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S. E. Dorris, B. Ma, M. Li, B. L. Fisher, R. E. Koritala, R. Erck,
D. E. Miller,[†] and U. (Balu) Balachandran

Energy Technology Division
[†]Materials Science Division
Argonne National Laboratory
Argonne, IL 60439

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FABRICATION OF BIAXIALLY TEXTURED TEMPLATES FOR COATED CONDUCTORS BY INCLINED SUBSTRATE DEPOSITION*

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Energy Technology Division
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Argonne National Laboratory
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ABSTRACT

YBCO-coated conductors will enable the development of smaller, lighter, more-efficient electric power devices that can be operated at temperatures approaching that of liquid nitrogen. The critical current density (J_c) of YBCO films on flexible metallic substrates has been significantly improved by epitaxially growing the YBCO on biaxially textured template films. Inclined substrate deposition (ISD) offers the potential for rapidly producing high-quality biaxially textured buffer layers that are suitable for YBCO-coated conductors. Using the ISD method, we have grown biaxially textured MgO films at deposition rates of 20-100 Å/sec. Electron microscopy of the ISD-MgO films revealed columnar grains topped off by MgO (002) planes, and X-ray pole figure analysis showed that the (002) planes are tilted with respect to the substrate normal, giving ISD-MgO films a roof-tile surface morphology. A small phi-scan full-width at half maximum of 10° was observed on ISD-MgO films deposited with a substrate inclination of 55° . YBCO films were grown on ISD-MgO buffered Hastelloy substrates by pulsed laser deposition. A sample that was $0.42 \mu\text{m} \times 0.45 \text{ mm} \times 1 \text{ cm}$ gave a transport J_c of 0.34 MA/cm^2 at 77 K in self-field. Details of ISD-MgO film fabrication and characterization, as well as the results of YBCO deposited on the ISD-MgO architecture, are presented.

INTRODUCTION

Smaller, lighter, more-efficient power devices such as motors, generators, transformers, transmission cables, and fault-current limiters that can be operated near the temperature of liquid nitrogen will be made possible by the development of YBCO-coated conductor technology [1-3]. The critical current density (J_c) of YBCO-coated conductors has been significantly improved by techniques that allow epitaxial growth of YBCO films on biaxially textured template films. With two of these techniques, ion-

beam-assisted deposition (IBAD) and inclined-substrate deposition (ISD), biaxially textured template structures have been fabricated on non-textured metallic substrates [3-5]. In comparison to the ISD process, IBAD of yttria-stabilized zirconia (YSZ) is relatively complicated and time-consuming, because an assisting ion source is necessary, and good in-plane texture requires a YSZ film thickness $>0.5 \mu\text{m}$ deposited at $1 \text{ \AA}/\text{sec}$. The ISD process using electron-beam evaporation of MgO produces well-textured films at significantly higher deposition rates ($20\text{-}100 \text{ \AA}/\text{sec}$), does not require an assisting ion source, is easier to accomplish, and does not depend on the recrystallization properties of the metallic substrates [4-6]. This paper describes the microstructure and surface morphology of ISD-MgO thin films on Hastelloy C276 (HC) substrates, and it shows the effect of substrate inclination on the biaxial alignment in these films. In addition, the paper presents the superconducting properties of YBCO films that are deposited on biaxially textured, ISD-MgO-buffered HC substrates.

EXPERIMENTAL

Mechanically polished HC, 0.1 mm thick \times 10 mm long \times 5 mm wide, was used as the substrate for ISD of MgO. ISD-MgO films were grown at high deposition rates ($20\text{-}100 \text{ \AA}/\text{sec}$) by electron beam (e-beam) evaporation using fused MgO from Alfa-Aesar (99.95% metals basis) as the target material. The substrate inclination (shown in Fig. 1 as the angle between the substrate normal and the evaporation direction) was varied from 10 to 70° in order to study its effect on the film texture. During the deposition, oxygen was introduced to maintain an operating pressure of 2×10^{-5} torr, while the substrate temperature was maintained at $<50^\circ\text{C}$. Surface roughness of the films was measured by atomic force microscopy (AFM). The surface roughness of as-deposited ISD MgO films was reduced by depositing a homoepitaxial layer of MgO at $700\text{-}800^\circ\text{C}$ with $\theta = 0^\circ$.

