Flux Pinning Enhancements of YBa$_2$Cu$_3$O$_{7-x}$ with Nanosize Magnetic Additions

M A Sebastian$^1$, N A Pierce$^2$, I Maartense$^1$, G. Kozlowski$^2$, T J Haugan$^3$

$^1$University of Dayton Research Institute, Dayton, OH 45469 USA
$^2$Wright State University, Dayton, OH 45435 USA
$^3$U.S. Air Force Research Laboratory, Aerospace Systems Directorate, WPAFB, OH 45433 USA

Author to whom correspondence should be addressed: sebastianm1@udayton.edu

Abstract. Different methods of flux pinning are being tested world-wide to enhance critical currents ($I_c$) of high temperature superconductor YBa$_2$Cu$_3$O$_{7-x}$ (YBCO) coated conductors exposed to high magnetic fields. Magnetic materials are interesting to consider as flux pinning additions because of their potential for very strong pinning strength. To our knowledge, there have been limited demonstrations of magnetic pinning additions to YBCO conductors. This paper describes the study of different M magnetic phase additions to YBCO including M = BaFe$_{12}$O$_{19}$, La$_{0.67}$Ca$_{0.33}$MnO$_3$, and other oxide phases. Nanosize additions were incorporated by depositing multilayer (M/YBCO)$_N$ films to minimize degradation of $T_c$, and testing volume % additions of M phase from 0.5 % to 5%. Results indicate that $T_c$ onsets are depressed with magnetic additions, however in some cases interestingly without degrading the transition width. With optimization of magnetic additions a 50% increase of critical current density has been obtained, for low magnetic fields of $< 10,000$ Oe at 65K to 77K. Microstructural and superconducting properties are summarized, including SEM analysis.

1. Introduction

Increased current density for YBaCuO$_{7-x}$ (YBCO) thin films can be accomplished by adding nano-size insulating phases [1]. Research has focused on addition of many single and multi-phases as inclusions of nanoparticles and nanorods such as: Y$_2$BaCuO$_5$ (Y211), Y$_2$O$_3$, Ba$_2$YTaO$_6$ (BYTO), BaSnO$_3$ (BSO), Ba$_2$YNbO$_6$ (BYNO), BaHfO$_3$ (BHO), and BaZrO$_3$ (BZO) [2]–[10]. These nanosized inclusions (additions) can be classified as one, two, and three dimensional pinning centers [11]. Resulting defects pin the normal core of the vortices of the type II superconductor. However, core pinning is limited by the coherence length of high temperature superconductors, which is usually on the order of $\xi = 25\AA$. The condensation energy of the Cooper pairs within the core, $U$, is equated to the maximum pinning energy:

$$U = \frac{(H_c^2/8\pi)\pi\xi^2}{(\Phi_0/8\pi\lambda)^2}$$

$H_c$ = critical field
$\Phi_0$ = flux quantum
\( \lambda_L = \) London penetration depth

In contrast, with magnetic pinning, the goal is theoretically to pin the magnetic flux of the vortex, instead of just the core. In this case, the pinning is limited by the magnetic penetration depth \( \lambda \), which is typically 1500 Å. The magnetic pinning strength is related to the penetration depth according to equation [2].

\[
\text{Magnetic pinning strength} = MH(\pi\lambda^2) \sim \Phi_0 M \quad (2)
\]

This results in a much larger columnar volume of pinning for magnetic pinning versus the core pinning [12]–[15]. This larger columnar volume results in the magnetic pinning strength being 100-1000 times the core pinning strength (figure 1). When an external magnetic field is applied perpendicular to the film plane of a YBCO film containing magnetic dopants, an S-shaped hysteresis loop versus a square shaped loop, indicates the presence of domain wall movement and magnetic pinning in the film. With perpendicular anisotropy, the magnetic pinning scales with the saturation magnetization. With in-plane anisotropy, magnetic pinning due to vortex and domain wall interaction occurs below the coercive field [14].

