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# BIAXIALLY TEXTURED $YBa_2Cu_3O_{7-x}$ FILMS DEPOSITED ON POLYCRYSTALLINE FLEXIBLE YTTRIA-STABLIZED ZIRCONIA CERAMIC SUBSTRATES (POSTPRINT)

**C.V. Varanasi, J. Burke, R. Lu, J. Wu, L. Brunke, L. Chuck, H.E. Smith, I. Maartense, and P.N. Barnes**

**Power Generation Branch**
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C.V. Varanasi, J. Burke, L. Brunke, L. Chuck, H.E. Smith, and I. Maartense (University of Dayton Research Institute)
R. Lu and J. Wu (University of Kansas)
P.N. Barnes (AFRL/RZPG)

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# Biaxially textured $YBa_2Cu_3O_{7-x}$ films deposited on polycrystalline flexible yttria-stabilized zirconia ceramic substrates

C.V. Varanasi [a,*], J. Burke [a], R. Lu [b], J. Wu [b], L. Brunke [a], L. Chuck [a], H.E. Smith [a,c], I. Maartense [a], P.N. Barnes [c]

[a] University of Dayton Research Institute, Metals and Ceramics, 2645 Fifth Street, Dayton, OH 45469, USA
[b] Department of Physics and Astronomy, University of Kansas, Lawrence, KS 66045, USA
[c] Air Force Research Laboratory (AFRL), Wright-Patterson AFB, OH 45433, USA

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ABSTRACT

Biaxially textured $YBa_2Cu_3O_{7-x}$ (YBCO) films were grown on polycrystalline flexible yttria-stabilized zirconia (YSZ) ceramic substrates (Ceraflex) buffered with MgO and $LaMnO_3$ layers. These substrates were initially coated with silica glass to obtain a smooth surface and then biaxially textured MgO buffer layers were deposited by ion beam assisted deposition (IBAD-MgO). Lanthanum manganate (LMO) cap layers and YBCO layers were then deposited by the pulsed laser ablation method. Highly textured YBCO films with a full width half maximum (FWHM) of 6.75° in (110) phi scans and a FWHM ~ 5° in (200) omega scans were obtained. An initial deposition yielded samples with a $T_c$ > 88 K and a self-field magnetization $J_c$ of $2 \times 10^5$ A/cm$^2$ at 77 K. A secondary ion mass spectrometry (SIMS) depth profile of the samples indicated that with the present deposition condition, some La, Mn and Mg diffusion into the YBCO layers is possible and this may reduce the $J_c$ in the self-field. The yield strength (YS) of uncoated Ceraflex substrates was compared with that of metallic substrates and it was found that Ceraflex substrates can have a YS at least 4–5 times higher than the YS of biaxially textured Ni–5 at.%W substrates and ~1.5 times that of Hastelloy™ substrates.



## 1. Introduction

$YBa_2Cu_3O_{7-x}$ (YBCO) coated conductors are presently being developed using conductive metallic substrates such as Ni–5 at.%W or Hastelloy™ [1,2]. However, losses due to eddy currents appear when these conductors are used in ac applications. Non-conducting or highly resistive substrates can avoid this issue for ac applications [3]. In addition, for cryoelectronic applications, YBCO films deposited on thermally- and electrically-insulating substrates are also of interest. Earlier work showed that $YBa_2Cu_3O_{7-x}$ coatings can be grown on highly resistive flexible polycrystalline yttria-stabilized zirconia (YSZ) substrates with ion beam assisted deposition (IBAD)-YSZ layers for RF cryoelectronic applications [4].

Polycrystalline YSZ substrates such as Ceraflex are commercially available in ultra thin, tough, flexible thin sheet form [5]. Sheets as thin as 0.05 mm thick and as large as 200 × 200 mm square are readily available. These substrates also have high hardness and fracture toughness – three times higher in bending strength, two or three times in fracture toughness than alumina. Properties such as this would make the material suitable as substrates for coated conductors if the lengths can be increased.

Gnanarajan et al. reported that IBAD-YSZ layers and YBCO with a phi scan full width half maximum (FWHM) of 19° can be grown on Ceraflex substrates when coated with an additional silica coating to reduce surface roughness [6]. However, it is known that biaxial texture can develop in IBAD-MgO faster than IBAD-YSZ [7,8]. Initial work by Lu et al. indicated that highly textured IBAD-MgO with a FWHM of 9.3° can indeed be grown on properly prepared Ceraflex substrates using particular processing conditions [9,10]. Here, we present initial results which indicate that biaxially textured YBCO can be grown on the IBAD-MgO buffered Ceraflex substrates using lanthanum manganate (LMO) cap layers deposited by pulsed laser ablation (PLD). Results on this YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex architecture include biaxial texture of the layers, superconducting properties such as critical transition temperature ($T_c$), critical current density ($J_c$), microstructures and SIMS depth profile analyses of the coatings. Both Ceraflex 3Y with 3 mol% $Y_2O_3$ and Ceraflex 8Y with 8 mol% $Y_2O_3$ were used. In addition, mechanical properties of the Ceraflex substrates were measured and compared with published values of biaxially textured Ni–5 at.%W and Hastelloy™ substrates.

