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Fabrication of TFA-MOD YBCO Films Using the $Y_2Ba_1Cu_1O_x$ and $Ba_3Cu_5O_8$ Powders

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Abstract

We fabricated YBCO film using a TFA-MOD method. In order to enhance the reaction kinetics and to control the formation of the second phases, $Y_2Ba_1Cu_1O_x$ and $Ba_3Cu_5O_8$ powders were used as precursors (the so called "211 process").
The films were calcined at 460 ℃ and then fired at 750 ℃-800 ℃ in a 12.1% humidified Ar-O_2 atmosphere. We found that the microstructure varied significantly with the firing temperature. The textures of all of the films were similar and mainly biaxial. For the film fired at 775 ℃, the critical current was obtained to be 39 A/cm-width (corresponding critical current density is 2.0 MA/cm^2).

Keywords : critical current, microstructure, pole-figure, TFA-MOD process, YBCO coated conductor

1. Introduction

To obtain excellent phase purity and a strong biaxial texture in the YBCO coated conductors (CCs), many thin film deposition processes have been devoted such as co-evaporation, PLD, MOCVD, and metal organic deposition (MOD), etc. Among them, the MOD process has received a great deal of attention, because of wide flexibility to coating materials and non-vacuum approach. Recently, the MOD method using trifluoroacetic acid (TFA-MOD) is considered to be one of the most promising processes since it exhibited the highest critical current density (J_c) of 3.6×10^6 A/cm^2. In the TFA-MOD method, the acetates of Y, Ba, and Cu dissolved in TFA are used as the precursor. It is known to be difficult to depress the formation of the BaF_2 phase, unless the processing conditions are precisely controlled, with the result that this method has low reproducibility. To this end, it would be desirable to develop a new synthetic method involving the use of a more reproducible and stable reaction by selecting other precursors and/or solutions.

In the fabrication of YBCO bulk superconductors, $Y_2Ba_1Cu_1O_X$ (Y211) and $Ba_3Cu_5O_8$ powders have been used as the precursors (the so called "211 process") in order to enhance the reaction kinetics and to control the formation of the second phases. We observed that those powders were hydrated in TFA and successfully synthesiszed the YBCO phase by the TFA-MOD method using this "211 process"[1]. However, the processing variables such as solution concentration, humidity, calcining and firing temperature were not optimized. In this study, YBCO film was fabricated by a TFA-MOD route with the "211 process" and evaluated the effects of the firing temperature on the phase formation, texture formability, and critical current (I_c) of the YBCO film.

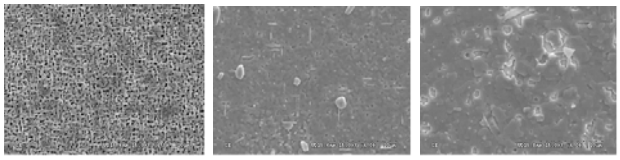
2. Experimental and Results

YBCO film was fabricated by the TFA-MOD method using the "211-process"[1] . The precursor powders were dissolved in TFA in a 1 : 2 molar ratio and refluxed at 80℃ for 6 h. The solutions were refined under decompression to yield a blue residue containing impurities. The residue was dissolved in methyl alcohol with a 2.2 M concentration. The gel film was deposited onto an (00*l*) $LaAlO_3$ (LAO) single crystal (0.4 cm × 1.4 cm) by the dip coating process. To evaluate the effect of the firing temperature on YBCO phase formation, the gel films were calcined at temperature of 460℃ in flowing 12.1% humidified oxygen and then fired at 750℃, 775℃, and 800℃ for 4 h in a 12.1% humidified Ar gas mixed with 1000 ppm oxygen. Subsequently, the fired film was heat treated at 450℃ for 10 h in dry oxygen to optimize the oxygen content in YBCO.

