Available online at www.sciencedirect.com
SCIENCE DIRECT
Materials Chemistry and Physics 94 (2005) 233–240
MATERIALS CHEMISTRY AND PHYSICS
www.elsevier.com/locate/matchemphys

Characterization and phase transitions of $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$–Ag composite powder obtained by spray pyrolysis

B.A. Marinkovic [a,*], P.M. Jardim [a], F. Rizzo [a], L. Mancic [b], O. Milosevic [b]

[a] *Departamento de Ciência dos Materiais e Metalurgia, Pontifícia Universidade Católica, Rua Marques de São Vicente, 225, 22453-900 Rio de Janeiro, RJ, Brazil*
[b] *Institute of Technical Sciences of Serbian Academy of Sciences and Arts, Knez Mihailova 35/IV, 11000, Belgrade, Serbia and Montenegro*

Received 3 January 2005; received in revised form 28 March 2005; accepted 20 April 2005

Abstract

$(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$–Ag (BPSCCO/Ag) composite precursor powder was prepared by spray pyrolysis. The as-prepared and thermally treated (750 °C (1 h) and 780 °C (3 h)) powders were characterized by transmission electron microscopy, ambient X-ray diffraction, In situ X-ray diffraction, magnetic susceptibility measurement and differential thermal analysis (DTA).

DTA curve exhibited two strong endothermic peaks above 790 °C, one very wide with the onset at 790 °C and the second at 840 °C. In situ X-ray diffraction measurements strongly supported the view that the first endothermic peak was due to Ca_2PbO_4 decomposition and a transient liquid formation through PbO–CuO–Ag eutectic. The second endothermic peak can be explained by onset of Bi-2212 peritectic melting that began at 840 °C with local formation of Bi-2201 intergrowths. The melting was complete at 875 °C.
© 2005 Elsevier B.V. All rights reserved.

Keywords: Superconductors BSCCO; Spray pyrolysis; Composite powder; Ag:(Bi,Pb)-2223 powder; In situ X-ray diffraction; Phase formation

1. Introduction

There are a few reports on metal/simple-oxide composite powders prepared by the spray pyrolysis route [1–3]. Majumdar et al. [2], for example, described the formation of Ag–CuO composite powder, while Matsumoto et al. [3] demonstrated viability of preparation of Ag–TiO_2 composite powder. Studies of metal/complex oxide composite powders, synthesized by spray pyrolysis, are even rarer. A study like this was performed by Tsudo et al. [4] on BPSCCO/Ag system. They studied the influences of the chemical composition of the precursor and the sintering conditions on the superconducting properties of (Bi,Pb)-2223 phase, such as T_c and J_c, and observed that the addition of Ag decreased both T_c and J_c.

Aerosol decomposition routes, such as spray pyrolysis, possess several advantages (chemical homogeneity of powder, uniform-sized particles, etc.) over conventional solid-state techniques [5]. The quality of the powders obtained by spray pyrolysis can be further improved by the addition of urea to the metal nitrates precursor solution [6]. The preparation of a BPSCCO/Ag composite powder containing chemically homogeneous, uniform-sized submicron BPSCCO particles, mixed with well dispersed silver could be useful in fusion-reformation process for obtaining Bi-2212 and (Bi,Pb)-2223 bulks, tapes or films. This is due to the important role that silver plays in the reduction of oxygen losses upon peritectic melting [7] of these two superconducting phases. The minimization of oxygen losses on melting should help the reformation of Bi-2212 and (Bi,Pb)-2223 during the solidification process [7,8]. On the other hand, it has been reported [4,9] that the addition of high contents of silver can decrease the critical temperature of oxide superconductors. Since Jin et al. [10] showed that the addition of 20% of silver did not affect the critical temperature of BSCCO phases, this weight percent of silver was chosen in the present work.

* Corresponding author. Tel.: +55 21 3114 1240; fax: +55 21 3114 1248.
E-mail address: bojan@dcmm.puc-rio.br (B.A. Marinkovic).

0254-0584/$ – see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.matchemphys.2005.04.051

Presented here is a report on the characterization of BPSCCO/Ag composite powders as-synthesized by spray pyrolysis and after a rapid thermal treatment at 750 °C (1 h) and 780 °C (3 h). Afterwards, an in situ synchrotron X-ray diffraction study of the phase transitions that occur in the precursor powder was performed before the final conversion to (Bi,Pb)-2223 phase. The results of this study are supplemented by differential thermal analysis (DTA).

