Nanotechnology as a way to overcome the rapid $J_c$ fall with HTS film thickness

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Abstract. We have carried out a comprehensive study of a relation between the nanostructure and electromagnetic properties of the films prepared by pulse laser deposition on LaAlO$_3$ substrates using YBCO targets with BaZrO$_3$ additives. HREM studies revealed that depending on the deposition conditions BZO can precipitate as nanorods normal to the substrate or as BaYZr$_x$O$_y$ nano-pancakes. BZO nanorods are formed at lower laser power and higher substrate temperature. Their lattice is rotated by 3-4° to provide matching with matrix without dislocations. In a contrary, the nano-pancakes are surrounded with a great number of dislocations. Mechanisms of the nanostructure formation have been analyzed. Embedding of a proper kind of nanoparticles into HTS films leads not only to a certain increase of the critical current density $J_c$ but to a substantial weakening of its field and thickness dependences compared with pure YBCO films. Negative curvature of I-V-curves (in double-log scale) for YBCO (BZO) films is an evidence of strong pinning on extended defects, such as nanorods and threading dislocations, and/or collective vortex pinning.

1. Introduction

Comprehension of factors controlling superconducting critical current density $J_c$ in high-temperature superconducting (HTS) materials is rapidly developing recent years. Epitaxial YBa$_2$Cu$_3$O$_{7-δ}$ (YBCO) films and coatings remain the most promising for applications, but their utility is still severely limited by fast decay of $J_c$ with the film thickening and in strong magnetic field $H$, especially applied normally to the substrate, that is, along the $c$-axis of YBCO.

This problem is partially solved by the incorporation of nanoparticles into YBCO matrix (mostly BaZrO$_3$ (BZO), also Y$_2$O$_3$, Y$_2$BaCuO$_x$, etc.) [1, 2]. Such defects serve as additional pinning centres and do not deteriorate the texture of a superconductor. The nanoparticles undoubtedly improve the field behaviour of the films. They retard $J_c$ decrease with the film thickening and reduce the exponent $α$ that characterizes the power-law decay $J_c \propto H^{α}$, from 0.5–0.6 in undoped YBCO down to 0.30 [1, 3]. At first, investigators used to introduce as many different particles as they could. However, it was comprehended soon that numerous factors have an influence on the efficiency of particles.

Perovskite BZO nanoparticles seemed to be the most effective, because they were found to self-assemble into columnar defects, called nanorods, aligned along the $c$-axis [1-6]. However, more

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nanorods are formed at higher substrate temperatures, while sometimes the best performance was observed at lower temperatures.

The best results appeared recently are obtained with a mixture of splayed columnar defects and small random nanoparticles [7]. The very high critical current density was assumed to arise from columnar defects providing large pinning energy, while splay and nanoparticles inhibit flux creep. A proper combination of these structures synergistically maximizes in-field $J_c$. This knowledge can be used to produce thick films with remarkable $J_c(H)$ and nearly isotropic angle dependence.

There is an alternative mechanism, which preserves strong pinning in thick BZO doped films. It is based on the concept of dominant pinning at the out-of-plane edge dislocations in low-angle boundaries between single crystal domains [8]. Indeed, rather large diameter of the nanorods about 10 nm is not mandatory for a large pinning energy. Diameter of much thinner defects, such as edge dislocation cores, is just comparable with the coherence length in $ab$-plane, $\xi_{ab}(77 \text{ K}) = 4 \text{ nm}$, and can provide sufficient pinning. Moreover, the density of dislocations can be extremely high [9] well exceeding a reachable number of artificial pinning centres, such as nanorods. Therefore, a conclusion has been made that the nanoparticles serve mainly as a mean, which helps to preserve the dense dislocation structure in thick YBCO films [10].

This idea is confirmed by the analysis of $J_c$ thickness dependences for pure and doped YBCO films [11]. In this work the decrease of critical current density with film thickening is attributed to the dilution of dislocation nanostructure. A high temperature during the deposition results in a relaxation of the dislocation structure in undoped films. Thus, a slower $J_c$ decrease with thickening of multilayers and films containing nanoparticles can be interpreted as an increase of characteristic time of dislocation crawling due to their pinning on extra interfaces and nanoparticles.

The both mechanisms, the synergetic combination of different type defects and the retardation of evolution of the dislocation structure, take place beyond controversy and both completely recognize that the film growth kinetics plays a prime role. Therefore, the present work is aimed to investigate carefully properties and nucleation of BZO nanoparticles, to clear up conditions and mechanisms of their formation as well as an influence on the critical current density.

