Biaxially Aligned Template Films Fabricated by Inclined-Substrate Deposition for YBCO-Coated Conductor Applications

Beihai Ma, Meiya Li, Rachel E. Koritala, Brandon L. Fisher, Alison R. Markowitz, Robert A. Erck, Steve E. Dorris, Dean J. Miller, and U. (Balu) Balachandran

Abstract-Inclined substrate deposition (ISD) has the potential for rapid production of high-quality biaxially textured buffer layers, which are important for YBCO-coated conductor applications. We have grown biaxially textured MgO films by ISD at deposition rates of 20-100 Å/sec. Columnar grains with a roof-tile surface structure were observed in the ISD-MgO films. X-ray pole figure analysis revealed that the (002) planes of the ISD-MgO films are tilted at an angle from the substrate normal. A small o-scan full-width at half maximum (FWHM) of ≈9° was observed on MgO films deposited at an inclination angle of 55°. In-plane texture in the ISD MgO films developed in the first 0.5 µm from the interface, then stabilized with further increases in film thickness. YBCO films deposited by pulsed laser deposition on ISD-MgObuffered Hastelloy C276 substrates were biaxially aligned with the c-axis parallel to the substrate normal. Tc of 91 K with a sharp transition and transport J_c of 5.5 x 10⁵ A/cm² at 77 K in self-field were measured on a YBCO film that was 0.46-µm thick, 4-mm wide, 10-mm long.

Index Terms—YBCO thin film, Coated conductor, Inclined substrate deposition, Biaxial texture

I. INTRODUCTION

 $2^{\rm ND}$ generation coated conductors and superconducting wires are promising for high-current-carrying applications and other electric power devices operating at temperatures that approach liquid nitrogen [1-3]. Textured template films or buffer layers are needed for deposition of biaxially aligned $YBa_2Cu_3O_{7\text{-}\delta}$ (YBCO) films to overcome weak links and, therefore, to achieve high critical current density (J_c) in the

Manuscript received August 5, 2001.

This work was supported by the U.S. Department of Energy (DOE), Eergy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-Eng-38.

B. Ma, R. E. Koritala, B. L. Fisher, R. A. Erck, S. E. Dorris, and U. Balachandran are with the Energy Technology Division, Argonne National Laboratory, Argonne, IL 60439 USA (corresponding author: Beihai Ma; phone: 630-252-9961; fax: 630-252-3604; e-mail: bma@anl.gov).

M. Li is with the Energy Technology Division, Argonne National Laboratory as a postdoc fellow.

A. R. Markowitz is with the Energy Technology Division, Argonne National Laboratory as a co-op student. She is a student in the Materials Science Department, Northwestern University, Evanston, IL 60208 USA.

D. J. Miller is with the Materials Science Division, Argonne National Laboratory, Argonne, IL 60439 USA.

YBCO films on metallic substrates [4]. Several techniques, including ion-beam-assisted deposition (IBAD), rolling-assisted biaxially textured substrates (RABiTS), and inclined-substrate deposition (ISD), have been developed in recent years [5-9]. When compared with IBAD and RABiTS, the ISD process produces textured films at high deposition rates (20-100 Å/sec) and is independent of the recrystallization properties of the metallic substrates [9].

We grew biaxially textured MgO thin films on mechanically polished Hastelloy C276 (HC) substrates by ISD with an electron beam (e-beam) evaporation system. Yttriastabilized zirconia (YSZ) buffer layers, ceria cap layers, and YBCO films were subsequently deposited on ISD-MgObuffered metallic substrates by pulsed laser deposition (PLD). Surface morphology was investigated by scanning electron microscopy (SEM), and surface roughness was measured by atomic force microscopy (AFM). The crystalline orientation of the films was studied by transmission electron microscopy (TEM). X-ray pole figures, as well as ϕ - and ω -scans, were used to analyze texture. In this paper, we discuss the growth mechanism, microstructure, and dependence of biaxial alignment of ISD MgO thin films on film thickness; we also report the orientation relationships and superconducting properties of YBCO fabricated using ISD MgO architecture on polished HC substrates.