* Corresponding author. Tel.: +1 937 255 4738; fax: +1 937 656 4095.
*E-mail address:* chakrapani.varanasi@wpafb.af.mil (C.V. Varanasi).

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## 2. Experimental

### 2.1. Mechanical properties of ceraflex substrates

The Ceraflex substrates are obtained from Marketech International Inc. [5]. Ceraflex 3Y strips also have good elasticity and high flexibility and are capable of bending to a radius of 8 mm. The Ceraflex 3Y substrates were sliced to a nominal tensile specimen size of 50.8 × 3.34 × 0.09 mm. To prevent damage during the slicing operation, the substrates were hot-wax mounted and sandwiched between two microscope glass slides. The glass-sandwiched substrate was fed into a slow-speed, water cooled/lubricated, diamond-impregnated, slicing wheel rotating at ~100 rpm at a 0.25 mm/s feed rate. After slicing, the test specimens were unmounted and cleaned in toluene to remove the wax and further cleaned in acetone and methanol, and then dried. After cleaning, the tensile specimens were optically inspected at 50× for machining damage and edge chipping.

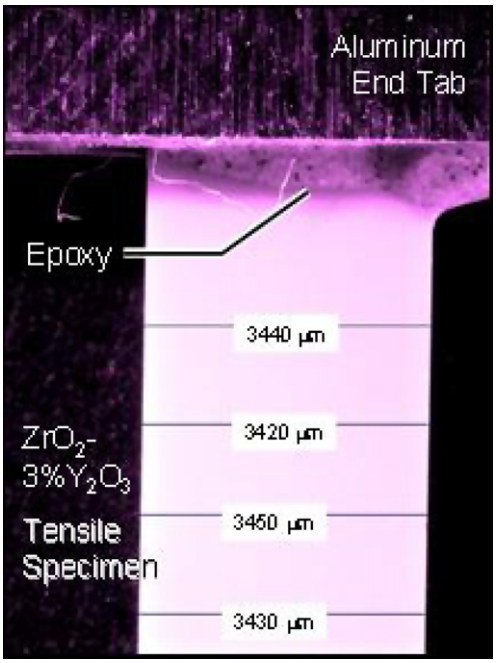


**Fig. 1.** Optical macrograph of a Ceraflex tensile test specimen in aluminum tabs. (Width measurements are also shown.)

The Ceraflex tensile specimens were then tabbed with 12 × 12 × 0.762 mm (nominal) aluminum plates at the ends to prevent crushing of the thin ceramic specimens in the tensile grip. Each end of Ceraflex tensile specimen was glued with a general-purpose two-part epoxy and sandwiched with two aluminum plates as shown in Fig. 1. The epoxy was allowed to cure for ~24 h to achieve maximum shear strength. The Ceraflex specimen tensile gage length after end tabbing was approximately 25 mm.

The width and thickness of the samples were measured before testing. The thickness was measured using a micrometer with debris-free flat anvils to prevent any surface damage to the tensile specimen. The width was measured optically using a calibrated microscope to prevent damage to the edge of the thin specimen. Typical variation in the sample widths is shown in Fig. 1.

The tabbed Ceraflex tensile specimens were mounted on an aligned tensile wedge grip (Instron Model 2716-015) on a Universal testing system (Instron Model 4486 SN C8825) with a 1 kN load cell (Instron Load Cell SN: UK881). The tensile extension rate was 0.0212 mm/s (0.05 ipm). A total of three tensile specimens were tested to failure. After testing, the remnants of each test specimen were photographed to show that the fracture occurred between the end tabs as shown in Fig. 2. A simple tensile testing method originally developed for textured metallic substrates [11] was used to test these thin ceramic substrates.

### 2.2. Buffer layers (MgO, $LaMnO_3$) and YBCO layers

The Ceraflex 8Y substrates were initially spin coated with methyl siloxane polymers to make smooth $SiO_2$ coatings as described in detail elsewhere [6]. A thin amorphous $Y_2O_3$ buffer layer (15–40 nm thickness) was deposited by e-beam deposition and pre-exposed in $Ar^+$ for 1–2 min on the initial $SiO_2$ layer. MgO of 9–11 nm thickness was deposited by IBAD at 1.5 Å/s, at room temperature using $Ar^+$ beam operating at 750 eV and 10 mA. An ion-to-atom ratio of ~0.9 was maintained during deposition. A homo-epitaxial MgO layer of 100–200 nm thickness was grown at 0.5–1.0 A/s, at 300–500 °C. In-situ real-time reflection high-energy electron deflection (RHEED) was used to monitor the texture development during IBAD-MgO and homo-epitaxial MgO depositions. A detailed discussion of the deposition conditions and characterization of these layers have been discussed elsewhere [10]. In the present