Several research groups have harnessed this additional pinning strength by ferromagnetic, antiferromagnetic, and ferrimagnetic additions to YBCO films. Chen Feng Tsai et al. compared and contrasted the addition of antiferromagnetic Fe\(_2\)O\(_3\) as a buffer layer for a YBCO film, as a multilayer composite with YBCO, and as a cap layer on top of YBCO film with and without the addition of CeO\(_2\). Their research found that the magnetic nanoparticles provided pinning in addition to the conventional defect pinning [16]. The majority of other research involved addition of soft and hard ferromagnets and ferrimagnets. Garcia-Santiago et al. produced YBCO films with hard ferromagnetic BaFe\(_{12}\)O\(_{19}\) (BFO) bilayer via pulsed laser deposition (PLD), utilizing a yttria-stabilized-zirconia (YSZ) buffer layer to decrease lattice mismatch and chemical reactivity. When compared to a YBCO film, the irreversibility line (IL) of the bilayer YBCO-BFO film shifted upward and above the IL of the YBCO film. The results explained by Bulaevskii’s theoretical approach are that “the effective magnetic field \( B \) in the YBCO layer is a sum of the applied magnetic field \( H \) and the demagnetizing field \( 4\pi M \) in the BFO layer: \( B = H + 4\pi M \). The magnetic domain walls are pinned if the applied field is lower than the coercive field, which is of the order of 5kOe for the BFO layer.” The stripe magnetic domain structure pins the vortices until the BFO film saturates at 10 kOe. The pinning enhancement
of a factor of two was estimated by comparing the ratio of the irreversibility fields for the YBCO-BFO film versus the YBCO film [12]. S. Wimbush et al. utilized mixed targets of 1-5 at. % iron oxides with YBCO to produce films by PLD. Results obtained showed that ferromagnetism and superconductivity were both present in the films at 77 K, and that the additions increased current density [17]. (Y1Fe5O12 (YIG) is ferromagnetic, and has been studied by several research groups. M. Gasmi et al. varied the concentration of YIG from 1-20 wt. % in YBCO powder pellets that were sintered. Magnetometer results showed that for an applied field \( H \) less than a threshold value, \( H^* \), superconducting domains dominate. The analysis showed that \( H^* \) decreased with increasing YIG concentration. This result is indicative of the YIG nanoparticles increasing the number of ferromagnetic domains in comparison with the YBCO superconducting domains. \( H^* \) also was found to decrease with temperature. When the applied field is greater than \( H^* \), the superconductor spin domains align with the applied field, forming a ferromagnetic phase along with the ferromagnetic domains on the surface. When the field is below \( H^* \), the spins in the superconducting domains reform Cooper pairs [18]. Q. Jia et al. also produced YBCO films with polycrystalline YIG substrates using YSZ and CeO2 buffer layers [19]. A. Bobyl et al. investigated a structure of YIG film on microstrip transducers and YBCO film, where the YBCO film acts as a shield for magnetostatic wave propagation [20]. Colossal magnetoresistance (CMR) compounds of \( La_{2/3}Ca_{1/3}MnO_3 \) (LCMO) and \( La_{0.6}Sr_{0.3}MnO_3 \) (LSMO) have also been studied with YBCO films. J. Chen et al., sputtered 8 layers of YBCO/LCMO. It was found that a decrease in the thickness of the LCMO layer, resulted in a decrease of the magnetoresistance of the multilayer film. At low temperatures, the multilayers were superconducting, while they were ferromagnetic at high temperatures [21]. H. Habermeier et al. used PLD to deposit bilayers of LCMO and bilayers of \( SrRuO_3 \) with YBCO on \( SrTiO_3 \) (STO) substrates. Magnetizing perpendicular to the film surface was found to increase current density. Current density was also found to increase with increasing field after zero field cooling, in contrast to the usual decrease in current density with increasing field seen in YBCO films [14]. J. Albrecht studied the effect of decoupling YBCO and LCMO with a STO space layer. It was found that the critical current density is affected by the magnetization of the LCMO layer, with significant magnetic pinning contributions shown. Substrate choice had an impact on induced strain, which affected the temperature dependence of the magnetic pinning contribution [22]. J. Huang researched LSMO + YBCO bilayers with CeO2, and found increased magnetic pinning at lower temperatures [23]. A. Meledin also investigated YBCO thin films with 6 mol. % MnFe2O4 (MFO) produced by chemical solution deposition, which resulted in 3D inclusions and lamellas within the YBCO film. These nano-inclusions resulted from reactivity and decomposition of the MFO with YBCO, and serve as artificial pinning centers [24].