II. EXPERIMENTAL PROCEDURE

HC coupons (≈5 mm wide and 10 mm long) were mechanically polished to a mirror finish with 0.25-um diamond paste for use as substrates. A surface roughness of ≈3 nm was measured by AFM. A schematic illustration of the experimental setup is given in Fig. 1. MgO thin films were grown from an MgO source by e-beam evaporation. Fused lumps of MgO (Alfa Aesar, 99.95% metals basis, 3-12 mm pieces) were used as the target material. The substrates were mounted on a tiltable sample stage above the e-beam evaporator. A substrate inclination angle α (substrate normal with respect to the evaporation direction) of 55° was used in this study. Oxygen flow was introduced into the system during film deposition. The base pressure of the vacuum system was 1 x 10^{-7} torr, which rose to \approx 2 x 10^{-5} torr during deposition. A quartz crystal monitor was mounted beside the sample stage to monitor and control the deposition rate. High -----

deposition rates of 20-100 Å/sec were used, and the substrate temperature was maintained between room temperature and 50°C during deposition. After the deposition of ISD films, a thin dense layer of MgO was deposited at a zero-degree inclination angle at elevated temperature (≈ 700 °C). This layer of dense MgO film has the same crystalline texture as the ISD MgO film, and is thus referred to as a homo-epitaxial MgO layer.

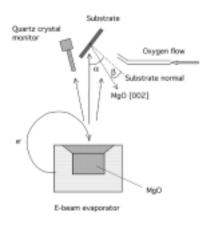


Fig. 1. Schematic illustration of experimental setup for ISD MgO.

YSZ, CeO₂, and YBCO films were deposited by PLD using a Lambda Physik LPX 210i excimer laser, with a Kr-F₂ gas premixture as the lasing medium. Commercial targets (Superconductive Components, 99.999% pure), 45 mm in diameter and 6 mm thick, were used. Substrates were attached to a heatable sample stage with silver paste and heated to a high temperature (700-800°C) during deposition. The size of the laser spot focused at the rotating target was ≈12 mm², which produced an energy density of ≈2.0 J/cm². The distance between the target and the substrates was ≈7 cm. The desired oxygen partial pressure was obtained by flowing ultra-high-purity oxygen through the chamber.

The superconducting critical transition temperature (T_c) and J_c were determined by the inductive method and confirmed by the transport method at 77 K in liquid nitrogen. Crystalline texture was measured by X-ray diffraction pole figure analysis using Cu- K_α radiation. In-plane texture was characterized by the FWHM of ϕ -scans for the MgO (002) reflection (2 θ = 42.9°), and out-of-plane texture was characterized by the FWHM of ω -scans at the MgO [001] pole for the same reflection. SEM and AFM were utilized to study morphology and surface roughness.

III. RESULTS AND DISCUSSION

Plan-view SEM (Fig. 2a) shows a roof-tile structure for the ISD MgO film deposited at room temperature with $\alpha=55^\circ.$ Columnar grains were observed on the cross-sectional fracture surface (Fig. 2b). The MgO grain size increased as the film grew up to a thickness of $\approx\!0.5~\mu m;$ thickness of film; it then stabilized at a grain size of $\approx\!0.2~\mu m$ without a noticeable change in size when the film grew further. The root-mean-

square (RMS) surface roughness was measured as 29 nm on an as-deposited ISD MgO film by tapping-mode AFM. E-beam evaporation of MgO at 700°C with a zero-degree inclination angle produced a smoother, dense homo-epitaxial layer (shown in Figs. 2c and 2d). RMS surface roughness was improved to ≈9 nm after the deposition of homo-epitaxial MgO films.

