

The spatial distribution of temperature and oxygen deficiency in spark-plasma sintered superconducting Bi-based materials



E. Govea-Alcaide^a, J.E. Pérez-Fernández^a, I.F. Machado^b, R.F. Jardim^{c,*}

^a Departamento de Ciências Básicas, Facultad de Ciencias Técnicas, Universidad de Granma, Apdo. 21, P.O. Box 85100, Bayamo, Cuba

^b Departamento de Engenharia Mecatrônica e Sistemas Mecânicos, Escola Politécnica, Universidade de São Paulo, 05508-900 São Paulo, SP, Brazil

^c Instituto de Física, Universidade de São Paulo, CP 66318, 05315-970 São Paulo, SP, Brazil

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ABSTRACT

Pre-reacted powders of $(\text{Bi-Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ (Bi-2223) were consolidated by using the spark plasma sintering (SPS) technique under vacuum and at different consolidate temperatures T_D . X-ray diffraction patterns revealed that the dominant phase in all SPS samples is the Bi-2223 phase, but traces of the $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{10+x}$ (Bi-2212) phase were identified. We have found that the transport properties of SPS samples depend on their oxygen content because the SPS process is performed under vacuum. Simulations by using the finite element method (FEM) were performed for determining the actual temperature in which powders are consolidated. From these results we have inferred that SPS samples are oxygen deficient and such a deficiency is more marked near the grain boundaries, suggesting the occurrence of grains with core-shell morphology. We also argued that the width of the shell depends on the consolidation temperature, a feature corroborated by the FEM simulations.

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The spark plasma sintering (SPS) is an effective, unconventional method for promoting densification of powders by the simultaneous action of a high direct electric current through graphite dies and uniaxial pressure [1]. In high- T_c cuprate superconductors, the use of SPS may be a difficult task because the process occurs under vacuum, and the general physics properties of these materials are very sensitive to the oxygen content. Such an oxygen-dependent features are much less pronounced in materials belonging to the Bi-Sr-Ca-Cu-O (BSCCO) system [2], making the application of the SPS method useful for promoting densification of these families of high- T_c cuprates superconductors. However, up to now there are few studies regarding the use of SPS in the BSCCO system. In the last decade, a study of the influence of the SPS conditions on the formation of $(\text{Bi,Pb})_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (Bi-2212) and $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ (Bi-2223) phases was conducted [3]. The authors obtained SPS samples under different consolidation temperatures T_D and times and the net result was the production of samples composed of extra phases, as inferred from X-ray diffraction analysis. Moreover, a recent study on SPS Bi-2223 samples suggested that T_D not only has a strong influence on the phase composition but also in the oxygen content of the Bi-2223 ceramics [4]. These results indicated that one of the most important challenges of any SPS process, even performed under vacuum, is to

determine precisely the consolidation temperature in which the powder is subjected during the brief time interval of the SPS process.

Within this scenario, the main motivation of this work is to disclose the intricate balance between T_D , phase composition, and the oxygen content of samples prepared by the SPS method. For this purpose, we have consolidated pre-reacted powders of the $\text{Bi}_{1.65}\text{Pb}_{0.35}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ compound by spark-plasma sintering. The finite element method (FEM) was used for optimizing the consolidation temperature in order to produce single phase materials. The SPS samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), temperature dependence of the electrical resistivity, and voltage-current characteristics.

The schematic drawing of the consolidation system is displayed in Fig. 1(a). It is composed of two inconel electrodes, six graphite spacers, the die with two plungers, and the sample, the latter located in the center of the apparatus. For each of the above domains, the electro-thermal process is described by a two coupled partial differential equations: one related to the charge conservation law and the other one associated with the heat transfer process [6,7]:

$$\nabla \cdot J = 0, \quad (1)$$

$$\rho c_p(T) \frac{\partial T}{\partial t} - \nabla \cdot (k \nabla T) = q_i. \quad (2)$$

Here, T is the temperature, ρ is the density, $q_i = JE$ is the heat loss by Joule effect, $J = E/\rho_e(T)$ is the electric current density, $\rho_e(T)$ is

* Corresponding author.

