

BSCCO Superconducting Powder by SHS

Soh Deawha*, Cho Yongjoon*, Korobova N.**

Myongji University, Korea*. Combustion Problems Institute, Kazakstan**

Abstract

The BSCCO superconductor materials of using Self-propagating High-temperature Synthesis (SHS) were studied. Mechano-chemical activation - as a pre-treatment of the reactants mixture - strongly influences the kinetic parameters, the reaction mechanism, and the composition and structure of the final product. In this paper as an effort for fabricating the SHSed BSCCO superconductor powder SHS method was considered to application in the synthesis of superconducting materials.

Key Words : BSCCO, SHS, Mechano-chemical activation, superconducting materials

1. Introduction

BSCCO is a major material for HTS devices. The properties of this materials are very dependent on the ratios of superconductor phases and the consequential microstructure produced. BSCCO powder has been synthesized by different methods.

There are two standard methods being used for preparing powders of copper ceramic superconductors.

Solidstate reaction method and Coprecipitation method. First method involves mixing of carbonate or oxide powders followed by ball milling. The length of this process depends on homogeneity required but usually varies from 2 to 10 hours. This is repeated until the desired composition is obtained. In the second method the nitrates of the individual constituent are used.

These methods have some problems, including requiring a large input of energy, use expensive equipment, long sintering times and high temperature reaction vessels.

The SHS method commonly uses mixed powders of metals and metal oxides[1,2]. The

reactions are typically very fast (a few seconds) and can produce high temperatures. Self-propagating high temperature synthesis is a process that enables the rapid formation of the materials by utilizing the energy given out in a very exothermic solid-state reaction [3,4].

In this paper we report about BSCCO powder preparation by SHS.

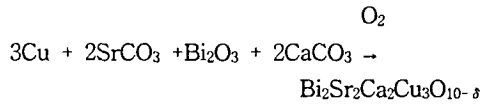
2. Experimental part

All preparations were conducted in an air atmosphere on a supporting ceramic tile and initiated by the application of a heated filament (typically at 700 °C). Reagents were purchased from Aldrich Chemical Co and used as supplied. Powder X-ray diffraction (XRD) patterns were obtained with a Siemens D5000 diffractometer using Co monochromatic radiation. Combustion rate and temperature measurements were taken by optical pyrometer and platinum-rhodium [Pt/PtRt (13 % Rt)] thermocouples. The phase composition of the samples were analyzed by X-ray diffraction (XRD).

(a) Preparation of SHSed samples

All the reagents were ground together in stoichiometric ratio and pressed into pellets of 20 mm diameter and 10 mm thickness by application of an uniaxial pressure of 0,5-0,7 GPa. The reaction was initiated in air by a heated filament. This produces a self-propagated reaction of 5~7 s duration and in some instances a partly fused product. The pellets were annealed at 900-880 °C for 1.5 h in air and had quenched procedure in order to induce superconductivity (Fig. 1). This produced an orange-yellow propagation wave that proceeded through the solid at 0.1-0.2 mm/s (Fig. 2).

On practice we had [5]



Where Cu is the fuel, Bi₂O₃ is an active filler, and O₂ is a gaseous oxygen.

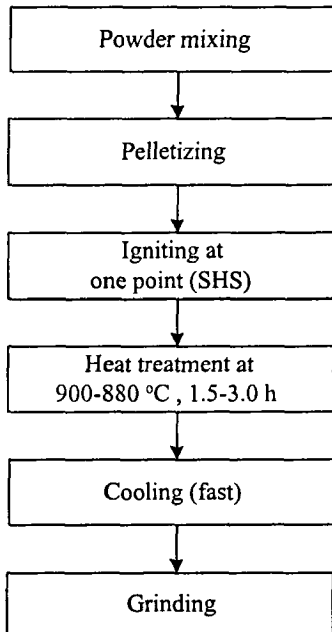


Fig. 1. Simple block diagram of SHS process.

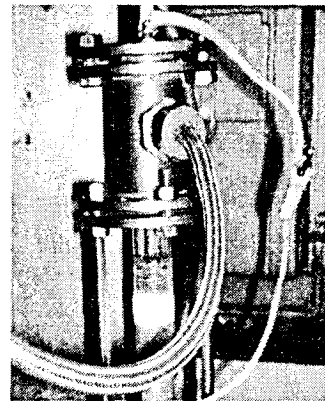
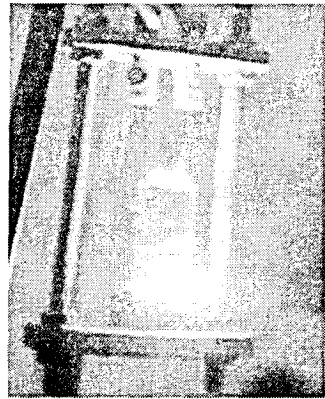


Fig. 2. SHSed BSCCO samples.

(b) Heat-treatment and Quenching conditions
- Seria 63

Heat-treatment; 900-880 °C (duration 1.5 h),

cooling up to 540 °C.

