 h total annealing time at 363 K. The inset shows a zoom on the transition temperature for the measurements relative to the as-grown and 6 h-annealed sample. For each step the corresponding T_c, the resistivity value at 275 K and the c-axis parameter are reported in Table 1

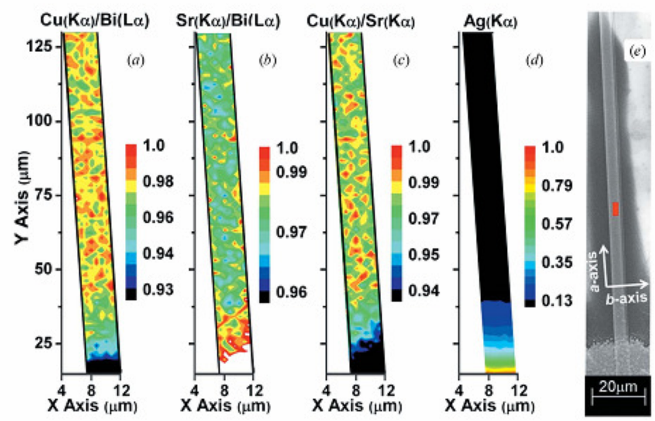

FIG. 2. Panels (a), (b) and (c) show Cu(Kα)/Bi(Lα), Sr(Kα)/Bi(Lα) and Cu(Kα)/Sr(Kα) normalized XRF count ratios, respectively. Panel (d) clarifies the Ag distribution. Panel (e) shows the SEM image of the same sample region: the red rectangle defines the beam dimension. The a- and b-axes are also shown, with the c-axis pointing out of the plane of the paper.

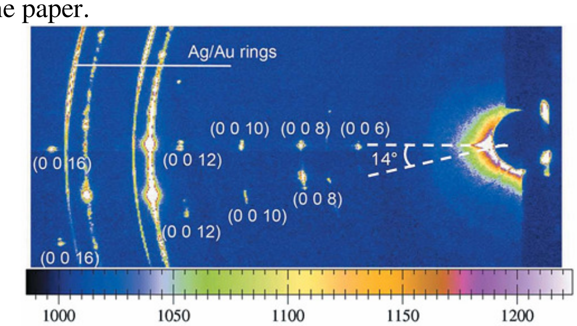

FIG. 3. μ-XRD image of the as-grown Bi-2212 crystal mounted on the chip. Two main series of (00l) reflections are visible with an angle of 14° between them. Diffraction rings from the Ag/Au contacts are also visible

Results from this experiment have been published in a recent paper. [3]

[1] M. Truccato, C. Lamberti, C. Prestipino and A. Agostino, "Evidence of ion diffusion at room temperature in micro-sized crystals of the $Bi_2Sr_2CaCu_2O_{8+\delta}$ superconductor", *Appl. Phys. Lett.*, **86** (2005) 213116.

[2] S. Cagliero, A. Agostino, E. Bonometti and M. Truccato, "Crystalline instability of Bi-2212 superconducting whiskers near room temperature ", *Supercond. Sci. Technol.,* **20** (2007), 667–671.

[3] S. Cagliero, A. Piovano, C. Lamberti, M.M. Rahman Khan, A. Agostino, G. Agostini, D. Gianolio, L. Mino, J.A. Sans, C. Manfredotti, and M. Truccato, *J. Synchrotron. Radiat.,* (2009) doi:10.1107/S0909049509036802 *in press.*