



Ultrafine nanocrystal precursor induced J_c increase of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ films prepared using advanced low-fluorine solution



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ABSTRACT

High-performance $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) films were obtained on LaAlO_3 (LAO) substrate using an advanced low-fluorine solution with Y-TFA and fluorine-free Ba and Cu salts as precursors. Macropore-free precursor films were obtained using a rapid pyrolysis with the heating rate of $5^\circ\text{C}/\text{min}$ from 200°C to 310°C . In the precursor film, CuO and $\text{Ba}_{1-x}\text{Y}_x\text{F}_{2+x}$ (BYF) were formed as intermediate phases. Ultrafine nanocrystalline CuO particles were found embedded inside the crystalline BYF matrix, leading to the formation of high-quality YBCO film. The obtained single-coated YBCO films showed a better in-plane and out-of-plane texture, and a higher critical current density (over $8\text{ MA}/\text{cm}^2$ at 77 K , OT), compared with the YBCO films produced from precursor consisting of larger CuO particles.

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1. Introduction

$\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) coated conductors have attracted tremendous attention because it works in high magnetic fields at 77 K [1]. To fabricate YBCO coated conductors, several physical and chemical approaches have been developed. Regardless of what techniques are being used, the goal is to produce long-tape conductors with a high critical current (I_c) and/or high critical current density (J_c) [2]. Since the discovery of YBCO, the metalorganic deposition (MOD) has been extensively utilized to fabricate YBCO films and coated conductors due to its low-cost and easy scale-up [3,4]. Metalorganic deposition using trifluoroacetates (TFAs) as metal precursors in an organic solvent (TFA–MOD) is a well-established route for YBCO coatings due to its high repeatability of fabrication of YBCO film with J_c over $1\text{ MA}/\text{cm}^2$ [5–7].

In the MOD process, a solution is firstly prepared, and then the precursor films are obtained by pyrolysis of the solution derived gel films. After that, the pyrolyzed precursor films are converted to YBCO phase through a firing process. In the multiple processes involved in the MOD route, there are many factors influencing the superconductivity of final YBCO films, including the chemistry of the precursor solution, the physical and chemical properties of gel film and pyrolyzed film, and the parameters including the gas flow rate, water pressure, temperature, etc., during the pyrolysis and firing process. All of these bears great influence on the final YBCO properties [8–10]. From the viewpoint of materials science,

it is well known that the macro-properties of any material were depended on its internal microstructures (micro-properties). The superconductivity of YBCO film is dependent on its phase structure and texture, which can be tuned by the parameters including the gas flow rate, water pressure, temperature, etc., during the firing process. However, when these parameters are fixed, the properties of final YBCO film are straightforwardly influenced by the properties of the precursor film, and hence by the precursor solution. In the past decades, much work has been done to optimize the TFA–MOD routes, so as to prepare YBCO films with a higher J_c [11–15]. However, little work has been carried out to illustrate the relationship between the properties of precursor and the final YBCO films.

In this paper, we tried to optimize the pyrolysis process so as to obtain precursor film with superior properties. The precursor film was derived from our newly developed advanced low-fluorine solution [16]. Through an optimized pyrolysis process, precursor films with ultrafine nanocrystalline CuO inside the BYF matrix were obtained. We found that this precursor film contributed to the improvement of texture and superconductivity of the final film. YBCO films with J_c over $8\text{ MA}/\text{cm}^2$ were obtained on LAO substrate. We also found that the solution chemistry showed great influence on the properties of the pyrolyzed film, and hence the properties of the final YBCO films.

