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Title: PREPARATION OF HIGH QUALITY YBa(2)Cu(3)O(7-DELTA)
THICK FILMS ON FLEXIBLE NI-BASED ALLOY SUBSTRATES
WITH TEXTURED BUFFER LAYERS

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Preparation of High Quality $YBa_2Cu_3O_{7-\delta}$ Thick Films on Flexible Ni-based Alloy Substrates with Textured Buffer Layers

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Abstract--High current $YBa_2Cu_3O_{7-\delta}$ (YBCO) thick films on flexible nickel substrates with textured buffer layers were fabricated. Highly textured yttria-stabilized-zirconia (YSZ) buffer layers were deposited by using ion beam assisted deposition (IBAD). Pulsed laser deposited YBCO films were not only c-axis oriented with respect to the film surface but also strongly in-plane textured. The in-plane mosaic spread of YBCO films was ~ 10°. A critical current density of $8x10^5$ A/cm^2 was obtained at 75 K and zero field for thin YBCO films. It was also demonstrated that thick YBCO films with a high critical current and excellent magnetic field dependence at liquid nitrogen temperature can be obtained on flexible nickel substrates by using the textured buffer layers. Issues encountered in producing the films were discussed.

I. INTRODUCTION

Critical current density (J_c) is the single most important property of the high temperature superconductors (HTS) for use in most applications. The low J_c values of these superconductors were a big disappointment of the early years of HTS research. Controlling the materials at the atomic scale becomes critical in producing HTS wires and tapes for bulk applications. In the last few years, researchers around the world have focused their efforts mainly on the oxide powder in tube (OPIT) process for making wires and tapes [1]. In a typical OPIT process, oxide powder is packed and sealed in an Ag tube and then the tube is swaged, drawn and rolled flat. Finally the tape is annealed at temperatures close to the melting point of the superconductor. Of the three major families of HTS, YBCO, Bi-Sr-Ca-Cu-O (BSCCO) and Tl-Ba-Ca-Cu-O (TBCCO), the best wires to date have been made in the BSCCO and TBCCO systems since a platelike morphology can be obtained in both superconductors using the OPIT process [1]. The drawing and rolling process not only densifies the powder but also produces texture in the materials during mechanical deformation. The slightly textured superconductors with platelike morphology are not weak linked, thus carrying high J_c [1]. In the last few years, tremendous progress has been made in producing long HTS wires and tapes using OPIT. However, only limited success has been achieved in making an uniform microstructure of the right superconducting phase over long lengths [1]. Furthermore, the intrinsic irreversibility lines of both BSCCO and TBCCO limit the applications of both superconductors to either low temperatures (<35 K) and high magnetic fields, or 77 K and small magnetic fields (<1 Tesla).

Very littler effort was made to produce YBCO wire and tapes due to the serious weak-link problem in the system. YBCO is the material of choice for applications at high temperatures and in high magnetic fields. Melt-textured processing did yield good YBCO conductors [2], but it will be very difficult to scale up the process for long lengths. One possible method for producing long wires and tapes other than OPIT is the thick film process. As is well known, YBCO thin films on single crystal substrates have J_c values over 10^6 A/cm^2 at 77 K. Extending the thin film technology to produce a thick film on a flexible substrate may open a new possibility for fabricating long YBCO wires or tapes. However, three limiting factors need to be considered. The first one is that high quality thick films have to be demonstrated since YBCO thin films flip their orientation at a thickness of ~ 5000 Å from c-axis oriented to a-axis oriented even on single crystal substrates [3]. The second one is that a textured buffer layer is needed to promote textured growth of YBCO films so that weak-links can be greatly reduced in number. The last one is how to scale up the deposition processes for producing YBCO thick films in long lengths.

