DEPOSITION OF BIAXIALLY TEXTURED YTTRIA-STABILIZED ZIRCONIA BY ION-BEAM-ASSISTED DEPOSITION*

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Deposition of Biaxially Textured Yttria-Stabilized Zirconia by Ion-Beam-Assisted Deposition *

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***Abstract*—Biaxially textured yttria (8 mol%)-stabilized zirconia (YSZ) thin films were deposited on randomly oriented Hastelloy C and Stainless Steel 304 at room temperature as a buffer layer for subsequent deposition of oriented $YBa_2Cu_3O_x$ films. The 0.16-1.3 μm thick YSZ films were deposited by e-beam evaporation at rates of 1.2-3.2 Å/sec. Biaxially textured films were produced with an Ar/O_2 ion beam directed at the substrate during film growth. X-ray diffraction was used to study in-plane and out-of-plane orientation as a function of ion-bombardment angle, film thickness, ion-to-atom flux ratio, and substrate material. In-plane and out-of-plane average-misorientation angles on these YSZ films that were deposited by ion-beam-assisted deposition were as low as 17 and 5.4°, respectively, on as-received substrates.**

I. Introduction

Coated conductors are candidates for the next generation of high-temperature superconducting wire and tape and are fabricated from long lengths of flexible polycrystalline metal substrates on which YBCO films have been deposited by vapor deposition. Intergranular weak links are a major hurdle in the fabrication of YBCO-coated conductors, and methods must be developed to reduce misorientation between current-carrying grains [1]. The average misorientation between grains (referred to as texture) can be measured by four-circle X-ray diffraction techniques. Because coated conductors are vapor-deposited epitaxially, the texture in the YBCO is determined by the underlying material on which it is grown.

Ion-beam-assisted deposition (IBAD) is a promising method for the fabrication of long lengths of coated conductors and enables the deposition of highly oriented template layers on randomly oriented metal substrates [2], [3]. These template layers are also known as buffer because they fulfill the dual functions of diffusion barrier between the metal substrate and YBCO conductor film [4] and of texture template for the epitaxially deposited YBCO films. IBAD has been used to deposit highly textured buffer layers of yttria-stabilized zirconia (YSZ) on polycrystalline metal substrates using an assisting off-normal low-energy ion beam incident on the substrate during deposition [2].

Several theories have been proposed for the mechanisms by which IBAD creates crystallographic texture in thin films. One such theory proposed by Bradley et al. [5], states that the difference in sputtering yields between crystallographic orientations creates a preferred orientation. Grains oriented with open channeling directions that are parallel to the bombarding ion beam will suffer less resputtering, and eventually overgrow grains that are less-favorably oriented, and begin to dominate the texture. More recently, Ressler et al. proposed a theory based on anisotropic damage between planes as a texture development mechanism in IBAD oxide films [6].

II. Experimental Procedure

YSZ films were deposited by electron beam evaporation. During deposition, the substrates were bombarded by an assisting 300-eV ion beam of Ar/10% O_2 ions at varying angles by using an 8-cm Kaufman-type DC ion source with molybdenum grids. The ion beam was neutralized with a tungsten filament at 110% of the ion beam current to ensure complete neutralization [7]. A calibrated quartz-crystal monitor (QCM) was used to determine the gross deposition rate. A schematic diagram of the deposition configuration is shown in Fig. 1.

The ion flux was measured with a faraday cup at normal incidence to the ion beam. An electron screening grid on the faraday cup was negatively biased to 20 V to repel the neutralizer electrons and thus accurately measure the ion flux. The surface ion current was calculated by multiplying the measured ion current by the sine of the angle. Angular

