LA-UR- 01-2099

Approved for public release;
distribution is unlimited.

Title: Development of the IBAD MgO Process For HTS Coated Conductors

Author(s): James R. Groves, Paul N. Arendt, Stephen R. Foltyn, Quanxi Jia, Terry H. Holesinger, Harriet Kung, Raymond F. DePaula, Paul C. Dowden, Eric J. Peterson, Liliana Stan, and Luke A. Emmert, MST-STC

Submitted to: ISTEL 2001--Honolulu, Hawaii, June 24-27, 2001. Session S2-2

This is an extended abstract.

Los Alamos
NATIONAL LABORATORY

DEVELOPMENT OF THE IBAD MgO PROCESS FOR HTS COATED CONDUCTORS

James R. Groves, Paul N. Arendt, Stephen R. Foltyn, Quanxi Jia, Terry G. Holesinger, Harriet Kung, Raymond F. DePaula, Paul C. Dowden, Eric J. Peterson, Liliana Stan, and Luke A. Emmert
Material Science and Technology Division, Los Alamos National Laboratory
Los Alamos, New Mexico, 87545 USA

ABSTRACT

We discuss our progress toward depositing IBAD MgO as a template for subsequent deposition of $YBa_2Cu_3O_{7-\delta}$ (YBCO) high temperature superconductors on metallic substrates. We have refined the process by improving substrate preparation and by using reflected high-energy electron diffraction (RHEED) to monitor the growth of IBAD MgO films. Here we present results on our work to optimize the IBAD MgO process for continuous processing of meter lengths. High quality IBAD MgO has been deposited on moving metal tape for use in coated conductor fabrication. By incorporating optimized processing parameters, we have been able to deposit films on moving metallic substrates with in-plane mosaic spreads near 8°. The subsequent pulsed laser deposition (PLD) of 1.5 μm thick YBCO films has resulted in superconducting transport critical current densities >1MA/cm^2 (75K, SF) on small area samples.

INTRODUCTION

High temperature superconductors (HTS) on flexible metal tapes are of recent interest for power transmission applications throughout the world. To date, only bismuth-based HTS wire has been produced in sufficient length to be used in fabrication of wire for electrical applications. However, YBCO has better irreversibility in magnetic fields than bismuth-based superconductors but suffers from a need for good grain alignment to achieve high superconducting performance[1]. To overcome this shortcoming, much effort has been focused on developing methods by which one can texture underlying layers or substrates for subsequent heteroepitaxial deposition of YBCO.

Two techniques have achieved marked success in the effort to produce YBCO coated conductors: roll-assisted biaxial texturing of substrates (RABiTS) and ion-beam assisted deposition (IBAD). In the case of RABiTS, the substrate material (pure nickel or a nickel-based alloy) is imparted with biaxial texture during mechanical deformation and subsequent heat treatments and has been successfully integrated with high performance YBCO[2]. In contrast, IBAD relies on bi-axial texturing of a template layer applied to any substrate (single crystal, polycrystalline- metal or ceramic). The most commonly used material to date has been yttria-stabilized zirconia (YSZ) and subsequent depositions of YBCO on biaxially oriented YSZ has resulted in world record performance for both short (<10 cm) and long lengths (1 meter) of coated conductors[3, 4].

A major criticism of IBAD YSZ in the production of lengths of coated conductors has been the length of time required and thickness needed (0.5 to 1 μm) to develop sufficient in-plane texture for deposition of YBCO with high performance superconducting transport properties[5].

Wang et al., showed that another material, MgO, could develop comparable texture using the IBAD process in only 10 nm[6]. This translates to a process that is ~100× faster per meter than IBAD YSZ.

EXPERIMENT

The substrates used in this study were either nickel-based alloys Haynes 242, Inconel 625 or silicon wafers (silicon substrates were used for TEM sample preparation and for optimization of IBAD processing parameters to reduce the effect of roughness). All metal substrates were 1 cm wide (Inconel 625 and the Haynes 242 were 0.1 mm and 0.5 mm thick, respectively). Before deposition, the metal substrates were mechanically polished with 1 μm diamond paste to an average surface roughness of 2 nm. The silicon substrates required no surface polish and the native oxide layer was not removed. An ~5 nm thick amorphous layer was deposited on the substrate surface using electron beam deposition with a silicon nitride source. A background partial pressure of nitrogen (6.7×10^{-3}Pa) was introduced into the deposition chamber during the silicon nitride coating process. A subsequent layer of MgO was deposited on the amorphous SiN_x layer using IBAD. Argon ions were accelerated to 750 eV with a current density of 100 mA/cm^2 using a 22 cm × 2.5 cm Kaufman ion source. The ion source incidence angle is at 45° relative to the substrate which corresponds to the MgO <110>. Concurrently, an electron beam evaporator provided the MgO vapor flux of 0.15 nm/s during the IBAD growth. The ion to atom ratio was kept constant at 0.7, which reduced the effective deposition rate by 30% to 0.1 nm/s due to resputtering. The vapor flux and the ion fluence were monitored with a quartz crystal microbalance (QCM) and a Faraday cup, respectively. All IBAD depositions were performed at room temperature. A homoepitaxial deposition of another 100 nm of MgO was then added at 500°C. This thickness was needed to allow the MgO film texture to be quantified by X-ray diffraction.