We have previously reported that high quality YBCO thick films can be obtained on single crystal yttria-stabilized zirconia (YSZ) substrates with a thin CeO_2 buffer layer under appropriate deposition conditions [4]. C-axis oriented YBCO films can be obtained even for a film thickness over 6 μm. It was also found that the zero field J_c at liquid nitrogen temperature decreases rapidly from $5x10^6$ A/cm^2 for a thin YBCO film to ~$2.5x10^6$ A/cm^2 at a thickness of 1 μm. However, the J_c levels off at a value of $1x10^6$ A/cm^2 for film thicknesses over 3 μm. On the other hand, there were a number of reports of depositing buffer layers on metal substrates [5], which can easily be obtained in long lengths as compared to flexible poly-crystalline YSZ substrates. Depositing buffer layers, mostly YSZ, on metal substrates such as Hastelloy (a Ni-based alloy) or stainless did not result in any textured films as a result of random nucleation of the buffer layers. One exception was using ion-beam assisted deposition (IBAD), in which a YSZ buffer layer was deposited in combination with irradiation from an ion beam directly on the substrates during deposition. With IBAD, the YSZ buffer layers are highly in-plane textured as demonstrated by Iijima et al. [6] and Reade et al. [7]. As a result, the YBCO thin films were also highly textured and had excellent superconducting properties on the IBAD deposited YSZ buffer layers. In this

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paper, we present our results on thick YBCO films on Ni substrates with an IBAD deposited YSZ buffer layer.

II. YBCO THICK FILMS ON SINGLE CRYSTAL YSZ SUBSTRATE

Single crystal (100)-oriented YSZ with 9.5 mol % Y_2O_3 was used in our study on making high quality YBCO thick films. One disadvantage of YSZ is that it has a poor lattice match with YBCO. As a result, while c-axis oriented YBCO thin films may be readily obtained on YSZ, they generally exhibit a mixture of in-plane orientations-the two predominant ones being YBCO [100] ǁ YSZ [100], and YBCO [100] ǁ YSZ [110] [8]. Since high-angle grain boundaries between these two orientations can dramatically reduce J_c [8], it is necessary to control the in-plane texturing in addition to having c-axis orientation for useful conductors. By depositing a thin buffer layer of CeO_2, the high-angle grain boundaries can be easily eliminated. The resulting in-plane orientation is YBCO [110]ǁ CeO_2 [100]ǁ YSZ [100] [9].

Film deposition of both CeO_2 and YBCO was made using a pulsed laser deposition (PLD) system employing a XeCl excimer laser (308 nm, 20 ns) [4]. A 1000 Å thick CeO_2 film was deposited at a substrate temperature of 800-900 °C in 200 mTorr O_2. The CeO_2 was (100) oriented and highly ordered, with a Rutherford backscattering ion beam minimum yield of 2% in the channeling mode at 2.2 MeV ion beam energy. For the YBCO deposition, the oxygen pressure was held at 200 mTorr and the substrate temperature was 800 °C. The deposition rate was 0.1-0.2 µm/min at a pulse repetition rate of 20 pps. When the desired thickness was reached the sample was cooled to room temperature in 250 Torr O_2. Transport critical current measurements were made at liquid nitrogen temperature (75 K) and self field on bridges ~ 100 µm wide and 2-3 mm long, using a 1 µV/cm criterion. Results are shown in Fig. 1 [4]. In the same figure, the critical current is calculated from the J_c assuming a film width of 1 cm. It is clear that J_c rapidly decreases as the film thickness increases but saturates to a value of 1×10^6 A/cm^2 for film thicknesses over 3 µm. Beyond 3 µm, the calculated critical current increases linearly with the thickness. From the calculation, a 6 µm thick and 1 cm wide film can carry ~ 600 A, which is a very high current value. The reason that J_c is still ~ 1×10^6 A/cm^2 for films beyond 3 µm is that the films are highly c-axis oriented. Fig. 2 shows a transmission electron microscopy (TEM) micrograph of a thick YBCO film. The image is a region 2.5 µm away from the YBCO/CeO_2 interface. It is very clear that the YBCO film is highly ordered even at this thickness with the c-axis oriented perpendicular to the film surface. X-ray measurements indicated that the ratio of c-axis to a-axis oriented material in the films was over 100:1 [4]. The J_c decrease is neither the result of a transition to a-axis growth nor a loss of in-plane texture, but instead is probably due to discontinuities, which constrain the current-transport cross-section, or some other type of disorder that increases the film resistivity. Nonetheless, the high J_c and the trend beyond 6 µm indicate that thin-film deposition techniques are suitable for the fabrication of thick film conductors. The data in Fig. 1 is also the basis for working on the thick films on flexible substrates.

