

Preparation of $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ thick films on MgO by chemical solution deposition

Masashi Seki, Shigeaki Yasuike, Toshinori Tokuda and Satoru Kishida

Tottori University, Koyama-Minami, Tottori 680-8552, Japan

FAX: 81-857-31-0880, e-mail: b98t3036@maxwell.ele.tottori-u.ac.jp

We prepared $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ ($n=2$:Bi-2212) thick films on MgO (100) substrates by a chemical solution deposition of acetic acid metal salt using a partial melt-process. From the X-ray diffraction results, we found that the optimum maximum sintering temperature (T_{max}) and period for obtaining c-axis-oriented Bi-2212 thick films were 910°C and 15min, respectively. The film showed a metallic temperature dependence of resistance in the normal state, and its T_c (critical temperature) was about 88K. The T_c of the Bi-2212 thick film annealed at 700°C was about 90K.

Key words: BSCCO thick film, melt-process, MgO, chemical solution deposition, CH_3COOH

1. INTRODUCTION

$\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$ (Bi-based) superconductors [1-4] are known to be one of potential materials for applications, for example, filters, rf devices and so on. Especially, the fabrications for rf superconducting devices operating in the frequency of micro-wave region require high quality thick films with low surface impedance on dielectric substrates. Recently, the superconducting filter system is expected as a front end of receiver for a base of mobile communication. Because, the filter promises to have low loss and abrupt decay characteristics. As a result, superconducting filters and antennas are expected to have high performance [5, 6]. In order to apply superconducting materials into power electronics, it is necessary to prepare high quality thick films.

So far Bi-based thick films have been prepared by various methods, coevaporation [7], magnetron sputtering [8], laser ablation [9], spray pyrolysis [10],

screen printing [11] and ion implantation [12]. Especially the method of a chemical solution deposition is known to be low cost. However, there are no reports on the preparation of Bi-based thick films by chemical solution deposition.

In this paper, we prepared Bi-based thick films on the MgO(100) substrates by a chemical solution deposition of acetic acid metal salt using a partial melt-process.

2. EXPERIMENTAL

We dissolved Bi:Sr:Ca:Cu=2:2:1:2 mixed powders into a solution of 100% CH_3COOH . The mix powders were prepared from BiOCH_3COO , $\text{Sr}(\text{CH}_3\text{COO})_2 \cdot (1/2)\text{H}_2\text{O}$, $\text{Ca}(\text{CH}_3\text{COO}) \cdot \text{H}_2\text{O}$ and $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$. Thick films were prepared by painting the solution on MgO substrates with a size of $10 \times 10 \times 0.5\text{mm}^3$. They were dried at 180°C for 30min, and were calcined at 500°C for 5min. By the process, the CH_3COOH in the thick film

is completely decomposed at evaporated. The dried thick films were sintered at the maximum temperatures (T_{max}) from 880 to 980°C for the periods of 1-60min in air, cooled down at the rate of 40°C/h from the T_{max} to 850°C in air, and held at 850°C for 2h followed by sudden cooling down to room temperature. The temperature progressing is shown in Fig. 1.

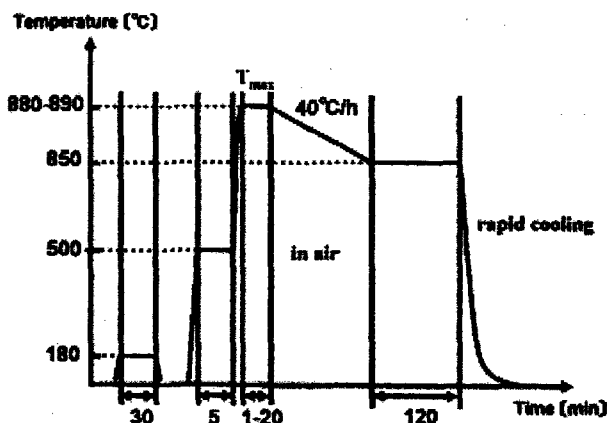


Fig.1 temperature progressing for partial melt-processing

Crystal structure, superconducting phase and crystallinity of the thick films were investigated by X-ray diffraction (XRD). The Scanning electron microscope (SEM) image of the films was observed. The resistance-temperature (R-T) characteristics and electron micro-probe analysis (EPMA) of the thick films were measured in order to clarify surface morphology, superconducting property and chemical composition, respectively.