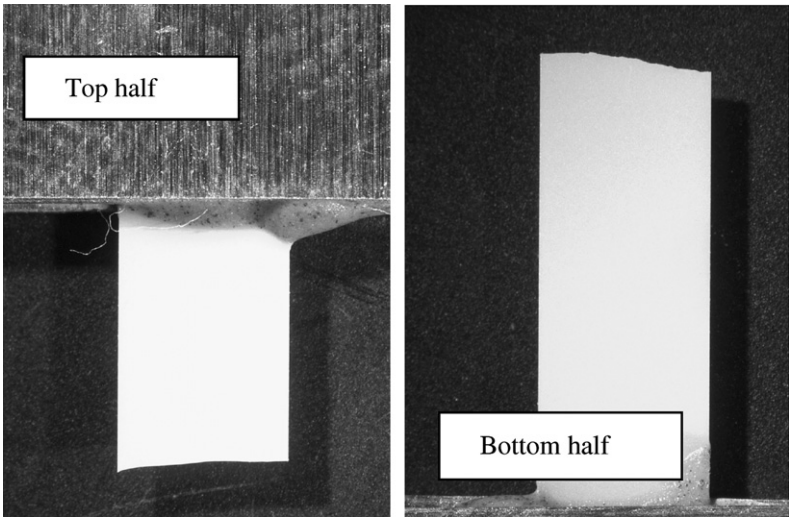


**Fig. 2.** Optical macrograph of a tested Ceraflex tensile test specimen at the end of the test showing the fractured tensile gage section: (a) top half and (b) bottom half.

**Table 1**
Summary of yield strength measurements

| Material | Sample ID | Nominal gage length, mm | Displacement rate, mm/min | Yield strength, MPa | UTS, MPa | Width, mm | Thickness, mm |
|---|---|---|---|---|---|---|---|
| $ZrO_2$–3%$Y_2O_3$ | 1 | 25 | 1.270 | 650 | 650 | 3.442 | 0.090 |
| | 2 | 25 | 1.270 | 860 | 860 | 3.384 | 0.090 |
| | 3 | 25 | 1.270 | 850 | 850 | 3.343 | 0.090 |
| | Average ± Standard deviation | | | 790 ± 120 | 790 ± 120 | | |
| Ni–5%W | | | | 163 (Ref. [11]) | | | |
| | | | | 176 (Ref. [12]) | | | |
| Hastelloy™ | | | | 464 (Ref. [12]) | | | |

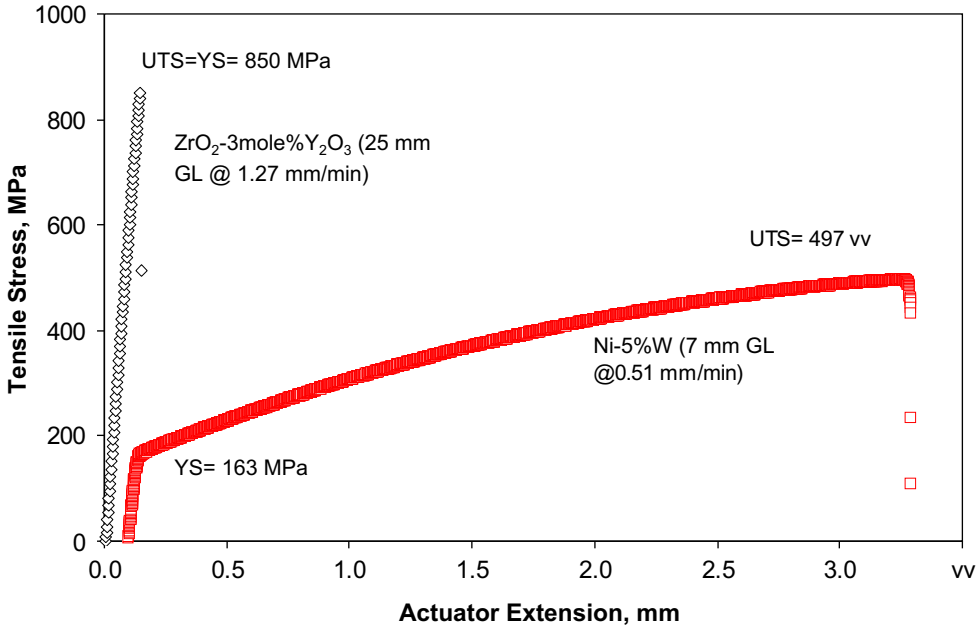


**Fig. 3.** Stress-extension curves of a Ceraflex substrate compared with a stress-extension curve of a biaxially textured Ni–5 at.% W substrate.

