FABRICATION OF INTRINSIC JOSEPHSON JUNCTIONS STACKS AND LOOSELY BOUND ATOMS IN SUPERCONDUCTING OXIDES

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Introduction

Josephson junctions (JJs) consist of two superconducting materials separated by a thin insulating barrier and their DC polarization enables to produce electromagnetic radiation in the THz range by means of voltages on the order of a few mV [1], which could be easily integrated in solid state devices with considerable potential applications, e.g. in the fields of security and environmental monitoring [2]. However, a major limitation is represented by the fact that single junctions typically irradiate in the pW power range, so that stacks of many in-series junctions are required to increase the emitted power. Single crystals of superconducting cuprates like Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ (Bi-2212) and YBa$_2$Cu$_3$O$_{7-δ}$ (YBCO) give a good opportunity to fabricate such arrays of JJs, provided that the current is forced to flow along the lattice c-axis. Ordinary photolithography and Focused Ion Beam (FIB) machining are widely used to shape these materials into the desired structures. However, both methods involve some etching techniques that can critically affect the crystal doping state. In this study we show that Bi-2212 crystals undergo some underdoping process during the JJ stack fabrication and investigate the possible influence of the etching methods.

Experimental

We have prepared JJ stacks out of Bi-2212 whiskers by means of a double-side fabrication technique. Bi-2212 whiskers have been grown via the glassy plates route by annealing at 862°C for five days in O$_2$ flow. They have been mechanically removed from the plates and glued to sapphire substrates. About 100 nm of Au has been e-gun evaporated on one side of the crystals immediately after cleaving them. On the same side, we have FIB-etched about 150 nm deep trenches across the whole crystal width. Subsequently, the crystals have been cleaved for the second time, then about 200 nm gold was evaporated on the freshly exposed surfaces. Standard photolithography was used to pattern the electrical contacts, including Ar milling and O plasma etching for about 4 h and 2 h, respectively. Additional trenches have been etched on the crystal top surface with various depths, up to a maximum of about 50 nm. Electrical characterization of the JJ stacks has been performed on a cryogen-free system by Cryogenic Ltd equipped with PXI multi-components including FPGA and low-noise preamplifiers.

In order to check the possibility that some heating taking place during the fabrication affects the doping state of the material, a parallel experiment has been carried out on pristine crystals. Four whiskers with regular surfaces have been selected at the optical microscope from the synthesized batch and have been positioned onto an amorphous support for XRD analysis. The crystals have been placed with their c-axis perpendicular to the plane of the substrate. They have undergone an ageing process consisting of a series of thermal treatments at 90°C in air in steps of 1, 6 or 15 hours. XRD data have been acquired on a Philips X’Pert diffractometer in theta-theta geometry with CuK$_\alpha$ radiation and have been analyzed with the program Fityk (GPL).

Results and Discussion

Fig.1 shows the IV characteristics measured at $T$=6.2 K for a JJ stack with cross section area of 21.1×2.5 μm$^2$ that was obtained by FIB-etching the crystal top surface for about 50 nm. The ratio between the overall voltage drop and the branch spacing indicates that about 75 JJs are present in the stack, which corresponds to a stack height of about 115 nm, according to the crystal structure. The inset of Fig.1 shows the temperature behaviour of the electrical resistance of the stack, which corresponds to the typical curve of the c-axis resistivity. According to Ref. [3], the ratio $R_{\text{MAX}}/R(285 \text{ K})=3.45$ implies that the oxygen stoichiometry is $x=0.23$ and therefore the stack is in the slightly underdoped regime.

Another method that is commonly used to determine the doping level in Bi-2212 is represented by the measurement of the c-axis lattice constant via XRD. We have obtained $c =30.547(8)$ Å for the pristine crystals of the batch, which in the case of whiskers corresponds to the highly overdoped regime [4]. Therefore, it is apparent that some underdoping process takes place during the stack fabrication.
Several possibilities exist in this respect. For instance, we have already shown that in FIB-machined microbridges fabricated out of YBCO crystals the normal state resistivity increases and the critical temperature slightly decreases with decreasing the bridge width [5]. This observation implies some role of laterally straggled Ga ions that are implanted in the material and could locally change its doping level because of a chemical effect. Another possible origin is represented by the production of secondary electrons during the interaction of the ion beam with the material. Indeed, we have recently shown that irradiation of Bi-2212 by a 17-KeV photon beam significantly affects the conductivity properties of this material and that such effect is inherently different from local heating, being very likely related to the electron knock-on mechanism for the interstitial O atoms, which are very loosely bound (≤2 eV) [6]. However, a detailed assessment of such possibilities requires XRD characterization of the stacks with submicrometric resolution, which is presently under way.

In the present paper we have investigated the possible effect induced by prolonged sample heating during the Ar milling and O plasma stages of the fabrication. XRD data show that since 3 h ageing an additional secondary phase domain can be detected in the patterns. Fig. 2 shows the evolution of the c-axis lattice constants both for the main phase (open squares) and for the secondary phase (full circles). It is apparent that, while the main phase does not significantly change, the secondary one undergoes an oxygen depletion process, with the c-axis value at 66 h ageing time that corresponds to a slightly underdoped state [4]. This is very similar to what happens during the stack fabrication. However, the observed timescale of this process seems too short for such effect to be the dominant one during the only 6 h long heating that has taken place for the stack production.

**Fig. 1** IV curve of a stack of about 75 JJs at T=6.2 K.Inset: R vs T behavior of the same stack for I=5 μA.

**Fig. 2** Time evolution of the c-axis parameter for the crystals aged at 90°C (see text).

**Conclusions**

We have successfully fabricated JJ stacks out of Bi-2212 whiskers and have shown that the crystals change their doping state during the process. Heating of the pristine crystals has been shown to induce the same effect, but on a much longer timescale. The role of the modifications induced by the Ga-ion beam is probably of major importance, but its nature has to be further clarified.

**References**