



Conference on the Application of Accelerators in Research and Industry, CAARI 2016,
30 October – 4 November 2016, Ft. Worth, TX, USA

Patterned electron beam exposures of YBCO – towards local control of doping

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Abstract

Local control of dopant profiles and ordered doping in high T_c superconductors have the potential to greatly increase the transition temperature, T_c . We report on experiments where we used focused electron beams to locally modulate the oxygen dopant concentration in commercial YBCO films (100 nm on LaAlO_3). Patterned exposure of YBCO samples to 10 keV electrons and fluences in the 10^{20} e⁻/cm² range led to increases of T_c of ~0.4 K, comparable to earlier reports from broad beam exposures in a similar fluence regime. We discuss our results in relation to concepts of local oxygen depletion and chain ordering induced by ionizing radiation and outline possible processing paths to implement a form of modulation doping in YBCO by patterning with intense, short excitation pulses (e. g. of MeV protons).

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Peer-review under responsibility of the Scientific Committee of the Conference on the Application of Accelerators in Research and Industry

Keywords: high T_c superconductivity, electron beams, doping

1. Introduction

The quest to increase the transition temperature of superconductors remains an exciting field of basic and applied research following the discovery of cuprates over 30 years ago [Bednorz] and recently with observations of very high

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transition temperatures for conventional superconductivity in sulfur hydride under high pressures [Drozdov, Gor'kov]. In this article we report on an experimental study of yttrium barium copper oxide (YBCO, $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, $x \approx 0.4$) where we attempted to affect the transition temperature by acting on the oxygen dopant sub-system using focused beams of 10 keV electrons. Tolpygo et al. have reported increases of T_c by up to 2 K following broad beam exposure of thin films of YBCO to 40 keV electrons with an optimal fluence of 5×10^{20} electrons/cm² [Tolpygo]. T_c converged back to the nominal value of 91 K when this optimal fluence was exceeded. The observed changes were explained as due to chain oxygen disordering (formation of vacancies and interstitials in specific lattice positions) which disrupts the conductivity of chains and decreases the hole doping in CuO_2 planes. Intriguing effects of oxygen chain order induced by beams of 20 keV electrons were reported by Seo et al. [Seo], where electron bombardments was found to instigate the collective hopping of oxygen atoms either from an interstitial site to a vacant chain site or by reshuffling of chain segments to extend the average length of chains without changing the overall oxygen content. In contrast to these studies with relatively low electron beam energies, irradiation induced T_c suppression due to formation of lattice defects by electron beam energies well above the displacement threshold were reported e. g. by Giapintakis et al. [Giapintakis]. Recently, formation of (transient) ordered dopant structures were reported from x-ray beam direct writing with lanthanum copper oxides [Poccia].

Also recently, Wolf and Kresin have proposed that ordering of oxygen dopants in YBCO films could lead to large increases in T_c [Wolf]. We now paraphrase the leading arguments of Wolf and Kresin from their article [Wolf]: “Dopants play a dual role in YBCO. On the one hand, they provide the (de-localized) charge carriers (holes) that support superconductivity. On the other hand, oxygen atoms are scattering centers responsible for pair breaking. When oxygen is added to under-doped YBCO, the mixed-valence state of the in-plane Cu leads to plane-chain charge transfer and the appearance of a hole, initially on the Cu site. Because of diffusion, the hole enters the system of de-localized carriers responsible for the metallic and, correspondingly, for the superconducting behavior as pairing of such holes causes superconductivity. But the added oxygen ion and corresponding in-plane Cu also form a defect with pair-breaking impact. The statistical nature of doping leads to a random distribution of dopants and at relatively low doping levels the spatial distribution can be rather broad. As a result, regions with smaller number of dopants can have larger values of T_c . These regions can form superconducting “islands” inside of the normal matrix which have been observed up to temperatures $T_c^* > T_c$.” We can now ask whether we can implement a form of modulation doping in cuprates, where optimally doped regions can provide the charge carriers that would then conduct more freely up to temperatures $T_c^* > T_c$ in adjacent under-doped regions. Below, we report on our first attempts to implement this idea in stripe like patterns of adjacent regions of optimally doped and under-doped YBCO formed using beams of low energy electrons.

2. Experiment

We purchased samples with thin films (100 nm) of YBCO on LaAlO_3 (100) with a nominal T_c of 90 K. (MTI Corporation, Richmond, CA). Sample dimensions were 10 mm x 10 mm x 0.5 mm. We then sputter deposited Ag/Ti/Au (100 nm/20 nm/ 400 nm) contacts in an electrode layout for four-point probe measurements. Four electrode patterns were deposited per sample. We then exposed areas between the electrodes to 10 keV electrons in an FEI Strata 235 dual beam FIB. The pressure in the vacuum chamber was in the low 10^{-6} Torr range. We selected an electron beam energy of 10 keV guided by simulations using the CASINO program [CASINO] which showed that at this energy electrons deposit most of their kinetic energy in the 100 nm YBCO film. We measured electron beam current values with a home built Faraday cup. Figure 1 shows examples of line patterns from electron beam exposure of samples i) and iii). Sample i) was exposure to an electron beam with a current of 8.6 nA for 10 minutes per line and a total of six lines. The scanning electron micrograph images taken after line patterning show the exposed lines with an apparent width of about 0.75 μm , much larger than the nominal electron beam spot size, which is of order 10 nm at these elevated current levels. We attribute the contrast to changes in the surface composition as a result of high fluence electron exposures. Further, vibrations and drift can contribute to line broadening during extended exposures. With a line width of 0.75 μm , an upper bound of the electron fluence per line is 6×10^{20} electrons/cm² for sample i). Sample iii) was exposed to a 1 nA electron beam also with six lines and 10 min per line. The apparent line widths were 0.5 micron and the upper bound of the fluence per line is then 2.4×10^{20} electrons/cm². The instantaneous local fluence is likely higher, but the 0.5 to 0.75 μm apparent line widths also reflect the range of secondary electrons.

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