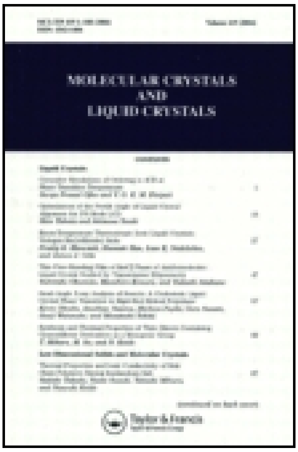

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FABRICATION OF Bi-Sr-Ca-Cu-O SUPERCONDUCTOR FROM NITRATE SOLUTIONS

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Abstract The Bi-based superconductor was fabricated employing the nitrate solution method. The phase development and super conducting property were examined by changing the composition and heat-treatment conditions. The optimal nominal composition was found to be Bi/Sr/Ca/Cu=2/2/2/3.5 with Tc(0)=54.5K.

INTRODUCTION

Since the Bi-Sr-Ca-Cu-O superconductor with high Tc was discovered by Maeda et al.[1], a number of studies on its fabrication, characterization and properties have been reported[2-6]. It should be notified that the properties of the Bi-Sr-Ca-Cu-O system is markedly sensitive to the preparation conditions. The homogeneity of the sample is one of the most important factor to dominate the superconducting properties. However, it is largely difficult to obtain homogeneous composition and structure by the solid state reaction mainly due to kinetic problems. In contrast, the powder prepared from the solution method is thought to be finer and more homogeneous than the solid state reaction[7-8]. In this study, fabrication from nitrate solutions was examined in order to obtain the Bi-Sr-Ca-Cu-O system with homogeneous texture and composition, which may lead to improvement of superconducting properties.

EXPERIMENTAL

The starting materials Bi_2O_3, $SrCO_3$, $CaCO_3$ and CuO were mixed to give the nominal compositions of

Bi:Sr:Ca:Cu=2:2:1:2, 2:2:2:3, 2:2:2.5:3.5, 2:2:2:4 while the total amount was determined to give 10 mmol of the superconducting phase. The mixture was dissolved in ca 150 ml of diluted nitric acid at ca.70°C and stirred for 30 min. Subsequently, 50 mmol of citric acid and 22.5 mmol of ethylene glycol were added to the solution and kept at 80 °C. The treatment was continued to release NO_x until it became sol. After gelation by heat-treatment, the resultant sample was crushed. At the next stage, it was calcined at 800°C for 5 hours in a stream of air . It was crushed again and employed for DTA and TG analyses. The sintering temperature was determined to be below the melting temperature by 5°C based on the DTA and TG results. The powder was pressed to make pellets, followed by firing at prescribed temperatures. For a part of the samples, calcination was conducted twice in order to improve the homogeneity of the sample composition. The samples employed in this study are summarized in Table I together with the firing conditions.

Table I Samples employed in this study

Sample No.	Composition Bi:Sr:Ca:Cu	Calcination temp. (°C)	Re-calcination temp. (°C)	Sintering temp. (°C)
A-1	2:2:1:2	800	---	850
A-2	2:2:1:2	800	835	853
B-1	2:2:2:3	800	---	862
B-2	2:2:2:3	800	838	862
C-1	2:2:2:3.5	800	---	860
C-2	2:2:2:3.5	800	838	857
D-1	2:2:2:4	800	---	860
D-2	2:2:2:4	800	835	857

The samples at various stages were examined by X-ray diffraction and scanning electron microscope (SEM). After the electrodes were attached to the sample by evaporating gold, they were connected to copper lead wires with a silver paste. The resistivity was measured employing the d.c. four probe method for the samples placed in a cryostat. The current level was maintained at

50 mA and the voltage drop was determined by averaging the values measured the forward and reverse directions. The temperature was measured using an Au(Fe)-Chromel thermocouple.

