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**CONTROLLED INTRODUCTION OF FLUX PINNING  
CENTERS IN  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  FILMS DURING PULSED-  
LASER DEPOSITION (POSTPRINT)**

**C. Varanasi**

**University of Dayton Research Institute**

**P.N. Barnes, J. Burke, J. Carpenter, and T.J. Haugan**

**Mechanical Energy Conversion Branch  
Energy/Power/Thermal Division**

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## Controlled introduction of flux pinning centers in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films during pulsed-laser deposition

C. Varanasi

University of Dayton Research Institute, Dayton, Ohio 45469

P. N. Barnes, J. Burke, J. Carpenter, and T. J. Haugan

Air Force Research Laboratory, Propulsion Directorate, 1950 Fifth St. Bldg 450,  
Wright-Patterson AFB, Ohio 45433-7251

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To introduce controlled random inclusion of nanometer-sized nonsuperconducting particulates in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  films for flux pinning enhancement, a special pulsed-laser-ablation  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  target with a  $\text{Y}_2\text{BaCuO}_5$  sector was made and the films were deposited on  $\text{LaAlO}_3$  substrates. Initial results showed that the films consist of 10–20 nm-sized precipitates. In a  $0.5 \mu\text{m}$  thick film, a transport critical current density ( $J_c$ )  $> 3 \text{ MA/cm}^2$  at 77 K in self-field was measured. Magnetization  $J_c$  at 77 and 65 K showed significant improvements in these films with fine precipitates as compared to regular  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  films ( $> 10$  times increase at 9 T, 65 K). © 2005 American Institute of Physics. [DOI: 10.1063/1.2143112]

Introduction of flux pinning centers in melt textured  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (YBCO) is known to improve the critical current density ( $J_c$ ) of these materials significantly.<sup>1</sup> Approaches to obtain significant enhancements by incorporating nanometer sized particulates that act as flux pinning centers in YBCO thin films has also been recently demonstrated.<sup>2</sup> The importance of this work is underscored by research in several groups.<sup>3–6</sup> Significant improvements in  $J_c$  were demonstrated in pulsed laser deposited (PLD) YBCO films with  $\text{Y}_2\text{O}_3$ ,  $\text{Nd}_2\text{O}_3$ , and  $\text{BaZrO}_3$ <sup>3,4,7–9</sup> particulates. In the PLD method, particulates are generally introduced by two methods. One approach uses a single YBCO target prepared with finite amounts of pre mixed second phase materials.<sup>3,4</sup> The other approach is to use two different targets of YBCO and a second phase, and during deposition to switch them alternatively in a periodic fashion resulting in the introduction of island growth pinning centers in a layered fashion.<sup>7–9</sup>

When a single premixed target is used, it offers little control on the content, size and distribution of the pinning centers through the thickness of the films formed during the PLD deposition process. Also, the second phase selected needs to be nonreactive with YBCO at the sintering temperatures (900–950 °C) of the target which is often higher than the growth temperature of the films (750–780 °C).<sup>10,11</sup> On the other hand, if two separate targets are used, the second phase particles can be included in a more controlled fashion. A disadvantage of the dual targets, other than the necessity of alternating between targets, is that the pinning centers generally are arranged in a lamellar fashion, causing structured pinning centers with a more directional dependence of critical current density, controlled by the separation of the nanoparticulate layers. However, a random distribution of pinning centers in the films without the layered structure may be desired to avoid any preference for a given magnetic-field orientation. This is especially relevant since in superconducting coil applications, the magnetic field will be present at a variety of angles to the coils.<sup>12</sup>

A YBCO target made with a sector of the second phase material (Fig. 1) can be rotated at fixed speed during laser ablation. This will result in an YBCO film deposited along

with the second phase particles. When rotational speed is combined with a preferred laser raster control for a fixed angle sector, this approach offers a greater flexibility to process YBCO films with the second phase particulates that can be varied in terms of the content, and distribution along the thickness of the films. The particle size can be varied by the substrate temperature, chamber pressure and laser power density, etc. Additional control can be obtained by selecting a different sector angle. This technique allows separation of the respective constituents for a more controlled introduction of random nonlayered particulates. Also, since this method uses a single target as opposed to two different targets, it allows continuous fabrication to make long length coated conductor tapes. This novel method to introduce controlled random inclusion of particulates in YBCO films is presented here and the initial results on the films processes by this method are discussed.

A special pulsed laser ablation YBCO target with an  $\text{Y}_2\text{BaCuO}_5$  (Y211) sector was made and placed in a Neocera PLD chamber. Instead of the sector of Y211 specifically being inserted in a wedge shaped groove cut out in an YBCO target, a thin sector shape was taken from a disc of Y211 and attached on to the top of the YBCO target using silver paste. A Lambda Physik excimer laser (Model No. LPX 300, wavelength  $\lambda=248 \text{ nm}$ , KrF) was used to deposit the films at 780 °C using a 4HZ repetition rate. All the films in the present study were made using an YBCO target with an Y211 sector of  $\sim 30^\circ$ . The target was rotated at a speed of 15–20 rpm to obtain an approximate ratio of 11 pulses of YBCO to one pulse of Y211 during growth of the composite Y211/YBCO nano particle film.