E-mail address: rjardim@if.usp.br (R.F. Jardim).

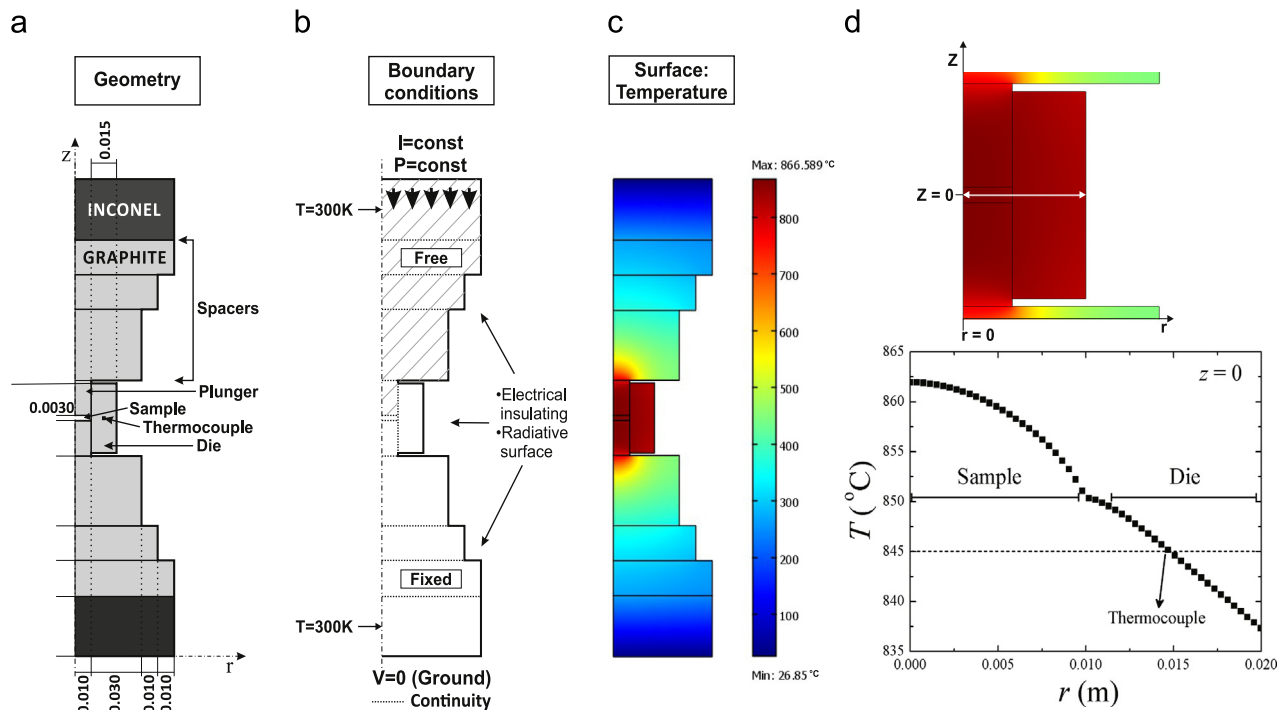


Fig. 1. (a) Schematic drawing of the consolidation system, (b) boundary conditions, (c) simulation by FEM of the temperature distribution of the whole system (see details in the text), and (d) expanded view of the temperature distribution of the die-sample region and the radial temperature profile for $z=0$.