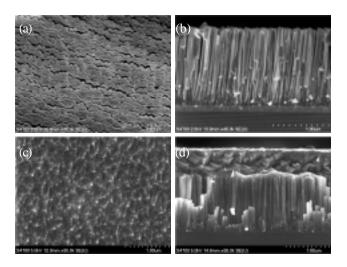


Fig. 2. (a) Plan view and (b) cross-sectional SEM images of ISD MgO film deposited at room temperature with $\alpha = 55^{\circ}$; (c) Plan view and (d) cross-sectional SEM images of MgO film after depositing additional layer of MgO e-beam evaporated at 700°C with $\alpha = 0^{\circ}$.

Typical X-ray pole figures of an ISD MgO film deposited with an inclination angle of 55° are shown in Fig. 3. Unlike the YSZ films prepared by inclined-substrate PLD [10], where the (001) planes are nearly parallel to the substrate surface, the [001] axis of the ISD MgO buffer layer is tilted away from the substrate normal. The asymmetric distribution of the pole peaks reveals that the MgO (001) planes have a tilt angle β toward the deposition direction. These ISD MgO films exhibit good texture; distinct in-plane alignment can be seen by the well-defined poles for not only the [001] axis but also the [010] and [100] axes in Fig. 3. Out-of-plane alignment was characterized by ω -scan; data were taken at the [001] pole.

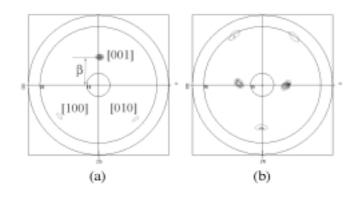


Fig. 3. (a) MgO (002) and (b) MgO (220) pole figures for an ISD MgO film deposited at room temperature with $\alpha = 55^{\circ}$.

The tilt angle, as determined from the chi angle value of the [001] reflection in the MgO (002) pole figure, was \approx 32°. TEM/selected area diffraction on the ISD MgO films confirmed that the top surface of an MgO columnar grain was terminated with a (002) plane; and MgO [002] was \approx 32° with respect to the substrate normal [11,12]. Figure 4 shows the ϕ -and ω -scan patterns for MgO (002) after homoepitaxially growing a 0.5- μ m-thick MgO layer on a \approx 1.5 μ m ISD MgO film at elevated temperature. FWHMs of 9.2 and 5.4° were observed in the MgO (002) ϕ -scan and ω -scan, respectively.

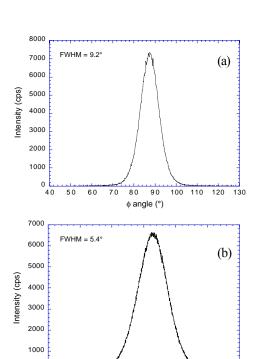


Fig. 4. MgO (002) (a) ϕ -scan and (b) ω -scan patterns after homoepitaxial growth of 0.5- μ m-thick MgO layer on ISD MgO film at elevated temperature.

20

ω angle (°)

25

15

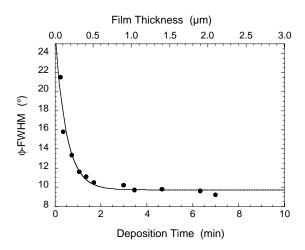


Fig. 5. Texture development in the ISD MgO film.

To study the thickness dependence of texture development in ISD MgO films, we first deposited ISD MgO films of various thicknesses followed by deposition of a $\approx\!0.5~\mu m$ homoepitaxial MgO layer at 700°C. Figure 5 shows the inplane texture as a function of ISD MgO layer thickness. In the first 0.5 μm , the ϕ -scan FWHM decreases rapidly with increasing film thickness; it then stabilizes at $\approx\!9^\circ$. Only $\approx\!1.5$ min is required to fully develop the desired texture at a deposition rate of 50 Å/sec. Furthermore, texture in the ISD MgO films has good tolerance to film thickness.