IBAD film growth was monitored in situ using Reflected High-Energy Electron Diffraction (RHEED) by collecting a spot intensity versus time (I vs. t) curve that used the reflections corresponding to the (002) and (022) planes. Images were captured using kSA400 software (k-Space Associates, Ann Arbor Michigan). All patterns were taken at a beam energy of 40 keV.

Pulsed laser deposition (PLD) was then used to heteroepitaxially deposit subsequent buffer and YBCO layers. These depositions were done at substrate temperatures of 730-770 °C. Two buffer layers were used in this work. The first layer was 50 nm of YSZ followed by 20 nm of yttria. Both of these were deposited at a rate of 0.5 Å/sec. These buffer layers were used to obtain improved lattice matching with the final YBCO films. The YBCO films were nominally 1.5 μm thick and were deposited at a rate of 20 Å/sec.

Metal samples were then patterned into micro-bridges with nominal dimensions of 250 μm wide by 5 mm long. Superconducting transition temperatures and transition widths were measured using an inductive probe. Transport critical current (I_c) and critical current density (J_c) were measured at liquid nitrogen temperature (75 K) and self field using a 1 μV/cm criterion.

RESULTS

The majority of our work has focused on optimizing IBAD MgO for subsequent YBCO deposition on technically important polycrystalline nickel-based super-alloys. To date, we have

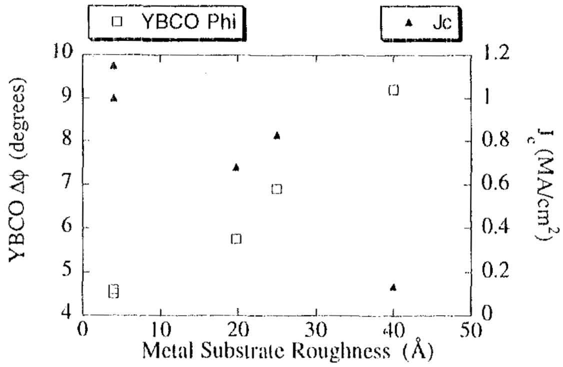

Figure 1. Plot of the effect of metal substrate roughness on critical current density and YBCO $\Delta\phi$.

deposited several YBCO films ($\geq$ 1.5 mm) on small-area metal substrates with IBAD MgO template films that have achieved over 1 MA/cm^2[7].

Several factors influence the deposition of high quality IBAD MgO films. One of the most critical is substrate roughness. As shown in figure 1, there is a direct correlation between reduction in metal substrate surface roughness and final YBCO in-plane texture (a measure of which is the full-width at half-maximum value taken from a peak in a ϕ-scan hereafter denoted by the symbol $\Delta\phi$) as well as the corresponding critical current density (J_c).

Another factor that affects good quality IBAD MgO is the extent of in-plane orientation distribution. Plan view TEM coupled with dark-field imaging has revealed that there are some irregularities in biaxial orientation between MgO grains deposited by IBAD. Although silicon is extremely smooth (<0.3 nm roughness), Figure 2 shows, in a dark field image, a distribution of in-plane orientations indicated as changes in contrast across the illuminated area.

Improvements in both surface finishing and optimization of deposition parameters for IBAD MgO has resulted in a remarkable improvement in both superconducting transport properties and in-plane texturing of final YBCO films. Typical values of critical current density $\geq$ 1 MA/cm^2 are now achieved on small area samples and YBCO $\Delta\phi$ values between 5° and 6° are routinely deposited on these template layers.

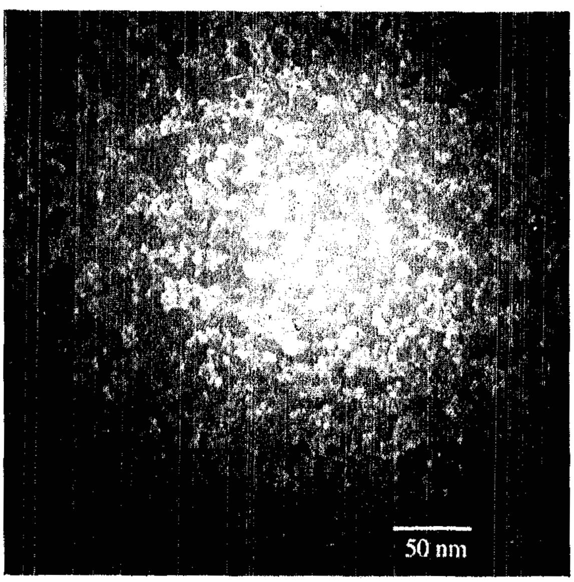

Figure 2. Dark-field TEM image of IBAD MgO (~10 nm thick) as-deposited on a silicon substrate. Changes in the contrast (from light to dark) indicate a decrease in relative orientation to the diffracted beam image.