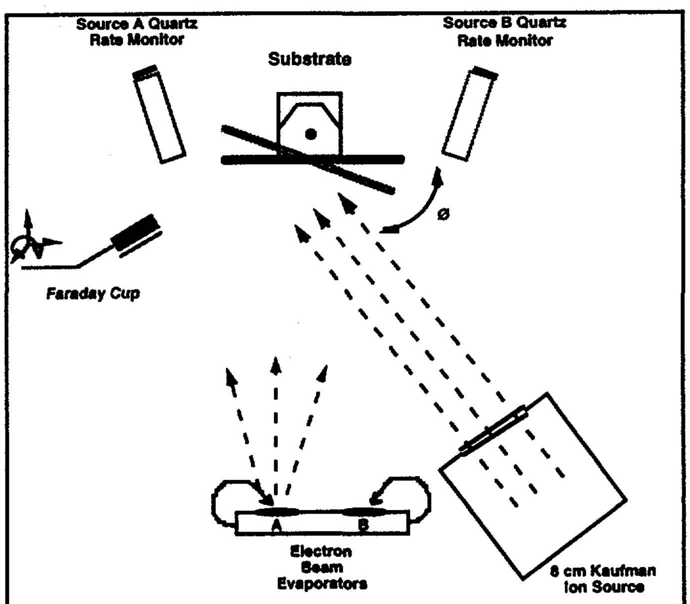

Fig. 1. Schematic representation of ion-beam-assisted electron beam evaporator chamber.

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adjustment of the incident ion beam was achieved by a substrate platen that allows rotational motion (0°-90° ± 2°), as shown in Fig. 1. IBAD YSZ films were deposited under ion bombardment at angles between 29 and 48° with respect to the substrate parallel to a gross thickness of 1 µm, determined by the QCM. A gross thickness of 1 µm yielded a net thickness of 0.65 µm at a bombardment angle of 45°, as determined by profilometry on a partially shielded silicon control sample. All provided thickness values are gross values.

The evaporation charge was fabricated by uniaxially pressing 8 mol% yttria-stabilized zirconia powder into 1.9-cm-diameter pellets, followed by sintering at 1600°C for 4 hrs. in ambient atmosphere. After firing, the pellets were crushed into 3-mm pieces and placed in an evaporation hearth.

As-received and polished Hastelloy C and SS 304 were used as substrates. Some Hastelloy substrates were polished with diamond paste to a final grit size of 0.1 µm. All substrates were ultrasonically cleaned in acetone and methanol before deposition and affixed to the substrate platen with conductive silver paste. The substrate platen, with the substrates, was placed into a load-lock chamber and evacuated to facilitate removal of residual solvent.

The base pressure of the IBAD chamber before evaporation and ion bombardment is 1 x 10^{-7} Torr, which rises to an operating pressure of 9 x 10^{-5} Torr during deposition. All depositions were performed at room temperature; however, during IBAD, the substrate temperature measured by a thermocouple on the substrate platen, rose from 25 to ≈200°C because of ion bombardment. Before all depositions, the substrates were subjected to a 500eV-Ar^+-beam etch for 5 min.

Biaxial texture was characterized with a four-circle X-ray diffractometer using CuKα radiation. Out-of-plane texture was determined by the full-width-at-half-maximum (FWHM) of omega scans of the YSZ (200) reflection, and in-plane texture was measured by the FWHM of phi scans of the YSZ (111) reflection.

III. RESULTS AND DISCUSSION

A. In-Plane Texture as a Function of Ion Beam Bombardment Angle

As-received Hastelloy, SS and polished Hastelloy substrates were used to investigate the effects of ion bombardment angle on biaxial texture. A constant ion-to-atom flux ratio (*r* value) of 2.8 was maintained by adjusting the ion current density while maintaining a constant deposition rate of 1.6 Å/sec. All films were (200) biaxially oriented with the (111) pole reflection in the direction of the ion beam. Fig. 2 shows a theta-2theta X-ray scan for an IBAD YSZ film with an assisting ion beam angle of 35°.

The out-of-plane FWHM showed no clear angular dependence, and values ranged from 5 to 10°. However, the out-of-plane texture of the polished substrates was on average 2° better than that of the unpolished substrates, as would be expected from a smoother growth surface. No clear difference was noted between the out-of-plane texture of the SS 304 and Hastelloy C substrates. Fig. 3 shows a typical omega scan for an IBAD YSZ film deposited on as-received SS.

