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Characterization of nano-composite M-2411/Y-123 thin films by electron backscatter diffraction and in-field critical current measurements

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Abstract. Thin films of nano-composite Y-Ba-Cu-O (YBCO) superconductors containing nano-sized, non-superconducting particles of $Y_2Ba_4CuMO_x$ (M-2411 with M = Ag and Nb) have been prepared by the PLD technique. Electron backscatter diffraction (EBSD) has been used to analyze the crystallographic orientation of nano-particles embedded in the film microstructure. The superconducting $YBa_2Cu_3O_7$ (Y-123) phase matrix is textured with a dominant (001) orientation for all samples, whereas the M-2411 phase exhibits a random orientation. Angular critical current measurements at various temperature (T) and applied magnetic field (B) have been performed on thin films containing different concentration of the M-2411 second phase. An increase in critical current density J_c at $T < 77$ K and $B < 6$ T is observed for samples with low concentration of the second phase (2 mol % M-2411). Films containing 5 mol % Ag-2411 exhibit lower J_c than pure Y-123 thin films at all fields and temperatures. Samples with 5 mol % Nb-2411 show higher $J_c(B)$ than phase pure Y-123 thin films for $T < 77$ K.

1. Introduction

The experimentally determined critical current density J_c of high-temperature superconducting (HTS) $YBa_2Cu_3O_7$ (Y-123) thin films is lower than the de-pairing current density predicted from Ginzburg-Landau theory. Various attempts have been undertaken to modify the chemical composition, microstructure and crystallinity of HTS thin films in order to increase the measurable J_c value. Periodic arrays of sub-micrometer holes or particles and columnar defects, for example, are effective in creating artificial pinning centers and in increasing the J_c value of HTS layers.

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Enhanced critical current densities are achieved also in nano-composite thin films consisting of a Y-123 matrix and nano-particles that are dispersed in the matrix. Nano-particles of various different materials such as $BaZrO_3$, $BaTiO_3$, Y_2BaCuO_5 (Y-211), Y_2O_3 and yttria-stabilized zirconia have been investigated [for example, see references 1, 2, 3].

Here we report on the characterization of nano-composite Y-123 thin films that contain $Y_2Ba_4MCuO_x$ (M-2411, M = Ag and Nb) nano-particles as secondary phase. Electron backscatter diffraction (EBSD) and transport measurements in magnetic field are employed to characterize the M-2411 / Y-123 nano-composite and pure Y-123 films.

2. Experimental

The HTS thin films were grown by pulsed laser deposition (PLD) on (001) single-crystal MgO substrates. A KrF UV-excimer laser (λ = 248 nm) was employed for ablation of ceramic targets at a pulse repetition rate of 10 Hz. Films were deposited in oxygen gas background (pressure 0.7 mbar) and post-annealed *in situ* after deposition (oxygen gas pressure 700 mbar). The ceramic targets used consisted of Y-123 as matrix material and of $Y_2Ba_4MCuO_x$ (M-2411, M = Ag and Nb) nano-particles embedded in the matrix. The M-2411 phase has a double-perovskite cubic structure and is chemically stable up to a high temperature (melting point above 1700 °C) [4, 5]. The content of secondary phase in ceramic targets was varied from 0 to 5 mol %. The thickness of films was ~ 200 nm for all samples as measured by atomic force microscopy (AFM). For electrical transport measurements the thin films were patterned into tracks of width 100 µm and length 1.0 mm by photo-lithography and wet-chemical etching. Au/Ag contact pads were evaporated onto the films after in-situ plasma cleaning of their surfaces. As-grown, unpatterned thin films were used for EBSD characterization.

The EBSD system consists of a FEI dual beam workstation (Strada DB 235) equipped with a TSL OIM analysis unit. The Kikuchi patterns were generated at an acceleration voltage of 20 kV and recorded by means of a DigiView camera system.

Angular critical current measurements at various temperature (T) and applied magnetic field (B) were performed using a 2-axis goniometer capable of rotating and tilting the sample inside of a 6 T magnet system in a helium flow cryostat. For transition temperature measurements, a current of 100 nA was applied to the sample and the voltage drop across the sample was measured. The current was then reversed to eliminate any thermal voltage effects. For the critical current measurements, the current was increased and the voltage drop across the sample measured until it reached 2 µV. The I-V curves obtained were analysed using a 0.5 µV criterion to determine the value of J_c.

3. Results and discussions

The M-2411/Y-123 nano-composite films reveal increased J_c values in self-field depending strongly on the composition of the target used for film deposition [6, 7]. For M-2411/Y-123 bulk superconductors, a linear increase of J_c with M-2411 content is observed [4].

Table 1. T_c, self-field J_c, and pinning force density f_p of Y-123 based thin film samples.