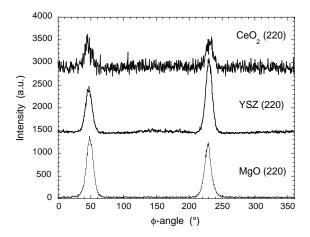


Fig. 6. (220) φ-scans for MgO, YSZ, and CeO₂ showing cube-on-cube epitaxial relationship.

YSZ and CeO₂ films were epitaxially grown on top of the homoepitaxial MgO film by PLD at elevated temperatures (700-800°C). Both CeO₂ and YSZ layers have a cube-on-cube epitaxial relationship with the MgO film underneath. (220) φ-scans plotted in Fig. 6 clearly showed a layer-by-layer epitaxy for MgO, YSZ, and CeO₂ films. Details of the epitaxial growth of YSZ and CeO₂ on MgO films will be reported elsewhere [12].

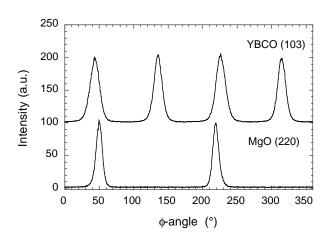


Fig. 7. ϕ -scans for MgO (220) and YBCO (103).

YBCO films deposited on YSZ- and CeO₂-buffered ISD MgO substrates were biaxially textured. FWHMs of 12° and 9° were measured from YBCO (103) and MgO (220) φ-scans, respectively, as shown in Fig. 7.

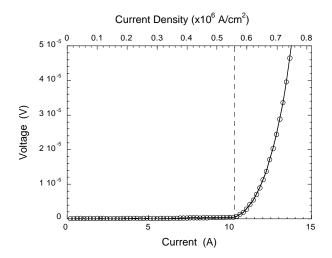


Fig. 8. Transport J_c of YBCO on ISD MgO measured at 77 K in self-field

The YBCO c-axis was parallel to the substrate normal, as illustrated by four evenly distributed peaks on the YBCO (103) ϕ -scan pattern. This revealed that the YBCO ab-plane was siting on a (112) plane parallel to the substrate surface. A unique orientation relationship, YBCO[100] // MgO[111] and YBCO[010] // MgO[110], was observed between the YBCO and ISD MgO template films. YBCO coated conductors fabricated by ISD MgO architecture exhibited a sharp superconducting transition with $T_c = 91$ K. As shown in Fig. 8, transport $J_c = 5.5 \times 10^5$ A/cm² at 77 K in self-field was measured on a sample that was 0.46- μ m thick, 4-mm wide, 1-cm long.

IV. CONCLUSIONS

Biaxially textured MgO films were successfully grown by the ISD method, which is much more time efficient for fabrication of buffer layers than is the IBAD YSZ process. MgO films grown by the ISD process contained columnar grains whose surfaces were terminated by (002) planes. Planview SEM revealed a roof-tile structure. The surface roughness and biaxial texture of the ISD MgO thin films were significantly improved by deposition of an additional thin layer of MgO at elevated temperature. FWHMs of 9.2 and 5.4° were observed in the MgO (002) φ-scan and ω-scan, respectively. Texture in the ISD MgO films developed rapidly in the first 0.5-µm of film growth, and then stabilized at FWHM ≈9° when the films grew thicker. CeO₂- and YSZbuffer layers were epitaxially grown on ISD MgO templates by PLD. YBCO films deposited on YSZ and CeO2 buffered ISD MgO substrates were biaxially textured with 12° FWHM measured on YBCO (103). A unique orientation relationship with YBCO[100] // MgO[111] and YBCO[010] // MgO[110] was observed between the YBCO films and ISD MgO template layers. YBCO coated conductors fabricated with ISD MgO architecture exhibited a sharp superconducting transition with $T_c = 91$ K and transport $J_c = 5.5 \times 10^5$ A/cm² measured at 77 K in self-field.