2. Samples

The superconducting films were manufactured by dual-beam pulse laser deposition [12] onto single crystal LaAlO$_3$ substrates. Two identical optical lines of Q-modulated Nd:YAG lasers (wavelength 1064 nm) and optical amplifiers were used. The pulse energy reached 250 mJ at 25 Hz repetition rate. Pure stoichiometric YBa$_2$Cu$_3$O$_{7-\delta}$ targets as well as doped with 1.5 and 3.0 mass% of BaZrO$_3$ were used. The targets were made by powder sintering of corresponding compounds prepared by solid state synthesis [13]. The substrate temperature was ranged from 750 to 800ºC. All other deposition conditions, such as gas pressure and composition (100 mTorr, 3:1 Ar/O$_2$ mixture), geometry of deposition, laser pulse energy and repetition rate were constant. The rate of deposition was about 25 nm/min. Film thickness was 150-300 nm. The superconducting transition temperature does not depend significantly on doping and is about 87 K.

A series of more than 50 samples at both transverse and planar orientations have been prepared for transmission (TEM) and high-resolution electron microscopic (HREM) studies. The phase composition and crystallographic orientation of surface layers were established by electron backscattering diffraction (EBSD).

3. Nanostructure of YBCO/BZO films

At first, the surface of doped YBCO films has been studied by TEM and EBSD. Numerous roundish particles are observed near the surface, from very small about 2 nm in diameter to very big up to 100 nm (figure 1a). The texture analysis evidences that the particle orientation absolutely coincides with YBCO matrix. The crystal structure of the particles is mainly orthorhombic, but about ~5% are cubic (SrZrO$_3$ type).
EBSD shows that the particles cover 70–90% of the surface. Such a large amount suggests that the particles segregate at the surface. Probably, they just have not enough time to form more equilibrium structure by volume diffusion. Indeed, a layer located at a certain distance below the surface and shown in figure 1b contains less number of particles. In addition, the composition of particles can differ from BaZrO$_3$. They are obviously depleted with zirconium.

The nanorods similar to those described in [1, 5-7] are seen in figure 2 showing TEM cross-section images of the films deposited at 765 and 800ºC. The typical diameter of nanorods is 5–20 nm. Like in other published pictures the dark and bright strips seen in figure 2 are not separate particles, but they are moiré fringes due to lattice mismatch [7, 14].

It is worth to remind general features of moiré fringes. Superposition of two grids of straight lines, we suppose those are atomic layers, would produce fringes in the perpendicular direction (vertical) if periods of the grids are the same but orientations are slightly different. The opposite case, that is the orientations are the same but the periods are different, results in horizontal fringes. Probably, this is the case in figure 2a indicating that the nanorods of different composition are absolutely aligned with the YBCO matrix.

If both orientations and periods of the line grids generating the moiré pattern are different a system of splayed lines is obtained. An angle between grids $\alpha$ and the difference in their periods $\Delta a/a$ can be easily calculated from the observed pattern: $\alpha = \arctg (a/d)$, $\Delta a/a = a/(d \tan \beta)$, where $d$ is the observed moiré fringe spacing and $\beta$ is their splay angle. For TEM image the period of generating grids can be assumed to be a distance between planes with heavier atoms, i.e. Ba and/or Y. Then, for YBCO the grid period is $c/3$ and $\Delta a/a = 5–7\%$ and $\alpha = 3–4^\circ$. These values are used to draw a model pattern shown in figure 3a.

HREM images show that the lattice parameter of the nanorods is larger than in YBCO. So, it is 0.413 ± 0.004 nm. It is slightly shorter than for pure BaZrO$_3$, for which $c = 0.419$ nm. Thus, the nanorods are compressed along the $c$-axis and their crystal lattice is inclined by 3–4º. It is clear that this happens to provide better matching with the matrix. As a result, any mismatch dislocations are not being formed in $ab$-plane at the interface between nanorod and YBCO matrix.

**Figure 1.** (a) Top surface of the film deposited using a target with 3% of BZO at 800ºC. (b) A layer approximately 50 nm below the surface of the same film.
Figure 2. Cross-section TEM images of the films deposited using a target with 3% of BZO showing (a) nanorods with aligned lattice and (b) nanorods with inclined crystal lattice. Arrows show direction perpendicular to the moiré fringes.