2. Experimental

Composite particles of Ag:(Bi,Pb)-2223 stoichiometry were synthesized from a mixed nitrate solution with the appropriate ratio of the cations necessary to produce the (Bi,Pb)-2223 phase (Bi:Pb:Sr:Ca:Cu = 1.8:0.2:2:2:3). The nominal concentration corresponds to 100 g of mixed metal oxides accomplished through dissolving of nitrates into 1000 ml of a 5 wt% HNO_3 water solution with 2 wt% urea and 20 wt% Ag (in the form of $AgNO_3$). The precursor solution characteristics (density, pH, viscosity and surface tension) were measured and used to calculate [11] the average droplet size (2725 nm) and the aerosol droplet density ($<10^{14}$ droplets m^{-3}). The solution was atomized using an ultrasonic atomizer with the working frequency of 1.7×10^6 Hz. Aerosol decomposition was carried out in a tubular flow reactor in argon gas at temperatures up to 840 °C. The aerosol flow rate was determined by the argon flow rate (1.7×10^{-6} m^3 s^{-1}) and the reactor geometry (quartz tube characteristics: inner diameter 3.6×10^{-2} m, length 6×10^{-1} m), therefore the residence time of a droplet/particle at the maximum reaction temperature was 5 s. The powder was analyzed in two states: as-prepared (collected at the end of the high temperature tubular flow reactor) and thermally treated in air at 750 °C (1 h) and 780 °C (3 h). The heat-treated powder can be used as the precursor powder for conversion to (Bi,Pb)-2223.

Particle structure was characterized by transmission electron microscopy (TEM) together with X-ray energy dispersive spectroscopy (EDS), using a JEOL 2010 operating at 200 kV. The TEM sample preparation procedure was ultramicrotomy. In this method, the powder is first embedded in a resin and then cut in an ultramicrotome (Reichert-Jung with a Diatome diamond knife), allowing the study of the particles internal structure.

Phase analysis of the as-prepared and heat-treated powders was completed using a Siemens D-5000 diffractometer. Cu Kα radiation and graphite monochromator were used with a 0.02 scanning step and a step time of 25 s. Magnetic susceptibility measurements were performed by an AC susceptometer Lake Shore 7000 (using magnetic field of 1Oe and frequency of 100 Hz) in the temperature range of 80–130 K.

DTA and in situ X-ray diffraction were used to study the reactions that precede the conversion of the composite precursor powder to Ag:(Bi,Pb)-2223. DTA of the heat-treated sample was carried out under static air atmosphere up to 900 °C in a Perkin-Elmer DTA-7. The heating rate was 10 °C min^{-1}. In situ X-ray diffraction experiments of the thermally treated powder were carried out at the XRD1 beam-line station of the Brazilian National Synchrotron Light Laboratory (LNLS). The powder was heated from room temperature up to 740 °C at a rate of 20 °C min^{-1}. From this temperature up to 830 °C, one pattern was taken every 5 °C. The exposure time for every pattern was about 10 min. From 830 °C a temperature–time schedule was applied up to the highest temperature measured of 875 °C, Fig. 1. One pattern was taken every 10 min in this temperature range. The step size was of 0.05° (2θ). The wavelength was 1.54068 Å ($\Delta\lambda$ = 0.001617), which was calibrated with silicon standard NBS640b.

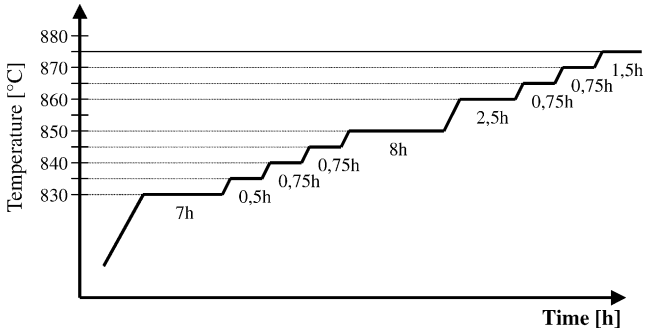

Fig. 1. Temperature–time schedule between 830 and 875 °C applied for in situ X-ray diffraction of the thermally treated powder.

Quantitative phase analysis of the room temperature patterns was carried out using the Rietveld method and the software Topas 2.1 [12].

3. Results and discussion

The X-ray diffraction patterns of the as-prepared and heat-treated powders (Fig. 2a and b) were analyzed quantitatively by the Rietveld method and the results are shown in Table 1. The as-prepared powder contains Bi-2212, Bi-2201, cuprate 14:24 ($Sr_{14-x}Ca_xCu_{24}O_{41}$), CuO and Ag, while the heat-treated powder consists mainly of Bi-2212 and some secondary phases, such as cuprate 14:24, Ca_2PbO_4 and Ag. The cuprate 1:1 ($Ca_{1-x}Sr_xCuO_2$) is present in a very small content (less then 1%). The amount of silver in the as-prepared powder is overestimated due to the presence of amorphous material that was not taken into consideration in the full

Table 1
Relative weight percentages of the phases present in as-prepared and heat-treated powders

	As-prepared	Heat-treated
Bi-2212 (%)	31.8	62.1
Bi-2201 (%)	17.5	–
14:24 (%)	19.2	14.1
CP (%)	–	6.2
1:1 (%)	–	0.8
CuO (%)	7.8	–
Ag (%)	23.7	16.8

Download English Version:

https://daneshyari.com/en/article/9782934

Download Persian Version:

https://daneshyari.com/article/9782934

Daneshyari.com