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FABRICATION OF HIGH CURRENT $YBa_2Cu_3O_{7-y}$ COATED CONDUCTORS USMG ROLLING-ASSISTED BIAXIALLY TEXTURED SUBSTRATES

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High critical current $YBa_2Cu_3O_{7-y}$ (referred to as YBCO) coated conductors were fabricated with a layer sequence of YBCO/YSZ/CeO_2/Ni. The cube (100) texture in the starting Ni substrates was obtained by cold rolling followed by recrystallization. A thin CeO_2 (Cerium Oxide) layer with a thickness of 100-200 A was grown epitaxially on the biaxially textured-Ni, substrates using an e-beam evaporation technique. This was followed by the growth of a thick (≤ 0.77 pm) YSZ (Yttria Stabilized Zirconia) layer using either e-beam evaporation or rf magnetron sputtering. The e-beam CeO_2 film had a dense microstructure. The microstructure of the e-beam YSZ film was porous whereas the sputtered YSZ film was dense. The YBCO films were grown by pulsed laser deposition on both e-beam and sputtered YSZ layers. A transport critical current density of- 1×10^6 A/cm^2 at 77 K was obtained for ~ 0.8 μm thick YBCO films on both YSZ surfaces in zero field. To demonstrate the quality and compatibility of the e-beam CeO_2 layers; YBCO films were also grown on CeO_2-buffered YSZ (100) single crystal substrates using e-beam co-evaporated Y-BaF_2-Cu precursors followed by a post-annealing process. A transport critical current density of over 1 $\times 10^6$A/cm^2 at 77 K was obtained on a ~ 0.3 μm thick YBCO film in zero field.

1. INTRODUCTION

For the development of coated conductors for high temperature (77 K), and high field applications, biaxially textured $YBa_2Cu_3O_{7-y}$ (referred to as YBCO) deposits have emerged as one of the leading candidates. Three approaches have been used to fabricate biaxially textured YBCO films. The first approach was to grow biaxially textured Yttria Stabilized Zirconia (YSZ) buffer layers on polycrystalline substrates including Ni-alloys such as Haynes 230 and Hastelloy C276 through an ion-beam assisted deposition (IBAD) process.[1-4] A high J_c of over 1×10^6 A/cm^2 at 75 K and zero field was obtained.[4] The second approach was to use the Inclined Substrate Deposition (ISD) technique where textured YSZ deposits were made using pulsed laser

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deposition on polycrystalline Ni-alloy substrates by placing the substrate at a certain angle during the deposition? The third approach was recently developed at Oak Ridge National Laboratory using the concept of Rolling-Assisted Biaxially Textured Substrates (RABiTS).[6,7] Our approach was to use rolling-induced texture to obtain biaxially textured Ni tapes and to deposit epitaxially on the tapes both buffer layers and superconductors to form a 'conductor. We demonstrated a J_c of 7 x 10^5 A/cm^2 at 77 K and zero field for films with a layer sequence of YBCO/YSZ/CeO_2/Ni.[7] In this architecture, all oxide layers were grown by pulsed laser deposition (PLD). There is a clear need to develop capabilities to deposit superconducting and buffer layers by alternative deposition techniques. Electron beam evaporation's and rf magnetron sputtering" techniques have been identified as scalable techniques for producing long conductors. In this paper, we report our demonstration of the growth of high current YBCO films on e-beam CeO_2 surfaces and also on both e-beam and sputtered YSZ-buffered Ni substrates