Figure 3. Model moiré patterns (a) produced by two systems of the same lines, relative difference of spacing $\Delta a/a = 5\%$, rotation angle $\alpha = 3^\circ$, (b) produced by two systems of lines, in which each third line is lighter than others, lines are not rotated and $\Delta a/a = 5\%$.

It should be noted that more complex pattern in figure 2a can also be easily modeled. For example, if each third line looks lighter than others the pattern shown in figure 3b is obtained. This is an absolutely natural assumption taking into account that an elementary cell of YBCO contains two atoms of barium and just one yttrium. An illusion of pairs of particles appears, but this is a moiré pattern. It can be distinguished from a real stack of particles, which indeed are observed sometimes but at higher magnification. Such a stack of particles is shown in figure 4.
Returning to nanorods, we can assert that their crystal structure can be either aligned or with the matrix or inclined by 3–4º. The nanorods often start from interfacial strongly damaged layer and finish at the free surface, but can also begin and end within the film like in figure 2 in the regions, where (001) planes are significantly deformed. The nanorods can be splayed from the normal direction as it was emphasized in [7].

It is worth to note that a strongly strained layer is seen at the interface with LaAlO$_3$ substrate for all deposition temperatures. The perfect crystal structure of the above layers indicates that the c-texturing is rather due to the strongly anisotropic perovskite structure of YBCO than to epitaxial mode of growth. The ability of the film to grow avoiding large-angle boundaries shows that the poorly ordered interfacial layer do not prevent transfer of the information about the substrate crystal orientation to the film growing above. This feature is extremely important for development of a technology for film deposition on a great variety of non-matching substrates.

Periodic changes of the contrast with a period of 30–50 nm seen in figure 2b are due to rotations of domains around the $c$-axis. The domains are unavoidably separated with low-angle dislocation boundaries and the nanorods are also predisposed to appear at these boundaries, especially at lower substrate temperatures. So, the dislocations may be involved in nucleation of the nanorods.

As we have seen already in figures 1b and 4, the doped films contained one more principally different type of nanoparticles but nanorods. They are planar and smaller in diameter, usually up to 3 nm in diameter. Such “nanopancakes” are arranged more or less regularly at 20–40 nm from each other, that is, at the distance comparable with the distance between low-angle boundaries.

The enlarged HREM image of an arbitrarily chosen nanopancake is shown in figure 5. The core of the particle has diameter of about 2 nm and it is surrounded with deformed area and dislocations aligned in $[001]$ direction, which are well seen after Fourier filtering. Analysis of the images estimates the number of such dislocations to be up 30–40 for each nanopancake. The larger nanorods do not form such a large number of dislocations. Lattice parameters $a$ of the nanopancakes and of the matrix usually coincide, but parameters $b$ are different. For the nanopancake it is 0.423 nm.

The observed total volume of nanoparticles is about 20%, that is, if such particles would consist of BaZrO$_3$ there would be a disagreement with the known contents of elements. This suggests that the composition of nanopancakes corresponds to Ba$_x$Y$_{1-x}$ZrO$_y$, where $y = 6$, as it was in [1] and [6]. Indeed, Ba$_2$YZrO$_6$ (BYZO) is a perovskite phase with $a = 0.425$ nm [6]. Actually, the particles are very small and their composition is variable. Therefore, it cannot be determined exactly by any technique.

In the film cross-section a single nanopancake is seen in figure 6 as brighter feature of 4–10 nm long and one elementary cell high. The planar nanoparticles can be assembled into stacks one over another or stepwise as it is shown in figure 4. The nanorods often start from the nanopancakes as in figure 7. This suggests that smaller planar particles are involved in nucleation of larger elongated nanorods. The $c$ parameter of the BYZO particle is slightly less than $c/3$ of YBCO, that is, BYZO the nanoparticles are compressed along the $c$-axis and the micro-strain is compensated by substitution of barium atoms (covalent radius 0.198 nm) with smaller yttrium atoms (0.162 nm).

Figure 4. Stacks of nanoparticles. Arrows show the stacking fault between the planar nanoparticles.
Figure 5. (a) HREM image of the BYZO nanopancake, [001] zone axis. (b) Position of [110] planes in the figure (a) determined by Fourier transformation. Extra half-planes, i.e., dislocations aligned in [001] directions, are well seen.

Figure 6. Cross-section HREM image of a single BYZO nanopancake.