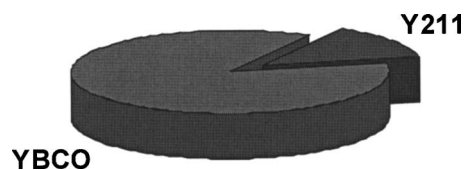


FIG. 1. Schematic diagram of a PLD target used in the present study showing a small sector made of Y211 in an YBCO target.

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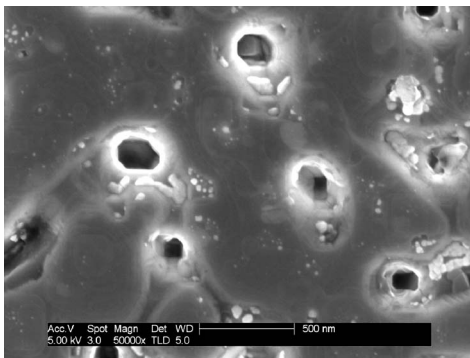
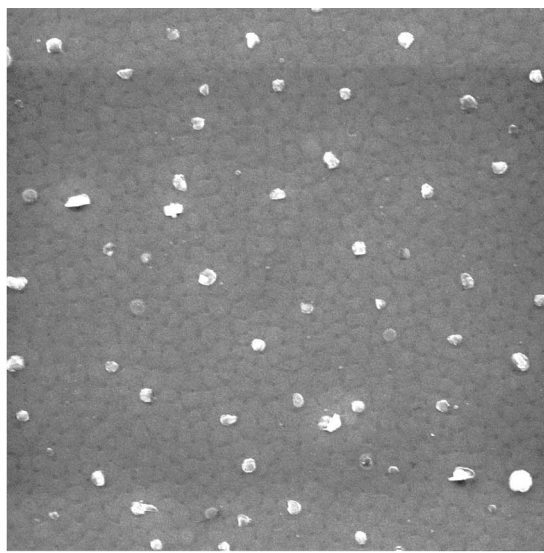
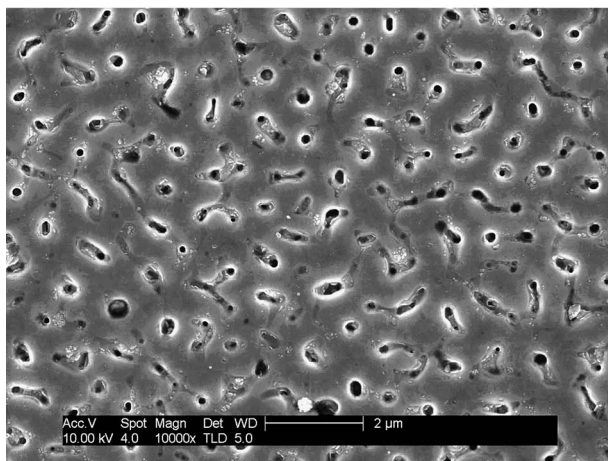


FIG. 2. Scanning electron microscope (SEM) micrograph showing the presence of nm sized precipitates (bright white precipitates) present in the YBCO film.

The film microstructure was studied using a SIRION (FEI Company) ultrahigh resolution field emission scanning electron microscope (FE-SEM). The critical transition temperature ( $T_c$ ) was measured by AC susceptibility and a vibrating sample magnetometer (VSM), Quantum Design PPMS system, was used to obtain magnetization  $J_c$  data at 77



(a)



(b)

FIG. 3. Scanning electron microscope (SEM) micrographs comparing the microstructures of (a) YBCO and (b) Y211/YBCO films grown in similar conditions on  $\text{LaAlO}_3$  substrates.

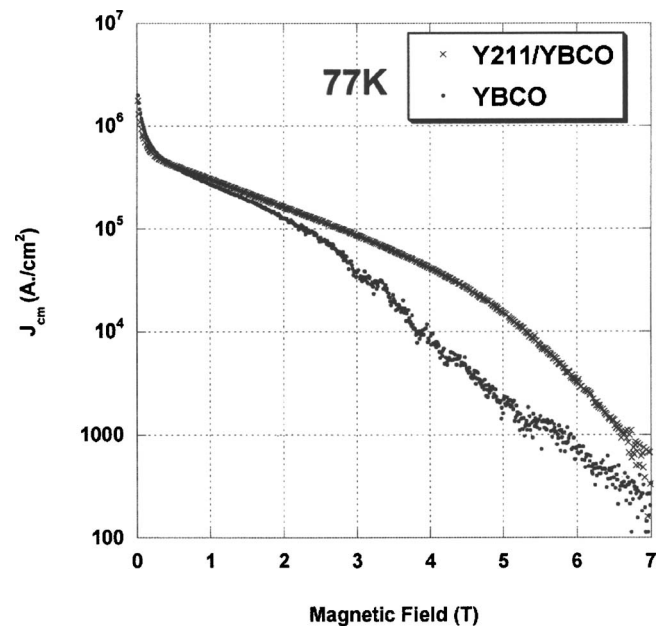


FIG. 4. Magnetization  $J_c$  measured at 77 K for a control YBCO and the sample with fine precipitates (Y211/YBCO) made using the pie shaped target.

and 65 K. Transport current measurements were taken on a  $0.5 \mu\text{m}$  thick, 4 mm long, 0.5 mm wide bridge sample. The thickness of the films was measured by using a profilometer and verified with cross-sectional SEM.