Thin layers of YSZ and ceria were deposited on top of the MgO-buffered substrates to improve the lattice match with YBCO. Buffered substrates were attached with silver paste to a heatable sample stage, and YBCO films were deposited by pulsed laser deposition (PLD) using an excimer laser system (Lambda Physik COMPex 201). During deposition of YBCO, the substrate temperature was controlled at $700\text{-}800^\circ\text{C}$ and the oxygen partial pressure at $200\text{-}300 \text{ mtorr}$.

Surface morphology and crystalline orientation of the films were investigated with a Hitachi S4700 field-emission scanning electron microscope (SEM) and a Philips CM30

transmission electron microscope (TEM). AFM was done with a Digital Instrument NanoScope. Textures were studied by X-ray diffraction pole figure analysis with Cu-K radiation using a Bruker D8 Discover X-ray diffractometer (XRD) and a GADDS (General Area Detector Diffraction Solution) detector. The in-plane texture of the films was characterized by measuring the full-width at half-maximum (FWHM) of ω -scans for the MgO (002) reflection; the out-of-plane texture was characterized by the FWHM of MgO [001] ω -scans. Inductive and transport methods were used to measure the superconducting critical transition temperature (T_c) and critical current density (J_c). Transport measurements were made on samples that had been coated with silver (thickness = 2 μm) by e-beam evaporation and then annealed in flowing high-purity oxygen for 2 h at 400°C.

RESULTS

Figure 2 shows SEM images of an ISD MgO film that was deposited at room temperature with an θ of 55°. The "roof-tile" structure of the film is evident in the plan-view (Fig. 2a), while the cross-sectional view shows that the film contains columnar grains that are nearly perpendicular to the substrate surface (Fig. 2b). For thin (<0.25 μm) films, the MgO grain size increased with the film thickness, but the grain size (0.1 μm) was nearly independent of thickness for film thickness >0.25 μm .

The faceted nature of ISD MgO films is clearly shown in the TEM images (Fig. 3) of a film grown with $\theta = 55^\circ$. In these images, the columnar grains are oriented horizontally, while the substrate runs in the vertical direction, so the (002) planes atop the MgO columns are tilted with respect to the substrate. The in-plane texture develops during deposition due to the combined effect of fast growth along the MgO {200} plane and self-shadowing due to deposition at an inclined angle [8, 9]. Because maximizing the (002) faces can decrease the surface free energy, the {200} plane is also the equilibrium crystal habit, as confirmed by the cubic morphology exhibited in the MgO film [4]. During inclined deposition, the {200} plane rotates toward the vapor source, so that the (002) surface grows faster than other crystalline faces.

The growth of tilted (002) facets on top of the MgO columns results in a sawtooth appearance in the cross-sectional view (Fig. 3) and gives rise to an RMS roughness of 28 nm. However, the roughness is decreased significantly (to 10 nm) by depositing a 0.5- μm -thick homoepitaxial MgO layer at 700°C with $\theta = 0^\circ$. Figure 4 shows plan and cross-sectional SEM images of an MgO film after deposition of a homoepitaxial layer.

In addition to obviously improving the surface smoothness, the homoepitaxial layer provides a denser structure. X-ray analysis showed that the homoepitaxial layer also sharpened the biaxial texture of the film. After deposition of homoepitaxial MgO, the FWHM of an MgO (002) ω -scan decreased by 2° , while the FWHM of the χ -scan decreased by 1° .