The microstructures were observed by scanning electron microscopy (SEM, XL-30, ESEM-FEG). The texture of the films was measured by four incomplete pole figures. The critical current (I_c) was also measured by a standard four-probe method with a 1 μV/cm criterion at the temperature of liquid nitrogen (77 K) in a zero field.

In order to evaluate the effect of the firing temperature, the calcined films were fired at 750℃, 775℃, and 800℃

for 4 h in 12.1% humidity. Fig. 1 shows the highly magnified (× 4,000) SEM images of corresponding YBCO films. It can be observed that all of the films had a crack-free surface, while the morphology was significantly varied with the firing temperature. For the films fired at 750℃, the grains were very fine and pores were frequently observed. As the temperature increased, the film became denser and the grains grew further. At the temperature of 800℃, the surface became irregular and second phase particles formed on the films. These particles were found by EDX to consist of Ba and Cu rich phases.



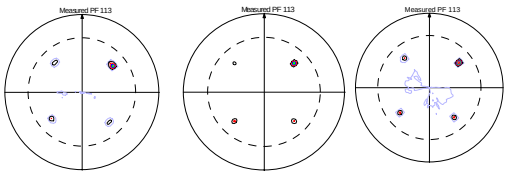
(a) (b) (c)

Fig. 1. SEM micrographs of the films fired at (a) 750℃, (b) 775℃, and (c) 800℃

It is also to be noted that the grain orientation with respect to the substrate depended on the firing temperature. For the films fired at 750℃, there were needle-shaped grains which appeared to be a-axis grain. The amount of this a-axis grain decreased as the firing temperature increased to 775℃, and it then disappeared at 800℃, suggesting that the grains mainly consisted of c-axis grain.

Fig. 2 shows the (113) pole-figures of the films fired at 750-800℃. The textures of all of the films were similar and mainly biaxial. For the films fired at 750℃, the major texture was biaxial and other minor components were present, as shown in Fig. 2 (a). Based on the SEM analysis shown in Fig. 1, it would appear that the minor texture component partly resulted from the presence of a-axis. For the films fired at 775℃, a sharp and strong biaxial texture formed. As the firing temperature was increased to 800℃, the minor texture components reappeared. Fig. 3 shows the variations of the I_c for the films with the firing temperature. It can be seen that the I_c significantly improved as the firing temperature increased from 750℃ and reached peak value at 775℃ and then decreased slightly as the firing temperature increased further. The highest I_c of 39 A/cm-width (the corresponding J_c is about 2.0 MA/cm^2) at 775℃ is probably attributed to such factors as the enhanced phase purity and the texture, and the moderate film density and grain size.

In this study, we fabricated YBCO films by a new TFA-MOD method which involved dissolving Y211 and $Ba_3Cu_5O_8$ powders in TFA. We found that a sharp and strong biaxial texture was formed, resulting in an I_c of 39 A/cm-width.



(a) (b) (c)

Fig. 2. (113) pole-figures of the films fired at 750℃, 775℃ and 800℃

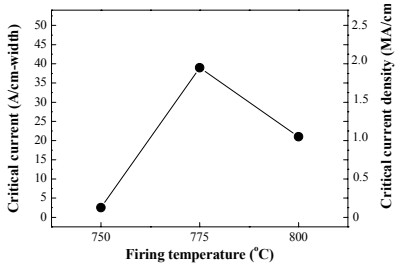


Fig. 3. Dependence of the critical current on the firing temperatures.

3. Summary

We successfully fabricated YBCO films using the "211 process" approach to the TFA-MOD method and investigated the effects of the firing temperatures on the phase formation, texture evolution, and critical current. We observed that the microstructure varied significantly with the firing temperature. As the firing temperature increased, the grains grew further and the film became denser. The degree of texture and phase purity also varied with the firing temperature. The highest I_c of 39 A/cm-width (the corresponding J_c is 2.0 MA/cm^2) was obtained for the film fired at 775℃.

4. References

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