

# Effects of Calcination on Compositions of (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> High Temperature Superconductor Powders Synthesized by the Solution Combustion Technique

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## Abstract

Applications of (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (where RE is a rare-earth element) high temperature superconductors extend over a wide range of energy-related industries such as power cables, electrical motors, and flywheel energy storage systems. Difficulty in synthesizing the superconductor powders, however, is one of the crucial factors that hinder their potential for industrial utilization. In this study, YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> superconductor powders were synthesized by a solution combustion synthesis technique. Corresponding metal nitrate were employed as reactants, while either sucrose or urea was used as combusting fuels in the powder synthesis process. Experimental results from x-ray diffraction indicated that YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> powders with composition suitable for practical applications could be achieved when the combusted powders were subjected to subsequent heat treatment. The results also revealed that combusting fuel had a significant effect on powder composition. Formation of superconductor powders and secondary phases with respect to heat treatment will be discussed.

Keywords: Superconductors, Combustion Synthesis, Powder, Sucrose, Urea

## **1. Introduction**

Discovery of (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (RE123) high temperature superconductors has opened the door for industrial applications such as power cables, electrical motors, and flywheel energy storage systems [1]. This is due to their unique capability in withstanding high magnetic fields, while carrying significant critical current densities. One of the major challenges in exploiting the RE123 superconductors in practical applications is the problem related to chemical inhomogeneity and impurities of the superconductor powders. It has been reported that uncontrolled synthesis process of Y123 can cause formation of non-superconducting phases, such as Y<sub>2</sub>Ba<sub>4</sub>O<sub>7</sub>, BaCuO<sub>2</sub>, Y<sub>2</sub>Ba<sub>2</sub>O<sub>5</sub>, BaCO<sub>3</sub> and Y<sub>2</sub>BaCuO<sub>5</sub> (Y211) [2-3]. Presence of secondary phase such as BaCO<sub>3</sub> has also been reported to suppress critical current density in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (Y123). In addition, superconductor powders with coarse particles are unfavorable for fabrication processes of both bulk and thin film superconductors. Synthesis of superconductor powders is, therefore, one of the

important goals in the field of superconductor processing. Solution combustion synthesis is a self-propagating synthesis

technique which requires small amounts of energy input compared to other synthesis techniques, such as solid state reaction. Additionally, solution combustion synthesis is a simple, rapid and effective technique often employed in production of ceramic powders with small particle size and chemical homogeneity. This study is, therefore, aimed at synthesizing RE123 superconductor powders with chemical composition required for practical applications. To further the development of the solution combustion synthesis processing, effects of post-combustion heat treatment, the calcinations process, on the composition of the RE123 powders such as YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> are investigated. X-ray diffraction was employed as a characterization technique to determine chemical compositions of the powders.

## 2. METHODOLOGY

### 2.1 Powder synthesis

The (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (RE: rare-earth element) superconductor powders were synthesized by solution combustion synthesis technique. The technique involved preparation of aqueous solutions containing metal nitrate and combustion fuel. A nitrate compound of rare-earth element (Y(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, Er(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O, and Sm(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O), Ba(NO<sub>3</sub>)<sub>2</sub> and Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (Aldrich) with stoichiometric ratios of RE:Ba:Cu = 1:2:3 were used as oxidizing compounds for the synthesis of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> powders. Either sucrose or urea, acting as combusting fuels in the powder synthesis process, was added to the prepared nitrate solution. Low heating temperature of less than 400°C was applied to the solution to initiate combustion. Upon completion of the combustion reaction, the products were collected and ground into powders with a mortar and pestle. Subsequently, the powders were calcined at 900°C for 4 hours.

### 2.2 Characterization

Composition of the powders were investigated by X-ray diffractometer (Phillips X'Pert), over angles ranging from 20° to 60° in 2θ, with a step size of 0.01° and a scan rate of 1.3 °/min.

## 3. RESULTS AND DISCUSSION

### 3.1 Compositions of uncalcined powders

Compositions of as-synthesized powders were identified by x-ray diffraction analysis. The analysis was conducted by matching the 2θ positions of prominent peaks from diffraction results with the patterns obtained from JCPDS database. The x-ray analysis showed that various compounds, primarily (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (RE123), (RE)BaCuO<sub>5</sub> (RE211), BaCuO<sub>2</sub> and BaCO<sub>3</sub>, were evident in the powder products, as shown in Fig. 1, 2 and 3. Formation of BaCO<sub>3</sub> was believed to be attributed to a reaction between Ba<sup>2+</sup> ions from Ba(NO<sub>3</sub>)<sub>2</sub> and carbon from sucrose or urea, while RE211 and BaCuO<sub>2</sub> may be formed as a result of imprecise stoichiometric mixing of RE<sup>3+</sup>, Ba<sup>2+</sup> and Cu<sup>2+</sup>. High concentration of secondary phase, specifically BaCO<sub>3</sub>, often results in the deterioration of superconductor properties. The results, therefore, suggested that an additional processing step is required to obtain superconductor powders with composition suitable for practical applications.