3. RESULTS AND DISCUSSION

Fig.2 shows XRD patterns of the thick films prepared at $T_{\text{max}}=880-980^\circ\text{C}$ for 5min in air using a partial melt-process. As shown in the figure, the thick films were a mixed phase of Bi-2212 and Bi-2201. When the Bi-2212 thick films were prepared changing the T_{max} in

the temperature range of 880 to 980°C, the thick films were c-axis-oriented. The Bi-2212 (0010) peak intensity of the thick film prepared at 880°C was low. However, the peak increased with the temperature and then, it was saturated at about 910°C. Although the figures were not shown, the intensity ratio of the Bi-2201 (006) to the Bi-2212 (008) XRD peaks (Bi-2201/Bi-2212 intensity ratio) increased with the temperatures. This may be caused by the evaporation of constituent elements. From the results, we found that the optimum T_{max} for obtaining c-axis-oriented Bi-2212 thick films were 910°C.

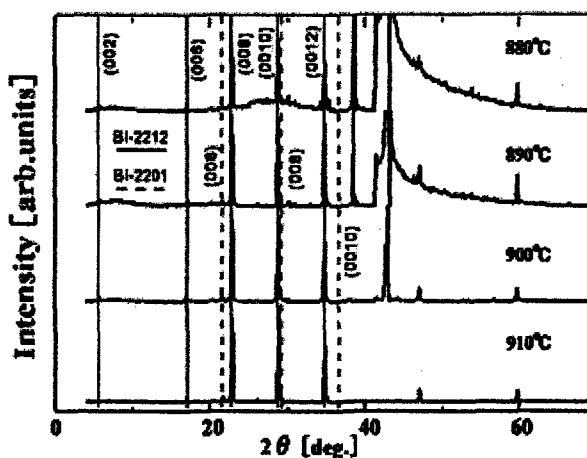


Fig.2 X-ray diffraction patterns of the Bi-2212 thick films prepared at 880-910°C (T_{max})

Fig. 3 shows XRD patterns of the Bi-2212 thick films prepared at 910°C for various periods. As shown in the figure, the thick films were a mixed phase of Bi-2212 and Bi-2201 for 1 to 20min. The Bi-2212 (0010) peaks of the thick films prepared for 5 to 15min were stronger than that prepared for 1 or 20min. This indicated that the thick films had high quality crystallinity. The Bi-2201/Bi-2212 intensity ratio of the thick film for 15min was the smallest among the thick films prepared for 5 to 15min. From the results, we found that the thick film for 15min was approximately Bi-2212 single phase.

Although figures were not show, the SEM images of the thick films were observed. The results indicated that the surface of the thick film prepared at 15min was approximately flat.

From the results, we found that the optimum period and temperature for obtaining the c-axis-oriented thick films with high crystallinity and were 15min and 910°C, respectively.

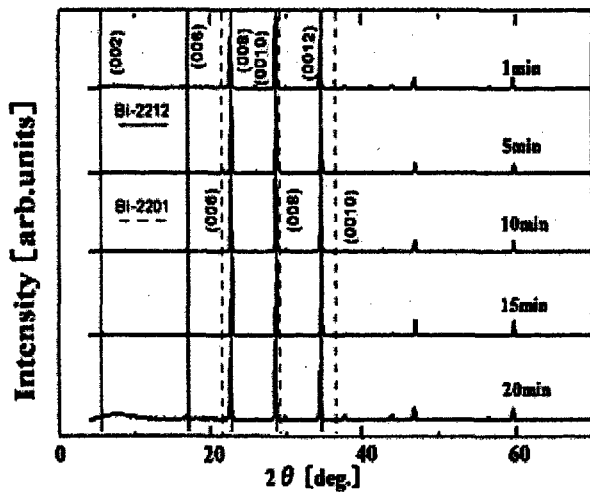


Fig.3 X-ray diffraction patterns of the Bi-2212 thick films prepared at 910°C (T_{max}) for various periods

