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Magnetization and critical current density of superconducting granules doped with NiO nanoparticles

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A series of bulk superconducting $YBa_2Cu_3O_{7-\delta}$ (YBCO) samples doped with antiferromagnetic NiO nanoparticles and subjected to 18 h high-temperature annealing have been synthesized. For YBCO samples doped with 8, 12, and 23 nm nanoparticles, the characteristic magnitudes ΔM of the magnetic hysteresis loops were compared and the optimal size of NiO nanoparticles was determined. The effect of the concentration of NiO nanoparticles with an average size of 8 nm on the magnetic hysteresis loops of YBCO and the critical current density at $T = 4.2$ K has been studied. An increase in the critical density of intragranular currents is achieved by adding 0.5 wt.% of NiO nanoparticles.

Keywords: magnetic hysteresis, magnetic pinning, RE-123, solid-phase synthesis, granular superconductor, critical current density.

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Owing to their capacity to carry critical currents of several thousand amperes at temperatures below 20 K in strong magnetic fields, second-generation high-temperature superconducting tapes based on $(RE)Ba_2Cu_3O_{7-\delta}$ are crucial for modern energy and research installations [1–4]. Planned megaprojects of particle accelerators and fusion reactors are imposing ever-increasing demands on the current-carrying capacity of superconductors [5]. The strategy used to enhance magnetic flux pinning and critical current density is the introduction of artificial pinning centers at the growth stage [6–8], replacement of the rare earth element or alteration of its stoichiometry [9], and irradiation of tapes with various ions [10,11]. The introduction of magnetic nanoparticles into a superconductor may be the most effective method for pinning enhancement [12–15], since magnetic defects provide a significantly stronger interaction with Abrikosov vortices than non-magnetic pinning centers. The technology of magnetic pinning for second-generation superconducting tapes is only nascent at present. The main challenges are the introduction of magnetic nanoparticles into the multilayer structure of tapes and minimization of the possible negative impact on the superconducting layer.

Certain features of influence of magnetic nanoparticles on the properties of superconductors may be investigated using the existing solid-phase synthesis technology. We have already determined that rapid sintering of powdered $YBa_2Cu_3O_{7-\delta}$ (YBCO) with ε-Fe_2O_3 and NiO nanoparticles provides an opportunity to obtain samples with an increased critical intragranular current density [16,17]. Since this method may be ill-fitted for introduction into the 2G tape technology, it is necessary to examine the ways to enhance magnetic pinning during long-term annealing of samples.

The studies of the properties of superconductors doped with nanoparticles in [16,17] were carried out at the nitrogen boiling temperature. However, the efficiency of application of high-temperature superconductors increases significantly at temperatures of 4.2–20 K. At these temperatures, the coherence length and the effective size of pinning centers are smaller than at 77 K. Therefore, enhanced pinning may be achieved with the use of smaller nanoparticles.

In the present study, we report the results of examination of the influence of concentration of ultra-small antiferromagnetic NiO nanoparticles on the critical current density of superconducting granules in polycrystalline YBCO at $T = 4.2$ K.

The preparation of $YBa_2Cu_3O_{7-\delta}$ followed the standard procedure of solid-phase synthesis from Y_2O_3, $BaCO_3$, and CuO powders. NiO nanoparticles with average sizes of 8, 12, and 23 nm were prepared at the Boreskov Institute of Catalysis (Siberian Branch, Russian Academy of Sciences) [18]. Their size was determined in [18] based on microphotographic images obtained using a high-resolution transmission electron microscope. Samples were prepared by adding an alcohol suspension of nanoparticles to YBCO powder dispersed in alcohol. Alcohol was then evaporated over low heat on a hot plate. Dry powder was ground thoroughly and subjected to hydrostatic pressing (~ 100 MPa). The obtained pressed pellets were heated in a furnace to a temperature of 920 °C and annealed at this temperature for 18 h. The samples were then held for 10 h at a temperature of 400 °C. A series of samples with the NiO nanoparticle concentration varying from 0 to 6 wt.% ($0 \le x \le 0.06$) were prepared. All samples had the same temperature of the onset of the superconducting transition above which magnetization M becomes positive: $T_c \approx 93$ K. Magnetic hysteresis loops were measured using a vibration magnetometer constructed in the laboratory of strong magnetic fields (Kirensky Institute of Physics, Siberian Branch, Russian Academy of Sciences) at $T = 4.2$ K within a magnetic field range of ± 7 T.

The comparison of magnetic hysteresis loops for YBCO samples with 1 wt.% nanoparticles 8, 12, and 23 nm in size allowed us to determine which size of NiO nanoparticles ensures the most efficient pinning of magnetic flux at $T = 4.2$ K. The magnitude of trapped magnetic flux is proportional to the distance between the branches of magnetic hysteresis loops measured when the field increases and decreases (ΔM). The values of ΔM in field $H = 0$ for samples with different sizes of NiO nanoparticles are listed in Table 1.