2. Experimental

In the present work, the precursor solution was prepared using fluorine-free Ba and Cu salts, and Y-TFA salt as precursors, as reported elsewhere [16]. Compared with our early developed low-fluorine solution, amine species were avoided and the fluorine content was further decreased in this solution. We called this solution

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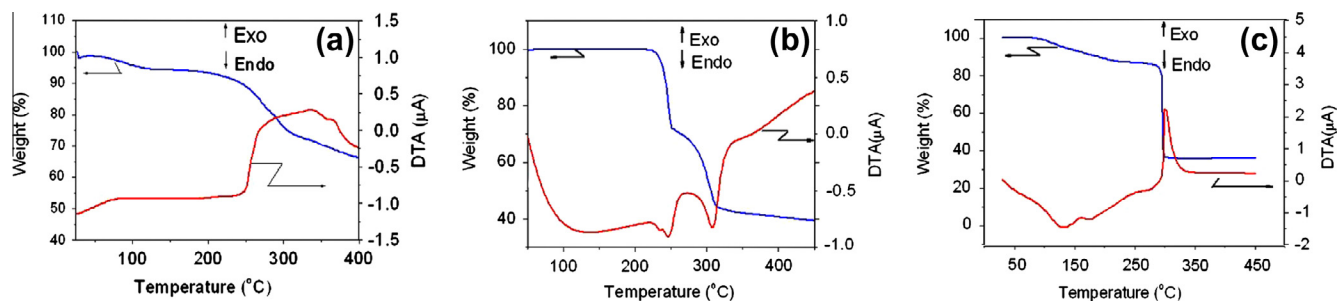


Fig. 1. TGA–DTA curves of different gels derived from (a) fluorine-free Ba-solution, (b) fluorine-free Cu-solution, and (c) fluorinated Y-solution.

as ‘advanced low-fluorine solution’. Briefly, the advanced low-fluorine solution was prepared by mixing the Y-TFA solution, and fluorine-free Ba and Cu solutions in a controlled stoichiometry of Y:Ba:Cu = 1:2:3.3. Another kind of TFA-containing solution (our early developed low-fluorine solution) as the reference solution was also prepared using Ba-TFA and fluorine-free Y and Cu salts as precursors [13]. Both of the solutions have the same metallic ion concentration of 1.2 mol/l. Using the advanced low-fluorine solution and the reference solution, the YBCO gel films were cast by dip-coating on the LAO substrates with size of 10 mm × 12 mm. The withdrawal speed was controlled at 3 mm/s. The coated YBCO gel films were put in a glass tube with diameter of 75 mm, and then pyrolyzed, fired and post-annealed to prepare YBCO superconducting films under controlled atmosphere. First, they were decomposed by increasing the temperature from room temperature to 450 °C in a humidified oxygen gas atmosphere. The heating rate was set at 10 °C/min from room temperature to 200 °C, and 15 °C/min from 310 °C to 450 °C. During 200–310 °C, different heating rates were used during the experiments. After being pyrolyzed at 450 °C for 10 min, the ‘precursor films’ were then heated to the maximum temperature of 800 °C (T_{max}) in a humidified O₂/N₂ gas flow for 70 min, and then in dry O₂/N₂ gas flow for another 20 min. During the firing process, the oxygen partial pressure was controlled at 1.3 vol.%, and the total gas flow was controlled at 2.5 l/min. Water partial pressure was controlled at 7.3 vol.% by setting the temperature of the water reservoir at 40 °C. After being fired at 800 °C, the samples were post-annealed at 450 °C in dry oxygen for 2 h. The thickness of single-coated films derived from both of solutions was about 200 nm.

The surface morphology of the precursor films pyrolyzed at 450 °C was imaged using a BX51 TRF Olympus microscope. The thermal decomposition of the precursor solution was investigated by thermogravimetric analysis (Netzsch STA 449 C). The 100 °C-dried gel was heated from room temperature to 500 °C at 10 °C/min under humidified O₂ atmosphere. 7000S-type X-ray diffractometer (XRD) with Cu K α radiation was applied to detect the orientation and the phase structure of the film. A Philips X-ray diffractometer (XRD) was used to investigate the texture of the YBCO films. The microstructure and texture of the film were observed using 3010F-type high-resolution transmission electron microscope (HRTEM). The film thickness was confirmed by the cross-section morphologies observed under the TEM. Additionally, the film was also characterized by a FEI Tecnai electron microscope, on which dark-field STEM were studied. To measure the J_c of the YBCO films, the as-prepared YBCO films were cut into pieces with a size of 2.2 mm × 2.5 mm. Multi-function Vibrating Sample Magnetometer (VersaLab-VSM, Quantum Design) was introduced to investigate their magnetization behaviors. The J_c related to the magnetic strength (H) was calculated from the M – H curves according to the Bean Model [17].

