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Effect of SiO_2 nano-particles and nano-wires on microstructure and pinning properties of $YBa_2Cu_3O_{7\text{-}d}$

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A B S T R A C T

A comparative study of the effects of nanosized silicon oxide nano-particle (NP) and nano-wire (NW) additions during the final processing stage on the microstructure and superconducting properties of polycrystalline $YBa_2Cu_3O_y$ (YBCO, or Y-123) was carried out. Samples were synthesized in air using a standard solid state reaction technique by adding nanosized entities up to 1 wt. %. Phases, microstructure, superconductivity, have been systematically investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and electrical measurements. TEM investigation shows the presence of inhomogeneities embedded in the superconducting matrix along with the presence of columnar defects in the case of SiO_2 nano-particles added, however nano-wires tend to agglomerate by entangling with each other in the inter-grain regions. The critical current density of the samples was examined using current–voltage, ac-susceptibility and magnetization measurements. The results are discussed in the framework of Bean's critical state model. Flux pinning force density is calculated and the possible pinning mechanisms prevalent in sintered samples are determined. The flux creep activation energy is determined in the light of vortex dynamics exhibited by the frequency dependence of the AC susceptibility. We have clear indication that a relatively low concentration of nanosized silicon oxide improves the pinning properties of Y-123 and we also validated the impact of the shape of nanosized inclusions on the superconducting properties.

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1. Introduction

The critical current density J_c is of great interest for potential applications of high-temperature superconductors (HTS). The effect of granularity/morphology on the superconducting properties of HTS bulk polycrystalline samples is among one of the greatest challenges in realizing applications of high temperature cuprate superconductors. For polycrystalline samples, the inter-granular critical current density is limited by weak links caused by grains boundaries and the intra-granular critical current is mainly limited by the thermally activated flux flow at high temperatures and high magnetic fields. To prevent the vortex motion and enhance the flux pinning strength it is necessary to introduce artificial pinning centers in a superconductor apart from those which occur naturally [1–5]. Artificial pinning centers (APCs) can be described as one-dimensional (1D) such as dislocations, two-dimensional (2D) such as grain boundaries and three-dimensional (3D) such as fine precipitates.

To increase the density of these defects, various techniques such as high energy ion irradiation [6], high energy ball milling [7], chemical doping and additives [8,9] have been reported in the literature. Chemical doping and additives in high T_c superconducting cuprates have attracted much attention compared to physical techniques because a suitable amount of nano-particles can produce mesoscopic and microscopic defects and help to improve the performance of the superconductor. Nanosized particles trapped within the superconducting grains may be able to modify the crystalline structure and generate crystal imperfections, structural defects such as dislocations, stacking faults, columnar defects etc. The nano-materials have to satisfy certain criteria to be acceptable as dopant; their presence should not affect the formation of the superconducting phase.

Various kinds of doping and additives, including metal [10] non-metal nano-particles [4,5,11] and carbon nano-tubes [9] have been used to improve both the inter-granular and intra-granular critical

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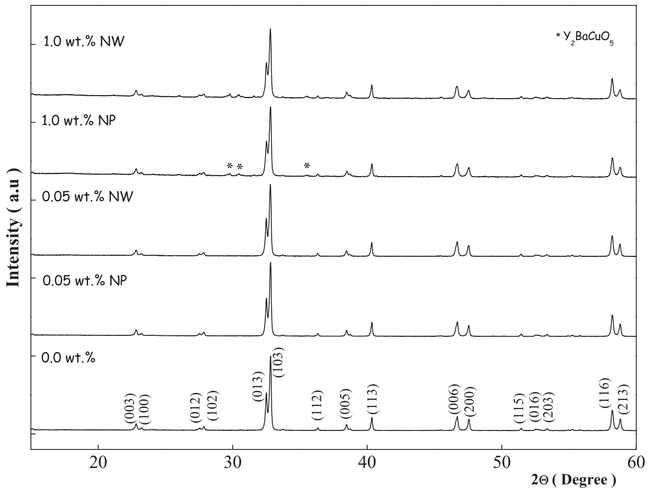

Fig. 1. X-ray powder diffraction patterns of samples added with various amounts of SiO_2 nano-particles (NP) and nano-wires (NW).

current densities in high temperature superconductors. There is general consensus in the literature that when nano-tubes such as carbon tubes are embedded in the superconducting matrix, they function as columnar defects [12–14]. Oxide perovskites ($BaZrO_3$, $BaSnO_3$, $BaNb_2O_3$, etc.) nano-additives have gathered the most interest as a class of materials for incorporation into YBCO. These additives improve the pinning ability by forming composites or acting as column defects comprised of self-alighted nanostructure such as nano-dots and nano-rods [12,15,16]. Si-based nano-materials in MgB_2 induce modifications in the microstructure and an enhancement of the pinning properties. However the effect of Si-based nano-materials in HTS ceramics, other than MgB_2, remains inconclusive and opposite results have been reported. Guo et al. [17] reported on the effect of nano-sized SiC addition on poly-crystalline Bi-2223 and they found that a small amount of SiC (0.15 wt.%) improved the critical current I_c and its behavior in magnetic field as a result of the enhancement in density, grain alignment, grain connectivity and flux pinning. Nano-sized SiO_2 addition shows a negative effect on T_{co} and J_c of $SmBa_2Cu_3O_7$