In the YBCO sample with NiO nanoparticles 23 nm in size, ΔM decreased by a factor of 1.1 relative to the ΔM value for undoped YBCO. In the YBCO sample with a NiO size of 12 nm, ΔM increased by less than 2% compared to undoped YBCO. In the YBCO sample with NiO nanoparticles 8 nm in size, ΔM increased by a factor of 1.1 relative to undoped YBCO. Further studies into the influence of the concentration of NiO nanoparticles on magnetization and critical current density were carried out for YBCO samples doped with NiO nanoparticles with an average size of 8 nm.

Figure 1, *a* shows the magnetic hysteresis loops measured at temperature $T = 4.2$ K. The dependence of ΔM on mass fraction of nanoparticles x is presented in Fig. 1, *b*. The sample with $x = 0.005$ has the maximum ΔM, which is 1.4 times greater than the value of ΔM for undoped YBCO. Since the $M(H)$ dependences for all samples are reversible above the critical superconductor temperature ($T_c = 93$ K), there is no need to take magnetic contributions into account when one analyzes superconducting hysteresis.

The critical current density was determined as $J_c(H) = 3\Delta M(H)/D$, where D is the characteristic scale of current circulation. The value of D in polycrystalline superconductors is determined by the size of granules and the parameters of their distribution [19–21]. It is also possible to estimate D from the asymmetry of magnetization hysteresis loops [22]. The values of magnetization with increasing field (M^+) are greater in magnitude than the magnetization values with decreasing field (M^-). The asymmetry of magnetization is attributable to the equilibrium magnetization of the surface layer with depth λ. The current circulation scale was determined as $D \approx 2\lambda/[1 - (\Delta M/2|M_m^-|)^{1/3}]$, where M_m^- is the minimum magnetization (i.e., the maximum diamagnetic signal value). The value of $\lambda = 0.15\,\mu$m was used in calculations. Scale $D = 4.2\,\mu$m was determined for undoped YBCO. As the NiO fraction increases, D grows to $6.8\,\mu$m at $x = 0.001$, but then decreases to $6.1\,\mu$m at $x = 0.06$. Note that the samples doped with nanoparticles of different sizes with $x = 0.01$ have the same D values.

Table 1. Values of ΔM obtained when YBCO was doped with NiO nanoparticles of different sizes ($x = 0.01$)

Sample	ΔM, emu/g ($H = 0$)
Undoped YBCO	35.2
YBCO + 23 nm NiO	31.9
YBCO + 12 nm NiO	35.8
YBCO + 8 nm NiO	38.9

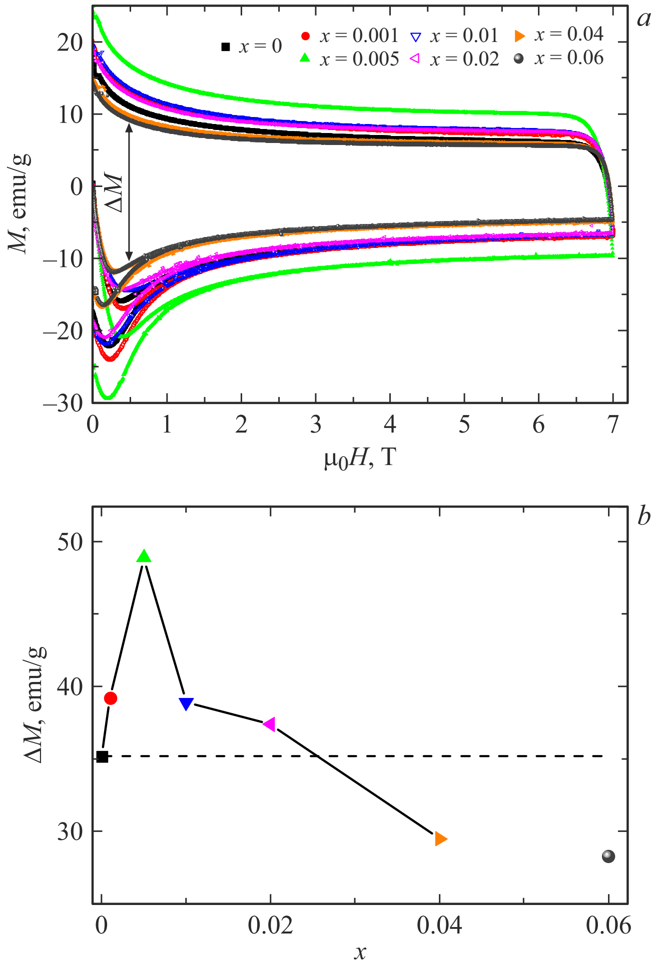

Figure 1. Magnetic hysteresis loops of samples with different nanoparticle concentrations at $T = 4.2$ K (*a*) and dependence of ΔM in field $H = 0$ on x (*b*). The dashed line indicates the ΔM value for undoped YBCO. A color version of the figure is provided in the online version of the paper.