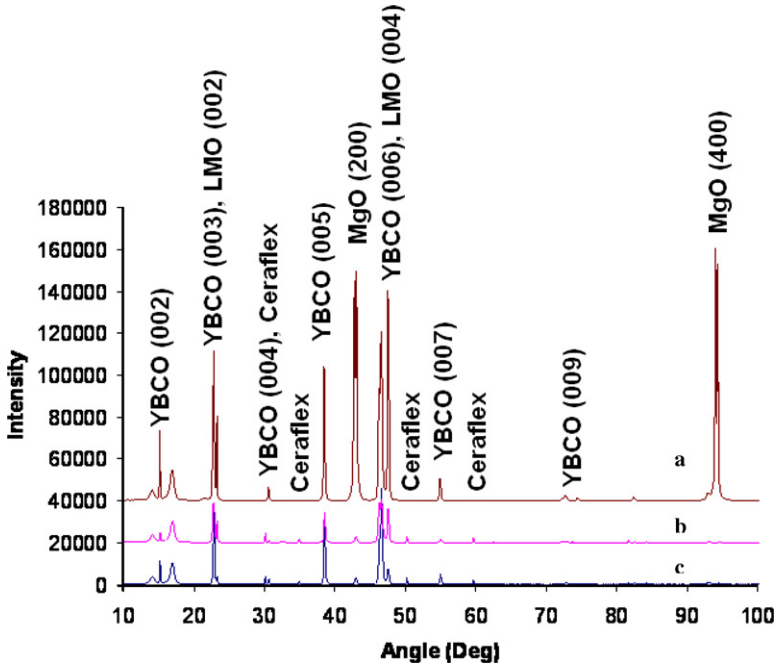


**Fig. 4.** $\theta$–$2\theta$ X-ray diffraction patterns of (a) YBCO/LMO/single crystal MgO, and (b) and (c) two different samples of YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex.

study, $LaMnO_3$ (LMO) buffer layer and YBCO layer deposition and characterization is presented.

A LMO cap layer was deposited by pulsed laser ablation (PLD) on the MgO buffered Ceraflex substrates. A YBCO film was then grown by PLD on top of the LMO cap layer. The YBCO/LMO layers were also grown on MgO (100) single crystal substrates for comparison purposes. All the PLD depositions were done in a Neocera chamber. A Lambda Physik KrF excimer laser (wavelength $\lambda = 248$ nm) was used to deposit films at an energy density of 2–4 J/cm$^2$. A layer of LMO of ~200 nm thicknesses was first deposited at 750 °C in an atmosphere of 300 m Torr of $O_2$ at 4 Hz for 15 min, and then a layer of YBCO of ~300 nm thickness was deposited at 780 °C in an atmosphere of 300 m Torr of $O_2$, at 4 Hz for 20 min. The films were annealed inside the chamber in 500 Torr of oxygen at 500 °C for 30 min. before cooling down to the room temperature.

The YBCO coatings were characterized for biaxial texture using a four circle X-ray diffractometer. Characterization of the film's microstructure was done using a scanning electron microscope (SEM); the critical transition temperature ($T_c$) was characterized by ac susceptibility and the critical current density ($J_c$) using a Quantum Design physical property measurement system (PPMS) vibrating sample magnetometer. Compositional sputtered depth profiles of the films were obtained with secondary ion mass spectrometry (SIMS) (CAMECA Model IMS4F7). The SIMS instrument employed $Cs^+$ primary ion bombardment at 5.5 keV and 42° incidence and detected positively charged atomic ions with a mass resolution of $M/dM = 600$. The analyzed area was circular with a 60 μm diameter.

## 3. Results and discussion

### 3.1. Mechanical properties

Tensile test results are summarized in Table 1. The average measured tensile yield and ultimate strength from three Ceraflex test specimens is 790 MPa with a standard deviation of 120 MPa.