Figure 5 shows the MgO (002) and (220) X-ray pole figures for a film (thickness = $1.5 \mu\text{m}$) that was deposited at room temperature with $\chi = 55^\circ$. The poles are sharp and well-defined for the [001] axis as well as the [010] and [100] axes, indicating good biaxial texture in the film. The pole peaks are not centered relative to the pole figure's origin because the MgO (00 l) planes are tilted with respect to the substrate normal, as shown in Fig. 3. Using the measured ω -angle for the [001] reflection of the MgO (002) pole figure, we determined that the tilt angle, θ , was 32° for the ISD MgO film deposited at room temperature with an inclination of 55° .

To identify the deposition conditions that provide the sharpest biaxial texture, we grew ISD MgO films at inclinations in the range of 10 - 70° , and measured the FWHMs of ω - and χ -scans for the films. Figure 6 plots the FWHMs of ω - and χ -scans for MgO (002) as a function of χ . Clear minima in the FWHMs were observed for $\chi = 30^\circ$ and $\chi = 55^\circ$. Depositions at an inclination of 55° resulted in the smallest FWHM for the ω -scans (12.2°) and gave a θ of 32° . Depositions at an inclination of 30° yielded the minimum FWHM for χ -scans (5.6°) and gave a tilt angle of $\theta = 22^\circ$. Even though the FWHMs for ω - and χ -scans are minimized at different inclinations, they both show local minima for either $\chi = 30^\circ$ or 55° , and the biaxial texture is good for either of these inclinations.

Because θ varies with the substrate inclination, it cannot be varied without influencing the texture of the MgO film. For the present work, ISD-MgO films were deposited with $\chi = 55^\circ$, giving a tilt angle of 32° . To sharpen the texture and reduce the surface roughness of the MgO template, a thin ($0.5 \mu\text{m}$) homoepitaxial layer of MgO was deposited with $\chi = 0^\circ$ on top of the ISD MgO layer. Because the lattice mismatch between YBCO and MgO is large (8.5%), various buffer layers were deposited on the MgO to reduce the mismatch with YBCO. The buffer layers were applied by either e-beam evaporation or PLD; YBCO was deposited by PLD.

The ω -scans for MgO (220) and YBCO (103) that were measured from YBCO on ISD-MgO-buffered HC indicate cube-on-cube biaxial alignment of the YBCO on MgO: YBCO [001] // MgO [001] and YBCO [100] // MgO [100] (or MgO [010]). Inductive

measurements on YBCO deposited on ISD-MgO-buffered HC showed a $T_c(\text{onset})$ of 90 K with a transition width of 2 K. Transport measurements (Fig. 7) indicated a J_c of 0.34 MA/cm² at 77 K in self-field on a sample that was 0.42 μm thick, 4.5 mm wide, and 1 cm long.

CONCLUSIONS

MgO films with good biaxial texture were grown on HC substrates by the ISD method. The MgO (002) ω - and χ -scans for as-deposited ISD-MgO films gave FWHM values of 12.2 and 6.3°, respectively, showing good in-plane and out-of-plane texture. SEM of the ISD-MgO surface revealed a roof-tile surface morphology, while a cross-sectional view showed a sawtooth structure in which columnar MgO grains were oriented perpendicular to the substrate surface and were topped by MgO (002) planes that were tilted with respect to the substrate. The sawtooth structure gives rise to a surface roughness of 28 nm for as-deposited ISD-MgO. The surface roughness was decreased to 10 nm by depositing a thin (0.5 μm) homoepitaxial MgO layer on the ISD-MgO at elevated temperature with 0° inclination. The homoepitaxial MgO layer also improved the biaxial texture, reducing the MgO (002) ω - and χ -scan FWHM values to 9.2 and 5.4°, respectively. YBCO deposited on ISD-MgO-buffered HC gave a sharp superconducting transition with a $T_c(\text{onset})$ of 90 K and a transition width of 2 K and a transport J_c of 0.34 MA/cm² at 77 K in self-field.