Ceramic targets used for PLD of HTS thin films	T_c [K]	J_c (77 K) [MA/cm^2]	J_c (65 K) [MA/cm^2]	f_p (1 T, 65 K) [GN/m^3]
Pure Y-123	88.7	1.98	4.96	17
Ag-2411 (2 mol %) / Y-123	89.9	4.18	8.42	31
Ag-2411 (5 mol %) / Y-123	84	0.199	1.35	5
Nb-2411 (2 mol %) / Y-123	89.6	2.76	7.05	20
Nb-2411 (5 mol %) / Y-123	87	1.89	6.28	24

Table 1 summarizes the critical temperature T_c, the self-field J_c at T = 77 K and 65 K, and the pinning force density $f_p = J_c(B, T) \times B$ measured at T = 65 K and B = 1 T (applied field oriented parallel to c-axis of layer, $H \parallel c$) of the HTS thin film samples. These results confirm our earlier data [6]. The pinning force density of sample Ag-2411 (2 mol %)/Y-123 is almost twice the force density of the pure Y-123 sample and three times higher than the value observed for NbTi measured at 4.2 K [1].

The in-field J_c measurements are shown on Figure 1. For all nano-composite films, except the sample Ag-2411 (5 mol %)/Y-123, the $J_c(B)$ measured for $H \parallel c$ is higher than for the pure Y-123 film (Figure 1a). The angular critical current measurements performed used the maximum Lorentz force configuration. This involves rotating the sample through an applied field with the field always perpendicular to the sample current. This configuration allows observation of *c*-axis pinning when the applied field is parallel to the *c*-axis (field angle $\theta = 0°$ and 180°). The sample Ag-2411 (2 mol %)/ Y-123 has a higher $J_c(\theta)$ for $H \parallel c$ ($\theta = 0°$ and 180°) than for $H \parallel a,b$ ($\theta = 90°$), as shown in Figure 1b. The anisotropy of this sample $J_c(0°) / J_c(90°) \approx 2$ is strikingly different as compared to all other films that show an anisotropy value less than 1.

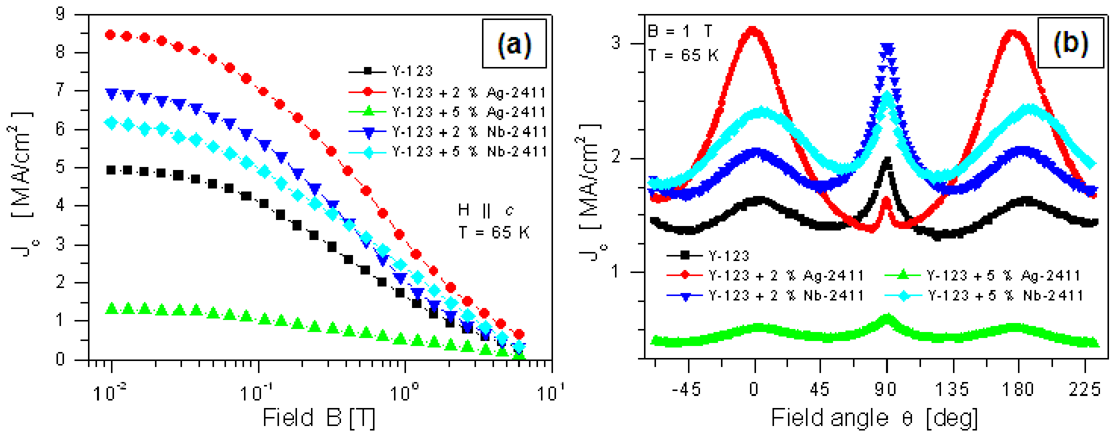

Figure 1. Critical current density of nano-composite M-2411 / Y-123 thin films measured at $T = 65$ K. J_c vs applied magnetic field B oriented parallel to c-axis (a). Angular dependence of J_c at $B = 1$ T (b).

Scanning electron microscopy (SEM) and EBSD images of pure Y-123 and of Ag-2411 (2 mol %)/ Y-123 thin film samples are shown in Figure 2. SEM images reveal much smoother surfaces of the nano-composite film (c) than for the Y-123 film (a). The EBSD phase contrast images are shown in Figures (b) and (d). The Y-123 phase is presented in red in both images. The film deposited from pure Y-123 ceramic contains Y-211 grains with a fraction of 15.5 % (shown as green in Figure 2b). The nano-composite film shows nanometer-scale Ag-2411 particles (shown as blue in Figure 2d) and no Y-211 grains. The Ag-2411 phase is uniformly distributed in the Y-123 matrix (2.6 % fraction) and the average Ag-2411 grain size is below 100 nm.