The in-plane texture of IBAD YSZ in this study shows a reproducible dependence on the angle of ion bombardment, as seen in Fig. 4. A minimization of the in-plane FWHM (phi) occurs at 35°, which corresponds to the (111)

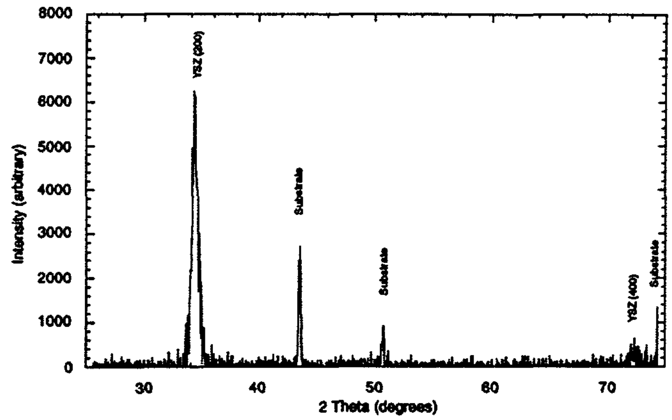

Fig. 2. Θ-2Θ X-ray scan for IBAD YSZ deposited on polished Hastelloy with assisting angle of 35°.

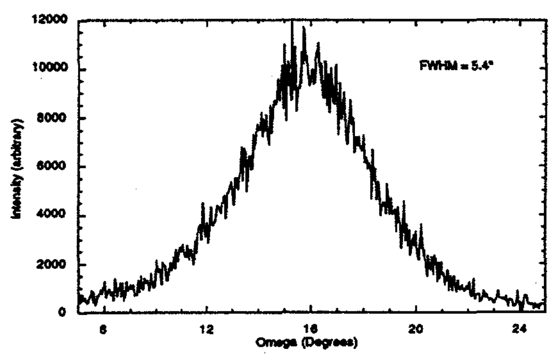

Fig. 3. (200) omega scan for IBAD YSZ film on as-received Stainless Steel.

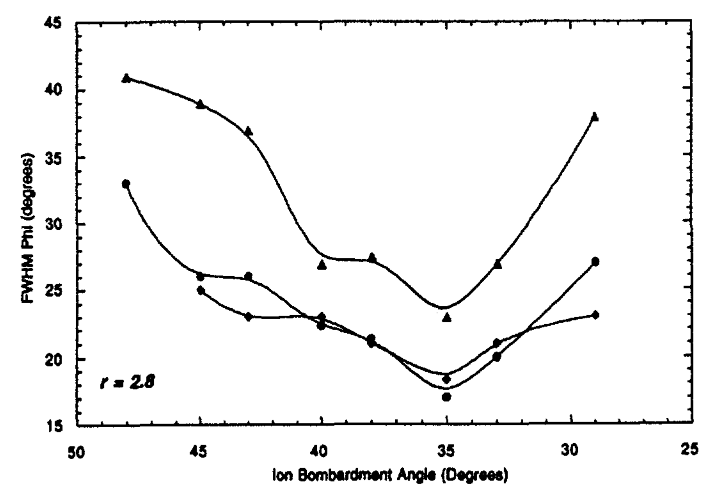

Fig. 4. Degree of in-plane texture as a function of ion bombardment angle from substrate parallel.

(● Hastelloy -C polished/0.1 µm) (▲ Hastelloy C-as received) (◆ Stainless Steel-as received)

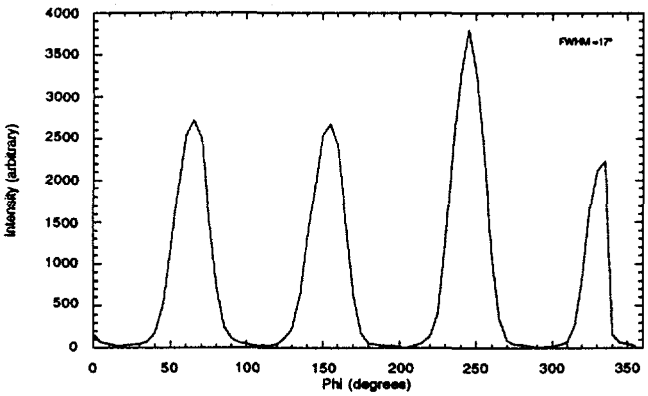

Fig. 5. X-ray phi scan for an IBAD YSZ film deposited on polished Hastelloy at 35°.