Several issues need to be considered when incorporating magnetic nanoparticles into YBCO thin films, such as whether to utilize a hard or soft magnet phase, crystal epitaxy, particle orientation, magnetic domain size, chemical reactivity, operation temperature, oxides necessary for the formation of YBCO, and feasibility. The choice of substrate determines whether the epitaxial films are grown in tension or compression [22].

### Table 1. Magnetic Properties

| Material                  | Magnetic Type | \( \sigma_s \) (Am\(^2\)/kg) @ 298K | \( \sigma_s \) (emu/g) @ 273K | \( M_0 \) (emu/cm\(^3\)) @ 77K | \( T_{\text{curie}} \) (K) |
|----------------------------|---------------|-------------------------------------|--------------------------|-------------------------------|------------------|
| \( La_{0.67}Ca_{0.33}MnO_3 \) | ferromagnetic | 3.5 \( \mu_H \)                      | 93                      | 113 (@100K)                  | 240              |
| \( SrRuO_3 \)              | ferromagnetic | 1.5 \( \mu_H \)                      | 20                      | 75                            | 160              |
| \( Y_1Fe_5O_{12} \)        | ferrimagnetic | 25 \( \mu_B \)                      | 75                      | 560                           |                 |
| \( BaFe_{12}O_{19} \)      | ferromagnetic | 75 \( \mu_B \)                      | 723                     | 560                           |                 |
| \( Fe \)                   | ferromagnetic | 197 \( \mu_B \)                     | 1740                    | 1043                          |                 |

\[25\] [26] [27] [28] [29] [30] [14] [32] [36] [37] [38]
Based on the magnetization properties and lattice mismatch found in Tables I and II, it was chosen to compare multilayer films of YBCO with the following additions: BaFe$_{12}$O$_{19}$, LCMO, SrRuO$_3$, and Y$_3$Fe$_5$O$_{12}$.

Table 2. Magnetic Compounds Lattice Parameters and Mismatch with respect to YBCO

| Material | Lattice Type | Lattice Parameters (nm) | Lattice Parameter of Epitaxy (nm) | a-b in Plane | Lattice Mismatch to Y123 a (%) | Lattice Mismatch c-direction to Y123 (%) |
|----------|--------------|-------------------------|----------------------------------|--------------|---------------------------------|-------------------------------------|
| YBCO$^a$ | Orthorhombic | a = 0.3825               | $\tilde{a}$ = 0.3855             |              |                                 |                                     |
|          |              | b = 0.3886               |                                  |              |                                 |                                     |
|          |              | c = 1.166                | $c' = 0.3887$                    |              | +0.04                           | -0.8                                |
| La$_{0.67}$Ca$_{0.33}$MnO$_3$$^b$ | Orthorhombic | a = 0.5457               | $\tilde{a}' = 0.3857$           | +1.95        | +1.1                            |                                     |
|          |              | b = 0.5451               |                                  |              |                                 |                                     |
|          |              | c = 0.7708               | $c'' = 0.3854$                   |              |                                 |                                     |
| SrRuO$_3$$^c$ | Pseudo-cubic | a = 0.393                | $a' = 0.41253$                   | +7.0         | +6.1                            |                                     |
| Y$_3$Fe$_5$O$_{12}$$^d$ | Cubic | a = 1.2376               | $a'' = 0.41680$                  | +8.1         | -0.05                           |                                     |
| BaFe$_{12}$O$_{19}$$^e$ | Hexagonal | a = 0.58945              | $a''' = 0.3869$                  |              |                                 |                                     |
|          |              | b = 2.3215               |                                  |              |                                 |                                     |