From the analysis of the inverse pole figure images (not presented here), we find that the Y-123 matrix is *c*-axis oriented and that the orientation of Ag-2411 nano-particles is random.

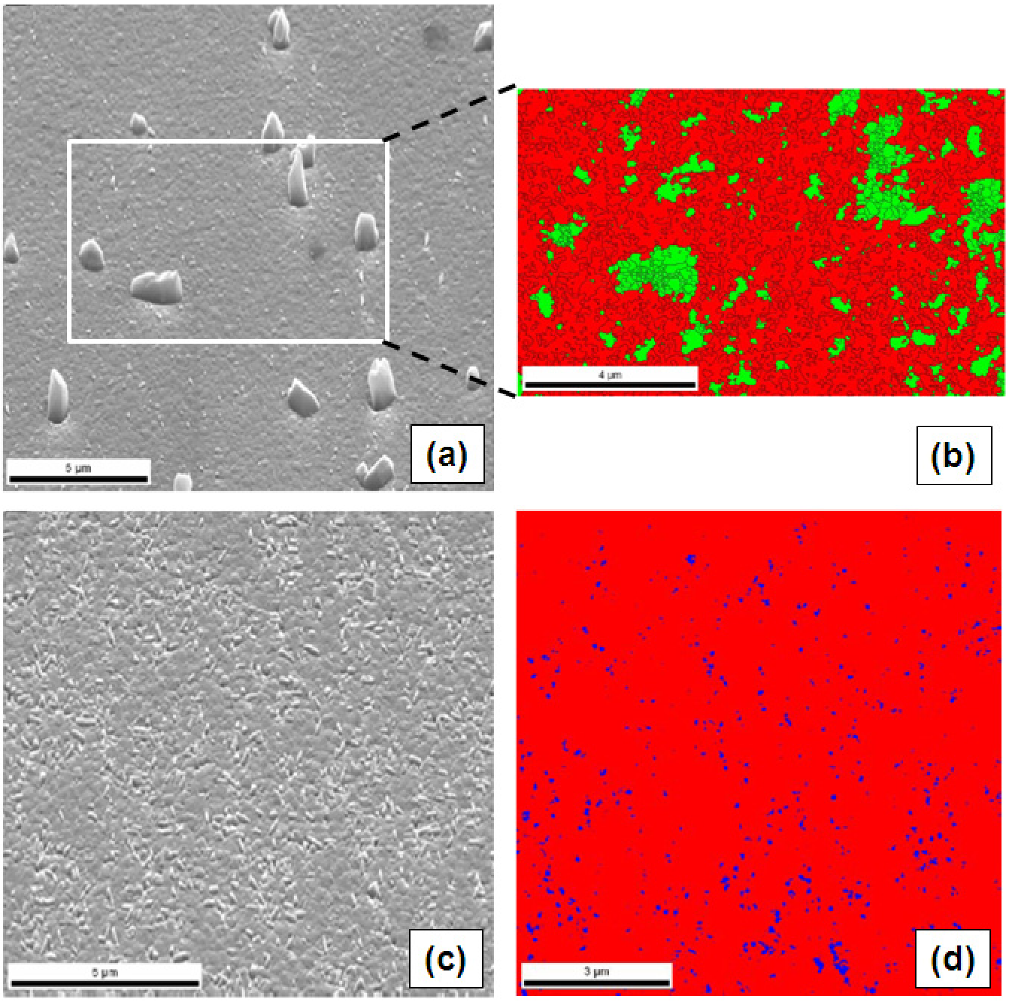

Figure 2. (a) SEM images of Y-123 film (a) and of Ag-2411 (2 mol %)/Y-123 film (c). Scale bars are 5 μm. EBSD phase contrast images of Y-123 film (b, scale bar is 4 μm) and of Ag-2411 (2 mol %)/ Y-123 film (d, scale bar is 3 μm). In the EBSD images the red, green and blue colours indicate the different phases of Y-123, Y-211, and Ag-2411.

The comparison of SEM and EBSD images indicates that the formation and clustering of Y-211 grains may be responsible for the relatively rough surfaces of Y-123 films. The smoother surface morphology, the absence of large fraction of Y-211, and the relatively homogeneous distribution of small Ag-2411 grains might be responsible for the increased critical currents of Ag-2411 (2 mol %)/ Y-123 thin film samples.

4. Conclusions

We have conducted angular critical current measurements and electron backscatter diffraction analysis of high-T_c superconducting M-2411/Y-123 nano-composite films and of Y-123 films. Layers fabricated by PLD from ceramic targets containing 2 mol % Ag-2411 or 2 to 5 mol % Nb-2411 show increased in-field J_c values. SEM and EBSD images show that nano-composite thin films are smoother and that the average grain size of nano-particles is smaller than 100 nm.

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