ACKNOWLEDGMENT

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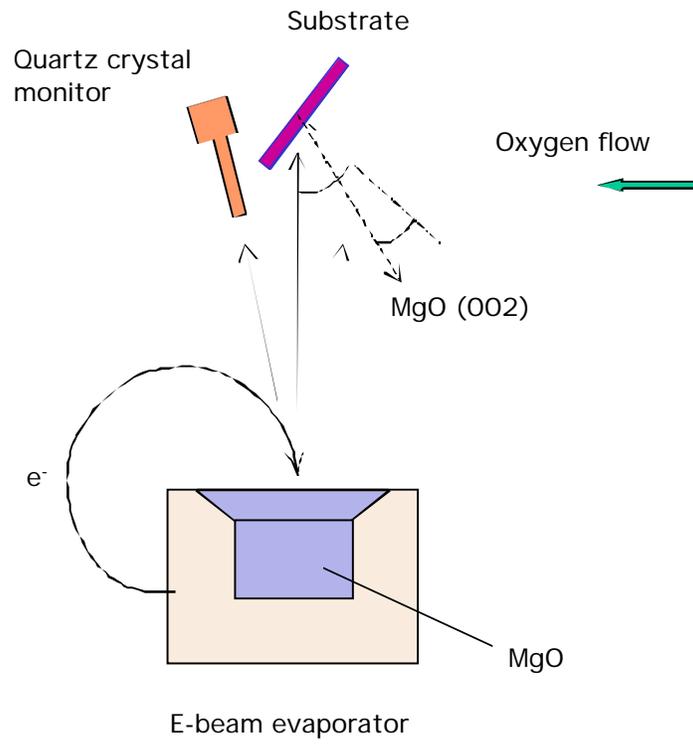
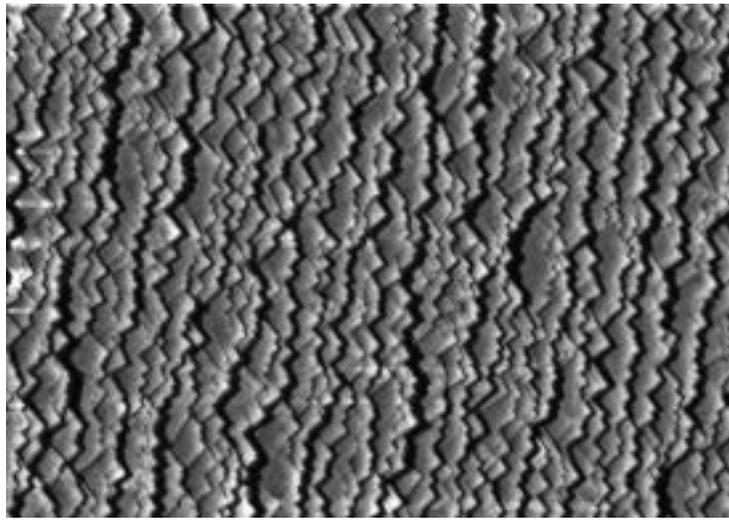
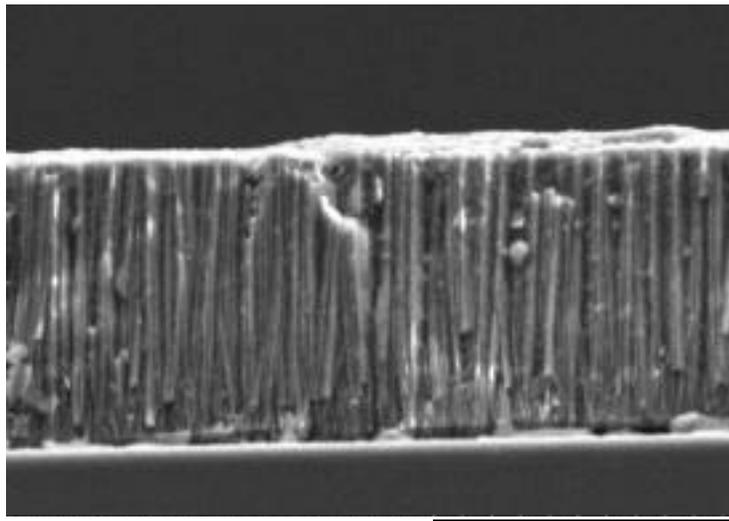


Fig. 1. Experimental arrangement for inclined substrate deposition (ISD) of MgO on HC substrate.



