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Magneto-conductivity fluctuation in YBCO prepared by sintering of ball-milled precursor powder

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HIGHLIGHTS

- Y-123 embedded by Y-deficient Y-123 nano-particles are obtained by ball-milling.
- Comparative study of the electrical magneto-conductivity fluctuation is reported.
- Occurrence of a pairing transition splitting related to Y-deficient Y-123.
- Y-deficient Y-123 increases the disorder and strengthen the pinning of vortices.
- The transition paracoherent-coherent is strongly affected by Y-deficient Y-123.

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ABSTRACT

We compare the electrical magneto-conductivity fluctuation between $YBa_2Cu_3O_y$ (Y-123) and $YBa_2Cu_3O_y$ embedded by an Y-deficient Y-123 nano-phase, generated by planetary ball-milling. Both samples were synthesized by solid state reaction with two stages of sintering at 950 °C and separated by energetic milling (milled sample) and hand grinding (unmilled sample). SEM analysis of the milled samples shows the appearance of spherical shaped nano-entities of Y-deficient Y-123 embedded in the Y-123 matrix. The magneto-conductivity data are analysed in terms of the temperature derivative of the resistivity and the logarithmic temperature derivative of the conductivity. The results show the occurrence of two-stage transitions: intragranular and intergranular, as well as a pairing transition splitting in the milled sample related to the presence of the Y-deficient nanoparticles. In the normal state, we identify a contribution of the Gaussian regimes for both samples. In the presence of a high magnetic field, the critical exponent near T_{co} of the milled material is higher than the one of the unmilled, indicating that the nanoparticles strengthen the effects of the order-parameter phase of individual grains of the Y-123 strongly disordered. This result fits well into a description based on a dirty limit of the general vortex-glass theory. In the paracoherent state, a crossover occurs and a small temperature range appears where the fluctuation conductivity may be governed by others mechanisms related to vortices dynamics. This temperature range is smaller in the milled sample.

1. Introduction

The preparation method of high temperature superconductor (HTS) materials and the nature of the chemical doping and additives can introduce additional defects in the form of single atoms, grain boundaries or phase separation in the superconducting matrix [1,2] which can locally depress the superconducting order parameter [3]. As a consequence, a strong enhancement of the granularity effects in transport and magnetic properties can be observed in HTS polycrystalline samples and lead to an increase of the disorder in the compound. Intergranular tunnel barriers

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influence both the superconducting and the normal state properties. The investigation of the impact of nano-sized inclusions in superconductors has been of great interest because a suitable amount of nanoparticles can produce mesoscopic and microscopic defects and help to improve the performance of practical superconductors. Studies of the excess conductivity on composites of YBCO with metal, semiconductor or magnetic entities have been reported [4–6]. However to our knowledge no data have been published regarding the effect of superconductor material inclusions in Y-123 on the excess conductivity. Earlier, we have shown that high energy ball-milling is an alternative way to facilitate the formation of an optimal microstructure with a high grain boundary density and lattice strain, which is expected to enhance the magnetic flux pinning and to improve the critical current in external magnetic fields [7,8]. The milled sample is composed by Y-123 and Y-deficient Y-123. The microstructure of the milled sample is characterized by a high density of interfaces between the Y-deficient Y-123 nano-phase and the Y-123 matrix. As thermodynamic fluctuation regimes of HTS are sensitive to the pairing symmetry, it is interesting to provide more evidence on the effect of Y-deficient Y-123 embedded in Y-123 on the magneto-conductivity fluctuation. One consequence is the enhancement of phase fluctuation effects in the resistive transition of the milled sample which could be regarded as another model system for studying the coherence transition. From conductivity measurements and fluctuation induced conductivity analysis, it is possible to separate the granularity at microscopic and mesoscopic levels. As the temperature decreases, we first observed a pairing transition and then a coherence transition. In HTS, the paraconductivity associated with thermal fluctuations of the order parameter depends both on the internal defects in the structure and on the extrinsic properties of samples such as the grain morphology. In the mean field region and according to the Aslamazov–Larkin (AL) theory [9], the excess conductivity is expressed as $\Delta\sigma = A\varepsilon^{-\lambda}$, where $\varepsilon = (T - T_c)/T_c$ is the reduced temperature and λ is an exponent related with the conduction dimensionality. At lower temperatures, the resistivity is described by a percolation-like process related to the activation of weak links between the superconducting grains [10,11]. Closer to the zero resistance temperature T_{co}, studies of the specific heat [12,13], penetration depth [14], and electrical conductivity [15–17] reveal the effect of critical fluctuations on a transition belonging to the three-dimensional 3D-XY universality class. In this regime, approaching zero resistance temperature, the conductivity diverges as a power law of the reduced temperature $(T - T_{co})^{-S}$. In most of previous reports [18–25] the conductivity critical exponents $s \approx 3$, $s \approx 4$ and $s \approx 5$ have been found near the coherence transition. Hence a study of the electrical excess conductivity due to thermodynamic fluctuations is very important to understand the intrinsic superconducting characteristics. It can provide useful information to resolve some microscopic and mesoscopic discrepancies between Y-123 embedded by an Y-deficient Y-123 nano-phase and the Y-123 matrix.