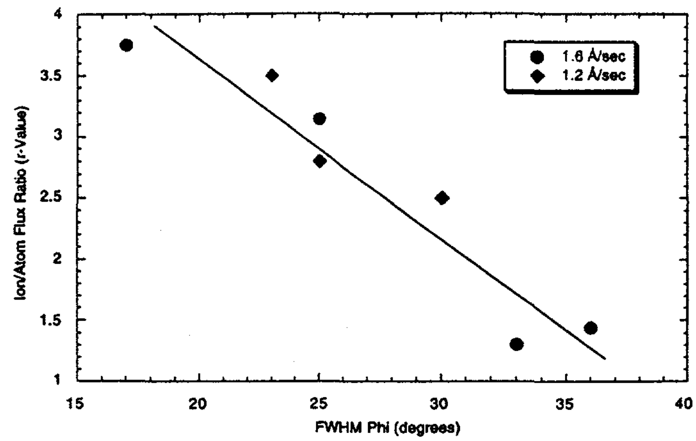

Fig. 7. The effects of ion/atom ratio (r value) on in-plane orientation of IBAD YSZ films.

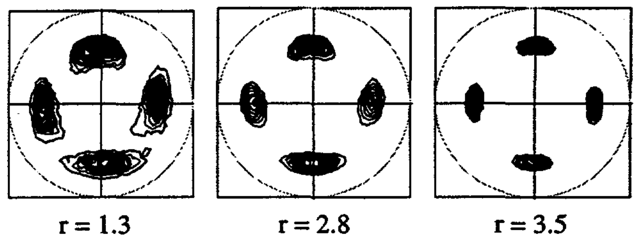

Fig. 8. (111) X-ray pole figures for IBAD YSZ films deposited with three different r values to gross thickness of 1 μm.

channeling angle for cubic YSZ. This channeling-angle effect has been well documented by other researchers [8], [9] and lends support to the selective resputtering theory [5].

The in-plane texture of IBAD YSZ on as-received SS substrates equals that of the polished Hastelloy substrates. Fig. 5 shows a phi scan for an IBAD YSZ film deposited on polished Hastelloy at 35°, with a FWHM of 17°. This is an important result because scale-up production of IBAD YSZ tapes for coated conductor applications would benefit from fewer processing steps. The reason for this effect is not completely understood, and more studies are in progress to determine a possible mechanism.

B. In-Plane Texture as a Function of Flux Ratio

The in-plane texture was studied for ion/atom flux ratios (r value) between 0 and 3.8 by varying the ion flux and the atomic flux for a bombardment angle of 35° to a gross thickness of 1 μm. YSZ films deposited without ion assistance were largely amorphous, with broad low-intensity (111) and (200) peaks (Fig. 6). IBAD with a 300-eV ion beam and flux ratios between 1 and 0.5 produced mixed orientations, dominated by (111) grains. The ions are presumably providing enough surface mobility to the adatoms of YSZ to preferentially grow with a <111> normal to the surface. This preferred <111> growth has been observed in sputtered YSZ films that undergo substrate bombardment from ions that escape the target sputtering plasma [10], [11].

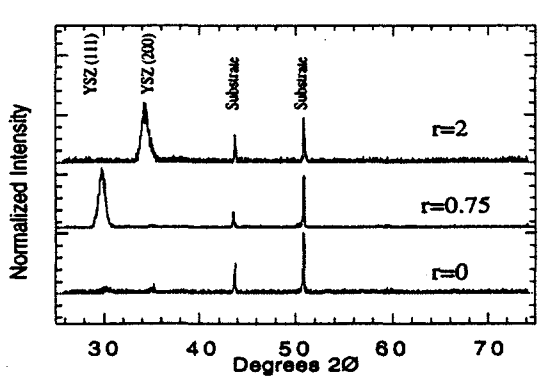

Fig. 6. Θ-2Θ X-ray scans of YSZ films for various ion/atom flux ratio (r value) on as-received Hastelloy substrates.

Films with a (200) biaxial orientation were observed when the r value exceeded 1. Fig. 7 shows that the in-plane texture obtained by (111) phi scans for the biaxially-oriented films show a steady improvement in the in-plane texture as the r value increases.

