

Oxide precursor-based MOD processing of YBCO thin films

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Abstract—A low cost MOD processing using YBCO oxide powder as a starting precursor was employed for fabrication of YBCO thin films. YBCO oxide is advantageous over metal acetates or TFA salts which are popular starting precursors for conventional MOD-TFA process. YBCO thin films were prepared by oxide-precursor-based MOD process and annealing condition was optimized. The YBCO thin film annealed at 780°C shows no transport I_c and poor microstructure. However, the YBCO thin film annealed at higher temperature shows improvement in microstructure and current transport property. In order to improve critical current, YBCO thin film was prepared by double coating method. YBCO thin film prepared with double coating approach shows enhanced superconducting performance ($I_c > 100 \text{ A/cm-w}$).

1. INTRODUCTION

YBCO($\text{YBa}_2\text{Cu}_3\text{O}_{7.5}$)-based coated conductors (CC), produced by deposition of superconductor on biaxially textured flexible substrates are currently promising HTSC wire architecture for power electric application due to their high critical current density at liquid nitrogen temperatures ($J_c > 1 \text{ MA/cm}^2 @ 77\text{K}$). Many efforts are in progress to develop long HTSC wire with high current carrying capacity by coated conductor architecture [1-3]. Among them, MOD-TFA is one of the most promising processes for fabricating YBCO CC with high critical current. MOD-TFA is non-vacuum process and it produces YBCO thin films with relatively high J_c [4-6]. Since, however, conventional MOD-TFA processing requires relatively expensive metal acetates or trifluoroacetates as starting precursors, cost-effective precursors are required for economical processing.

In this study, YBCO oxide powders are employed as starting precursors for MOD processing and effect of annealing temperatures on microstructures and superconducting properties of YBCO thin films were investigated. In addition, it was shown that fabrication of YBCO thin film with high critical current could be realized by this oxide-precursor-based MOD process using double coating method.

2. EXPERIMENTALS

YBCO powders were employed as starting precursor for MOD processing of YBCO thin films. YBCO powders were dissolved in aqueous solution of TFA (trifluoroacetic

acid) and refluxed for a few hours and the mixed aqueous solution of YBCO powders and TFA was distilled to remove residual impurities such as acetic acid, water, etc. Distilled residue appeared to be blue gel and dried gel was diluted with methanol and filtered to produce final coating solution.

(100)-oriented LaAlO_3 single crystals were used as substrates for film growth. All substrates used in this study were cleaned with isopropanol and deionized water.

YBCO gel films were deposited by dip coating and dried on hot plates. Dried gel films were calcined at the optimized temperature and atmospheres. Annealing of calcined precursor films was performed in wet Ar/O_2 atmosphere to convert oxyfluoride to YBCO. Ag layer was deposited on YBCO surface by RF magnetron sputtering (KERI). Texture and microstructure of grown YBCO thin films are characterized with XRD, SEM, Raman spectroscopy, etc. Critical current was measured with conventional 4-probe method (77K, Self field).

3. RESULTS AND DISCUSSIONS

3.1. Effect of annealing temperatures

MOD precursor solution prepared with YBCO powders shows good adhesion to substrates. Fig. 1 presents of fully

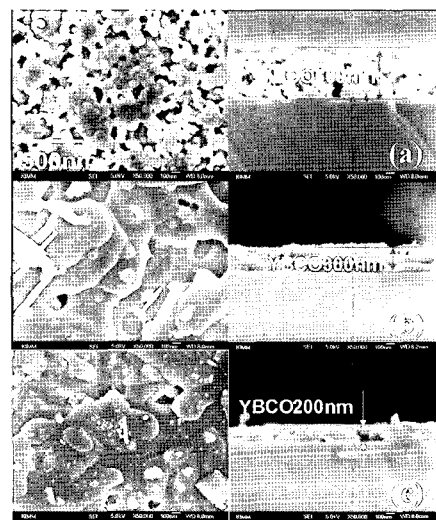


Fig. 1. Microstructures of YBCO thin films prepared with YBCO oxide-precursor-based MOD method annealed with different temperatures: (a) 780°C, (b) 800°C and (c) 810°C.

processed YBCO thin films annealed at various temperatures. Films were deposited on LAO substrates (10mmx10mm).