2. EXPERIMENTAL ASPECTS

The 125 μm thick as-rolled Ni substrates were cleaned initially with both acetone and methanol, and recrystallized to the desired (100) cube texture by annealing at 800 °C for 2 h in a high vacuum furnace. An AIRCO Temescal CV-14 10-kW power supply with three e-beam guns was used for the deposition. The biaxially oriented Ni substrates were mounted directly on a heating block inside the e-beam chamber. After the vacuum in the chamber had reached a base pressure of 1 x 10^{-6} Torr, a mixture 4% H_2 and 96% Ar (Forming Gas) was introduced until the pressure inside the chamber reached ~ 1 Torr. The Ni substrates were then annealed at ~ 650 °C for 60 minutes. During CeO_2 deposition, the chamber was pumped out and maintained at a pressure of 2 x 10^{-5} Torr using a mixture of 4% H_2 and 96% Ar. The CeO_2 was deposited by evaporating a Cc metal source and the temperature of the Ni substrate was reduced to 625 °C. To understand the chemistry of the oxide formation, a Dycor Quadruple Gas Analyzer was mounted in the e-beam system. The background H_2O pressure was around 1 x 10^{-5} Torr. The deposition rate was 2-4 Å/sec with the operating pressure of 2 x 10^{-5} Torr, and the final thickness was varied from 100-200 A. During the deposition, the H_2O pressure slowly fell to 2 x 10^{-6} Torr. The measured partial pressure of O_2 was in the range of 10^{-7} – 10^{-8} Torr during deposition. The H_2O pressure present in the chamber is sufficient to oxidize the film to form stoichiometric CeO_2. Following deposition, the substrate was cooled in a pressure of ~ 2 x 10^{-6} Torr to ≤ 100 °C before exposure to atmosphere. The CeO_2 was also deposited on YSZ (100) single crystal substrates at 625 °C using similar deposition conditions

The electron beam evaporation technique was used to deposit YSZ directly on the CeO_2-buffered Ni substrates."' Yttria Stabilized Zirconia was used as the source. The YSZ layers were grown at a temperature of- 625 °C with a total pressure of ~ 2 x 10^{-5} Torr using a mixture of 4% H_2 and 96% Ar. The YSZ deposition rate was 2-5 Å/sec, and the final thickness was varied from 500-5000 A. YSZ films were also grown on e-beam CeO_2-buffered Ni substrates by rf magnetron sputteting as has been described in detail elsewhere." The CeO_2-buffered Ni substrate was mounted on a heating block inside the sputter system. Prior to heating the substrate, the sputter chamber was evacuated to a pressure of ~ 1 x 10" Torr. The chamber was then back-tilled to a pressure of 10 mTorr with a mixture of 4% H_2 and 96% Ar. The substrate was heated to ~ 780 °C and annealed at 780 °C for IO min prior to sputtering. After annealing, YSZ was sputter deposited at 780 °C for ~ 2 h with an on-axis YSZ target located ~ 5 cm from the substrate. The plasma power was 75 W at 13.56 MHz. The resulting YSZ film was smooth and dense, and its thickness was estimated to be approximately 0.77 μm.

The pulsed laser deposition technique was used to grow YBCO at 780 °C and 185 mTorr O_2 on both e-beam and sputtered YSZ-buffered substrates as described elsewhere." Precursor YBCO films were grown on CeO_2-buffered YSZ (100) substrates, YSZ (100). and $SrTiO_3$ (100) substrates by electron beam co-evaporation of Y, BaF_2, and Cu at a combined deposition rate of ~ 5 Å/sec. Details of the experimental conditions were, reported earlier.[12] During evaporation, the substrates were held at ~ 100 °C, while the initial pressure of 2 x 10^{-6} Torr rose to 6 x 10^{-6} Torr. Tantalum crucibles were used for the Cu, Y and BaF_2 sources. Steady BaF_2 evaporation rates were obtained by covering the crucible with a matching lid with a 3-mm orifice. Thermionics 150-0010 3-kW e-beam guns were used for the evaporation.

