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Effect of humidity on microstructure and properties of YBCO films prepared by Electron Beam Coevaporation

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YBCO superconducting films were prepared by Electron Beam Coevaporation method. All the YBCO films were annealed at 760°C in humidity range of 2.3%–9.5%. Microstructure of the YBCO thin films was analyzed by means of X-ray diffraction (XRD) and scanning electron microscopy (SEM). Superconducting properties of the YBCO films were measured by electromagnetic induction method. XRD results showed that *c*-axis-oriented grains existed in the YBCO films. Morphologies of the YBCO films showed that all the films had a smooth and crack-free surface. YBCO films prepared at 7.3% humidity condition showed J_c value of 4.6 MA cm^{-2} at 77 K in self-field.

YBCO, superconducting films, coevaporation

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1 Introduction

YBCO films have been intensively studied owing to their excellent electrical property [1–3]. Compared with metal, YBCO thin films have more advantages in the application for microwave devices. It makes the devices smaller, lighter, and with higher quality factor and lower insertion loss.

YBCO films can be prepared by using various methods such as magnetron sputtering, vacuum evaporation, chemical vapor deposition (CVD), liquid phase epitaxy (LPE), pulsed laser deposition (PLD) and metal organic deposition (MOD) [4–10]. Among these methods, evaporation offers high uniformity over large area and high volume deposition rates, which are essential for a low cost production and widespread industrial use in future.

In the present work, we developed an electron beam coevaporation system to fabricate YBCO films [11,12]. The growth of 300 nm YBCO films was investigated at different temperatures [13]. The used conditions that lead to high J_c values for 300 nm thick YBCO films were not adequate for the growth of the thicker YBCO films in our system. We discovered a pre-heat treatment of the precursor film before the conversion process suppressed secondary phase formation, leading to the improvement of the homogeneity of the YBCO films.

2 Experimental details

Precursor films of stoichiometric cation composition $Y_1Ba_2Cu_3$ were deposited on (100) $LAlO_3$ (LAO) substrates by coevaporation of Y, BaF_2 and Cu sources. The Y metal and BaF_2 were deposited using 10-kW e-beam guns and an 8-kW e-beam gun was used to deposit Cu metal. The substrate temperature during deposition was 300°C and the oxygen background pressure was 1–5×10^{-3} Pa. Deposition rates for the three cation materials were measured by three identical Inficon quartz crystal monitors (QCM).

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The temperature profile for the conversion process is shown in Figure 1. Post deposition was performed in a quartz furnace. Typical conditions for the intermediate anneal were temperature, 400°C, duration, 0.5 h. Precursor films were converted at 760°C for 2 h. The oxygen partial pressure (p_{O_2}) was fixed at 100 mTorr. The total gas flow was fixed at 200 sccm using electronic mass flow controllers and was humidified by bubbling through a heated water bath. The dry oxygen treatment was carried out at 500°C for 0.5 h in the oxygen gas flow.

The YBCO films were studied by X-ray diffraction (XRD) to evaluate phase identification. Scanning electron microscope (SEM) of YBCO films was carried out by mean of a Hitachi S-4100 SEM equipped with energy dispersive spectroscopy (EDS) detector. Cation concentrations of precursor films were based on sensitivity factors derived from inductively coupled plasma (ICP) measurement. The critical current (J_c) measurement and T_c were carried out by four-probe method to evaluate superconducting properties.

3 Results and discussion

Figure 2 shows surface and cross-section SEM images of precursor films. Precursor thin films have stoichiometric compositions and homogeneous thicknesses. The EDS results indicate that big particles contain all the three cations, Y, Cu, and Ba. The surface coarsening may be due to the incomplete reaction of Y and Cu precursors with oxygen. The thickness of precursor films was 650 nm. From cross-section SEM images of precursor films, we have found that the films have columnar grain structure.

Figure 3 shows XRD patterns of the YBCO films annealed at 760°C in Ar mixed with O_2 at different humidities (2.3%–9.5%). The figure shows that the YBCO (00l) plane is parallel to the surface in all the films indicating well-developed *c*-axis orientation. Almost pure YBCO (00l) is observed in the films. YBCO films annealed at humidity of 7.3% have the strongest intensity of YBCO (005).

We investigated full width at half maximum (FWHM) of the (005) ω scan and (103) φ scan of the YBCO films. The results are shown in Figure 4. YBCO films annealed at humidity of 7.3% have the best in-plane and out-plane textures.