3. Results and discussion

To optimize the pyrolysis process, it is necessary to carefully investigate the decomposition of the gel derived from different

solutions. The TGA–DTA curves of the gel derived from the barium solution, copper solution and the yttrium solution were shown in Fig. 1. The TGA–DTA curves of fluorine-free Ba-salt (barium lactic acetate) and Cu-salt (copper methacrylic acetate) used in the advanced low-fluorine solution indicated that their decomposition occurred around 250 °C, and finished over 300 °C (Fig. 1a and b). The Y-TFA decomposed promptly at 305 °C (Fig. 1c). Based on this, we set a low heating rate (lower than 10 °C/min) from 200 to 310 °C, and a high heating rate of 15 °C/min from 310 to 450 °C. We found that a high heating rate such as 15 °C/min from 200 °C to 310 °C led to the macro-pores in the precursor film (Fig. 2a). Using a low heating rate less than 10 °C/min from 200 to 310 °C, the inhomogeneities, such as blistering and surface damage generated by fluorinated gas bubbling, could be effectively eliminated in the precursor film (Fig. 2b).

After the gel films were pyrolyzed, ‘precursor films’ or ‘pyrolyzed films’ were obtained. XRD indicated that the BYF and CuO were formed in all the precursor film pyrolyzed with different heating rate, as shown in Fig. 3. The intermediate phases of BYF and CuO in the precursor were same to those prepared using other TFA solution routes [9]. However, the structure and morphology of these intermediate phases maybe discrepant under different heating rate during 200–310 °C. The mean sizes of the CuO crystallites were calculated according to the Scherrer formula. The particle size of CuO in the advanced low-fluorine solution derived precursor films pyrolyzed with heating rate of 0.5 °C/min, 2.5 °C/min, 5 °C/min, and 10 °C/min, was estimated to be 12 nm, 9 nm, 8 nm, and 13 nm, respectively. Both a low heating rate of 0.5 °C/min and a high heating rate of 10 °C/min led to precursor film with larger CuO particles. It was reported that a long pyrolysis process could lead to the coarsening of the CuO particles in the film, and that a much higher heating rate also made the Cu-salts in gel film decompose promptly and sublimate dramatically, which could lead to the aggregation of CuO particles especially on the film surface [18]. Thus, an optimal heating rate of 2.5 or 5 °C/min during 200–310 °C resulted in the precursor film with finer CuO particles.

It should be noted that the solution chemistry also influences the size of the CuO particles. Using the same heating rate of 5 °C/

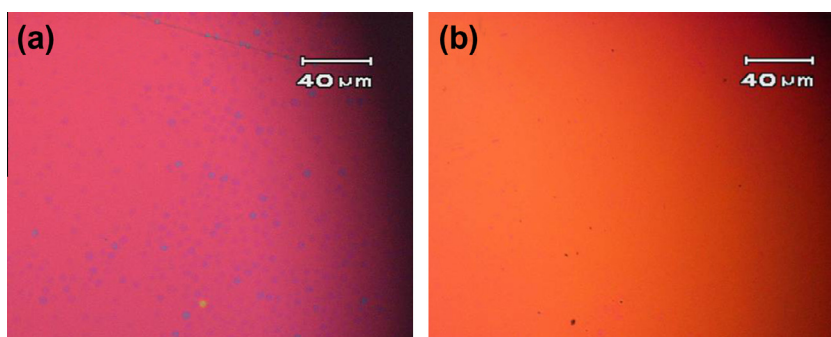


Fig. 2. Surface morphologies of precursor films pyrolyzed at 450 °C using a heating rate of (a) 15 °C/min from 200 °C to 310 °C; (b) 5 °C/min from 200 °C to 310 °C.

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