(a)



(b)

Fig. 2. SEM photomicrographs of ISD MgO film: (a) top surface and (b) cross section.

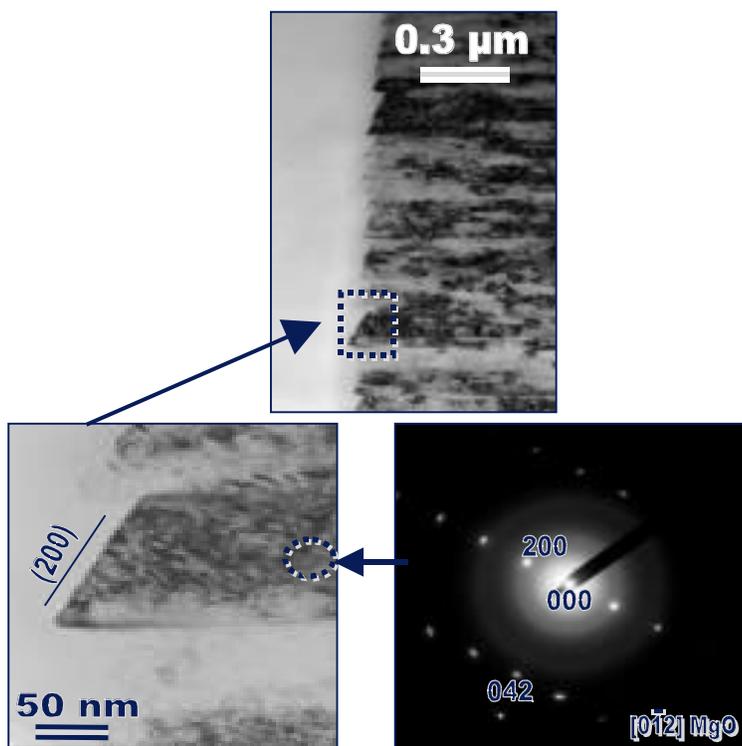
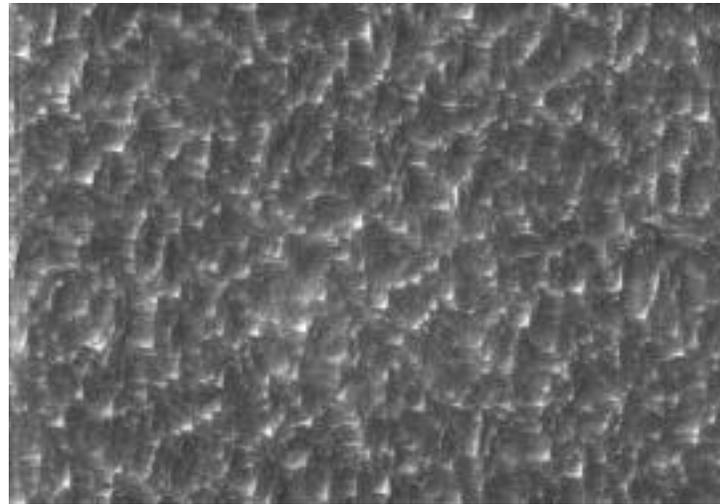
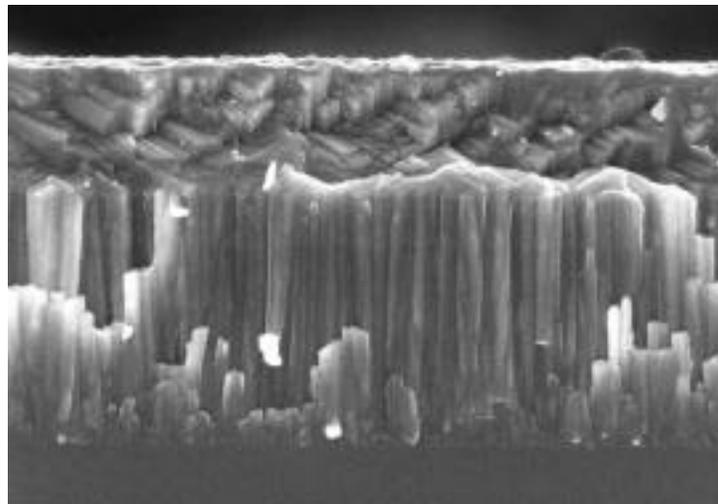