Figure 5 shows surface morphologies of the YBCO films annealed at 760°C in Ar mixed with O_2 at different humidities (2.3%–9.5%). It shows that all the films have a crack-free surface and consist of *c*-axis-oriented grains, i.e., the grains in which the *c*-axis of the lattice is normal to the substrate. On the other hand, the surface morphology changes with humidity. With humidity increasing (⩽7.3%), the film became denser. The YBCO films prepared in humidity of 7.3% were the densest in all the prepared films. There were some big particles in the YBCO films prepared in humidity of 7.3%, which were characterized by EDS to be YBCO. Lower humidity (2.3%) involves low conversion rates and inadequate HF release, which may cause incomplete reaction or disorder (such as *a*-axial-oriented grains and pores). On the contrary, when humidity is higher than 7.3%, high conversion rate of reaction (1) was obtained, which resulted in distorted growth front, i.e., when some *a*-axial-oriented grains nucleated in the film, there was no enough neighboring *c*-axial-oriented grains to fill the gap between *a*-axial nucleation centers. Therefore, pores are formed. Maybe there are very small *a*-axial-oriented grains in the YBCO films that is why we did not find *a*-axial orientation in XRD patterns.

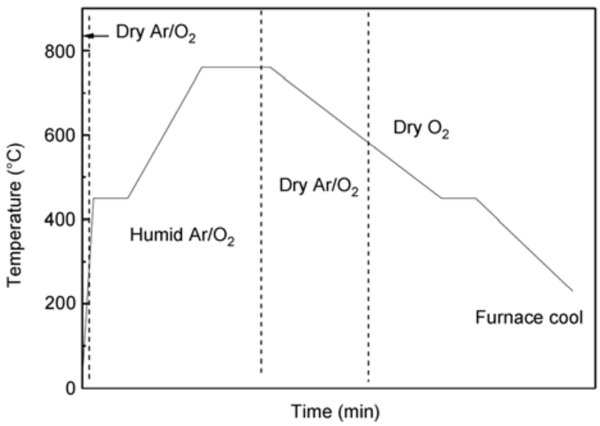

Figure 1 Typical temperature profile for the conversion process.

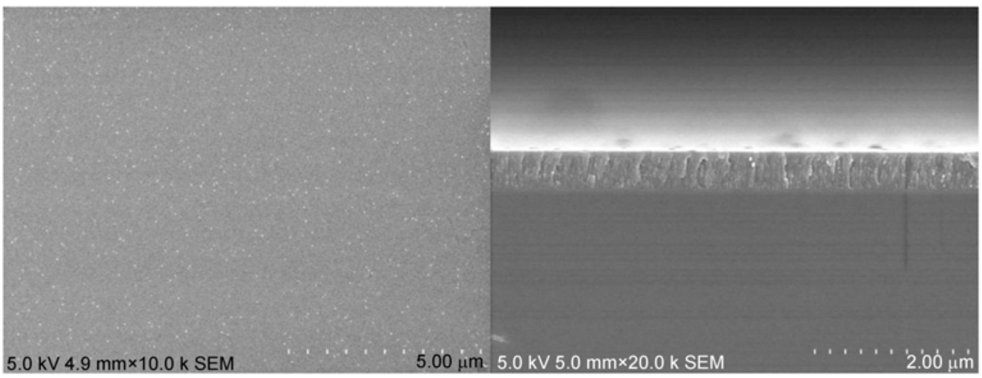

Figure 2 Surface and cross-section SEM images of precursor films.

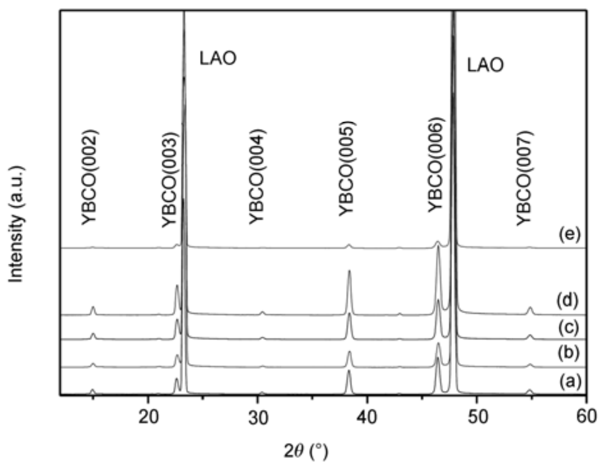

Figure 3 XRD patterns of the YBCO films annealed prepared in different humidities. (a) 2.3%; (b) 4.2%; (c) 5.6%; (d) 7.3%; (e) 9.5%.