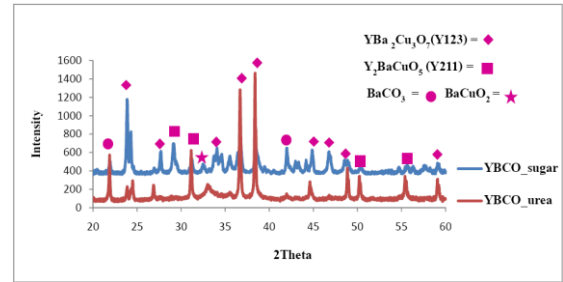


Fig. 1 X-ray diffraction pattern of uncalcined Y-BaCu-O powders, using sugar and urea as combustion fuels

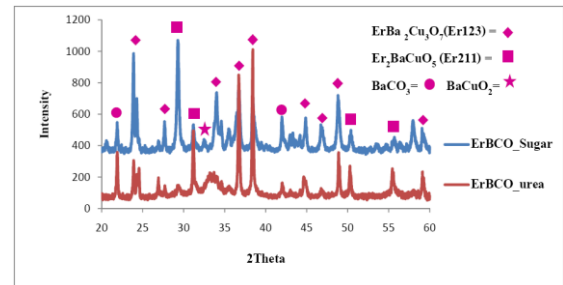


Fig. 2 X-ray diffraction pattern of uncalcined Er-Ba-Cu-O powders, using sugar and urea as combustion fuels

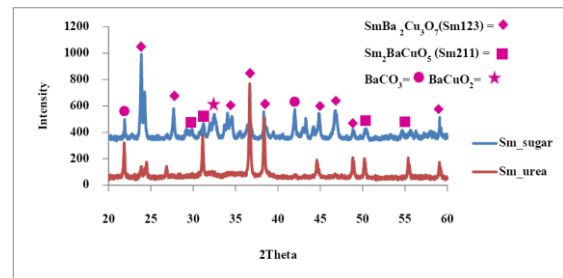
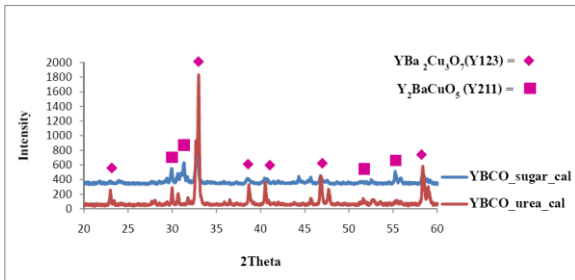


Fig. 3 X-ray diffraction pattern of uncalcined Sm-Ba-Cu-O powders, using sugar and urea as combustion fuels

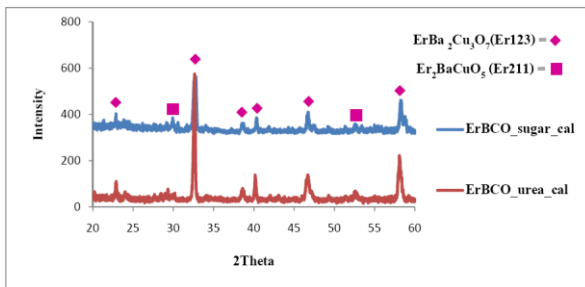
### 3.2 Compositions of powders subjected to calcination

To eliminate secondary phases which have detrimental effects on properties of superconductors, the as-synthesized powders were subsequently calcined at 900°C for 4 hrs. Compositions of calcined powders were also identified by x-ray diffraction analysis. Results from the x-ray analysis indicated that only RE123 and RE211 phases were present in the powder products, as shown in Fig. 4, 5 and 6. Elimination of BaCO<sub>3</sub> may be attributed to the high temperature heating, since BaCO<sub>3</sub> is unstable at temperature higher than 800°C. However, high temperature calcinations of 900°C may result in an unfavorable effect, since it can cause evaporation loss of Ba<sup>2+</sup> and Cu<sup>2+</sup> [4].

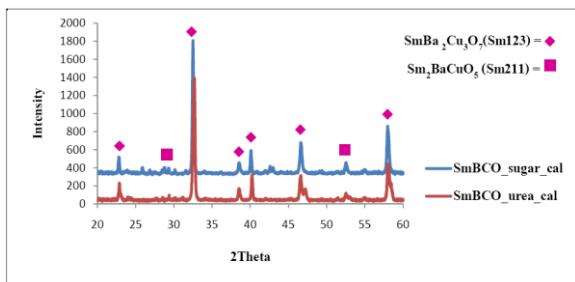
Minor loss of Ba<sup>2+</sup> and Cu<sup>2+</sup> may lead to formation of (RE)BaCuO<sub>5</sub> (RE211) instead of (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> (RE123). Different from BaCO<sub>3</sub>, the RE211 phase does not play a role in suppressing critical current density of the superconductors. On the contrary, the RE211 phase acts as flux pinning center which results in the enhancement of critical current density in superconductors [5]. Therefore, presence of RE211 phase in the powders is desirable for the use of RE123 in practical applications.



**Fig. 4** X-ray diffraction pattern of Y-Ba-Cu-O powders subjected to calcinations at 900°C



**Fig. 5** X-ray diffraction pattern of Er-Ba-Cu-O powders subjected to calcinations at 900°C



**Fig. 6** X-ray diffraction pattern of Sm-Ba-Cu-O powders subjected to calcinations at 900°C

#### 4. CONCLUSION

YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> high temperature superconductor powders were synthesized by a solution combustion synthesis technique. X-ray diffraction results indicated that the as-synthesized powders consisted

of (RE)Ba<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, (RE)BaCuO<sub>5</sub>, BaCuO<sub>2</sub> and BaCO<sub>3</sub>. To eliminate undesirable phase, especially BaCO<sub>3</sub>, the powders were subsequently calcined at 900°C. The results indicated that YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub>, ErBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> powders with composition suitable for practical applications could be achieved.

#### 5. ACKNOWLEDGMENTS

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