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Fine YBaCuO Powder Prepared by Sol-gel Method

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Abstract

In order to get proper original suspension for the electrophoresis process of making YBaCuO films, YBaCuO superconductor powder was prepared with the Sol-gel method. The composition of the powder was analyzed by X-ray diffraction, which was identified as $YBa_2Cu_3O_{7-x}$(Y123) phase. The form of YBaCuO single particle was showed to be spherical by scanning electronic microscope and its size was among 0.2-1 ㎛. The critical transition temperature (T_c) and the critical current density (J_c) were measured with the four-probe method. The T_c was about 91 K, and the J_c was 5-30 A/㎠.

Key Words : Sol-gel method, $YBa_2Cu_3O_{7-x}$ powder, Superconductivities

1. introduction

Scientists have tried many methods to gain YBaCuO films on metallic substrates with the aim of fulfilling the application of YBaCuO superconductor tape or in industry. Electrophoresis is one kind of technique that is not only simple but economic to get fine films on large area substrate [1].

During the electrophoresis process the original powder is required to be rather tiny particles. If the superconductor particles in the solvent are too large, on the one hand they will sediment quickly, and on the other the deposited films after sintering tend to crack which will lead to the decreasing their critical transport currents. Since well-distributed and compact films show better properties, we should try to make fine superconductor powder as original material in the electrophoresis.

There are two kinds of methods to prepare YBaCuO superconductor powder i.e. dry method and wet method. The dry method is also named solid phase reaction method and is commonly used [2]. The process begins with mixing three kinds of compound Y_2O_3, $BaCO_3$ and CuO, followed with repeatedly grinding and sintering. But with this kind of method the sizes of YBaCuO particles are more than 1um and distribute in a large scale. Also they are easily contaminated during grinding. The wet method suggests that the preparation is going in the liquid phase. Concretely it includes many kinds of method such as co-deposition, Sol-gel, sp raying and drying, nitrate resolving [3-5,]. Compared with the dry method, the size of ultimate powder of wet method is much smaller. However there are many aspects that need to be improved. For example, the process of co-deposition is much complicated and it is difficult to maintain the exact metal atom ratio of 1:2:3 while good sprayer is needed in the spraying method and a proper vertical height of liquid transportation tube should be chosen in case $Ba(NO_3)_2$ which is hard to dissolve would deposit and cause heterogeneous composition. It was proved that the YBaCuO samples made by citric method after the sintering have good toughness [6]. Therefore we prepared YBaCuO superconductor powder with Sol-gel method using citric acid solution matrix. This kind of Sol-gel method has the advantages of short reaction period and low sintering temperature. It

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is valuable reference in the process of preparing practical YBaCuO superconductor material.

2. Experiment

Y_2O_3, $Ba(NO_3)_2$ and CuO (all with the purity of A. R.) powders were weighed respectively according to the atom ratio of Y:Ba:Cu=1:2:3. Nitrate solutions of Y, Ba metal elements were prepared, which were together with $Ba(NO_3)_2$ added into the excess citric acid solution. The value of PH was adjusted by dropping NH_3H_2O. By the time $Ba(NO_3)_2$ would be completely dissolved and the citric solution was deep blue. Then the solution was put in muffle furnace and heated to get rid of the solvent. During this period there existed the transition from transparent liquid to gel. With the color of the gel growing deeper and deeper, it would turn to be black netted viscose. Finally it was burned into ashes by the heat. The final ashes were grinded into powder and sintered at the temperature of 880℃ with flowing O_2. The ultimate black powder was YBaCuO superconductor powder.

3. Results and discussion

3.1 The analysis of the composition of YBa-CuO powder

The ashes (1), sintered powder at 880℃ in oxygen atmosphere (2) and twice-sintered powder at 900℃ in air (3) were respectively analyzed by X-ray diffraction (showed as figure 1,2,3). As figure 1 showed, the composition of the ashes was very complex. They were just the middle production of the preparation. It meant that the burning of gel at low temperature (300℃) had just finished the decomposition and oxidation of citric acid complex compound. Because of the complexity and uncertain of organic reaction, the composition of ashes was not clear.

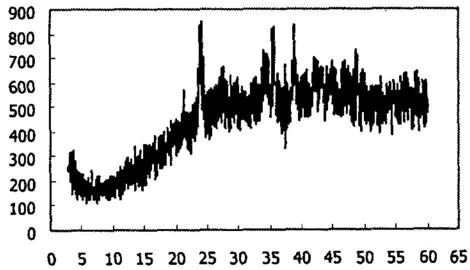

Fig. 1. The X-ray diffraction of the ashes at 200℃.

