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The Improvement of MTG Process for Preparation of YBCO superconductor

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Abstract
In the YBCO matrix, 10wt.% Y_2BaCuO_5 (211 phase) was added, the final 211 content of YBCO made by MTG process could reach about 20wt.% which was the optimum value for the critical current density. And also 10wt.% Ag was added in the matrix of YBCO, which was nearly about the saturate solubility of silver in textured YBCO. SmBaCuO crystal was grown by the melted-condensed process.

A $5\times5\times2mm^3$ single crystal of SmBaCuO was used to be the seed in the preparation of YBCO. It was proved the orientation of YBCO was the same as the orientation of the SmBaCuO seed. The oxygen absorption of bulk oriented YBCO was studied and the heat treatment of oxygen absorption would be in flowing oxygen, at 400℃ for about 24 hours. The magnetic hysteresis loops were measured by Vibrating Sample Magnetometer and the J_c was calculated by means of Bean's model.

Key Words : 211 phase, Silver, SmBaCuO seed, MTG, YBCO

1. Introduction

After the Melted Textured Growth proposed by S. Jin[1],some people have tried other improved methods[2,3,4,5]. The critical current density of the small textured YBCO sample has reached the application level [6,7]. The practical application needs large bulk YBCO with high J_c at liquid nitrogen temperature,but the large bulk YBCO do not have satisfactory flux pinning centers, and there are some microcracks and some pores in the bulk YBCO.

M.murakami showed that the dispersed fine Y_2BaCuO_5 (211 phase) particles can introduce the flux pinning centers in YBCO superconductor[6]. In the previous work[8] of the author it is known that the relation between 211 contents (W_{211} wt.%) of textured YBCO made by MTG and the numerous 211 contents (X_{211} wt.%) obeys equation $W_{211}=8.5\%+0.91X_{211}$.

If the numerous 211 contents (added in the YBCO matrix) $X_{211}=0$, the 211 contents of the textured YBCO will be 8.5%. In reference[8], it is also known that the optimum value of 211 content in the textured YBCO is 20wt.%, therefore if 12.6wt.% of 211 is added in the YBCO matrix, the 211 content in textured YBCO will be about 20wt.%. When 211 content is less than 20wt.%, J_c increases with increasing 211 content, J_c will reach the maximum at the point of 20wt.% of 211 content, when 211 content is larger then 20wt.%, J_c will decline with the increasing 211 content. According to above equation, if 12.6 wt.% of 211 content is added in the YBCO matrix, the 211 content in the textured YBCO will be about 20wt.%. In the previous work[9],it is shown that the melting point of YBCO decreases with increasing Ag content in the YBCO matrix. The melting points of YBCO with different Ag contents are used in the designing of MTG program for textured YBCO preparation.

In this paper, the solubility of Ag in textured YBCO was examined, which could be used to determine the silver content in the textured YBCO, and $SmBa_2Cu_3O_x$ crystals was prepared by the melted-condensed process, which was used as seeds to introduce the YBCO growth. The heat treatment for oxygen absorption of bulk YBCO was studied.

The above factors (the 211 content, the melting point of YBCO with different Ag contents, the solubility of Ag in textured YBCO, SmBaCuO seed and oxygen absorption conduction) were comprehensively considered to improve the MTG process for the preparation of bulk YBCO superconductors.

2. Experiment

2.1 The solubility of Ag in condensed YBCO

The added Ag contents in sintering YBCO and the analyzed Ag contents in condensed YBCO were shown in table 1. The analyzed Ag contents in condensed YBCO accord with the added Ag contents, if the added Ag contents is less than 14wt.%. The analyzed Ag contents are stable at 15wt.% when added Ag contents are larger than 16wt.%, it means that Ag content in YBCO is saturated, or the solubility of Ag in condensed YBCO is 15wt.%.

Table 1, Ag contents in the condensed YBCO

No.Samples	A4	A6	A8	A10	A12	A14	A16	A18
Added Ag contents	4.0	6.0	8.0	10.0	12.0	14.0	16.0	18.0
Analyzed Ag contents	4.3	6.2	8.1	9.2	12.1	14.2	15.1	15.2

2.2 Growth of $SmBa_2Cu_3O_x$ crystals

The melting point of SmBaCuO is 50℃ higher than that of YBCO. Therefore the single crystal of SmBaCuO can be used as seed crystals for preparation of YBCO.