Much of the IBAD MgO texture consistency has been due to application of RHEED as a process monitor during deposition. In this monitoring scheme, a spot pattern is obtained as the IBAD MgO layer is deposited. Monitoring of spot intensity as a function of time (I vs. t curve) has been shown previously to allow one to determine at what point the deposition should be stopped to achieve the best textured films[8]. Initially, films deposited on 1 cm by 1 cm silicon wafers were used to calibrate the RHEED I vs. t curve with X-ray diffraction measurements of the in-plane texture. Film growth was stopped at various times along the I vs. t curve and analyzed with XRD for texture. The results of one of these experiments are shown in figure 3. Briefly, we found that halting the deposition at the time at which the spot intensity reaches its maximum value results in the best $\Delta\phi$ value.

The next major challenge has been to deposit these films on continuously processed lengths.

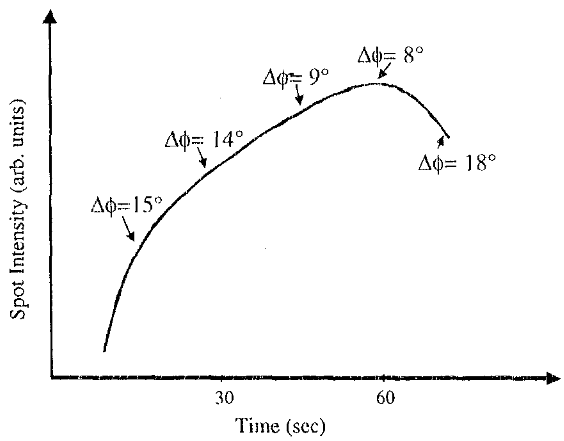

Figure 3. XRD Δϕ values for IBAD MgO films deposited at thicknesses corresponding to the annotated times along the RHEED I vs. t curve.

Initial depositions on meter long substrates produced samples that were superconducting from end to end but had I_c's of 1.2 and 2.5 A, respectively[9]. However, these early tapes had YBCO average Δϕ values of 18.5° and 23.2°, respectively. Analysis revealed that these early tapes suffered from the problems previously discussed (i.e., high substrate roughness, lack of RHEED monitoring, etc.).

Recently, we have incorporated RHEED monitoring to fabricate longer length samples (~100 cm) and improve Δϕ. The IBAD MgO had Δϕ ≤ 14° along the length of the sample. Some sections (over 10 cm long) had Δϕ values approaching 8° as shown in figure 4. We have incorporated improved substrate polishing and optimized IBAD deposition parameters through the use of the RHEED I vs t technique to achieve the improved bi-axial texture shown in this example.

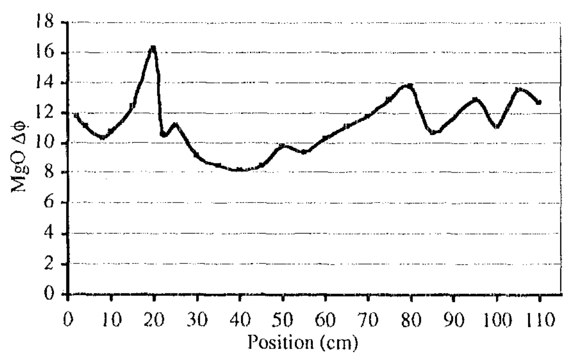

Figure 4. XRD Δϕ values for IBAD MgO films deposited on a continuously processed meter length of 1 cm wide nickel-based alloy I625.

CONCLUSION

Here, we have demonstrated that high quality IBAD MgO can be deposited on moving tape. Many parameters influence the IBAD MgO texture. The most prominent is substrate surface roughness. Improvement in substrate polishing coupled with in situ RHEED I vs. t curve monitoring has resulted in the optimization of processing parameters and ultimately in the deposition of highly textured (Δϕ ~8°) IBAD MgO on meter long nickel-alloy substrates. Further, small area samples, subsequently deposited with > 1 μm thick PLD YBCO, have achieved J_c >1MA/cm^2 (75K, SF).

REFERENCES

1. D. Dimos, et al., *Phys. Rev. Lett.*, 61, 219-222 (1988).
2. A. Goyal, et al., *JOM*, 51, 19-23 (1999).
3. S. R. Foltyn, et al., *IEEE Trans. Appl. Supercond.*, 9, 1519-1522 (1999).
4. S. R. Foltyn, et al., *Physica C*, 341-348, 2305-2308 (2000).
5. D. K. Finnemore, et al., *Physica C*, 320, 1-8 (1999).
6. C. P. Wang, et al., *Appl. Phys. Lett.*, 71, 2955-2957 (1997).
7. J. R. Groves, et al., *JMR Rapid Comm*, (2001 in press.
8. J. R. Groves, et al., *Physica C*, (2001) in press.
9. D. E. Peterson, et al., Los Alamos National Laboratory Superconductivity Technology Center-Coated Conductor Tape Development, *Superconductivity for Electric Systems Programs 2000 Annual Peer Review*, August 2000.