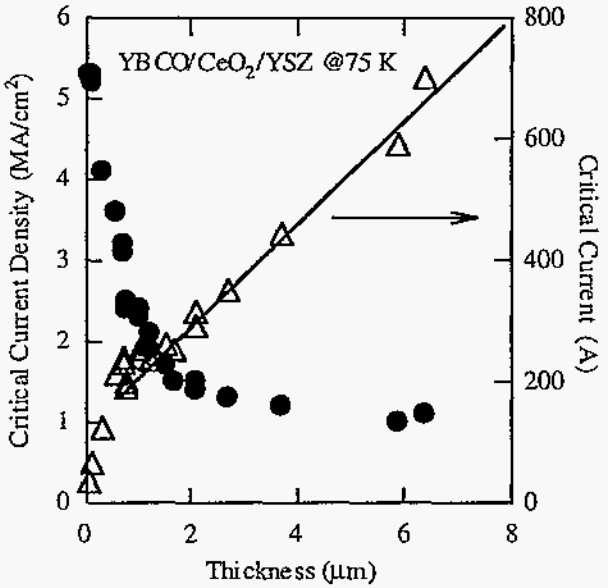

Fig. 1. Critical current density vs. thickness for YBCO films on YSZ with a CeO_2 buffer layers. A total of 22 samples were measured for the 14 points shown. The average J_c is shown in the figure . The right axis is for critical current calculated assuming 1 cm wide samples.

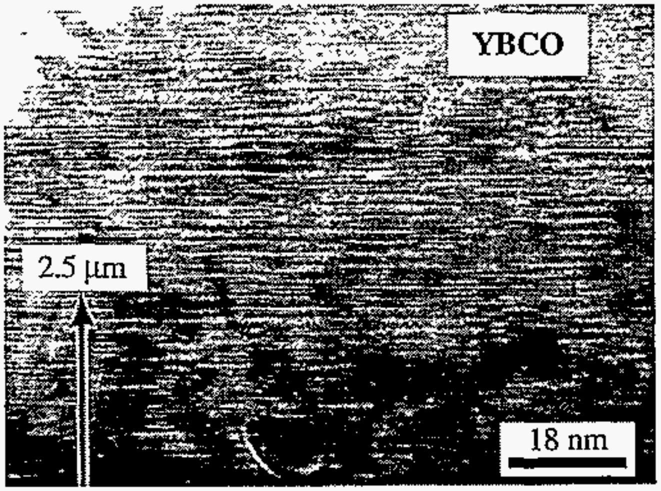

Fig. 2. TEM micrograph taken at 2.5 µm away from the YBCO/CeO_2 interface. The figure shows that the YBCO maintains the c-axis orientation perpendicular to the film surface even at this thickness.

III. ION BEAM ASSISTED DEPOSITION

Ion beam assisted deposition (IBAD) has been used for decades for hard coatings, optical film coatings and so on

[10]. IBAD was used to deposit biaxially aligned Nb films at room temperature on amorphous silica substrates [11]. In the experiment, a glancing 200 eV Ar^+ beam was used to irradiate the film as it was deposited. The textured direction of the Nb film was shown to align with the direction of the incident ion beam. Variation of sputtering yield with ion beam direction with respect to the crystal structure was proposed as the mechanism for texturing [11]. As mentioned in the introduction, Iijima et al. [6] and Reade et al. [7] reported the preparation of biaxially aligned YSZ films on Ni-based Hastelloy substrates using dual ion beam deposition and ion assisted pulsed laser deposition, respectively.

We used a dual ion beam sputtering deposition for depositing YSZ films on Ni and Ni-based alloy substrates as shown in Fig. 3 [12]. The ion sources are manufactured by Ion Tech with a sputter source diameter of 5 cm and an assisting source diameter of 2.5 cm. The angle between the substrate normal to the assisting ion beam is ~55°. Argon is used in both ion guns. The total pressure during deposition is 1×10^{-4} Torr in which the oxygen partial pressure is $1.5\text{-}2.5\times10^{-6}$ Torr. A ceramic YSZ target was used and the films were deposited at room temperature. The YSZ deposition rate was controlled at ~0.4 Å/s.