The SmBaCuO crystal was growth by melting-condensed method. The $5\times5\times2$ mm^3 single crystal were cleft from the grown crystal.

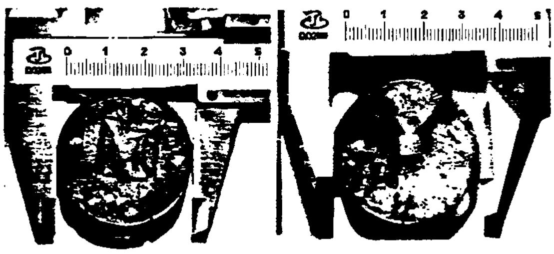
2.3 Preparation of textured YBCO by MTG method

MTG process for YBCO was taken place in a Ni-Cr resistance wire furnace with a constant temperature zone of 60mm,which was controlled by a Pt-PtRh thermocouple. The mixture of YBCO(80wt.%), Y_2BaCuO_5(10wt.%) and Ag (10wt.%) was placed on the high quality Alumina substrate. The sample was heated to 1080℃ in 2h, held at 1080℃ for 1h, reduced to 1030℃ in 20 min., and then reduced to 980℃ at the rate of 1～2℃ per hour, and then reduced to 400℃ at 100℃ per hour, The oxygen absorption was taken place at 400℃ under O_2 environment for about 24 hours.

3. Results and Discussions

3.1 The process of YBCO growth around SmBaCuO seed

The seed of SmBaCuO was put on the surface of melted YBCO at 1030℃～1050℃, at which YBCO stayed in melted state and SmBaCuO seed stayed in solid state. When the temperature of the system reached about 1010℃, the melting point of YBCO, YBCO start to crystalize around the SmBaCuO seed at the same orientation as SmBaCuO seed(see Fig.1). Fig.1(a) is the middle way of the YBCO growth process, Fig.1(b) is the final of YBCO growth.



(a) (b)

Fig.1 Growth process of YBCO crystal around SmBaCuO seed.
(a) The middle way of the growth process (b) The final of process

3.2 The orientation of YBCO made by MTG

In the test the oriented YBCO was introduced by SmBaCuO seed from YBCO melt. Therefore the position of SmBaCuO seed is quite important for the orientation of YBCO. The a-b plane (001) of the SmBaCuO seed which was carefully cleft from the bulk crystal should be paralleled with the surface of the YBCO melt. Fig 2(a) is the X-ray diffraction pattern of SmBaCuO seed, it is perfectly C-axis orientation. When the seed was used to introduce the YBCO growth, the orientation of YBCO was tremendously in agreement with that of SmBaCuO seed, which was proved by the comparision of X-ray difftractions of oriented YBCO made by MTG, see Fig.2(b), and of SmBaCuO seed, see Fig.2(a).

3.3 Study of Oxygen absorption of oriented bulk YBCO

The oxygen absorption of the oriented bulk YBCO has some different behavior due to its large volume and high density, in which oxygen diffusion is more difficult than that in small YBCO sample[10]. In the test, a 9.76g oriented YBCO sample was used under O_2 flow, the temperatures for oxygen absorption are 380℃, 400℃, 450℃, 500℃ and 550℃ respectively. A electronic balance with 10^{-4}g accuracy was used to record the weight change in oxygen absorption process. 400℃ is the best temperature for oxygen absorption among the results of test temperatures shown in Fig.3. It seems that after 1300 minutes(～22 hours), the oxygen absorbed in YBCO is still unsaturated. Therefore more than 50 hours should be used for oxygen absorption of oriented YBCO.