Fig. 3. Cross-sectional view of ISD MgO layer showing MgO (200) plane on top of MgO columnar grain and corresponding selected area diffraction pattern.

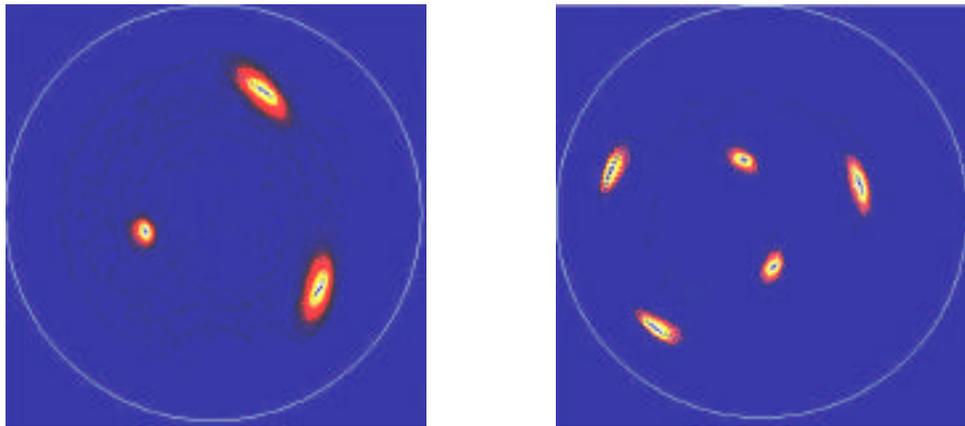


(a)



(b)

Fig. 4. SEM photomicrographs of ISD MgO topped with a homoepitaxial MgO layer: (a) top surface and (b) cross section.



(a)

(b)

Fig. 5. (a) MgO (002) and (b) MgO (220) pole figures for a 1.5- μm -thick film that was deposited at room temperature with $\theta = 55^\circ$.

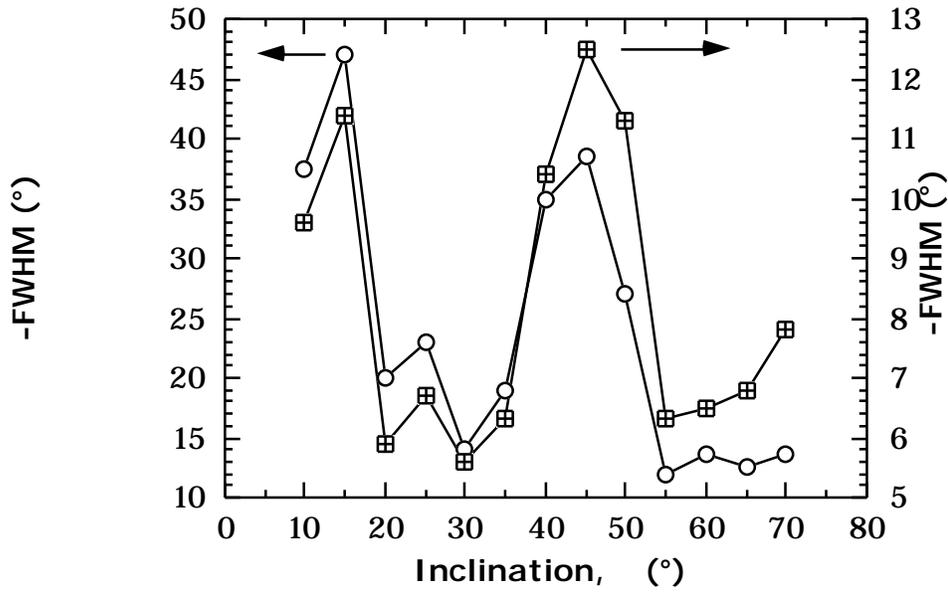


Fig. 6. Effect of inclination, θ , on FWHM values of MgO (002) ω - and χ -scans.