The films were analyzed by XRD. A Philips Model XRG3100 diffractometer with Cu K_α radiation was used to record powder diffraction patterns. For texture analysis, a Rigaku rotating anode X-ray generator was used, with a graphite monochromator selecting Cu K_α radiation, and slits defining a 2 x 2 mm' incident beam. A four-circle diffractometer was used to collect pole figures, ω scans, and ϕ scans. SEM micrographs were taken using a Hitachi S-4100 Field Emission Scanning Electron Microscope. The beam voltage was 15 kV. The thickness of the films was determined both by Rutherford Backscattering Spectroscopy (RBS) and Alpha-Step 500 (Tencor Instruments) Profilameter scans, The rcsistivity and transport critical current density, J_c was measured using a standard four-probe technique. The values of J_c were calculated using a 1 μV/cm criterion. The Ag contacts were made by sputtering. The YBCO films with Ag contacts were annealed in 1 atm O_2 at 500 °C for 30 min and slow cooled to room temperature in pure Or.

3. RESULTS AND DISCUSSION

The e-beam and sputtered buffer layer characteristics, and transport property measurements of the YBCO films are discussed below.

3.1. E-beam evaporated CeO_2 and YSZ layers

The XRD results from the θ-2θ scan, and also from ω and ϕ scans for 1000 A thick YSZ deposited on 200 A thick CeO_2-buffered Ni substrates revealed good epitaxial texturing. The FWHM for Ni (002), CeO_2 (002). and YSZ (002) were 7.4". 6.6". and 6.8". and that of Ni (202). CeO_2 (202), and YSZ (202) were 9.5°, 8.8°, and 8.5°, respectively. The CeO_2 (111) and YSZ (202) pole figures also demonstrated that the buffer layers were epitaxial with a single orientation. SEM micrographs indicated that the 200 A thick CeO_2 layers were smooth, dense, and columnar." On the other hand, the e-beam YSZ layers were porous and columnar." The porous microstructure of the e-beam YSZ layers could be due to the evaporation from an oxide source. It may be possible to change the microstructure by depositing YSZ at higher temperatures or by evaporating the respective metal sources. The CeO_2 and YSZ layers were crack-free. There seems to be thickness dependence with respect to crack formation in CeO_2 layers on Ni substrates. For example, thick (≥ 2000 A) CeO_2 films typically were cracked.

The θ-2θ scans on the thick YBCO film deposited on all e-beam YSZ/CeO_2/Ni substrates showed c-axis alignment. These films were 0.3 cm wide and 1 cm long. The ϕ scan for a 0.76 μm thick YBCO film on the RABiT substrate is shown in Figure 1. It shows good in-plane

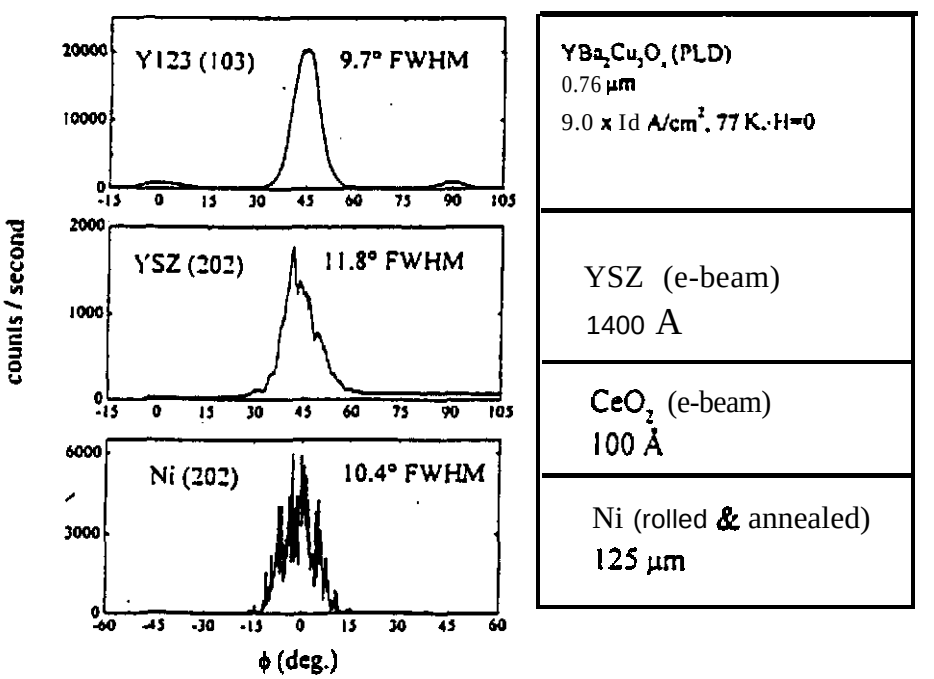

FIGURE 1
The ϕ scans for a 0.76 μm thick YBCO deposited on 1400 A thick YSZ-buffered Ni substrates.