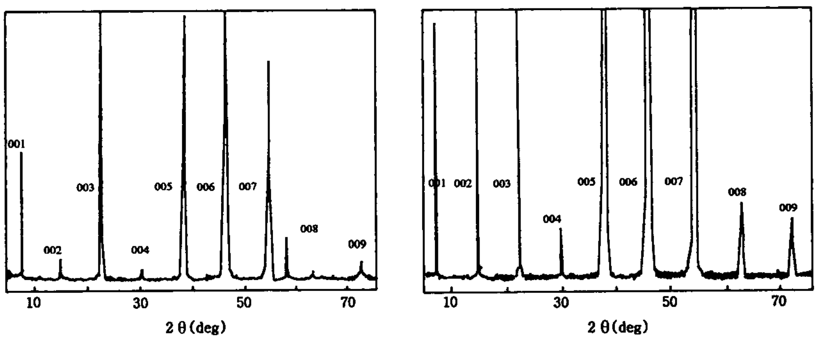


Fig.2 X-ray diffraction patterns of (a) SmBaCuO seed and (b) oriented YBCO

3.4 Critical current density of the oriented YBCO

The hysteresis loops of two samples, $8.34\times4.00\times1.62mm^3$ and $9.76\times4.44\times2.16mm^3$ after heat treatment at 400℃, under O_2, for 24hours, were measured by a Vibrating Sample Magnetometer (see Fig4). According to Bean's model, $J_c = 20\triangle Md^{-1}$ was used to calculate the critical current density(J_c).

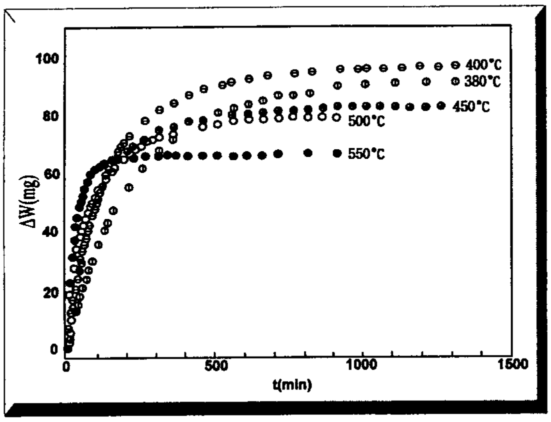


Fig.3 The weight changes in oxygen obsorption of oriented YBCO at various temperatures

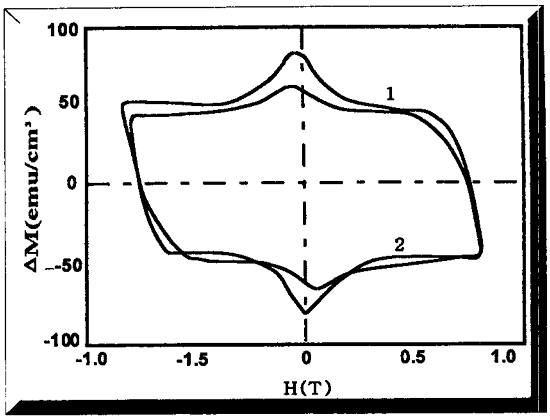


Fig.4 Hysteresis loops of oriented YBCO after heat treatment at 400℃ under O_2 flow and for 24 hours

From the hysteresis loops of oriented YBCO after heat treatment at 400℃ under O_2 flow and for 24hours, J_c of both samples were calculated 7×10^4A · cm^{-2},(0.01T, 77K). The magnetic levitation force of a oriented YBCO sample, 21mm in diameter, 10mm in height, is about 4.6N · cm^{-2} in liquid nitrogen.

4. Conclusions

4.1 The relation between added 211 contents(X_{211}) and calculated 211 contents (W_{211}) in textured YBCO is W_{211} = 8.5% + 0.91X_{211}. When 12.6wt.% 211 was added, the 211 content in textured YBCO will be approximately 20wt.% which is the optimum value in YBCO for the flux pinning center.

4.2 The solubility of Ag in condensed YBCO is about 15wt.%. The melting point of YBCO with different Ag content decreases with increasing Ag content.

4.3 The melting point SmBaCuO is 50℃ higher than that of YBCO and they have the same crystal structure, therefore SmBaCuO crystals can be used as seeds to introduce the growth of YBCO.

4.4 The optimum factors of heat treatment for oxygen absorption of bulk condensed YBCO are under O_2 flow at 400℃ for 50 hours.

4.5 The critical current density of a typical sample of oriented YBCO, which included 20wt.% of 211 phase and 10wt.% of Ag, is 7×10^4A · cm^{-2} (77K, 0.01T).

Acknowledgments
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