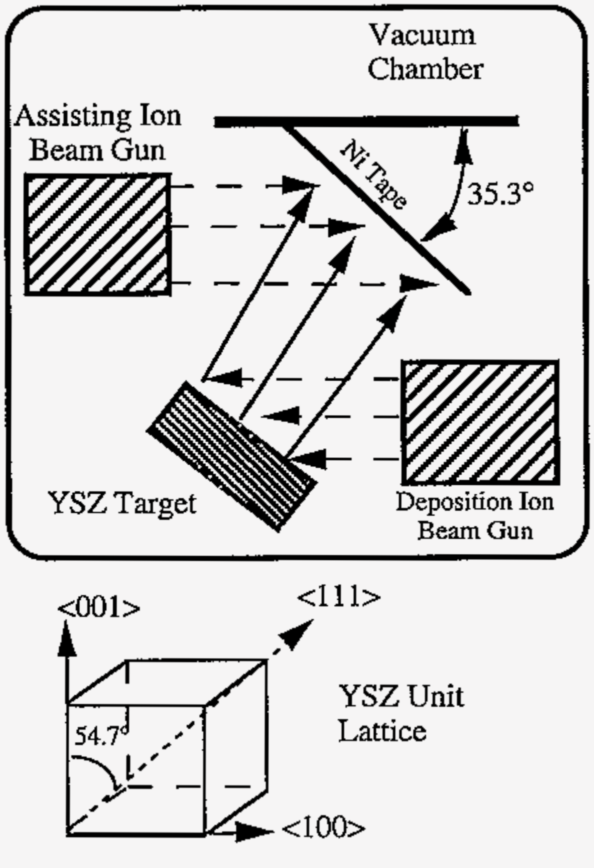

Fig. 3. Dual ion beam sputtering system for deposition of textured YSZ thin films on Ni.

The assisting ion gun was operated at a beam voltage of 250 V and current density of 150 mA/cm^2 on the samples, respectively. A typical YSZ thickness is 5000 Å-8000 Å as determined by a quartz thickness monitor. The substrates used in this study were either Ni or Hastelloy C-276. The reason why we and others use these substrates is that the materials have a good thermal expansion match with YBCO and relatively oxidation-resistant at high temperature. The substrates were electropolished followed by an ultrasonic cleaning in soap and water with a final methanol rinse and drying with nitrogen gas before loading into the deposition system. The RMS (Root-Mean-Square) surface roughness of the substrates was ~1000 Å with nominal peak to valley distances of 7500 Å.

With IBAD, the YSZ films on Ni were polycrystalline with diffraction peaks of (111), (200), and (220) observed in θ-2θ x-ray diffraction. Under the optimized IBAD deposition conditions, only (200) oriented YSZ films were found in the x-ray diffraction, indicating other oriented grains are eliminated [12]. Though preferential etching of misoriented materials was proposed as being responsible for inducing texture earlier, a mechanism based on growth instability of the differing orientations during ion bombardment was also suggested [13]. However, more detailed experimental results are needed to identify which mechanism is responsible for the texturing.

Fig. 4 shows an x-ray ϕ scan of (202) YSZ on Ni. In contrast to an angle independent value for a randomly oriented YSZ film, the IBAD YSZ film showed four peaks in 360°, as expected for a system with a four-fold symmetry. It should be noted that the peaks are very broad as compared to the ones for a YSZ single crystal. However, it is still very remarkable that the full width at half maximum (FWHM) of the peaks is 14° for a YSZ film on a polycrystalline Ni substrate. One of the (111) peaks observed in the ϕ scan always corresponds to the direction of the assisting ion beam.

IV. YBCO FILMS ON Ni

Both CeO_2 and YBCO films were deposited using parameters similar to the ones described in Section II. All the films were subsequently deposited without breaking the vacuum. These films were deposited in a separate chamber after YSZ deposition. After the deposition, no further heat treatment was carried out. In Fig. 5, an x-ray ϕ scan of a YBCO film on YSZ/Ni with a CeO_2 buffer layer is shown. Note that the x-ray diffraction intensity is plotted on a logarithmic scale. From the figure, it is clear that the CeO_2 buffer layer was grown on YSZ with a cubic-on-cubic match (the diffraction peaks from both (202) CeO_2 and YSZ are aligned with each other) [9]. However, a 45° rotation between the YBCO basal plane and that of CeO_2 is observed as expected from the lattice match point of view [9]. The lattice constants of YBCO a axis and b axis (3.82 Å and 3.89 Å, respectively) multiplied by $\sqrt{2}$ are very close to the lattice constant of CeO_2 (5.41 Å). The data also shows that both YBCO and CeO_2 only have one in-plane orientation.