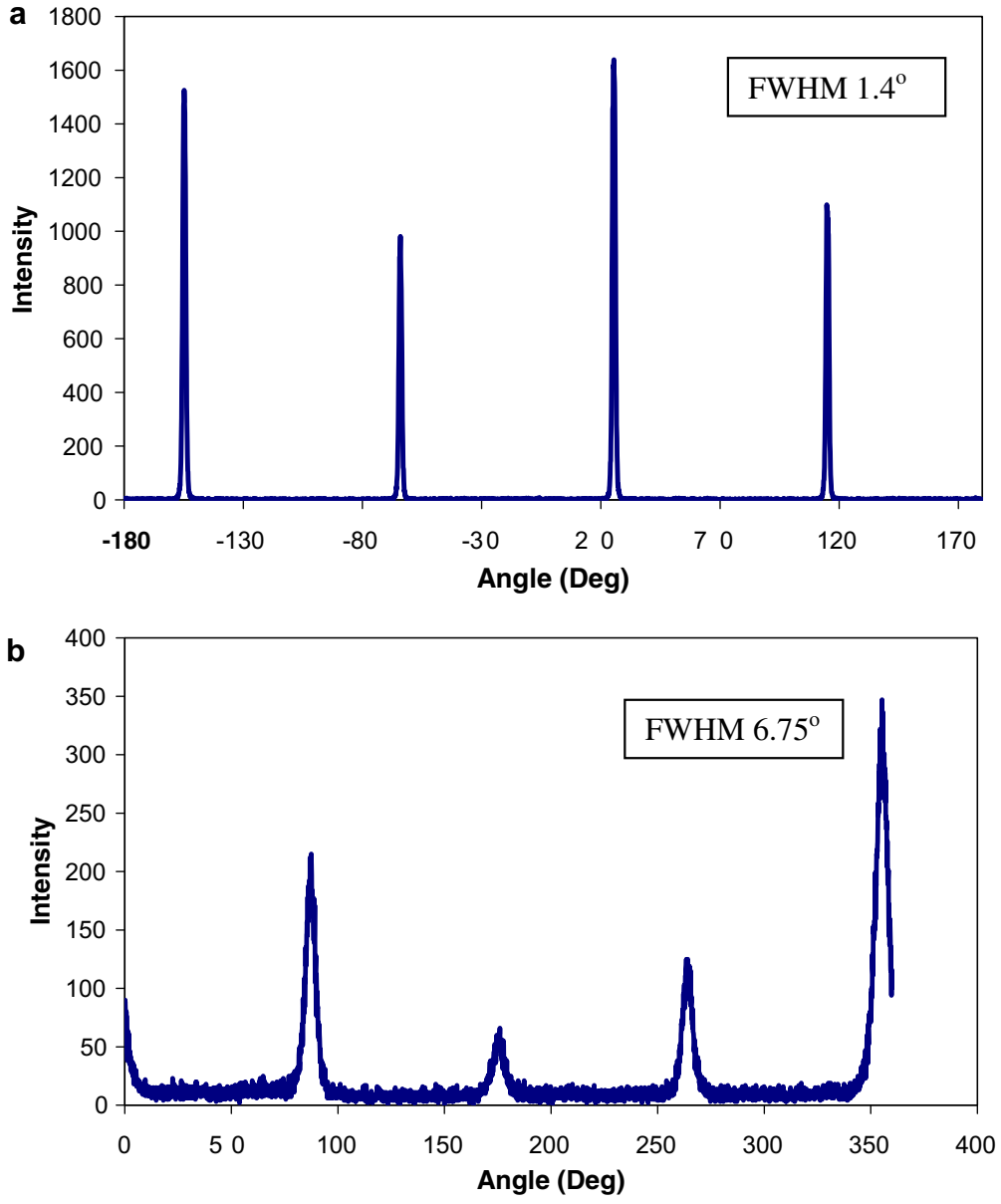


**Fig. 5.** Phi scans of (110) YBCO on (a) YBCO/LMO/single crystal MgO, and (b) YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex.

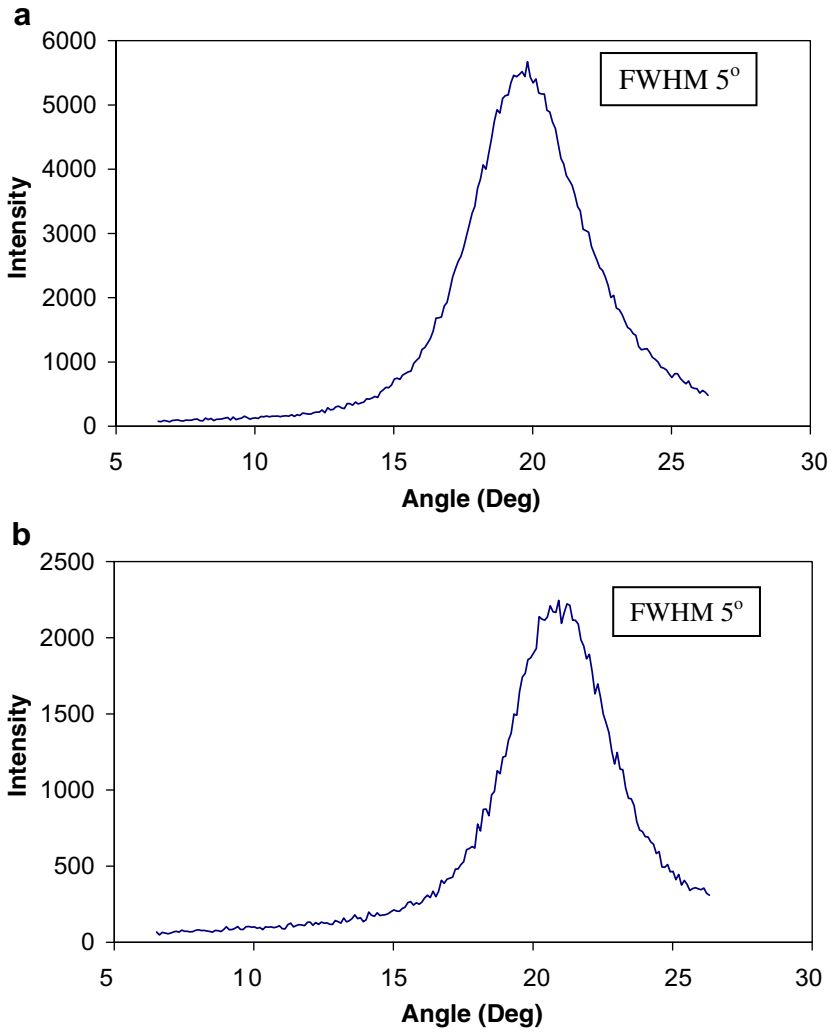


**Fig. 6.** (200) YBCO omega scans of YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex in (a) longitudinal and (b) transverse directions.