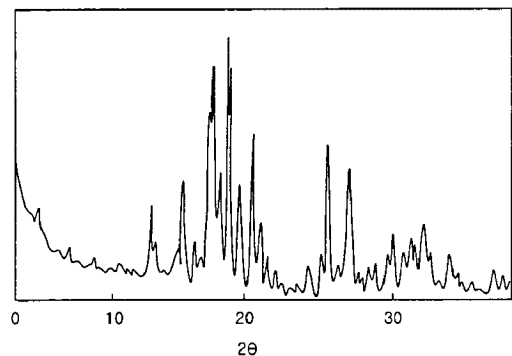
Quenching conditions: from 540 °C to the room temperature air.

- Seria 64

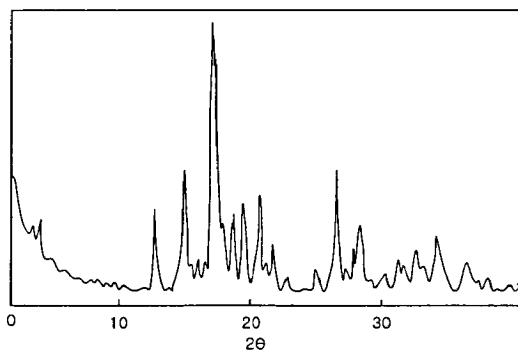
Heat-treatment: 900-880 °C (duration 3 h), cooling up to room temperature

3. Results and discussion

SHS synthesis of HTS materials is rapid and relatively easy to perform. The inhomogeneity was due to the short time during which the sample was at a high temperature. Because of the significant heat loss, the rapid pellet cooling did not enable complete homogenization.



(a) Seria 63



(a) Seria 64

Fig. 3. X-ray diffraction patterns of powders made by SHS.

X-ray powder diffraction pattern (Fig. 3) of the synthesized materials revealed that crystalline BSCCO (2212) phase was produced in samples. Seria 63 was characterized only by presence of BSCCO (2201) phase, but as you can see that the conditions were almost similar. In all samples we can find small amount of intermediates like $\text{Bi}_2\text{Sr}_2\text{CuO}_x$, $\text{Ca}_{2-x}\text{Sr}_x\text{CuO}_3$, $\text{Ca}_{0.4}\text{SrO}$, 6CuO_2 , $\text{SrCuBi}_2\text{O}_x$. In Seria 64 because of rapid quench we can find additional CuO .

Pre-heating the green pellet enhanced the homogenization of the product by increasing the combustion temperature as

$$T_c = T_0 + (-\Delta H)C_0 / c_p(T) \rho ;$$

where T_0 is the initial temperature, $-\Delta H$ the average heat of the reaction, C_0 the concentration of the limiting reactant, the conversion, $c_p(T)$ the mean specific heat. Pre-heating is often used to combust low exothermic mixtures, which are not combustible at room temperature. Pre-heating a sample in our experiments had two effects. First it increased the combustion temperature to 1100-1200 °C and prolonged the exposure time of the combusted sample to high temperatures. Both effects increased the homogeneity of the product.

4. Conclusions

Formation of SHSed BSCCO superconductor powder is a simple synthesis without expensive equipment and is time and energy efficient. The peculiarities was shown as following:

- 1) Process of Cooling after SHS synthesis should not be very fast.
- 2) The temperature of heat-treatment from which it will be cooling is a specific point or HTS material structure formation.

3) It is necessary to use non-stoichiometric mixing of starting materials because of vitalization of some components during the high temperature SHS process.

Acknowledgements

This work was accomplished by KISTEP grant of M6-0011-00-0043 for Intl. joint-research program.

References

- [1] Shin-Pei Matsuda, "Chemistry and processing of HTS", in Chemical processing of ceramics. Ed. by B. I. Lee, E. J.A. Pope, p. 445, 1994.
- [2] Y. Idemoto, K. Fueki, "Oxygen nonstoichiometry and valences of bismuth and copper in $\text{Bi}_{2.00}\text{Sr}_{1.88}\text{Ca}_{1.00}\text{Cu}_{2.14}\text{O}_y$ ", Physica C, Vol. 168, p. 167, 1990.
- [3] A. Makino, "Fundamental Aspects of the Heterogeneous Flame in the Self-Propagating High-Temperature Synthesis (SHS) Process", Progress in Energy and Combustion Science, Vol. 27, p. 1, 2001.
- [4] Soh Deawha, Korobova Natalya, "Peculiarities of SHS and Solid State synthesis of $\text{ReBa}_2\text{Cu}_3\text{O}_{7-x}$ Materials", J. of KIEEME, Vol. 15, No. 3, p. 275, 2002.
- [5] D. Soh, N. Korobova, "Synthesis of $\text{Bi}_{2+x}\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{4+2n+d}$ compounds by SHS", Proc. Summer Conf. of KIEEME, 2002, p. 94.