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ATOMIC STRUCTURE OF SUPERCONDUCTOR YBa$_2$Cu$_3$O$_{6.9}$ IN FIELD ION MICROSCOPE

G.A. MESYATS, N.N. SYUTKIN, V.A. IVCHENKO and E.F. TALANTSEV
Institute of Electrophysics, Ural Division U.S.S.R. Academy of Sciences, Sverdlovsk 620219, U.S.S.R.

Abstract - Surface images of superconducting phase YBa$_2$Cu$_3$O$_{6.9}$ and YBa$_2$Cu$_3$O$_{6.15}$ compound have been obtained using FIM technique. The ion image is shown to be produced by yttrium atoms mainly. The superconductor crystal lattice defects have been examined. The ion contrast given by a twin boundary is discussed taking into account the type of imaging atoms.

I INTRODUCTION

The atomic structure of superconducting YBa$_2$Cu$_3$O$_{7-x}$ ceramics has been investigated by FIM technique. Field ion microscopy makes it possible not only to visualize the pure surface of the specimen in a highly intensive electric field, but also to analyse the crystal lattice in bulk by controlled removal of atoms of the material at cryogenic temperatures. The aim of the present study has been to obtain field ion images of superconductors in atomic scale, to find out which of sublattices of the ordered compound produces the surface image, to observe possible structural defects.

Previously Kellog and Brenner /1/, Melmed et al. /2/ have studied superconducting ceramics in field ion microscope. The authors of /1/ could not observe a full ion image of the surface of the specimen due to the constructive features of the instrument (imaging atom probe). According to Melmed et al., only Cu-O chains are imaged in the field ion patterns of the surface.

II EXPERIMENTAL

Square rods of 1x1x10 mm cut out by a diamond saw from certified ceramics YBa$_2$Cu$_3$O$_{7-x}$ have been used as blanks for sample-emitters. One part of the samples with 0.1 x 0.15 had the superconducting transition temperature 92 K; the second part with 0.85 x 0.9 did not possess superconducting properties. Tip samples were obtained by electropolishing in 5-15% solution of perchloric acid in ethylene glycol monobutyl ether /1/. The atomically smooth surface was prepared in situ by field evaporation of atoms of the specimen from most protruding parts of the hemispheric emitter tip.
Residual gas pressure was down to $10^{-5}$ Pa. Solid nitrogen (63 K) served as a cooling agent. A gas mixture, with molecular nitrogen prevailing was used as an imaging gas. A microchannel converter with the channel plate diameter 56 mm and channel diameter 14 mm was used to intensify the image. Surface images were registered by a still camera in the process of slow field evaporation of atoms of the material itself. No hints of adsorption on the surface have been observed during the investigation of YBa$_2$Cu$_3$O$_{7-x}$ ceramics in field ion microscope, this being typical of the surface of pure metals under the same conditions. The use of hydrogen as an imaging gas led to the loss of resolution of individual atoms in the ion images of the superconductor; the faces at the main zone lines could not be seen distinctly.

III RESULTS AND DISCUSSION

Fig. 1a presents the surface ion image of the superconducting ceramics YBa$_2$Cu$_3$O$_{6.90}$. The image resembles the microstructure pattern given by a pure metal or an ordered solid solution as regards the regularity of the ring pattern. We deal with a complex compound, this implying that the surface is mainly depicted by atoms of one species. Computer simulation of ion image of individual sublattices of YBa$_2$Cu$_3$O$_{7-x}$ structure has shown that the image simulated for yttrium sublattice (Fig.2b) fits the one obtained experimentally (Fig.1) best of all. High tetragonality, $c/a = 3$, has led to the specific character of the ion image of the superconductor, i.e. the specific morphological development of poles /3/. Only the pole of cube (001) might be the most developed one, this being exhibited on the screen of the microscope. All the other cubic poles are developed like dodecahedron rings and like poles of higher indexes, such as (012) etc. The stereographic projection plotted for yttrium sublattice appeared to be in good agreement with the FIM image of superconducting ceramics.