$^a$ICDD 00-040-0159  $^b$[25]  $^c$[31]  $^d$[32]  $^e$ICDD 00-039-1433

2. Experimental Procedures

2.1. Target Preparation

The YBCO target was produced via solid state processing of dried commercial Nexans YBCO powder. The target was pressed utilizing a Carver die press with a 1.25 inch diameter die, and then sintered at 850°C for 60 hours and 920°C for 156 hours, achieving a final percentage of theoretical density of 93.4 %. BaFe$_{12}$O$_{19}$, La$_{0.67}$Ca$_{0.33}$MnO$_3$, Y$_3$Fe$_5$O$_{12}$, and SrRuO$_3$ were commercial targets purchased from SCI Engineered Materials Inc., STMC LLC JR Gaines, Kurt Lesker, and SCI respectively.

2.2. Thin Film Production via Pulsed Laser Deposition (PLD)

Multilayer composite films were produced via PLD with a KrF excimer laser ($\lambda = 248$nm) using the above targets and LaAlO$_3$ (LAO) and SrTiO$_3$ (STO) substrates. The BaFe$_{12}$O$_{19}$-YBCO composite films consisted of a layer of YBCO followed by a layer of BFO, repeating 28 times. PLD conditions were a heater block temperature of 790°C and a 300 mTorr O$_2$ atmosphere, with an energy of 650 mJ and pulse rate of 4 Hz. Pulses were varied and attenuating mirrors utilized to vary the thickness of the BFO layer, resulting in volume percent additions of 0.6, 1.4 and 2.7 vol. %. The LCMO-YBCO composite films were produced under the same conditions, with the exception of using a pulse rate of 2 Hz for the LCMO layers. Volume percent of LCMO additions were 0.8, 1.2, and 2.4%. The SrRuO$_3$ (SRO)-YBCO composite films repeated layers of YBCO and SRO 38 times under similar PLD conditions with the exception of a temperature of 800°C and a repetition rate of 2 Hz. with pulses and attenuating mirrors also varying the volume percent additions for SRO from 1.2, 2.4, and 4.7 vol.%.

The Y$_3$Fe$_5$O$_{12}$ (YIG)-YBCO composite films were similar to the SRO composites, with the exception of a temperature of 790°C. Volume percent additions varied from 3.6, 7.0, and 11.1 vol. %. All films were annealed for 30 minutes at 500°C and an oxygen atmosphere. All nominal vol.%’s were based on PLD calibration deposition rates.
2.3. Film Characterization

Film thickness was determined by etching the thin films with nitric acid and measuring the stair step thickness with a profilometer. Magnetic current density ($J_{cm}$) was measured with a Quantum Design Physical Properties Measurement System (PPMS) with a vibrating sample magnetometer (VSM) probe. The VSM data was attained for conditions of 77 K, 65 K, and 40 K with an applied field varied from 0-9 T for $H$ // $c$-axis of the films. The measured magnetic data was used to determine the current densities by utilizing a simplified form or the Bean Critical State Model: $J_{cm} = 30\Delta M / d a^3$ (A/cm$^2$), where $\Delta M$ is the change in magnetization, $d$ is the film thickness, and $a$ is the side length dimension of the square films [33]–[35].

Superconducting transition temperature ($T_c$) was attained via AC susceptibility measurements, with the amplitude varied from 0.025 Oe to 2.2 Oe, at a frequency of 4 Hz. Transport current density ($J_t$) was measured on microbridges 0.05 cm wide and 0.3 cm long, by a 4-point contact method with a 1µv/cm criteria. A FEI Sirion Scanning Electron Microscope (SEM) with ultra-high resolution at 5 kV and a spot size setting of 3 for nano-scale was used to obtain images of the films’ surface. X-ray diffraction analysis utilized the Bruker D8 Discover diffractometer with the following operation conditions: high resolution, Co tube 1.78897 Å, Power: 40kV, 35mA; 2-Theta: 5-80°. A Tescan Lyra FIB/SEM system was used to prepare a foil of YBCO/SRO sample for TEM examination by using TALOS TEM SYSTEM FSE under the acceleration voltage of 200 kV.