AC susceptibility measurements showed that all the films processed using the Y211 sector/YBCO target have a 90.5 K  $T_c$  onset with a sharp transition indicating that the incorporation of the second phase particles did not deteriorate the structure of YBCO films and are of good quality. Figure 2 shows a high resolution SEM photomicrograph of a film, which shows the presence of 10–20 nm sized spherical particles in the films. The spiral growth of YBCO grains can also be clearly seen; and evidently, the areas where the spirals fail to coalesce appear to form the pores visible in the

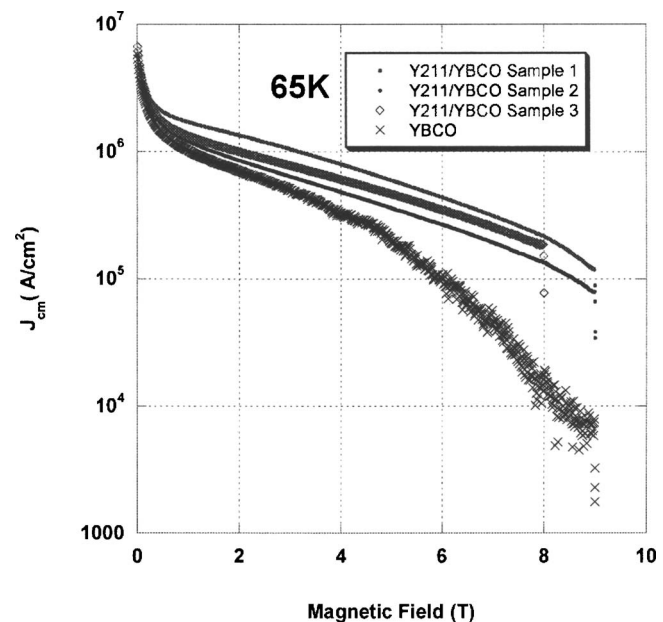


FIG. 5. Magnetization  $J_c$  measured at 65 K for three different Y211/YBCO samples compared to a standard YBCO film.

micrograph. The presence of 100–200 nm size pores were observed throughout the microstructure. The presence of the pores is likely due to the particular growth parameters of Y211/YBCO films as the regular pure YBCO films processed under similar growth conditions do not show this kind of porosity. Figure 3(a) shows lower magnification scanning electron microstructures of a YBCO film and Fig. 3(b) shows a Y211/YBCO film taken at the same magnification. It can be seen that the porosity is high in Y211/YBCO films as compared to pure YBCO films.

Figures 4 and 5 show the magnetization  $J_c$  data obtained at 77 and 65 K, respectively. For comparative purposes, data from a regular YBCO film on LaAlO<sub>3</sub> substrate is also plotted in these figures. It can be seen that the magnetization  $J_c$  is similar for both standard YBCO and YBCO/Y211 samples at lower fields up to 2 T for 77 K. However, as the magnetic field is increased, the  $J_c$  of the standard YBCO films falls more rapidly while the YBCO/Y211 films tend to better maintain higher  $J_c$  values indicating more effective pinning at higher fields in these preliminary films. However, significant pinning at a larger range of fields is clearly seen in the 65 K data presented in Fig. 5. In Fig. 5, data obtained from three different films of Y211/YBCO is compared to standard YBCO film. The pinning improvement can be clearly seen to occur in all the three film samples even up to 9 T, with one sample having more than 10 times (an order of magnitude) improvement in  $J_c$ . The transport current in 0.5  $\mu\text{m}$  thick films showed a  $J_c$  of 3.1 MA/cm<sup>2</sup> in self-field, 77 K. Since this is initial results only, optimization of the films should show improvement in the pinning strength, especially at lower fields for 77 K.

In summary, nanometer sized particulates were introduced by PLD into YBCO films using an YBCO PLD target with a second phase sector of Y211. Microstructure shows that YBCO films grown on LaAlO<sub>3</sub> substrates had nm sized particulates. Initial results indicate improvements in magnetization  $J_c$  at both 77 and 65 K especially at high fields in the composite Y211/YBCO films. An order of magnitude in-

crease in the magnetization  $J_c$  at 9 T, 65 K was obtained in the samples. The 0.5  $\mu\text{m}$  thick Y211/YBCO films have critical current density  $>3$  MA/cm<sup>2</sup> at 77 K, in self-field. Process optimization in terms of refinement of the particle size, content, and composition, can be anticipated to improve the properties further.

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