Microstructure of YBCO thin film annealed at 780°C contains a large number of pores and shows sparse distribution of grains. However, YBCO annealed at 810°C shows dense microstructures and contains only small number of pores. In addition, grain linkage of YBCO was improved by annealing at high temperatures. It was shown that microstructure of YBCO thin film was improved by annealing at higher temperature.

XRD result of YBCO thin films shows that all of YBCO thin films in this study prepared by oxide-precursor-based MOD process shows well-developed c-axis orientation as shown in Fig. 2.

An XRD pattern of YBCO thin film annealed at 780°C shows existence of non-superconducting secondary phases such as BaCuO₂ (Fig. 2(a)). Existence of 630cm⁻¹ peak in Raman spectrum of same specimen also indicates the existence of BaCuO₂ as shown in Fig. 3 [7].

(103) pole figure of YBCO thin film shows well-developed contours discretely spaced by 90° in phi angle indicating in-plane texture of YBCO thin film (Fig.4). Full width at half maximum of phi-scan ($\Delta\phi$) show improvement correlated with annealing at higher

Critical currents of fully processed YBCO films were measured by conventional 4-probe method. YBCO annealed at high temperature shows improved superconducting performance as shown in Fig. 5.

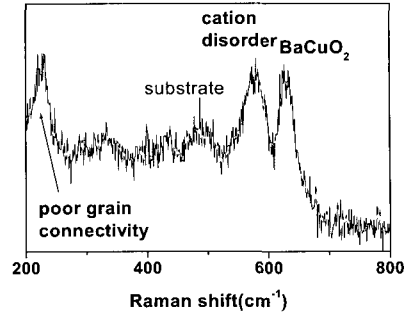


Fig. 3. Raman spectrum of YBCO thin film annealed at 780°C.

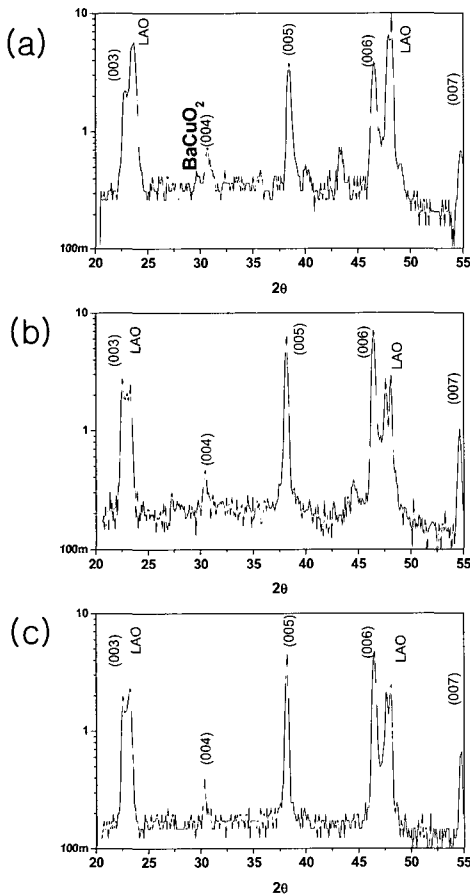


Fig. 2. 2θ-θ scan profiles of YBCO thin films annealed at different temperatures: (a) 780°C, (b) 800°C and (c) 810°C. temperature (Fig. 4(b)).