Bradley et al. showed that the degree of orientation of thin films under IBAD increases asymptotically to a critical value of r (r_c), where more material is sputtered than deposited [5]. It is, therefore, not surprising that the degree of in-plane texture should also increase to a critical value of r. Due to system limitations, we could not evaluate r_c, but a trend of improving in-plane texture with r value was observed. Fig. 8 shows a series of (111) pole figures for IBAD YSZ films deposited when the r values were 1.3, 2.8, and 3.5. In all cases, the (111) pole is oriented in the direction of the bombarding beam.

C. In-Plane Texture as a Function of Gross Film Thickness

The in-plane texture of IBAD YSZ films improves linearly with film thickness in the range between 0.3 and 1 μm gross film thickness. This effect was observed in IBAD YSZ films when r values were 1.3 and 2.8, as seen in Fig. 9, and confirms that the texture improves with film thickness, presumably as grains oriented with the ion beam overgrow the misoriented grains.

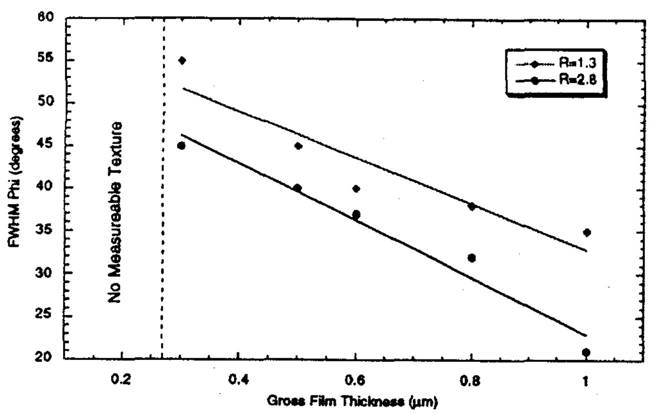

Fig. 9. In-plane texture as a function of gross film thickness.

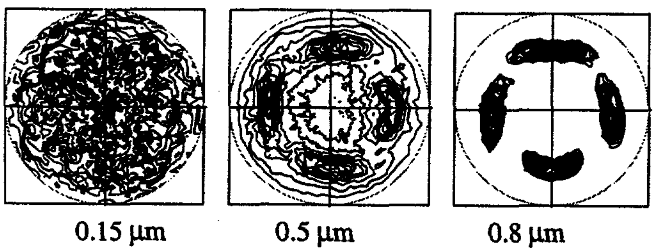

Fig. 10. (111) X-ray pole figures of IBAD YSZ films for various gross thicknesses when $r = 2.8$.

The in-plane FWHM of the surface texture of the IBAD YSZ films is significantly better than the bulk measured values, due to the X-ray penetration depth into the film (>1 µm). Knierim et al. studied IBAD YSZ films under glancing-angle X-ray diffraction and found that the surface layer FWHM was typically 9° smaller than the bulk [12]. Under optimal deposition conditions for YBCO, an IBAD YSZ film with an in-plane FWHM of 19° or less, which would mean a surface FWHM of less than 10°, should yield critical current densities > 10^6 A/cm^2 [13]. Therefore, deposition of an IBAD YSZ film under similar deposition conditions requires a gross film thickness of at least 1 µm (net thickness of 0.61 µm). Confirmation of this general rule is currently being investigated.

Fig. 10 shows the progression of texture of IBAD YSZ films as shown by (111) X-ray pole figures. Films < 0.3 µm show no discernible texture whereas, in thicker films, (111) poles are clearly visible that sharpen and grow in intensity as the film thickness increases.

IV. Conclusions

The texture of YSZ films deposited by the IBAD technique was investigated and found to depend on ion-bombardment angle, film thickness, ion-to-atom flux ratio, and substrate material. An ion-bombardment angle of 35° with respect to the substrate parallel produced a minimum in-plane texture, which was as low as 17° in a 1-µm-thick film. Higher ion/atomic flux ratios improved the in-plane texture. Additional work is needed to find a value of r_c at which texture no longer improves due to complete resputtering. This will require an optimized IBAD system capable of ion flux values greater than 350 µA/cm^2 at the substrate.

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