Figure 7. A nanorod starting from a BYZO nanopancake.

The earliest stages of nanopancake nucleation can be also traced. According to the theory [15], $\frac{1}{2}[110]$ partial dislocations are directly involved in the structural transformation into BYZO phase. Indeed, figures 8a-c show that CuO planes are perfect in pure YBCO, while the dislocations are formed in doped samples figures 8d-f. The transformation occurs by substitution of copper with barium in CuO$_2$ planes embracing yttrium atoms with following partial substitution of barium with zirconium.
It can be concluded from the analysis of more than 100 diffraction patterns and EBSD data that BYZO phase exists in tetragonal BaTiO$_3$-type modification ($a = b = 0.425$ nm, $c = 0.412$ nm). The amount of this phase is about 70 vol.%, BaZrO$_3$ phase share is about 25 vol.% and 5 vol.% is fcc ZrO$_2$-type phase. The last one is shown in figure 9. YBCO become tetragonal in the vicinity of fcc nanoparticles because the matrix loose oxygen leaving to the particles.

It should be emphasized that there are both the nanorods and the nanopancakes in doped YBCO films deposited at all substrate temperatures. However, the concentrations of two types of nanoparticles depend on the deposition temperature. We cannot describe this ratio quantitatively, but qualitatively the higher deposition temperature, the greater content of nanorods and fewer nanopancakes. This observation is consistent with the hypothesis that small planar particles promote nucleation of the nanorods as well as with the result of [7]. It has been shown there that formation of columns is constrained by kinetic energy. A higher growth temperature should induce more ordered longer and aligned nanorods due to higher mobility of atoms. If the process is controlled only by free energy, the opposite result would be expected. In other words, the growth is governed by kinetics rather than thermodynamics.

Another argument for the kinetic mechanism of the nanorod growth is that at lower deposition temperatures the nanorods, which number is significantly reduced, are arranged mainly at low-angle and antiphase boundaries. That is, the boundaries promote formation of nanorods.

Dominating tiny planar nanoparticles, which form numerous edge dislocations around them, over the nanorods at lower deposition temperatures seem to be responsible for higher critical currents and better performance in applied field. This is true until increasing disorder due to less equilibrium growth conditions does not result in a reduction of the superconducting transition temperature and in a violation of $c$-axis texturing, appearance of $a$-oriented phase, which is destructive for a superconducting current flow.

**Figure 8.** (a), (b) and (c) Images of the YBCO matrix free of nanoparticles. Arrows show copper chains in [100] projection. (d), (e) and (f) Beginning of BYZO phase nucleation. Arrow shows missing copper atoms. Edge dislocations are seen in (e) and (f). (b), (c), (e) and (f) are obtained by Fourier transformation (filtering).
Figure 9. BYZO pancake in (010) projection. The contrast is due to local variations of chemical composition. Vertical arrows delimit fcc modification of BYZO, horizontal arrows show the staking fault. Cubic BYZO phase lies below the left horizontal arrow.

The nanopancakes seem also to serve as obstacles for thermally activated crawling of dislocations and their annihilation. As a result, the $J_c$ decrease with thickness should be essentially reduced. Thus, just $\text{Ba}_{1-x}\text{Y}_x\text{ZrO}_y$ nanopancakes appear to be the main reason of the critical current enhancement in the doped films due to (1) additional flux pinning on dislocations around the particles and (2) effective suppression of fast thermally activated relaxation of the dislocation nanostructure.

4. Measurement of superconducting properties of YBCO/BZO films

The critical current densities at different orientations of applied field have been evaluated for pure YBCO films and films with BaZrO$_3$ additives by magnetic susceptibility measurements using the critical state model [16]. The results presented in figure 10 show that $J_c$ is almost constant at applied field below 10 mT and decreases rapidly at higher fields. It is seen from the figure 10 that the films with BZO nanoparticles have higher critical current densities despite superconducting transition temperature decrease. Such a decrease is insignificant because the most of Zr is within nanoparticles and do not contaminate YBCO matrix.

I-V curves of the YBCO/BZO films in applied magnetic field have been measured using etched 90-100 µm bridges. Contact resistance did not exceed 1 Ω. The bridges transit to the resistive state with a transport current increase well before other regions of the designed superconducting structure, providing reliable measurements of the bridge resistance by conventional 4-probe technique. The transport properties of the YBCO films with BZO additives are measured in the temperature range from 78 to 150 K at all orientations the applied field up to 1 T. The electric field is measured with the accuracy of about 0.1 µV/cm, that is enough for detailed I-V recording.

