

High-Current Y-Ba-Cu-O-Coated Conductor Using Metal Organic Chemical-Vapor Deposition and Ion-Beam-Assisted Deposition*

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Abstract--YBa₂Cu₃O_x (YBCO) films have been deposited on buffered metal substrates by metal organic chemical-vapor deposition (MOCVD). Nickel alloy substrates with biaxially-textured yttria-stabilized zirconia (YSZ) buffer layers deposited by ion-beam-assisted deposition (IBAD) were used. A liquid-static precursor delivery system was designed, constructed, and used in the MOCVD facility at Intermagnetics for the reported work. At 77 K, under self-field conditions, we achieved a critical current (I_c) of 97.5 A in YBCO film grown by MOCVD on an IBAD substrate. This I_c corresponds to a critical current density of 1.3 MA/cm² and 130 Å/cm width of tape.

Index Terms--MOCVD, IBAD, current density, biaxial-texture

INTRODUCTION

IGC-SuperPower has been developing a metal organic chemical-vapor-deposition (MOCVD) process for the fabrication of high-current YBa₂Cu₃O_x (YBCO) conductors with substrates prepared by ion-beam-assisted deposition (IBAD). MOCVD has several special features such as nonline-of-sight coating, separation of precursors from the deposition chamber, high deposition rates, and no target fabrication expense, all of which can be important for production of long lengths of YBCO conductor for electric power applications. The possibility of using YBCO to produce high-current tapes was first demonstrated with IBAD substrates [1], and only IBAD substrates have been used to produce a 100-Å YBCO tape in meter-long lengths [2]. Earlier, we reported that we had achieved both high (50-Å) critical currents (I_c) and high (>1 MA/cm² at 77 K) critical current densities (J_c) in YBCO films fabricated by MOCVD on IBAD substrates [3]. The performance of the MOCVD-prepared YBCO films in high magnetic fields was shown to be comparable to that of pulsed-laser-deposition (PLD)-

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(PLD)-prepared YBCO films. Here, we report the use of a liquid precursor delivery system, a doubling of the I_c to almost 100 A, and microstructure analysis of MOCVD-prepared YBCO films.

EXPERIMENTAL

The metal substrates used for MOCVD of YBCO consisted of biaxially-textured buffer layers of yttria-stabilized-zirconia (YSZ) deposited by IBAD at Argonne and Los Alamos National Laboratories (ANL and LANL), respectively. Polished Hastelloy-C and Inconel 625 metal substrates, ≈100 μm thick were used. The YSZ buffer layers were typically 0.5–1 μm thick. In the process used at ANL, an 8-cm Kaufmann-type DC ion source was used to produce a 300-eV assisting ion beam of Ar/10%O₂ that was directed onto the substrate surface at an angle of 35°. Electron-beam evaporation was used for YSZ deposition. The atomic deposition rate was 1.6 Å/s and the ion flux was 200 μA/cm², yielding an ion-to-atom flux ratio of 2.8. Details of the IBAD process used at LANL have been published in Ref. 4. Briefly, Inconel 625 substrates were polished to a surface roughness of 3 ±1 nm. The assisting ion was produced by a linear DC Kaufmann-type source, with beam dimensions of 2.5 x 23 cm. The atomic deposition rate was 0.5 Å/s, the assist ion energy was 200 eV, and the ion flux was 190 μA/cm². The YSZ-buffered metal substrates were cut into 1-cm-long, 4–8-mm-wide segments for YBCO deposition by MOCVD. Single-crystal YSZ substrates and SrTiO₃ substrates were also used for deposition, for comparison.

MOCVD of YBCO was conducted at Intermagnetics in a custom-built facility that has been described previously [3,5]. Tetramethyl heptanedionate (thd) precursors were used for Y, Ba, and Cu. YBCO films, 1 μm thick, were deposited within a temperature regime of 700–800°C under a reactor pressure of 1–5 torr and an oxygen partial pressure of 0.3–0.8 torr. After deposition, YBCO films were coated with silver pads, annealed at ≈400°C in oxygen.

The out-of-plane and in-plane textures of the YSZ buffer layers were examined by X-ray diffraction and pole figure measurements. The surface morphology of the YBCO films was studied by scanning electron microscopy (SEM), followed by compositional analysis by energy dispersive X-ray spectroscopy (EDS). The thickness of the films was measured by surface profilometry. The current density of the films was tested in liquid N in zero applied magnetic field. The samples were not patterned and J_c was measured across the entire width of the sample; continuous DC currents up to 100 A were used in the measurements.