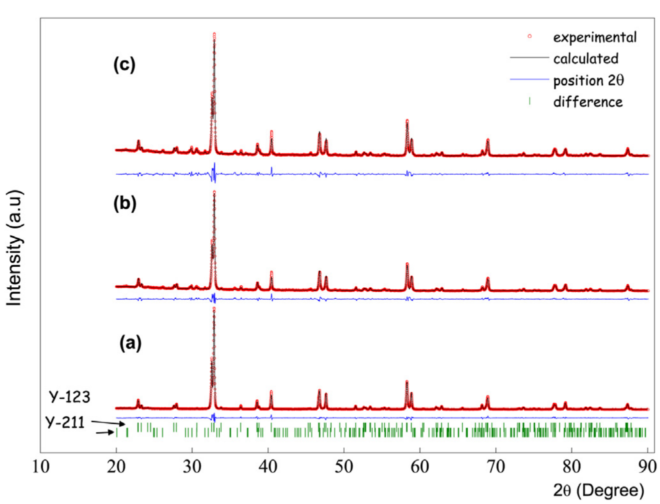

Fig. 2. Experimental and calculated XRD patterns of YBCO samples with (a) 0.0, (b) 1 wt.% SiO_2 NP and (c) 1 wt.% SiO_2 NW additions after Rietveld refinement. The dots are the measured X-ray diffraction data and the solid line is the calculated profile. The difference curve (observed results minus calculated ones) is plotted at the bottom. The tick marks below represent the positions of Bragg reflections.

materials due to the reaction between SiO_2 and the matrix [18]. Moreover, the substitution of small amounts of Si at Cu sites in the CuO_2 planes has been found to suppress the superconductivity in cuprate systems such as $(Cu_{0.5},Tl_{0.5})Ba_2Ca_2Cu_3O_{10\text{-}d}$ [19]. Little work is devoted to the effect of SiO_2 powder addition to YBCO [20,21]. Structural and micro-structural analyses show that SiO_2 addition during the initial sintering cycles are sticking on the surface of the YBCO grains and leave the Y-123 structure practically intact. SiO_2 degrades the width of the M–H loops and the H_{irr} (magnetic irreversibility field) and strongly affects the flux dynamics determined by the inter-grain regions of the samples. Nano-material additives may thus be more efficient during the final sintering cycles of YBCO superconductor, which adjust the local microstructure by generating efficient defects. Recently, we studied the effect of nano-particle SiO_2 addition during the final sintering cycles and we showed that a suitable amount of nano-sized SiO_2 particles does not affect the formation of the superconducting phase and does not alter T_{co}. Transmission electron microscopy observations show the presence of columnar defects and a Si-rich nanophase embedded in the superconducting matrix [21]. Though many papers were devoted to the study of the addition of nano-sized materials to HTS materials, the effect of the nano-sized shape on the properties of HTS compounds are very scarce [22]. The addition of nano-sized materials can also modify the coupling between grains by increasing the width and/or the degree of disorder and by the composition variation of the grain boundaries [23].

In this paper we report a comparative study of the effect of SiO_2 nano-material addition during the final sintering cycles of YBCO on the microstructure and superconducting properties. For this purpose, a series of specimens with different amounts of SiO_2 (0–1 wt.%) nano-particles and nano-wires were grown in air by the solid state reaction. Various superconducting parameters like critical temperature (T_c), global critical current density $J_{ct}(H)$, intra-grain critical current density$J_{cm}(H)$ and inter-grains critical current density $J_{c\,in}(H)$, flux pinning force density, flux creep activation energy (E_a), are evaluated and compared. We find that the shape of the nano-materials effectively controlled the grain microstructure, the grain coupling and hence the superconducting properties.

2. Experimental

The pure and nanometer size SiO_2 particles and wires added YBCO samples were prepared by the conventional solid–state reaction method under identical conditions. The single phase YBCO was synthesized by thoroughly mixing high purity of 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. 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. During the final processing stage, SiO_2 nano-particles (30 nm in size) and nano-wires (30 nm in diameter) were added to the precursor powder YBCO by mixing and hand grinding both powders in an agate mortar. The additional amount of SiO_2, varied from x = 0 to x = 1 wt.% of the total mass of the samples. The mixed powders were pressed into pellets at 750 MPa in the form of circular disks having 13 mm in diameter. The pellets were sintered at 950 °C for 8 h in air and then cooled to room temperature at a rate of 4 °C/min.

The structure and phase identification of the powder sample ground from the sintered pellets were examined by powder XRD using a Philips 1710 diffractometer with CuKα radiation in 2θ range from 20 to 60° with step size of 0.015° and step time of 10 s. Scanning electron microscopy (SEM) measurements were performed using an FEI Nano Lab 200. The transport properties of the samples were studied by measuring the electrical resistivity-temperature $\rho(T)$ using the four-probe technique. The pellets