texture. It is interesting to note that the thickness of the YSZ buffer layer was only 1400 A. As shown in Figure 1, the distribution of in-plane orientations of the YBCO was rotated 45° with respect to the underlying Ni in about 95 % of the film. The resistively determined critical temperature, T_c was around 87 K. The temperature dependence of J_c for 0.76 μm thick YBCO films on all e-beam buffer layers is shown in Figure 2. A maximum J_c of ~ 0.9 x 10^6 A/cm^2 was obtained at 77 K and zero field. At 75 K, a J_c of about 1 x 10" A/cm^2 was obtained. These results show that high J_c YBCO films can be grown on YSZ buffer layers with a relatively small thickness of 1400 A.

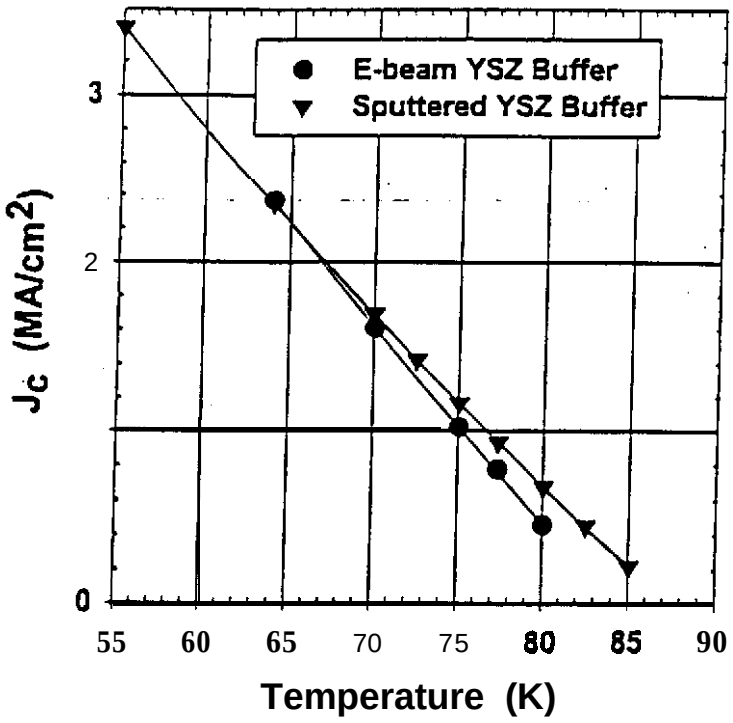

FIGURE 2
The temperature dependence of J_c values for ~ 0.8 μm thick YBCO films deposited on 1400 A thick e-beam YSZ (on 100 A thick e-beam CeO_2-buffered Ni) and 0.77 μm thick sputtered YSZ (on 100 A thick e-beam C&-buffered Ni) buffers.

3.2. E-beam evaporated CeO_2 and sputtered YSZ layers

The XRD results from the 0.77 μm sputtered YSZ layer deposited on CeO_2-buffered Ni substrates revealed good epitaxial texturing. The microstructure of the sputtered YSZ layers was dense, crack-free and well aligned. High current YBCO films were grown on a combination of thick sputtered YSZ buffers and thin e-beam CeO_2 buffers on textured-Ni substrates. The temperature dependence of J_c for a 0.76 μm thick YBCO film is shown in Figure 2. A maximum J_c of > 0.9 x 10^6 A/cm^2 at 77 K and zero field was obtained. From the texture analysis of this film, the distribution of in-plane orientations of the YBCO film was over 95 % cube-on-cube with

respect to the underlying Ni. The difference in the distribution of the YBCO orientations on all e-beam buffers (as from section 3.1) and a combination of sputtered and e-beam buffers (as from the present section) may be related to the microstructure, thickness, deposition technique, and the deposition temperature of the YSZ buffer layers. The detailed microstructure characteristics of all these buffer layers have been described elsewhere.[11,13,14]