In this paper we report a comparative study of the electrical magneto-conductivity fluctuation in the broadening region of the resistivity of Y-123 and Y-123 embedded by an Y-deficient Y-123 nano-phase. To identify power-law divergences in the conductivity, the data were analyzed in terms of the temperature derivative of the resistivity $d\rho/dT$ and the logarithmic temperature derivative of the fluctuation conductivity $-d\ln\Delta\sigma/dT$. Experimental results and discussion are presented to compare the mesoscopic and microscopic inhomogeneity effects on the critical exponents and the fluctuation dimensionality in both materials, sintered in the same conditions.

2. Experimental details

Details on the preparation of Y-123 polycrystalline samples and the effect of the planetary ball-milling have been reported elsewhere [7] and therefore we only give a brief description. The single phase Y-123 was synthesized by thoroughly mixing high purity Y_2O_3 (99.9%), $BaCO_3$ (99.9%) and CuO (99.9%) according to the chemical formula of Y:Ba:Cu = 1:2:3 (Y-123). This mixture of powders was pelletized and then calcined at 950 °C for 12 h in air in order to produce an oxide precursor without remainder of any carbonates. One part of the resulting oxide precursor was milled via the planetary milling technique and another part was ground by hand. In the present work, we milled the material by planetary ball-milling for 4 h with a speed of 600 rpm and a ball-to-powder weight ratio of about 5:2. Fig. 1 shows SEM and STEM micrographs of the milled oxide precursor. This figure shows that milling for 4 h fractured the precursor oxide lumps into fine particles. Fine particles are dispersed on the surface of the Y-123 matrix. The oxide precursors are then pressed uniaxially into pellets of 0.5 mm thick and 7 mm in diameter under a uniaxial pressure of 750 MPa. These pellets are sintered in air at 950 °C for 8 h. The samples prepared using ball-milling and hand grinding are referred to as “milled” and “unmilled”.

The structure and phase purity of the powder sample ground from sintered pellets were examined by powder X-ray diffraction (XRD) using a Philips 1710 diffractometer with CuK_α. The scanning electron microscopy observations were performed in a FEI Nano Lab 200. Specimens in the form of parallelepiped, adequate for resistivity measurements, were cut out from the sintered pellets. The resistivity experiments were performed with a low frequency AC technique based on a lock-in amplifier with a resolution of 2 nV. The temperature was measured with a Pt sensor allowing a resolution, better than 2 mK. Measurements were concentrated in the temperature range encompassing the superconducting transition. Electrical contacts were made using silver paint and the contact resistance value was approximately 0.5Ω. The temperature

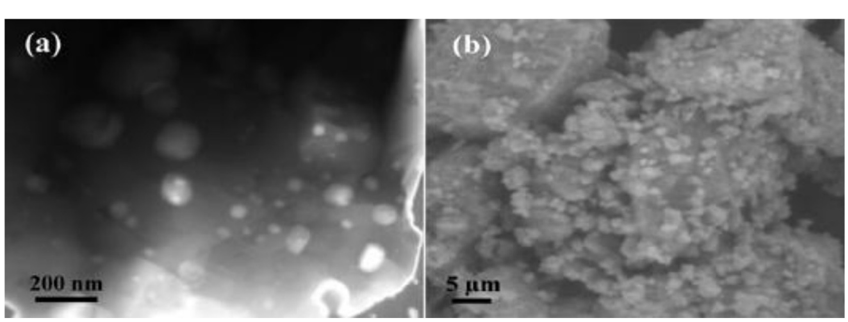

Fig. 1. STEM (a) and SEM (b) micrographs of the oxide precursor powder after ball-milling.

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