The smallest FWHM of the diffraction peaks from the (103) YBCO Phi scan is ~10° [14]. Though this value is still relatively large, it is significant to obtain YBCO with such a small in-plane mosaic spread on a randomly oriented Ni substrate, indicating that the majority of the grains have grain-boundary angles of less than ~5°. Based on the results of the bi-crystal thin film experiments by Dimos et al. [15], the critical current density across a grain boundary with a tilt angle of 5° will be about 20% that of the grain J_c. Therefore, J_c at liquid nitrogen temperature for the YBCO films on CeO_2/YSZ/Ni is expected to be $\sim 1\times10^6$ A/cm^2 assuming the intragrain J_c is 5×10^6 A/cm^2, which is the best J_c for YBCO thin films on single crystal substrates such as $LaAlO_3$ and $SrTiO_3$. Indeed, a 2800 Å YBCO thin film on CeO_2/YSZ/Ni has a J_c of 8×10^6 A/cm^2 at 75 K by a direct transport measurement with a J_c criterion of 1 μV/cm^2. The obtained J_c is fairly close to the estimated value based on the FWHM of the in-plane mosaic (the texturing quality of the film). It should be noted that the YBCO films were deposited on substrates with a fairly rough surface. Thick YBCO films were also made. Fig. 6 shows J_c vs. magnetic field for a 1.5 μm thick YBCO film with the magnetic field parallel to the c-axis of the superconductor. The YBCO film with a width of ~ 1 cm carried a critical current of 23 A [14] and 47 A at 75 K and 65 K, respectively. In comparison, data for Bi-2223 tapes [1] and Tl-1223 thick films [16] are also included in the figure. It is clear that the YBCO film has outperformed both Bi- and Tl-based superconductors at high magnetic fields. This is expected since the YBCO irreversibility line lies at a much high temperature than in the Bi- and Tl-based superconductors.

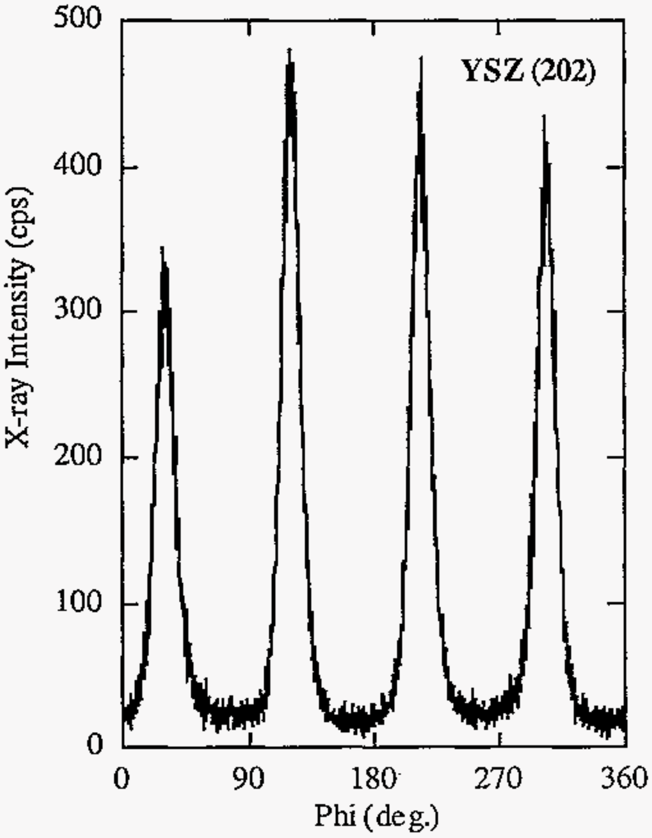

Fig. 4. X-ray Phi scan for an IBAD deposited YSZ film on Ni. The FWHM of the peaks is 14°.