LIQUID PRECURSOR DELIVERY

The schematic diagram in Fig. 1 shows the configuration of the MOCVD system at Intermagnetics. Precursor delivery is a three-step process. In the first step, a liquid precursor is pumped at a constant flow rate by a liquid-delivery pump. In the second step, the liquid precursor is flash-vaporized in a vaporizer maintained at a constant temperature. The vaporized precursor then flows in a carrier gas, such as Ar, through heated lines and is injected onto a hot substrate through a shower head. All components of the precursor delivery system have been designed and developed at Intermagnetics.

There are several advantages to liquid-precursor delivery when compared with solid-precursor delivery. The key advantages are listed in Table I.

The composition of YBCO films prepared by the liquid-precursor delivery system during 12 deposition runs showed that the film composition was reproducible within $\pm 5\%$ of the target composition.

CRITICAL CURRENT MEASUREMENTS

It was reported previously that I_c values of 50 A were achieved in YBCO films grown by MOCVD on IBAD substrates at 77 K under self-field conditions [3]. High values of I_c were also maintained in the presence of a magnetic field. At 75 K, with the magnetic field perpendicular to the c-axis ($B \perp c$), J_c values of 570,000 and 240,000 A/cm² were achieved in fields of 1 and 5 T, respectively. With the field parallel to the c-axis ($B \parallel c$), J_c

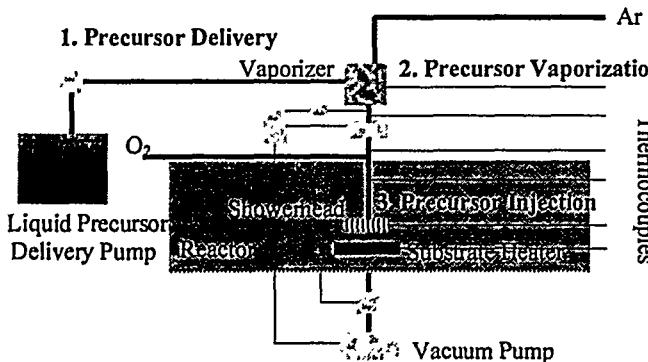


Fig. 1. Schematic diagram of the MOCVD system used at Intermagnetics.

TABLE I
COMPARISON OF SOLID- AND LIQUID-PRECURSOR DELIVERY SYSTEMS

Characteristic	Solid Precursor	Liquid Precursor
Technique	Precursors are individually sublimed separately from 3 ovens	Precursor is flash vaporized at a single location
Temperature Control	Difficult (film composition is sensitive to even 1°C change)	Single-point of control. Easier
Precursor Stability	Bad for long deposition periods	More stable in solution form
Precursor Vapor Pressure	Changes as precursor is depleted	Constant volume of precursor is vaporized. Vapor pressure is constant
Deposition Rate	Difficult to increase because of turbulence of carrier gas at high rates	Easily increased by increasing precursor flow rate.

values of 215,000 and 100,000 A/cm² were achieved in fields of 1 and 2.5 T, respectively. At 64 K, the J_c values were 1.2 MA/cm² and 770,000 A/cm² at 2 and 5 T, respectively, in the $B \perp c$ orientation. In the $B \parallel c$ orientation, the J_c values were 680,000, 520,000, and 300,000 A/cm² at 1, 2, and 5 T, respectively. Since that report was published, the I_c of YBCO films made by MOCVD on IBAD YSZ substrates has been doubled to almost 100 A, as shown in the current-voltage (I-V) curve in Fig. 2. The I_c of 97.5 A exhibited in Fig. 2 corresponds to a J_c value of 1.3 MA/cm² and translates to 130 A/cm width of film. These values compare very well with those achieved in YBCO films deposited on buffered-metal substrates by pulsed-laser deposition.

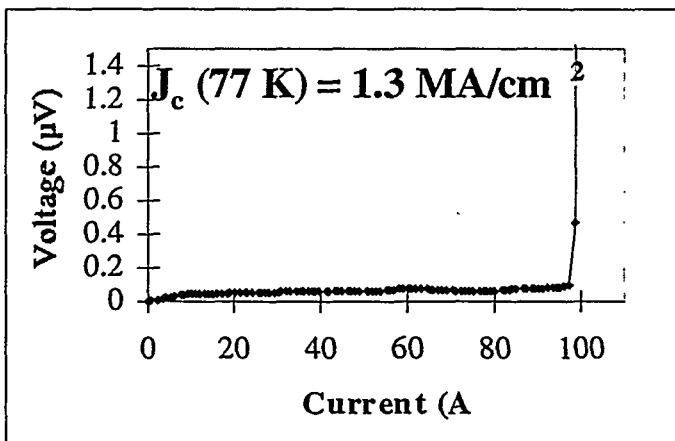


Fig. 2. I-V curve obtained from YBCO film deposited by MOCVD on IBAD substrate. The I_c is 97.5 A, which corresponds to 130 A/cm width of tape.