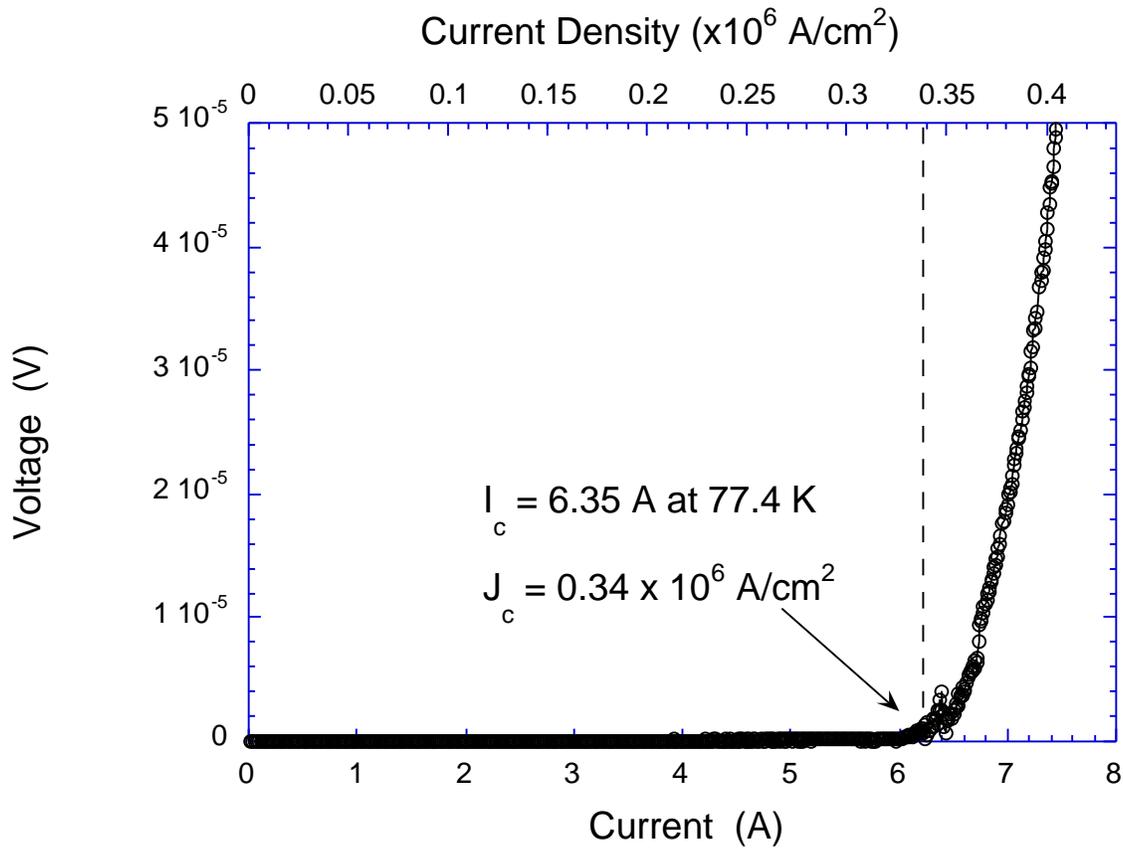


Fig. 7. Critical current measurement on a YBCO film deposited by PLD on MgO-buffered Hastelloy C276. Sample was $0.42 \mu\text{m}$ thick, 4.5 mm wide, and 1 cm long.