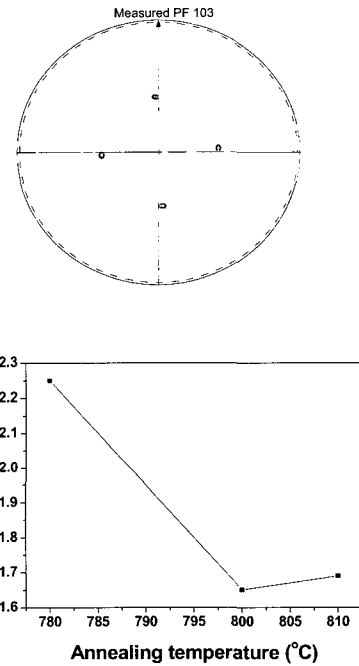


Fig. 4. (a) (103) pole figure and (b) in-plane orientation ($\Delta\phi$) of YBCO thin films.

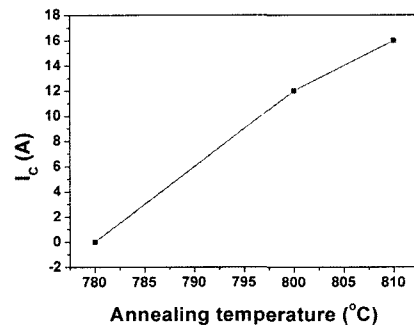


Fig. 5. Critical current of YBCO films as a function of annealing temperatures.

3.2. double coating

In order to improve critical current of YBCO thin films, multiple coating and calcinations method was employed. In this study, coating and calcinations process was repeated twice. Coating and calcination were repeated twice and calcined precursor film was annealed at 810°C. Size of specimen is 10mmx2mm. Fig. 6 shows microstructure of a fully processed YBCO thin film prepared by double coating process. Surface of a double-coated YBCO thin film shows dense microstructure and good grain.

In addition, the thickness of grown YBCO film was estimated to be 0.9 μ m and cross-sectional observation of YBCO thin film indicates dense and uniform microstructure (Fig. 7).

XRD characterization indicates trace of CuO phase, but well-developed c-axis orientation (Fig. 8)

Fig. 9 shows critical current of YBCO thin film prepared by multiple coating. Measured I_c value is about 105A/cm-w although real I_c in 2mm width is about 21A. The critical current density (J_c) was estimated to be about 1.2MA/cm². Hence, it was shown that specimen prepared by double coating improves current transport property of YBCO thin film.



Fig. 6. Microstructure of YBCO thin film prepared by double coating.



Fig. 7. Cross-sectional view of YBCO thin film prepared by multiple coating method.

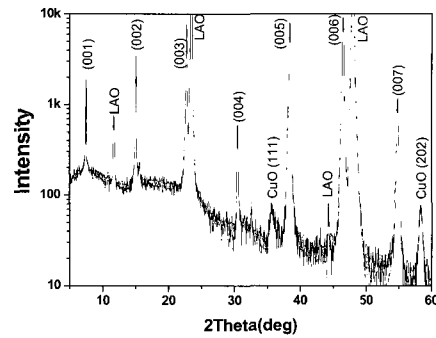


Fig. 8. 2 θ - θ scan of YBCO thin film prepared by multiple coating method.

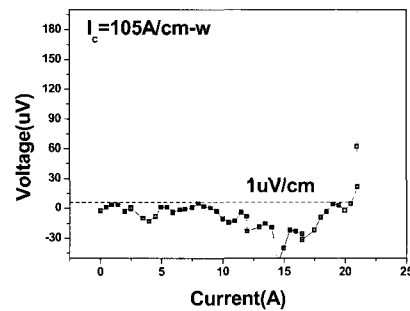


Fig. 9. Critical current of YBCO thin film prepared by multiple coating method.

4. SUMMARY

In summary, YBCO thin films were fabricated by low cost YBCO oxide precursor-based MOD process and effect of annealing condition was investigated. The YBCO thin film annealed at 780°C shows no transport I_c and poor microstructure. However, the YBCO thin films annealed at higher temperatures show improved microstructure and current transport properties. In this study, it was shown that double-coating approach is effective in improving critical current value. YBCO thin films prepared with multiple coating approach shows enhanced superconducting performance of $I_c = 105 \text{ A/cm-w}$ ($J_c = 1.2 \text{ MA/cm}^2$).

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