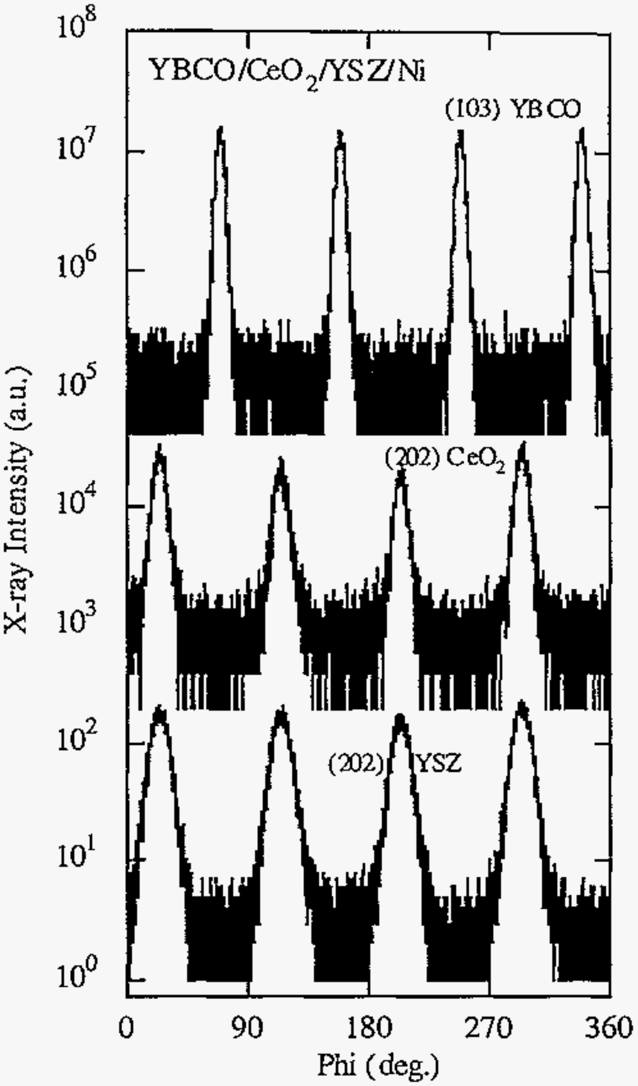

Fig. 5. X-ray ϕ scan of a YBCO on CeO_2/YSZ/Ni. Note that each plot is offset.

The J_c measured in the thick films was slightly less than expected, indicating further processing improvement in making YBCO films is needed. By any means, the J_c values are still very good. These values are directly related to the microstructures of the films. Fig. 7 shows scanning electron microscopy images of the YBCO surface and cross-section. The grains are very well connected with a typical grain size of ~1 μm. The well connected grains promote easy current flows through the grain boundaries.

Grain boundaries and voids are clearly visible in Fig. 7(a), indicating that the films were formed by island growth. As previously found, substrate temperature during deposition, deposition rate and final film thickness control the overall surface morphology [17]. The results show that there is still room for further optimization of the deposition parameters for YBCO. Fig. 7(b) shows that the YBCO films are very dense with internal structures. Detailed microstructural analysis is under way.

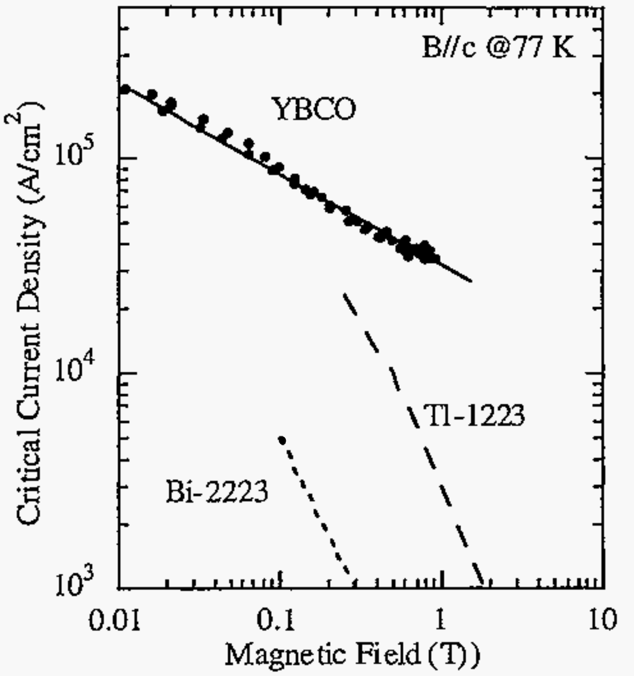

Fig. 6. Critical current density vs. magnetic field for a YBCO thick film, Tl-1223 thick film and Bi-2223 tape.