ACKNOWLEDGMENT

SEM/TEM analysis was performed in the Electron Microscopy Center for Materials Research at Argonne National Laboratory. This work was supported by the U.S. Department of Energy (DOE), Energy Efficiency and Renewable Energy, as part of a DOE program to develop electric power technology, under Contract W-31-109-Eng-38.

REFERENCES

- [1] D. K. Finnemore, K. E. Gray, M. P. Maley, D. O. Welch, D. K. Christen, and D. M. Kroeger, "Coated Conductor Development: An Assessment," *Physica C*, 320, 1-8 (1999).
- [2] Y. Iijima and K. Matsumoto, "High-Temperature-Superconductor Coated Conductors: Technical Progress in Japan," *Supercond. Sci. Technol.*, 13, 68-81 (2000).
- [3] J. O. Willis, P. N. Arendt, S. R. Foltyn, Q. X. Jia, J. R. Groves, R. F. DePaula, P. C. Dowden, E. J. Peterson, T. G. Holesinger, J. Y. Coulter, M. Ma, M. P. Maley, and D. E. Peterson, "Advance in YBCO-Coated Conductor Technology," *Physica C*, 335, 73 (2000).
- [4] D. Dimos, P. Chaudhari, and J. Mannhart, "Superconducting Transport Properties of Grain Boundaries in YBa₂Cu₃O₇ Bicrystals," *Phys. Rev.* B, 41, 4038-4049 (1990).
- [5] Y. Iijima, M. Kimura, T. Saitoh, and K. Takeda, "Development of Y-123-Coated Conductors by IBAD Process," *Physica C*, 335, 15 (2000).
- [6] C. P. Wang, K. B. Do, M. R. Beasley, T. H. Geballe, and R. H. Hammond, "Deposition of In-plane Textured MgO on Amorphous Si₃N₄ Substrate by Ion-Beam-Assisted Deposition and Comparisons with Ion-Beam-Assisted Deposited Yttria-Stabilized-Zirconia," *Appl. Phys. Lett.*, 71, 2955-2958 (1997).
- [7] A. Goyal, D. P. Norton, J. D. Budai, M. Paranthaman, E. D. Specht, D. M. Kroeger, D. K. Christen, Q. He, B. Saffian, F. A. List, D. F. Lee, P. M. Martin, C. E. Klabunde, E. Hardtfield, and V. K. Sikka, "High Critical Current Density Superconducting Tapes by Epitaxial Deposition of YBCO Films on Biaxially Textured Metals," Appl. Phys. Lett., 69, 1975 (1996).
- [8] M. Bauer, R. Semerad, and H. Kinder, "YBCO Films on Metal Substrates with Biaxially Aligned MgO Buffer Layers," *IEEE Trans. Appl. Supercond.*, 9, 1502 (1999).
- [9] B. Ma, M. Li, Y. A. Jee, B. L. Fisher, and U. Balachandran, "Inclined Substrate Deposition of Biaxially Textured Magnesium Oxide Films for YBCO Coated Conductors," *Physica C*, 366, 270-276 (2002).
- [10] K. Hasegawa, K. Fujino, H. Mukai, M. Konishi, K. Hayashi, K. Sato, S. Honjo, Y. Sato, H. Ishii, and Y. Iwata, "Biaxially Aligned YBCO Film Tapes Fabricated by All Pulsed Laser Deposition," *Appl. Supercond.*, 4, 487-493 (1996).
- [11] U. Balachandran, B. Ma, M. Li, B. L. Fisher, R. E. Koritala, R. Erck, and S. E. Dorris, "Fabrication by Inclined-Substrate Deposition of Biaxially Textured Buffer Layer for Coated Conductors," Proceedings of Materials Research Society Fall 2001 Meeting, Boston, Nov. 25-29, 2001.
- [12] B. Ma, et al., "Pulsed Laser Deposition of Biaxially Textured YBCO Films on ISD MgO Buffered Metal Tapes," to be submitted to Supercond. Sci. Tech., (2002).