Table 2 presents the obtained D values and the values of J_c in fields with a strength of 0 and 6 T. The approximate error of determination of J_c is $\pm 10^5$ A/cm^2. The field dependences of the critical current density of the samples are shown in Fig. 2, *a*. Figure 2, *b* presents the variation of J_c in $H = 0$ with increasing x.

As was expected from the ΔM data, the sample doped with 0.5 wt.% NiO (8 nm) has the maximum value of J_c. However, J_c for this sample is only 1.07 times greater than the corresponding value for undoped YBCO. The critical current density in the samples doped with 1 wt.% or more NiO decreases compared to undoped YBCO. In strong magnetic fields (6 T), J_c for the sample doped

Table 2. Sample parameters estimated from magnetic hysteresis loops

x	ΔM, emu/g ($H = 0$)	D, μm	J_c, 10^6 A/cm^2	
			$H = 0$	$\mu_0 H = 6$ T
0	35.2	4.2	16.0	6.0
0.001	39.2	4.5	16.6	6.1
0.005	48.9	5.6	17.1	6.9
0.01	38.9	6.8	10.9	4.1
0.02	37.4	6.7	10.5	4.0
0.04	29.5	6.6	8.5	3.2
0.06	28.3	6.1	8.9	3.3

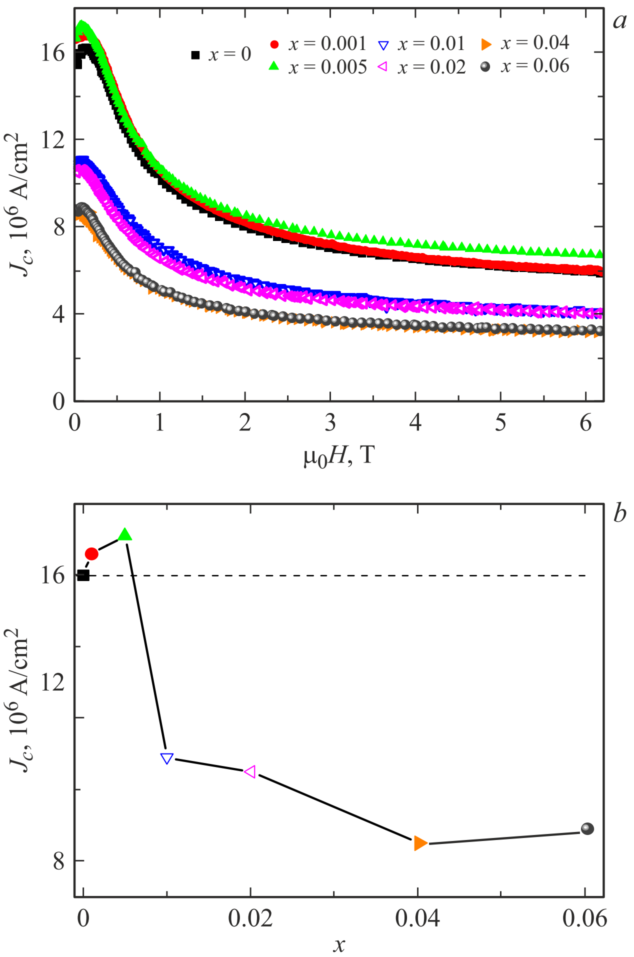

Figure 2. Field dependences of the critical current density at $T = 4.2$ K (a) and dependence of J_c in $H = 0$ on x (b). The dashed line indicates the J_c value for undoped YBCO. A color version of the figure is provided in the online version of the paper.

with 0.5 wt.% NiO (8 nm) is 1.15 times greater than the corresponding value for undoped YBCO. The critical current density increases due to the enhancement of surface pinning by NiO nanoparticles on the surface of granules and due to additional defects in the YBCO crystal lattice induced by the introduction of Ni [12,15].

The authors of experimental studies often use an increase in ΔM to support the conclusion that a specific type of dopant nanoparticles holds promise in the context of increasing the critical current density. However, we have demonstrated here that the growth of ΔM is not necessarily correlated with the variation of J_c. Thus, a correct comparison of J_c of different superconducting samples requires analyzing the characteristic structural dimensions of these samples [20,21].

We note in conclusion that a significant (1.4-fold) increase in ΔM was detected when YBCO was doped with ultra-small NiO nanoparticles (8 nm) with a concentration of 0.5 wt.%. The size of superconducting granules increases by a factor of 1.3, and the critical intragranular current density in this doped sample increases by a factor of 1.07 in zero field and 1.15 in strong magnetic fields.

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Conflict of interest

The authors declare that they have no conflict of interest.

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