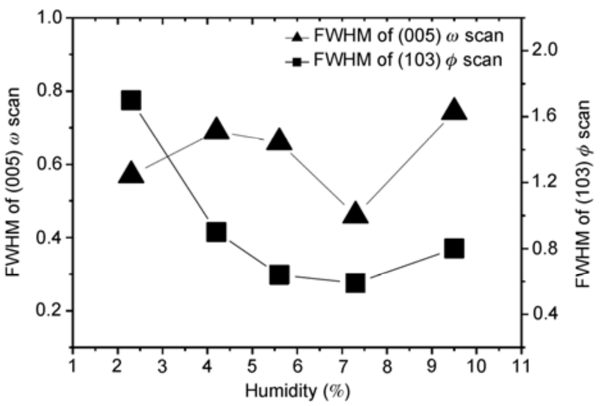

Figure 4 Full width at half maximum (FWHM) of the (005) ω scan and (103) ϕ scan of YBCO films.

$$Y+BaF_2+Cu+O_2+H_2O \rightarrow YBCO+HF \tag{1}$$

Figure 6 shows the J_c values of the YBCO films annealed at 760°C in Ar mixed with O_2 at different humidities (2.3%–9.5%). With increasing humidity in range of 2.3%–7.3%, the corresponding J_c value increased from 0.5 MA cm^{-2} to 4.6 MA cm^{-2} and afterwards decreased to 0.3 MA cm^{-2}. The trend of J_c values corresponds to XRD and SEM results. This means the better the texture of YBCO film is, the better superconductivity it provides.

4 Conclusion

We presented a promising approach for industrial scale-up using electron beam coevaporation of Y-BaF_2-Cu precursor thin films. Precursor thin films showed stoichiometric compositions and homogeneous thicknesses. We successfully prepared a series of YBCO films annealed at 760°C in Ar mixed with O_2 at different humidities (2.3%–9.5%). SEM study showed that all the YBCO films had crack-free

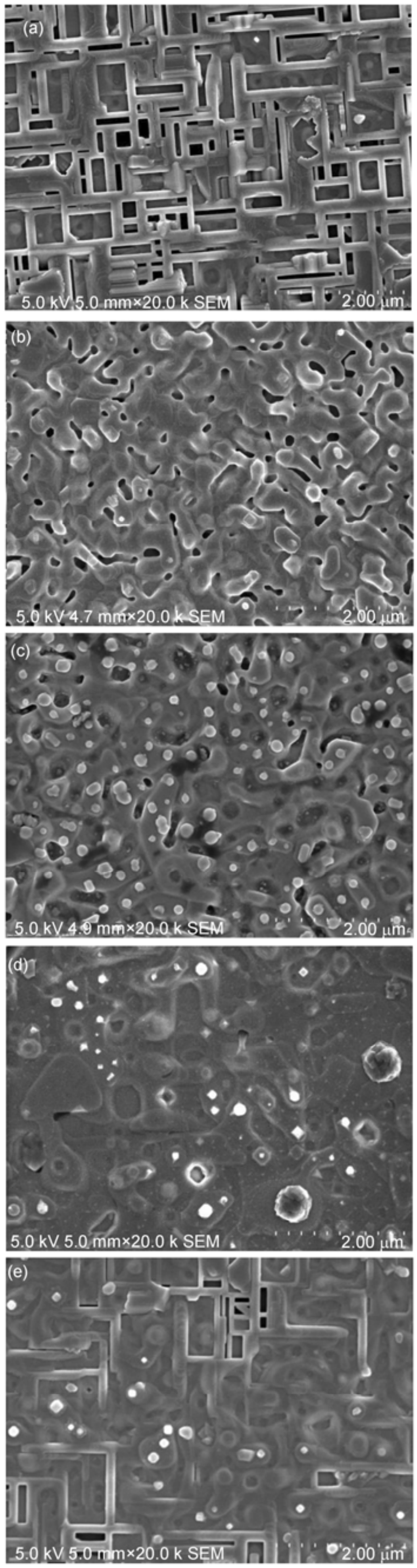

Figure 5 SEM micrographs of YBCO films prepared in different humidities. (a) 2.3%; (b) 4.2%; (c) 5.6%; (d) 7.3%; (e) 9.5%.

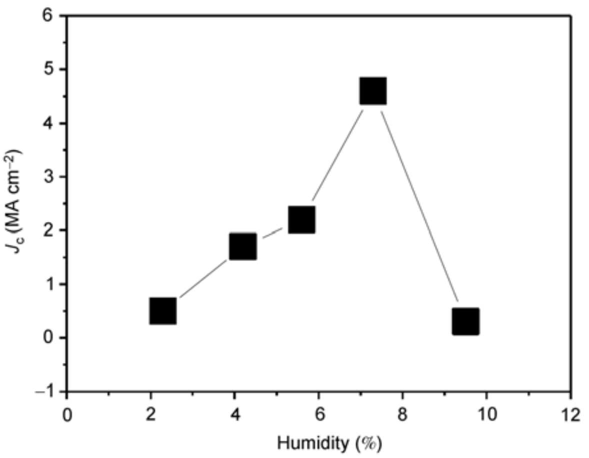

Figure 6 Dependence of critical current density (J_c) (77 K, 0 T) on humidity.

surface. We have found that the YBCO films prepared in humidity of 7.3% have the best texture and superconductivity.

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