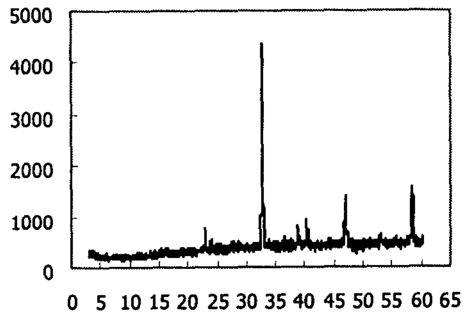

Fig. 2. The X-ray diffraction of YBaCuO powder at 880℃ in oxygen atmosphere.

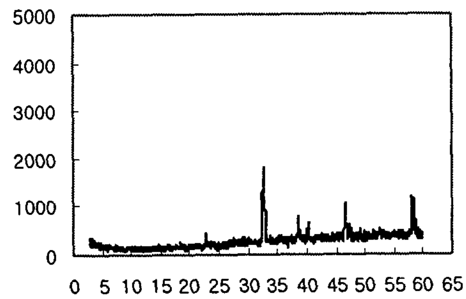

Fig. 3. The X-ray diffraction of YBaCuO powder at 900℃ in air.

Because the longer the sintering time was and the higher the sintering temperature was, the more completely the solid reaction went. So we dealt sample 3 with higher temperature and longer time in comparison with sample 2. From figure 2 and figure 3 it seemed that sample 3 contained less superconducting phase despite the special dealing with it. While sample 2 dealt in oxygen atmosphere had less foreign substance. It was because flowing oxygen can make carbon in the powder oxidized completely and take the gas production away. Thus the final composition of sample 2 was nearly pure Y123.

3.2 The size of YBaCuO powder

The form of YBaCuO powder was observed under scanning electronic microscope. As the picture showed, the particles were spherical and their sizes mainly distributed among 0.2-1㎛. It was hard for dry method to get such tiny particles. For with this kind of method the powder was mechanically grinded and the general size was among a large scale of 1-45 ㎛. It was too big as original powder for electrophoresis.

In the Sol-gel method the size of YBaCuO powder is affected by the addition amount of citric acid [7]. When the gel burns there exists oxidize-reduce reaction. The nitrate acts as oxidant while citric acid acts as reducing agent. If citric acid is excessively added, the oxidization would not finish completely. And less heat was given off which inhibits the agglomeration of product particles. Thus the ashes consist of smaller particles. Generally the amount of citric acid was added by the ratio $n_{citric}/n_{metal}=2\sim3$.

3.3 The superconductivity of YBaCuO powder

The samples were pressed YBaCuO strips (6 ㎜, 60㎜, 1㎜). After sintering at 880℃ for 3 hours they were dealt with oxygen annealing at 400℃ for 24 hours. The critical current density J_c and the critical transition temperature T_c of the samples were measured with four-probe method. The results were listed in table 1. That the J_c values were a little low may be caused by numerous weak links between tiny particles, which decrease the transport current through the samples.

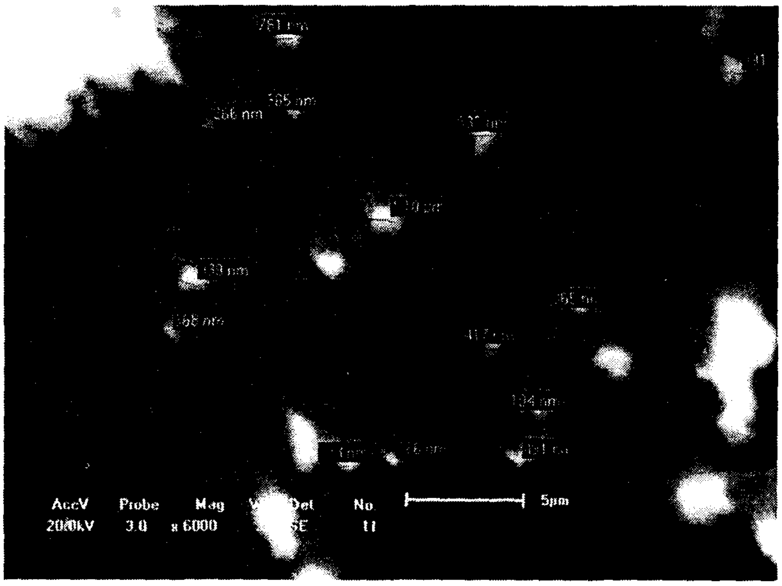

Fig. 4. SEM picture of YBaCuO superconductor powder.

4. Conclusion

YBaCuO superconductor powder was prepared with the Sol-gel method. Rather pure Y123 was gained. The size of Y123 powder was among 0.2-1 ㎛. At liquid nitrogen temperature the critical transition temperature (T_c) was about 91K, and the critical current density (J_c) was 5-30 A/㎠. The Sol-gel method is simple to operate, and it lasts a short time. It also lowers the sintering temperature of YBaCuO about 10 0℃.

Table 1. The superconductivity of sintered YBa-CuO strips.

samples	T_c(K)	J_c(A/㎠)
6#	89	0.26
9#		5.11
10#		28.96
12#	91	11.93
13#	93	5.95

Acknowledgements

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