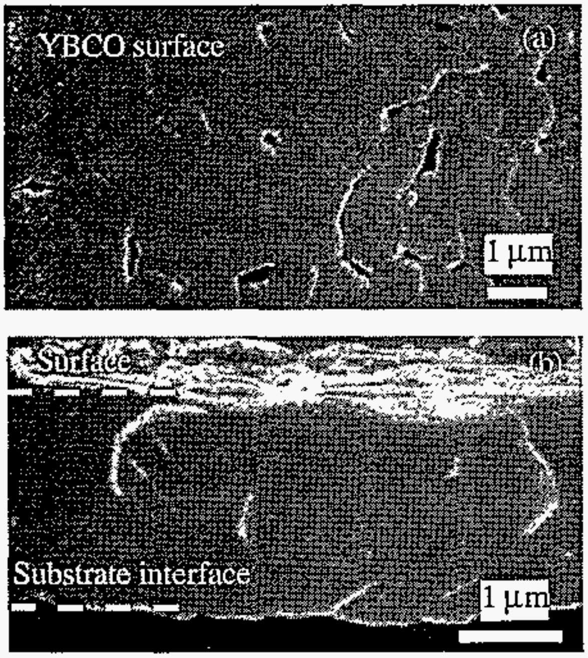

Fig. 7. Scanning electron microscopy images of a YBCO film. (a) surface and (b) cross-section.

V. FUTURE WORK

The biggest problem we found is from the grain boundaries in the Ni substrates. The substrate materials from various vendors are different. The grain boundaries contains gases such as sulfur from the original production of the Ni or Ni-based alloys. Furthermore, the boundaries also contain secondary-phase materials. Since the IBAD YSZ films were made at room temperature, the grain-boundary problem only becomes visible after YBCO deposition which was done at a high substrate temperature. Fig. 8 shows an optical micrograph of a YBCO film on CeO_2/YSZ/Ni. The grain boundaries from the substrates are imprinted through all the layers to the surface. The YBCO film has a good superconducting transition temperature of ~90 K, but a very low critical current density as a result of the grain boundaries. Furthermore, delamination of the film was observed in some areas (areas surrounded by the dark rings in the figure). We are currently trying to solve this very fundamental problem. Other issues such as adhesion between the oxides and metal substrate are also being studied.

Though the scale-up has not been done by us, researchers at Sumitomo have deposited both YSZ and YBCO films on a 1.1 meter long Hastelloy tape using pulsed laser deposition [18]. Since the YSZ films were not textured from the process, the obtained J_c is ~1.0-4.5x10^4 A/cm^2 at 77 K. This work demonstrated that thin film deposition techniques can be used to make long tapes, especially in this case of high temperature superconductors. Furthermore, assisting ion guns up to 1

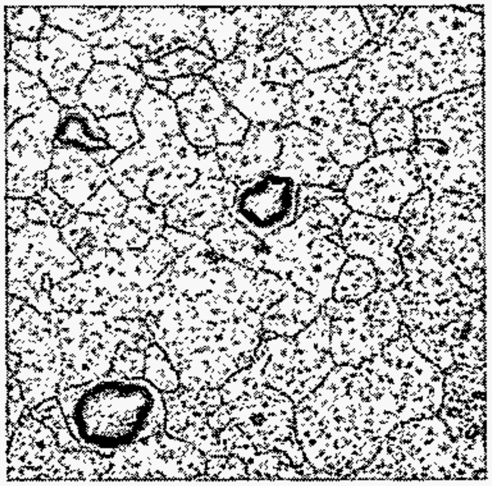

Fig. 8. Optical micrograph (200 X) of a YBCO thick film on CeO_2/YSZ/Ni, showing the grain boundary problem from the substrate. The dark rings are the perimeters of where the film is delaminated.

meter long are currently available. As long as the basic material problems can be solved, scale-up for making long wires and tapes will only be a matter of time.

VI. CONCLUSIONS

We have demonstrated that high critical current YBCO thick films can be obtained using textured buffer layers. Ion beam assisted deposition is capable of creating highly textured films on randomly oriented substrates. The processes used in the present experiment can be scaled up for the long lengths. Thick YBCO films on flexible substrates are expected to be useful for applications at liquid nitrogen temperature and in high magnetic fields.

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