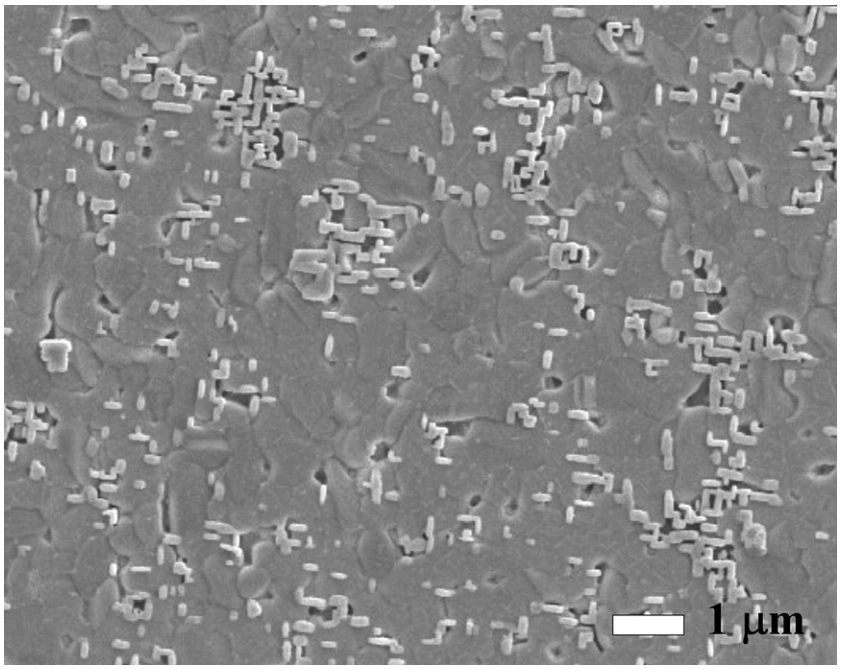


**Fig. 7.** Scanning electron micrograph of a YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample.

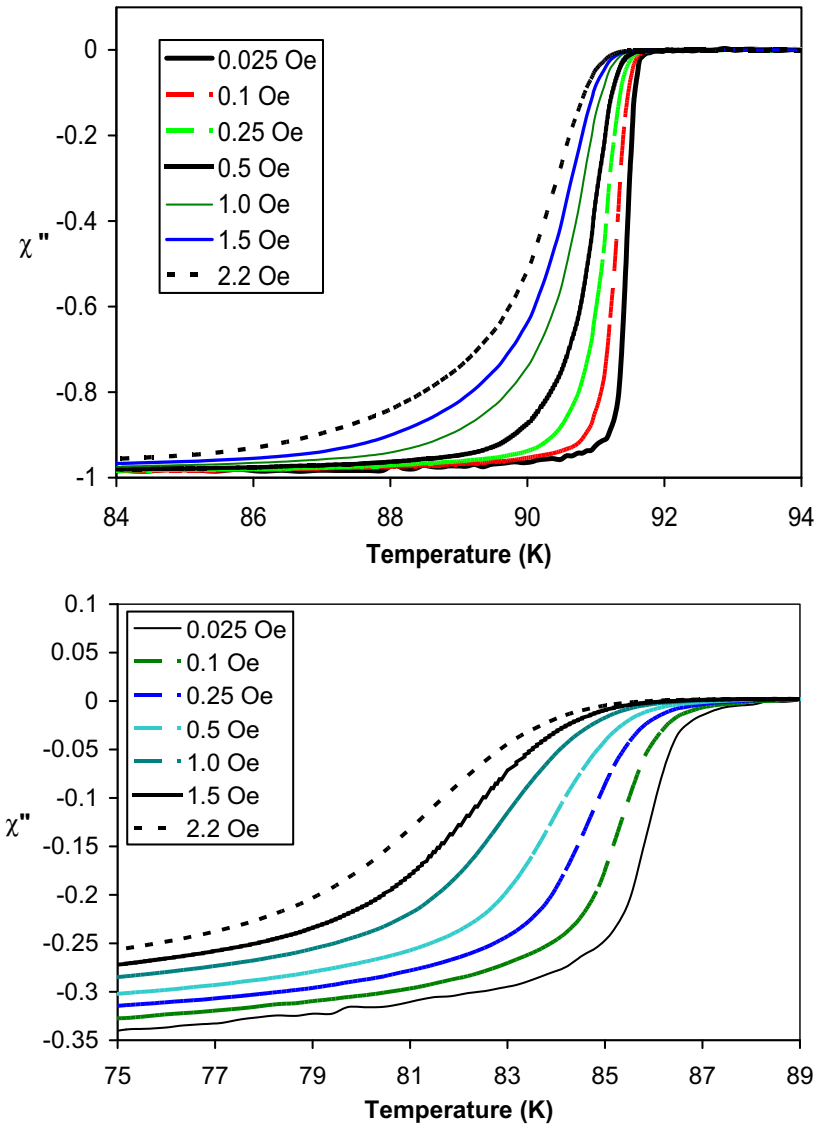


**Fig. 8.** AC susceptibility curves of (a) YBCO/LMO/single crystal MgO and (b) YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex.

It is not surprising that the yield strength and the ultimate strength are same in Ceraflex as it is a brittle structural ceramic material and deforms only elastically and not plastically. It may be of concern that the standard deviation is slightly high; however, this is due to the sensitivity of the ceramics to cracks and other defects and this behavior is expected.

Fig. 3 shows the stress-extension curves of a Ceraflex substrate as compared to previously reported data [11] of Ni–5 at.% W biaxially textured metallic substrate. The tensile yield strength of the Ceraflex substrate is higher than a similarly tested Ni–5 at.% W substrate showing a steep slope in stress extension curve as the load was applied. As expected, the Ni–5 at.% W substrate shows considerably more plastic deformation as compared to the Ceraflex substrates. Even so, the YS of the Ceraflex substrate was found to be almost five times higher than the YS of Ni–5 at.% W substrate. Also as compared to the published data of Hastelloy™ substrate [12], the YS data of ceraflex substrates were found to be 1.5 times higher than that of Hastelloy™ substrates as shown in Table 1.