3.3. E-beam co-evaporated YBCO precursors and post-annealing

The e-beam CeO_2-buffered YSZ (100), YSZ (100), and $SrTiO_3$ (100) substrates were pre-annealed at optimum temperatures and pressures of oxygen prior to the YBCO precursor deposition. The deposited precursor films were post-annealed in a flowing mixture of N_2, O_2, and H_2O with pO_2 = 200 mTorr and pH_2O = 20 Torr at 760 °C for 60 min. At the end of the hold the gas flow was switched to the dry conditions. The sample was then cooled to 500 °C for 30 min oxidation anneal in 1 atm of O_2. The high-temperature anneal under wet conditions resulted in conversion of the Y, BaF_2, Cu into $YBa_2Cu_3O_{7-y}$. Details of the'post-annealing process have been published elsewhere.[12] The temperature dependence of the resistivity for ~ 0.3 μm thick YBCO films on CeO_2-buffered YSZ (100) (referred as CeO_2/YSZ), YSZ (100), and $SrTiO_3$ (100) (referred as STO) substrates are shown in Figure 3. The room-temperature resistivity of the YBCO film on YSZ substrate was very high and the T_c also was suppressed to 85 K. On the other hand, the YBCO films on CeO_2/YSZ and STO substrates had low room temperature resistivities (230-280 microOhm.cm) and a T_c of about 90 K. The extrapolated values of the resistivity data (between 300 K and above T_c) below zero indicates the presence of predominantly cl YBCO films on STO and CeO_2/YSZ substrates. The relatively poor properties for films deposited directly on YSZ result from a reaction between the substrate and film. This reaction disables the growth of high quality cl YBCO films. A thin cap layer of CeO_2 (200-400 Å), however, prevents this unwanted reaction, leading to film properties which are comparable to those on relatively inert and well-matched STO (100) substrates. For example, a maximum J_c of over 1×10^6 A/cm^2 at 77 K and zero field was obtained on CeO_2-buffered YSZ. The θ-2θ scans of the YBCO films on CeO_2/YSZ substrates indicated the presence of a c-axis-aligned Elm. The presence of small impurity peaks (20 = ° and °), which may be indexed as belonging to $BaCeO_3$, indicate that there is some interaction between the CeO_2 layer and YBCO. The field dependence of J_c for 0.3 μm thick YBCO films on CeO_2/YSZ, and STO substrates are shown in Figure 4. From Figures 3 and 4, we conclude that CeO_2–buffered YSZ substrates provide good templates for ex situ grown YBCO films. Attempts are being made to grow YBCO films on RABiTS by this ex situ precursor approach.