RESUTS AND DISCUSSION

PHASE DEVELOPMENT

The sequential phase development for the prepared samples was similar each other, while the quantity of the

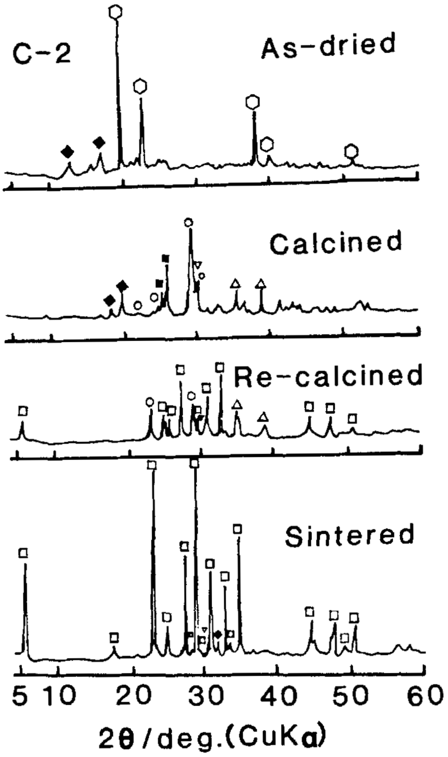

FIGURE 1 X-ray diffraction patterns for the samples at various stages. $Bi_2Sr_2CaCu_2O_y$(□), $Bi_2Sr_2CuO_x$(○), $Sr(NO_3)_2$(⬡), $SrCO_3$(■), CuO(△), Bi-Ca-O(▽), unknown phase(◆).

formed phases and the formation temperature depended on the composition. Figure 1 shows the X-ray diffraction patterns for C-2 obtained at various stages. The as-dried sample kept at 110°C consisted of nitrates and unknown phases. The latter is presumed to be citrates because strong exothermic peaks were observed around 280°C in the DTA chart which is considered due to the combustion of the citrates.The predominant phase for the calcined and re-calcined samples was the semiconducting phase $Bi_2Sr_2CuO_x$ while the low Tc phase $Bi_2Sr_2CaCu_2O_y$ could be found only in the re-calcined sample. After sintering the re-calcined sample, the low Tc phase was predominant but Bi-Cu-O, the semiconducting and unknown phases were also found as minority phases. From the SEM observation, it was revealed that the re-calcined and sintered sample was comprised of finer grains than the calcined and sintered one.

PROPERTY

The Tc(0) values for A-1 to D-1 and A-2 to D-2 are exhibited in Figs.2(a) and 2(b). Except for the A series of the samples, the re-calcined samples showed higher Tc(0) values than the calcined samples. As far as the conditions examined in this study are concerned, 40 hour was the most suitable period with regard to the Tc(0). It should be noted that the values are much lower than the reported values for the low Tc phase. Since the impurity phases existed in the sintered sample, it may responsible for the tailing at the lower temperature. However, the difference in Tc(0) between C-1 and C-2 is as large as 20K. It suggests that there would be large room to increase the Tc(0) values by choosing more suitable heat-treatment conditions.

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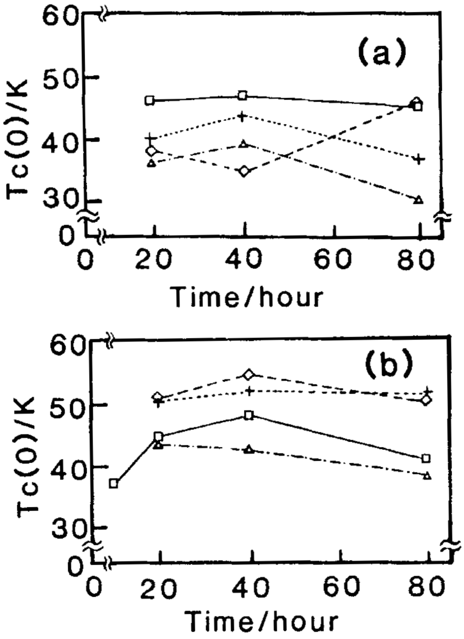

FIGURE 2 Relationship between Tc(0) and sintering period for A-1 to D-1 (a) and A-2 to D-2 (b). A(□), B(+), C(◇), D(△).

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