3. Results and Discussion

Magnetic nanoparticle addition effects on the onset critical temperature ($T_{c\text{-}onset}$) and the self-field current density ($J_{c\text{-}self\text{-}field}$) can be found in figure 2. Increased vol. % of BFO dramatically decreases $T_{c\text{-}onset}$ & $J_{c\text{-}self\text{-}field}$ and increases $T_c$ FWHM, compared to LCMO, SRO, and YIG, and may be the result of reaction products and strain development due to a higher degree of lattice mismatch with YBCO. Microstructure studies involved SEM, TEM, and XRD analysis. SEM imaging in Figure 3 depicts typical island growth and insulating phase nanoparticle charging. In Figure 4, TEM depicts very nice
epitaxial film growth and characteristic imaging of the YBCO film. Elemental analysis confirmed presence of the expected elements, (Ru and Cu are shown for example) and areas of the film that are copper rich. TEM was also in very good agreement with measured film thickness of 236 nm.

**Figure 3.** SEM micrographs of YBCO and multilayer magnetic addition/YBCO films.

Initial XRD analysis also indicates epitaxial growth of YBCO film identified by (00l) and substrate peaks. Magnetic addition peaks were minimal due to the low volume percent of the films analyzed (1.4-3.6%). Magnetic current densities, \( J_{cm} \), were attained at 77, 65, and 40K with the field ramped from 1 to 9T for each of the dopant systems, varying the volume percent. LCMO-YBCO 28 layer bi-layer films’ thickness averaged 347 nm, with YBCO layers ~ 11.0 nm/layer. From Figure 5(a) at 77K, 1.2 and 2.4vol.% LCMO films attained higher \( J_{cm} \) than an undoped YBCO film at fields < 1000 Oe and at fields <7000 Oe for 65K. At 40K, all three vol.% LCMO (0.8, 1.2, and 2.4) films attained \( J_{cm} \) higher than YBCO. BFO-YBCO 28 layer bi-layer films’ thickness averaged 300 nm, with YBCO layers ~ 11.0 nm/layer. From Figure 5(b) at 77K, the 1.4 vol.% BFO film attained higher \( J_{cm} \) for fields <5000Oe. BGO films of 1.4% and 0.6% performed better than YBCO films at 65K and 40K. Due to its poor results at 77K, 2.7 vol.% was not measured at lower temperature. YIG-YBCO 38 layer bi-layer films’ thickness averaged 254 nm, with YBCO ~6nm/layer. From Figure 5(c), at 77K, the multilayer films \( J_{cm} \) is higher than YBCO for fields > 10,000Oe, whereas all vol. % YIG outperform YBCO at 65K and 40K. SRO-YBCO 38 layer bi-layer films’ thickness averaged 240 nm, with YBCO
layer ~ 6nm/layer. From Figure 5(d), all vol.% SRO (1.2, 2.4, and 4.7%) outperformed YBCO film at 65, and 40K.

4. Conclusion
A comparison of the best vol.% film from each multilayer system studied shows that 3.6% YIG and 2.4 vol.% SRO outperform LCMO & BFO at fields greater than 10,000 Oe for all temperatures studied (77, 65, and 40K) (Figure 5(e)). The YIG and SRO films have very good Tc’s of 90K, with Tc FWHM of 1.6 and 1.8K, indicating good YBCO film structure. From magnetic hysteresis loops, Hsat is 3000 Oe for the YIG multilayer film and 2500 Oe for the SRO multilayer film. The “S” shaped hysteresis loop is indicative of domain wall movement and magnetic pinning in the films (Figure 6). The YIG (3.6%) and SRO (2.4%) multilayer films showed an increase of 25% performance of Jcm/Jc(YBCO) for H< 0.5T for 40-77K, and an increase of 35% and 70% respectively for Jcm/Jc(YBCO) for H>1T for 40-77K. This research shows the impact of combining magnetic and defect pinning in YBCO superconducting films.

Figure 6. Hysteresis Loops for SRO & YIG/YBCO multilayer films

Figure 5. Jcm vs magnetic field for YBCO multilayer films with varying vol.% (a) LCMO, (b) BFO, (c) YIG, (d) SRO, (e) comparison of vol.% dopant optimized multilayer films.

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