I-V curves measured in perpendicular applied field for a thinner 150 nm YBCO-1.5% BaZrO$_3$ film deposited at 800ºC are shown in figure 11. The I-V curves at parallel field orientation are very similar. Only expected weaker influence of the parallel field on the critical current could be noticed. More attention should be paid to a negative curvature of the I-V dependences by a power law $E = E_c(J/J_c)^\mu$ gives n ranging from n = 21 at $E = 10$ µV/cm down to n = 11 at 100 µV/cm (at 78 K in zero applied field). Such a law is expected for nonhomogeneous vortex flow, which is observed along the domain boundaries in films with low critical current density and bicrystals [17-21]. Therefore, the vortices seem to move homogeneously in the investigated YBCO/BZO films and domain boundaries do not contribute to a superconducting current limitation.

However, the I-V curves can be well approximated in the whole measurement range by an expression $E = E_c \exp(-(J/J_c)^\mu)$, which is typical for a collective vortex creep or a creep in superconductors with extended linear or planar defects. Figure 12 shows field dependences of the $\mu$ exponent for perpendicular and parallel orientations of the applied field. The $\mu$ exponent is known to depend on a pinning mechanism and on vortex creep regime [22]. Collective pinning on weak point-like centers usually gives $\mu \leq 1$. In the case of pinning on extended defects $\mu = 1$ [22-25] for thermally activated depinning of vortices and $\mu = 3/2$ for plane defects [26]. The experimental results in figure 12 show that in field high enough to neglect a self field of the transport current $\mu = 1$ for perpendicular field and $\mu = 3/2$ for parallel field. Thus, in perpendicular applied field vortex pinning and creep can be supposed to be controlled by extended $c$-oriented linear defects, such as dislocations and nanorods, while at parallel field orientation planar defects, such as interfaces and/or the film surface, are effective.
Figure 10. Field dependences of the critical current density $J_c$ for samples with different concentrations of BaZrO$_3$ additive and different orientations of applied field $H \parallel c$ and $H \parallel ab$.

Figure 11. I-V curves for YBCO+1.5% BaZrO$_3$ film bridge. $T_s = 800^\circ$C. Magnetic field is perpendicular to the film.

Figure 12. Field dependence of the $\mu$ exponent involved in the function $E = E_c \exp(-J_0/J)^\mu$ approximating I-V curves for the film with 1.5% BaZrO$_3$, $T_s = 800^\circ$C, at different orientations of applied field.

5. Conclusion
Epitaxial YBa$_2$Cu$_3$O$_{7-\delta}$ films prepared by pulse laser deposition on LaAlO$_3$ single crystal substrates using a target doped with BaZrO$_3$ contain two principally different types of nanoparticles – nanorods and nanopancakes.

The first ones are elongated normally to the substrate along the $c$-axis of YBCO. Their diameter ranges from 5 to 20 nm. The composition rather well corresponds to BaZrO$_3$. The nanorods are nucleated within YBCO volume and they are epitaxially coupled with YBCO matrix. Dislocations are not formed around this type of particles.

The second type nanoparticles are tiny thin planar Ba$_x$Y$_{1-x}$ZrO$_y$ inclusions of variable composition and crystal structure. Their diameter is about 2 nm. They are surrounded with a complex system of dislocations both in $ab$-plane and along the $c$-axis. The diameter of strongly strained area near the nanopancakes is about 5 nm. The nanopancakes can serve as nuclei for the nanorods.

The ratio between two types of particles depends on the substrate temperature and the deposition rate. The higher temperature and the slower deposition, the greater number of nanorods. At lower substrate temperatures the nanorods are attracted to low-angle boundaries between the single-crystal domains. This allows concluding that nucleation and growth of nanorods is kinetic controlled.

Dominating nanopancakes with edge dislocations around them at lower deposition temperatures seem to be responsible for higher critical currents and better performance in applied field due to
additional flux pinning on dislocations, reduced flux creep and the effective suppression of fast thermally activated relaxation of the dislocation nanostructure. Therefore, they are preferable as artificial pinning centres.

Thus, introduction of nanosized BaZrO$_3$ additives, which form defect structure of the film during deposition, is a very promising way to increase critical current density in HTS materials, in particular to overcome the rapid $J_c$ fall with HTS film thickness.

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