MICROSTRUCTURE ANALYSIS

The surface morphology of the films was examined by SEM. We found that the surface morphology of the films became rougher with increasing thickness in the range from 0.1 to 1 μm . A puzzling finding was that the rougher films almost always yielded a higher J_c than the smoother films. Figs. 3 and 4, respectively, show a relatively smooth film and a very rough film, both 1 μm thick. Contrary to expectations, the smooth film sustained an J_c of only 1 A, whereas the rough film exhibited an J_c of almost 100 A.

The cause of surface roughness in films of the same thickness, as seen in Figs. 3 and 4, could be that the rough films are usually off-stoichiometric. The off-stoichiometric compositions were deliberately chosen to achieve higher J_c .

The information obtained from the analysis of the surface morphology of the films does not provide complete details of the microstructure and its influence on the critical current. Therefore, steps have been taken to examine the cross-sectional microstructure of MOCVD-prepared YBCO

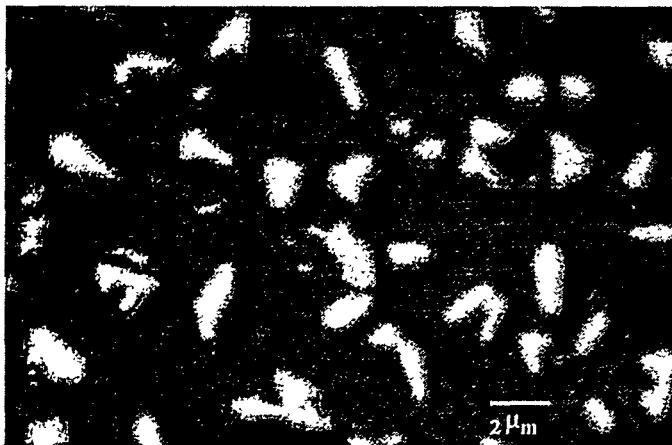


Fig. 3. Microstructure of a 1 μm YBCO film deposited by MOCVD on a IBAD substrate. In spite of the relatively smooth film surface, the film sustained an J_c of only 1.3 A.

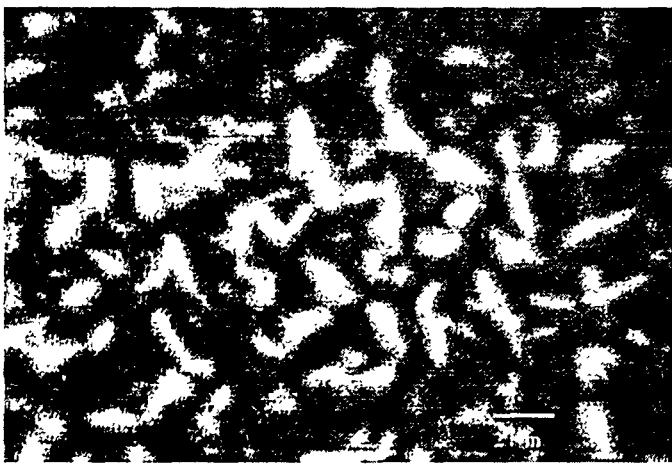


Fig. 4. Microstructure of a 1 μm YBCO film deposited by MOCVD on a IBAD substrate. In spite of the rough film surface, the film sustained an J_c of 97.5 A (data shown in Fig. 2).

films. Samples were fractured and their cross sections examined by SEM. Fig. 5 shows the microstructure of a YBCO film on an SrTiO_3 substrate; the film is dense and has a columnar structure. EDS spectra were obtained from various sections of the sample, and the YBCO and SrTiO_3 substrate regions were confirmed.

More detailed analyses will be performed and will include YBCO on metal substrates to increase our understanding of the relationship between microstructure and J_c .

CONCLUSIONS

YBCO films have been deposited by MOCVD on biaxially-textured YSZ buffer deposited by IBAD. A liquid-precursor delivery system was designed and constructed for use in the MOCVD facility. Through improvement to the MOCVD equipment and optimization of process conditions, J_c as high as 97.5 A (corresponding to a J_c of 1.3 MA/cm^2) was achieved at 77 K under self-field conditions. This J_c value corresponds to 130 A/cm-width of tape. Microstructure analysis of the tapes revealed a very rough surface morphology in the high- J_c films.

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Fig. 5. Fracture surface of a YBCO film on an SrTiO_3 substrate. A columnar grain growth is evident, and the film appears to be dense.