### 3.2. Texture quality of YBCO

Intensity $\theta$–$2\theta$ X-ray diffraction patterns of different YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex samples are compared with

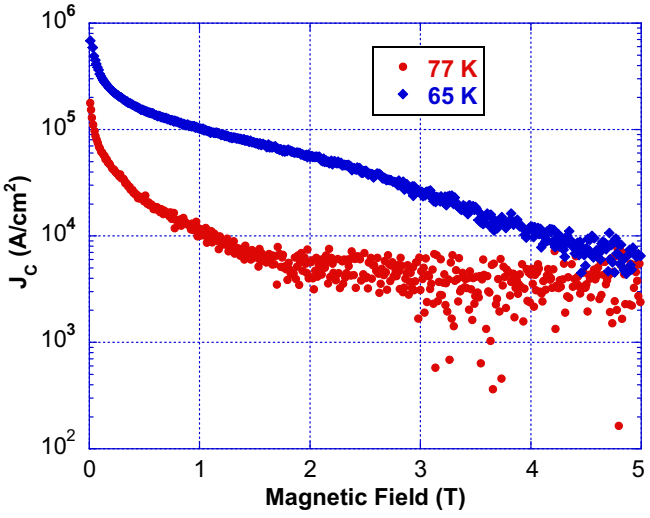


**Fig. 9.** Magnetization $J_c$ data as a function of applied magnetic field of YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample at 77 K and 65 K.

the diffraction pattern of a YBCO/LMO/MgO single crystal sample in Fig. 4. It can be seen that *c*-axis oriented YBCO layers are grown in both types of samples showing only (00l)-type peaks from YBCO. As expected, peaks corresponding to the polycrystalline Ceraflex substrates were evident only in the Ceraflex substrate samples.

Fig. 5a shows the (110) phi scans of the YBCO/LMO/single crystal MgO substrate. A very low FWHM of 1.4° observed for the YBCO films indicates the presence of high-quality biaxial texture. However, a (110) phi scan of YBCO on the YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample, as shown in Fig. 5b, does show that the FWHM is wider in these films. For this sample, a FWHM of 6.75° was measured. Although this value is greater than what was achieved on the single-crystal substrate, the FWHM is comparable to what is generally observed in YBCO films grown on textured metallic substrates [13]. Omega scans of YBCO (200) taken on these samples also show *c*-axis out of plane alignment with a FWHM of ~5° in both longitudinal as well as transverse directions as shown in Fig. 6a and b, again indicating that YBCO films with good biaxial texture can be grown on the Ceraflex substrates using the present architecture.

### 3.3. Microstructure of YBCO

Fig. 7 shows a scanning electron micrograph of YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample. It can be seen that YBCO grows with a typical plate-like microstructure. The average grain size was found to be 2–5 μm. In addition to the *a*–*b* oriented grains, some growth of *a*-axis grains was also observed in the films, which may account for the slight degradation of the observed $J_c$ as discussed later. Reduction in the amount of *a*-axis grains can be achieved by adjusting the deposition temperature during processing [14].

### 3.4. Superconducting properties of YBCO layers

The $T_c$ of the YBCO films measured by ac susceptibility showed that a high $T_c$, above 91 K, is possible in the YBCO/LMO/single crystal MgO sample as shown in Fig. 8a. The YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex samples (Fig. 8b) have a slightly lowered $T_c$, 88.5 K, and broader transitions compared to the YBCO/LMO/single crystal MgO. The presence of a broad transition width in the ac susceptibility data indicates that the YBCO films may have a lower $J_c$ [15]. The magnetization critical current density ($J_c$) of the YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex films measured at 77 K and 65 K are shown in Fig. 9. A self field $J_c$ of 0.25 MA/cm$^2$ at 77 K and $J_c$ close to 1 MA/cm$^2$ at 65 K self field was noted in these films using Bean's model and the measured magnetic moment. The observed reduction in $J_c$ in these films as compared to YBCO grown on single crystal substrates (typically >1 MA/cm$^2$) can be due to several factors, including the lower $T_c$ and *a*-axis grain growth observed in the films.

To further understand the reasons for the reduction in $T_c$ and $J_c$ in these films, compositional depth profiling was performed using SIMS. Fig. 10 shows a sputtered depth profile acquired from a YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample. In this figure, all elemental signals are plotted on a linear scale, with each signal normalized to its own maximum intensity, so as to best indicate composition. The interfaces in the sputtered profile show some broadening, or non-infinite slope. Both elemental inter-diffusion and roughness of the film surface and/or film interfaces can contribute to these interface slopes in the observed profile. While some roughness is expected, the signals from the YBCO elements show a second, even less steep interface slope at sputter times just beyond an inflection in the signals, at approximately 300 nm of sputter time. This secondary slope indicates elemental interdiffusion between the YBCO and LMO. Also, Mg is seen to diffuse from the MgO layer, as revealed by the broader slope of the Mg signal into LMO compared to the slope of the La and Mn signals into the MgO. Furthermore, the Mg has diffused well into the LMO layer, as evidenced by the substantial penetration of the Mg signal into the LMO layer, when compared to its lack of penetration into the $SiO_2$ layer. It can also be observed in the SIMS data that the relative ratio of La/Mn signals is not constant in the LMO layer. It is thought that this is not due to artifact of depth profiling but probably caused by Mg diffusion in to LMO and its substitution in the lattice.