the temperature dependence of the electrical resistivity, $c_p(T)$ is the heat capacity as a function of temperature, and $k(T)$ is the thermal conductivity. Values for each parameter are assigned for each material or domain in the geometry, i.e., the inconel, the graphite, and the Bi-2223 sample. Table 1 displays the parameters used in the FEM simulations for the Bi-2223 sample [4,9]. The parameters used for both the graphite and the inconel 600 are reported elsewhere [6,8]. The initial and boundary conditions used for solving Eqs. (1) and (2) are displayed in Fig. 1(b). The initial temperature was set to be 300 K and the heat losses by conduction and/or convection through the gas were neglected because the process occurs in vacuum. All the free surfaces exposed to the vacuum chamber have heat losses by radiation, given by $\dot{q}_{rad} = \sigma_s \epsilon (T_d^4 - T_0^4)$, where T_d is the temperature of the free surfaces, σ_s is Stefan–Boltzmann's constant, $\epsilon=0.3$ (0.69) is the graphite (inconel 600) emissivity, and $T_0=300$ K is the temperature of the wall of the chamber. The temperature of both the upper and lower inconel electrodes was 300 K and the electrical current density is injected from the top to the bottom. Finally, the electro-thermal equations (1) and (2) were solved by the FEM by using the COMSOL Multiphysics™ package.

Powders of $\text{Bi}_{1.65}\text{Pb}_{0.35}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ (Bi-2223) were prepared by the traditional solid state reaction method, as described elsewhere [4]. The final consolidation of the samples was performed in a SPS 1050 Dr Sinter[®] apparatus. Powders of Bi-2223 were placed inside a cylindrical graphite die, between two graphite plungers, as displayed in Fig. 1(a). The die was then placed inside the chamber of the SPS apparatus and sintering was performed under vacuum (roughly from 10 to ~ 30 Pa) and an uniaxial pressure along the z -axis of 50 MPa. In order to study the influence of the consolidation temperature, T_D , the samples were subjected to different temperatures $T_D=750$, 830, and 845 °C. The above samples were labeled as **S75**, **S83**, and **S85**. The heating rate was $\text{HR}=145$, 160, and 163 °C/min, and the dwell time was 5 min for all samples. Further details of the consolidation process employed for producing the samples are described elsewhere [4]. Additionally, for comparison reasons, ~ 4 g of the starting powder was cold

Table 1

Parameters of the Bi-2223 phase used in the FEM simulations [4,9]: ρ is its density, $\rho_e(T)$ is the electrical resistivity as a function of temperature, $c_p(T)$ is the heat capacity as a function of temperature, and $k(T)$ is the thermal conductivity.

Properties	Value	Units
ρ	5700	kg/m ³
$\rho_e(T)$	$0.8 \times 10^{-6} + 0.8 \times 10^{-8} T$	Ωm
$c_p(T)$	$131.6 + 0.77 T$	J/kg K
$k(T)$	$0.27 + 1.95 \times 10^{-3} T$	W/m K

pressed inside the SPS apparatus and the resulting pellet was sintered at 845 °C in air for 2400 min. This sample (**R84**) will thereafter be referred as a *reference sample*. Finally, the density, ρ , of all pellets was determined by the Archimedes method.

The phase identification was evaluated, in both powder and bulk samples, by means of X-ray diffraction patterns obtained in a Bruker-AXS D8 Advance diffractometer. These measurements were performed at room temperature using $\text{Cu K}\alpha$ radiation in the $3 \leq 2\theta \leq 80^\circ$ range with a 0.05° (2θ) step size, and 5 s counting time.

The temperature dependence of the electrical resistivity, $\rho(T)$, and the current–voltage characteristics $I \times V$ measurements were performed in a closed cycle cryogenics refrigerator ARS-4HW/DE-202N attached to a temperature controller Lakeshore model 331S. Typical dimensions of the samples were $t=0.5$ mm (thickness), $w=2$ mm (width), and $l=10$ mm (length). More details of these measurements are given elsewhere [4].

The optimum sintering temperature for producing single-phase Bi-2223 is $T_s \sim 845$ °C [5]. In addition to this, samples sintered at temperatures below 845 °C usually exhibit extra phases, as Ca_2PbO_4 , Bi-2201, and Bi-2212, and those sintered above T_s , due to the presence of liquid phase, are composed of at least two main phases: Bi-2223 and Bi-2212 [5]. Motivated by these experimental results, the conducted simulations of the SPS process were performed by assuming $T \sim 845$ °C in the position of the thermocouple, i.e., at $z=0$ and $r=0.015$ m (see Fig. 1(a)). Under these

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