According to Melmed et al. the FIM image of superconductor is depicted rather by atoms of Cu-O chains than by yttrium atoms. Using the com-

Fig. 1 Surface FIM images: a -YBa$_2$Cu$_3$O$_{6.90}$; b -YBa$_2$Cu$_3$O$_{6.15}$; c -single crystal YBa$_2$Cu$_3$O$_{6.60}$.
Fig. 2 a - field ion image of (001) region, $V = 11$ kV; 
b - computer simulated image of (001) region, only yttrium atoms are imaged.

Computer simulated ion images given either by yttrium atoms only or by atoms of Cu-O chains only /2/, we may predict FIM images of a twin boundary for both cases, Fig. 3.

Fig. 3 Computer simulation of a twin boundary in orthorhombic lattice of $YBa_2Cu_3O_7-x$: a - only yttrium atoms are imaged; 
b - only Cu-O chains are imaged.

From Fig. 3 it is evident that the contrast of a twin boundary for the first case due to the change of parameters of the unit cell sublattice for Cu-O ($a:b:c = 1:2:6$) must be similar to the image of a c-domain boundary in alloys of LIo-type structure, when this boundary crosses a superstructural pole, Fig. 4. A typical for ordered alloys contrast of a double step appears to be due to the parameters relation $a:b = 1:2$.

In the case when only yttrium atoms are imaged the double step contrast will not appear in the image of a twin boundary (Fig. 3a), for the relation of the parameters $a:b$ is $1:1$. Hence, the real ion contrast of a twin boundary must show which atoms give the FIM image of superconductor.
Fig. 4 A boundary of c-domains A and B in Pt-Co alloy: 
a -initial image; b -several dozens of atomic layers have been evaporated, $V = 7$ kV.

In ref. 1 a FIM image of a twin boundary is shown. No contrast of a double step is seen in the image. So, in our opinion and according to the experimental data obtained in /1/ only yttrium atoms are responsible for the surface image. We have obtained the micrograph of a twin boundary lying in the \{110\} plane (Fig. 5). No double step contrast is observed in the image.

Fig. 5 FIM images of structural defects in $\text{YBa}_2\text{Cu}_3\text{O}_6.90$.

- a - defect in the (001) region; b - a twin boundary in the \{110\} plane.

FIM micrograph of $\text{YBa}_2\text{Cu}_3\text{O}_{6.15}$ compound is presented in Fig. 1b. It is identical to the image of superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{6.90}$ Fig. 1a. It is usually assumed that Cu-O chains are responsible for the superconducting state of the ceramic compound. So, the practical identity of the images of superconductor and nonsuperconductor also confirms the imagibility of yttrium atoms.

Some authors consider (e.g., Cerezo et al./4/), that typical contrast of a superconductor surface Fig. 1a may be related to the emitter pre-
paration technique. We have obtained also the image of a single crystal \( \text{YBa}_2\text{Cu}_3\text{O}_{6.90} \) FIM specimen prepared mechanically by splitting. It is evident that the ion contrast of the ceramic compound surface does not depend on the way of emitter preparation, at least for the specimens used in the present study.

In accordance with all these considerations we can conclude that FIM image of the pure surface of \( \text{YBa}_2\text{Cu}_3\text{O}_{7-x} \) is formed mainly by yttrium atoms.

We have also analysed the structure of the superconductor in bulk in the process of field evaporation of atoms of the material itself. A single crystal was usually observed on the screen of the microscope with its regular ring pattern disturbed in some cases at low-index poles, Fig. 5a. Such an ion contrast corresponds to crystal lattice defects.

In addition to the regular ring pattern given by yttrium atoms the images show random enough distribution of brighter points. These may be atoms of other species positioned under the surface yttrium atoms affecting the image indirectly.

Thus, in the present study we have succeeded in obtaining well-formed FIM images both of superconducting and nonsuperconducting phases with atomic resolution, and identifying imaging atoms as yttrium ones and in observing the structure of the superconductor in bulk.

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