

Synthesis of Y–Ba–Cu–O superconducting material by citrate chemical route — some observations

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Abstract. We present an overview of our powder synthesis procedure and give some initial results concerning the superconducting properties of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$.

Keywords. $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$; citrate chemical route.

1. Introduction

The practical limitations of the standard ceramic technique make it difficult to achieve homogeneous fine particles of the oxide mixture, which is one of the pre-requisites for a good quality superconductor. Many workers (Chu and Dunn 1987; Dunn *et al* 1987; Fujiki *et al* 1987; Kini *et al* 1987; Sribata *et al* 1987; Wang *et al* 1987; Khurana *et al* 1989) have used the well-known chemical method for preparing the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ superconductor. This method is known to provide a very fine and highly homogeneous mixture of the respective oxide powders as a starting material for the fabrication of shaped structures like wire and thin films of superconducting materials. Our work involved use of the citrate process to prepare ultimate precursor for obtaining a superconducting material. We find that homogeneous powders with high surface areas are readily produced and that these powders may then be fabricated into dense ceramics.

2. Powder preparation and sintering

High purity nitrate salts of yttrium, barium and copper were used as starting materials. The salts were dissolved in distilled water and the individual nitrate solutions were mixed together in proper proportions with citric acid to form a uniform solution. One gram equivalent of citric acid was used for each gram equivalent of metals. Water was evaporated from the solution at 70°C until it became a viscous liquid which was further dehydrated in an oven at 120°C for 4 to 6 hours resulting in a solid precursor. The powder obtained was so fine that there was no need for the grinding process.

The solid precursor was fired at elevated temperatures to form the desired superconducting material. Figure 1 shows a differential thermal (DT) and thermogravimetric (TG) scan analyses of the dehydrated precursor powder in air. This behaviour is similar to that reported by Dunn *et al* (1987). The weight loss over the range 150 to 250°C arises primarily from decomposition of the nitrates and partially citrates while the weight loss above 300°C is from the organic constituent. The magnitude of the weight change is consistent with what is expected based on the starting materials and citric acid additions. The DT/TG analysis gives us an idea of the pre firing temperature

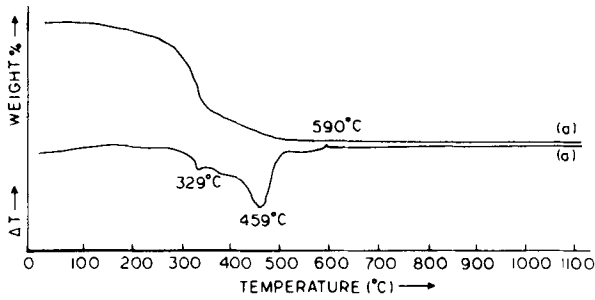


Figure 1. Thermogravimetric and differential thermal scan of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ precursor powder heated in air. Heating rate was $10^\circ\text{C}/\text{min}$.

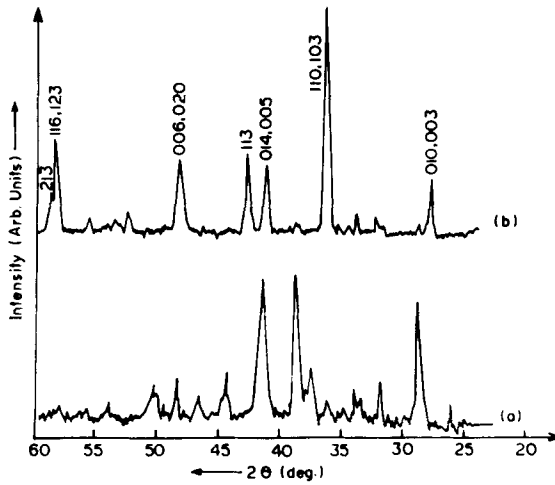


Figure 2. X-ray diffraction patterns at room temperature of (a) the precursor and (b) the superconducting sample obtained from the precursor.

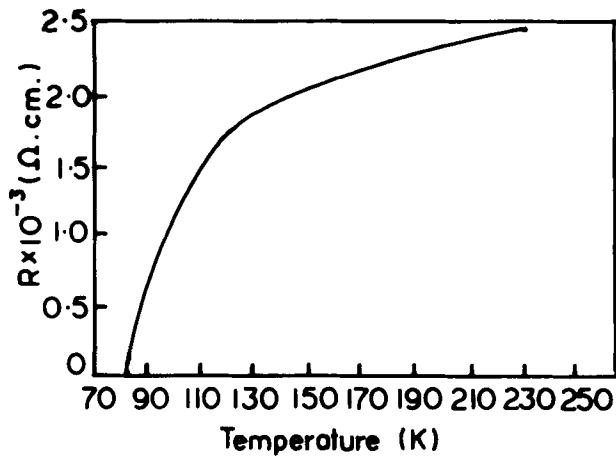


Figure 3. Representative d.c. resistivity data vs temperature.

of solid precursor. Thus, solid precursor powder was later pre-fired at 600°C in air for nearly 6 hours.

The pre-fired powder was pressed into pellets and sintered at various temperatures ranging from 750°C to 950°C at different time intervals. The samples sintered above 800°C started showing the presence of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ phase. The X-ray diffraction pattern of the superconducting $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ sample obtained by sintering at 800°C for 18 hours and its precursor is shown in figure 2. A typical plot of resistance against temperature of the superconducting sample sintered at 800°C is shown in figure 3.

However, the current density value obtained from such samples was not encouraging and requires further investigation.

References

- Chu C T and Dunn B 1987 *J. Am. Ceram. Soc.* **70** C-375
Dunn B, Chu C T, Zhou L W, Cooper J R and Gruner G 1987 *Adv. Ceram. Mater.* **2** 343
Fujiki M, Hikita M and Sukegawa K 1987 *Jpn J. Appl. Phys.* **26** L1159
Kini A M, Geiser U, Kao H C I, Carlson K D, Wang H and Monaghan M R 1987 *Inorg. Chem.* **26** 1834
Khurana B S, Tripathi R B, Khullar S M, Kotnala R K, Singh S, Jain K, Reddi B V, Goel R C and Das B K 1989 *J. Mater. Sci. Lett.* **8** 234
Sribata S, Kitagawa T, Okazaki H, Kimura T and Murakami T 1987 *Jpn J. Appl. Phys.* **27** 253
Wang X Z, Hentry M, Livage J and Roseman I 1987 *Solid State Commun.* **64** 881