Fig. 4 shows the R-T characteristics of Bi-2212 thick films annealed at (a) 0°C (as-deposition), (b) 500°C, (c) 600°C, (d) 700°C and (e) 800°C for 1h in oxygen gas. The resistance values of the film were normalized by that at 200 K. We defined T_c as the zero-resistance temperature. The T_c of the as-deposited thick film was about 88K. This indicated that the as-deposited thick film was optimally doped in oxygen content. Although T_c of the thick film annealed at 500°C was about 81K. This indicated the thick film becomes over doped. The T_c of the thick film increased with the temperature, becomes maximum at 700°C and then decreased. The T_c of thick film may be due to oxygen content.

Fig. 5 shows the I-V characteristics of the Bi-2212 thick

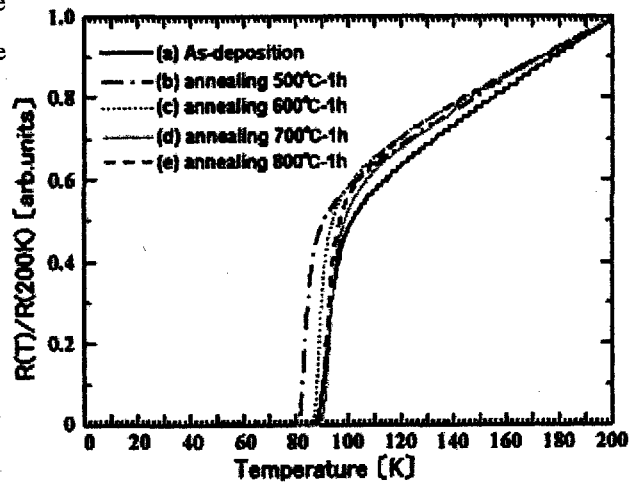


Fig.4 R-T characteristics of the Bi-2212 thick films annealed at (a) 0°C (as-deposition), (b) 500°C, (c) 600°C, (d) 700°C and (e) 800°C for 1h in oxygen gas

films annealed at 0-800°C for 1h in oxygen gas. The critical current (I_c) of the as-deposited thick film was about 180mA at 77K, 0T. The I_c of the thick film annealed at 600°C was about 50mA. From the results, we found that the I_c of the thick films were approximately constant for annealing temperatures from 600 to 800°C. The decrease of the I_c by annealing at

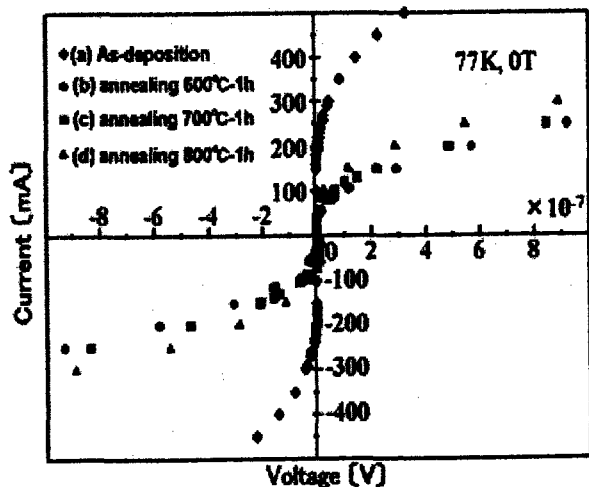


Fig.5 I-V characteristics of the Bi-2212 thick films annealed at (a) 0°C (as-deposition), (b) 600°C, (c) 700°C and (d) 800°C for 1h in oxygen gas

600°C-800°C was independence of the T_c of thick films. The decrease of the I_c may be due to weak coupling of the thick films, which is caused by annealing process.

4. CONCLUSIONS

We prepared the Bi-2212 thick films on the MgO(100) substrates by a chemical solution deposition using a partial melt-process. The solution of 100% CH_3COOH was used in this study. From the results, we found that optimum temperature (T_{max}) and period for obtaining c-axis-oriented Bi-2212 thick films were 910°C and 15min, respectively. The films showed a metallic temperature dependence of resistance in the normal state, and their T_c was about 88K. The T_c of the Bi-2212 thick film annealed at 700°C was about 90K.

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