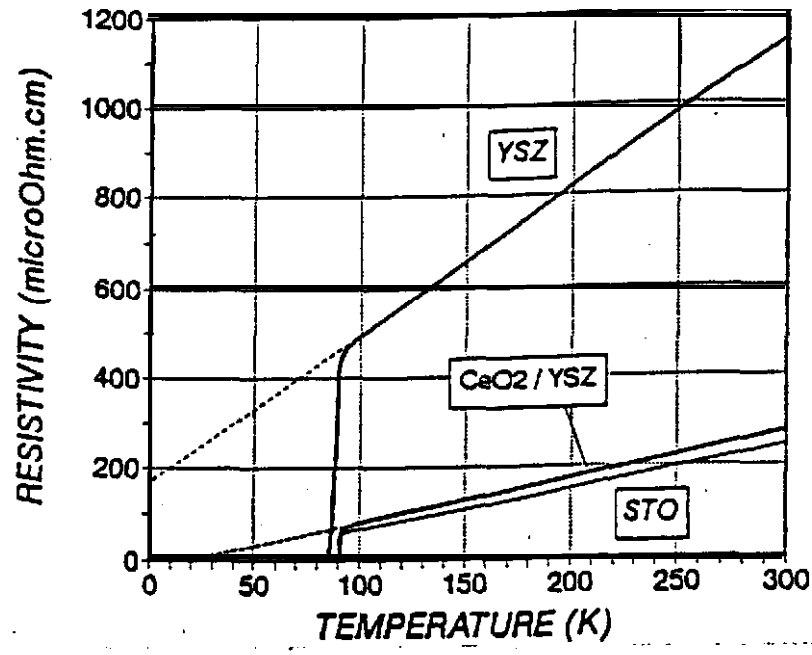

FIGURE 3
The temperature dependence resistivity for ~ 0.3 pm thick YBCO film on e-beam CeO_2-buffered YSZ (100). YSZ (100), and $SrTiO_3$ (100) (referred as STO) single crystal substrates.

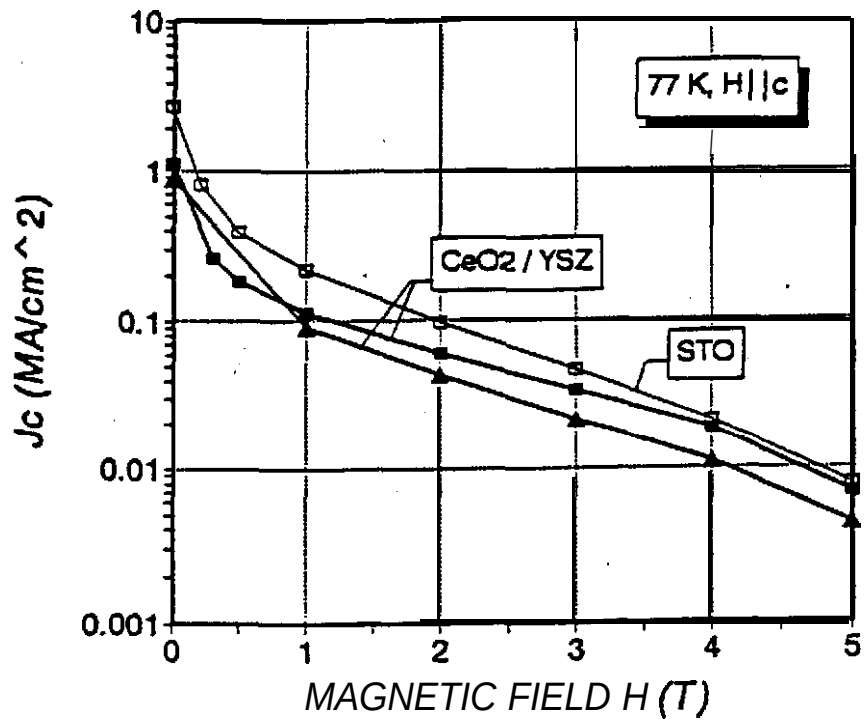

FIGURE 4
The field dependence of critical current density, J_c for ~ 0.3 μm thick YBCO film on e-beam CeO_2-buffered YSZ (100). and $SrTiO_3$ (100) (referred as STO) single crystal substrates.

4. CONCLUSIONS

We demonstrated that high current YBCO films can be grown on buffer layers deposited by both all e-beam and a combination of e-beam and rf magnetron sputtering techniques. The microstructure of the e-beam YSZ films was porous as compared to that of the dense sputtered YSZ films. The microstructure of the thin &beam CeO_2 films was smooth, crack-tie, dense and columnar. We have also demonstrated that high critical current YBCO films can be grown on e-beam CeO_2-buffered YSZ (100) single crystal substrates using e-beam co-evaporated precursors followed by post-annealing.

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