The diffusion of La and Mn into the YBCO matrix, especially via the grain boundaries, can potentially reduce the $T_c$ and $J_c$ of the YBCO films. Even though the biaxial texture of YBCO was shown to be good, elemental inter-diffusion causing chemical substitutions could adversely affect the superconducting properties. However, a LMO layer of similar thickness deposited on an MgO single crystal yielded high-quality YBCO using the same processing conditions. This suggests that the elemental interdiffusion is not contingent on the LMO chemical composition used or on the

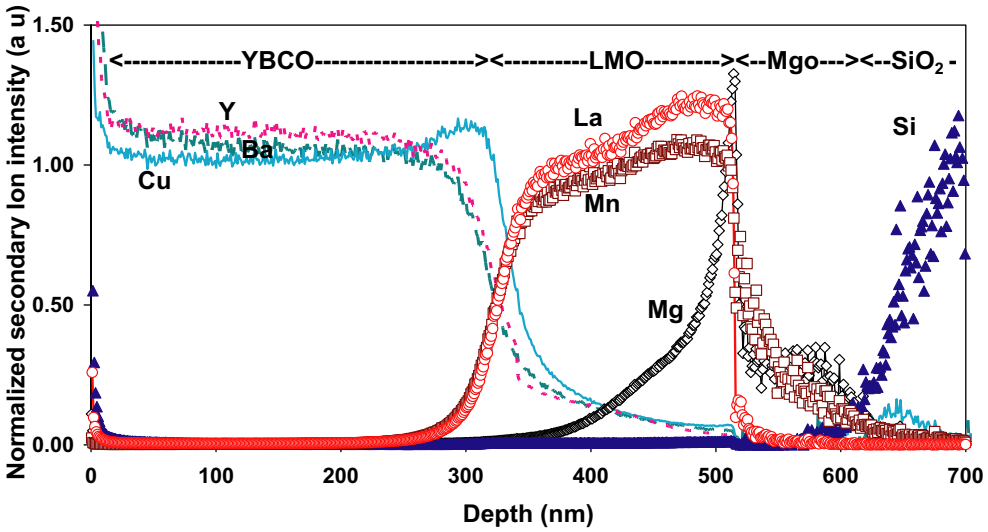


**Fig. 10.** SIMS depth profile analyses of a YBCO/LMO/IBAD-MgO/$Y_2O_3$/$SiO_2$/Ceraflex sample.

subsequent processing conditions used after the IBAD layer is deposited.

It is possible that the small grain size of the IBAD-MgO on these Ceraflex samples makes it more reactive, resulting in its interdiffusion into the LMO as compared to LMO on single crystal MgO deposition under the same deposition conditions. Different deposition methods like SuperPower Inc. processes have produced YBCO coated conductors of excellent quality using an intermediate LMO layer on top of a homo-epitaxial MgO layer [16]. Similar results have also been demonstrated by Oak Ridge National Laboratory [17]. It can then be expected that optimization of the deposition conditions should correct the interdiffusion issue, especially since the biaxial texture is good and high $J_c$ ($2.5 \times 10^5$ A/cm$^2$) was readily achieved on the initial Ceraflex sample with little optimization. Possible changes to the processing conditions used in the present work include making the LMO layer thicker or lowering the deposition temperature to reduce the observed interdiffusion. Importantly, the data presented here shows that highly textured YBCO with reasonable superconducting properties can be grown with little process optimization on polycrystalline flexible YSZ substrates using IBAD-MgO as a template layers and LMO as a cap layer.

It is expected that further refinement of the processing parameters can further improve the superconducting properties. The results discussed in the present report are believed to be useful not only for the coated conductor applications development but also for cryo-electronic applications or microwave filters. In other words, in the applications where a non-conducting substrate and a textured superconducting film are preferred, the MgO buffered ceraflex substrates can be very useful. The future research may include mechanical property testing of YBCO on Ceraflex substrates to determine if the properties of the conductors remain the same as the substrates or change due to the additional processing. Additionally, ac loss testing of the conductors made using Ceraflex substrates will be also useful, to determine the advantages of using these substrates over conducting metallic substrates.

## 4. Conclusions

Initial results for YBCO films deposited on flexible polycrystalline YSZ Ceraflex substrates with IBAD-MgO and $LaMnO_3$ cap layers show good promise. Highly textured YBCO with an in-plane FWHM of 6.75°, out of plane FWHM of ~5° was grown with a $T_c > 88$ K and a $J_c > 0.25$ MA/cm$^2$ at 77 K. Yield strength almost five times greater than Ni–5 at.% W and 1.5 times greater than Hastelloy™ was measured for the Ceraflex substrates. This demonstrates the feasibility of using Ceraflex as a substrate in YBCO coated conductors or electronic applications in instances where it is desirable to have a non-conducting substrate.

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