Control on phases formation in melt processed (Bi,Pb)-Sr-Ca-Cu-O samples due to Ag and PbO additions

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INTRODUCTION

In order to produce bulk HTS pieces, the basic and fundamental studies of those HTS superconducting material is extremely important. When studying and analyzing the behavior of a material it ir important also to consider , from a certain perspective, what will be the behavior of the same material when produced on a commercial scale and estimate the expenses for production in low, medium and large scale.

Bulk HTS pieces are produced for a variety of applications such as short-circuit current limiters, superconducting motors and generators, current leads and magnetic "levitators" for frictionless bearings, magnetic levitation trains (MAGLEV's) and flywheels for "energy storage".

METHODOLOGY

Manufacture of the Precursor Material of (Bi, Pb) -2223

Initially, a box furnace (JUNG) was mapped with a multimeter connected to a Cromel-alumel alloy thermocouple positioned in the center of the furnace box (site designated for heat treatment material placement). The furnace was heated from 27 °C to 850 °C (± 2 °C) and a table was constructed relating the values of the indicated temperatures in the digital display of the furnace box with the readings on the multimeter connected to the thermocouple. It was possible to verify that there is a real increase of 15 ºC in the readings, higher than the value indicated in the digital display of the oven.

After understanding of the furnace control, a mixture of the precursor oxides Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, CuO (Alfa Aesar, 99.99%) in the ratio of 1.84: 0.32: 1.84: 1.97: 3 respectively . These reagents, in the form of powder, were weighed in the proportions indicated, on a high performance scale analytical model of the manufacturer Ohaus. These powders were mixed and macerated in a pestle mortar, both of agate.

After these oxides had been mixed homogeneously, their mixture was taken to the box furnace, where it underwent a series of thermal treatments aiming the formation of the phase (Bi,Pb)-2223. Each treatment consisted of a heating from the ambient temperature, rate of 300 °C/h, until a certain temperature "TP" is reached. From this moment on, na isotherm was applied for a certain time interval t_P. Thereafter the material was cooled to room temperature to be subsequently macerated and mixed and then subjected to a further treatment cycle. A summary is shown in Figure 01. Samples of the material obtained at the end of each cooling, including the precursor material, were analyzed by X-ray diffraction (XRD) using a PANALYTICAL diffractometer, model X'PERT-PRO and also a BRUKER D8 Discover diffractometer.

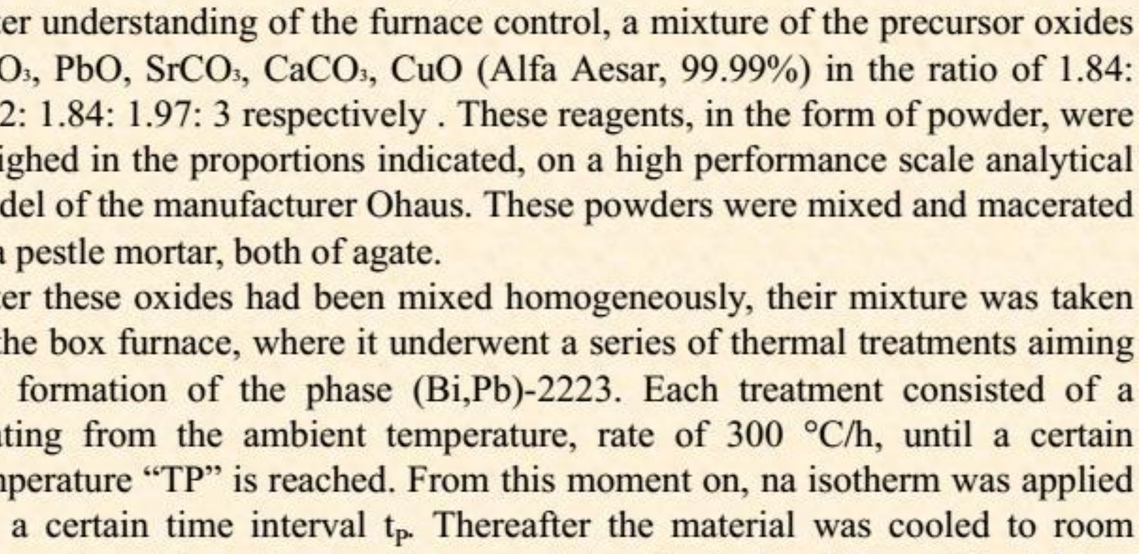

Figure 01. Schematic heat treatment process of the precursor phase (Bi, Pb) -2223. After the process shown in figure 01 D, the sample was formed and that last heat treatment was repeated.

RESULTS AND DISCUSSION

Irrespective of the amounts of Ag added to the blend, the start and end regions (Curves starting and ending at the same temperatures) of the peritoneal decomposition of the materials are close (~1 °C) as shown in Figures 02 and 03. It is possible to assume that the PbO may have induced a phase change region. However, it is possible to verify that Ag delimits the phase transformation energy during the cooling, as it is possible to observe in the DTA, due to the appearance of three peaks of energy that are accentuated according to the decrease in Ag content or increase in the percentage of PbO in mass. The reverse may be causing an increase in energy absorption (peaks appearing during cooling) and attenuating the surrounding regions at 872 °C and 840 °C and decreasing at the peak located near the temperature of 880 °C.

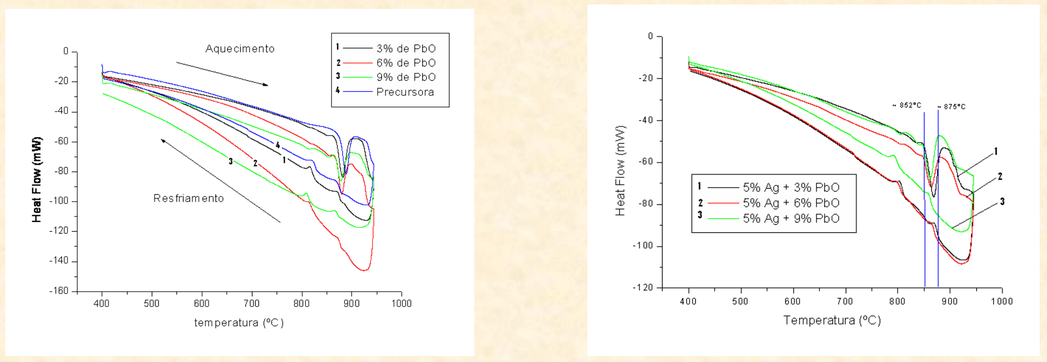

Figure 02: Comparative DTAs - show the behavior during the cooling and heating of Ag and PbO additives.

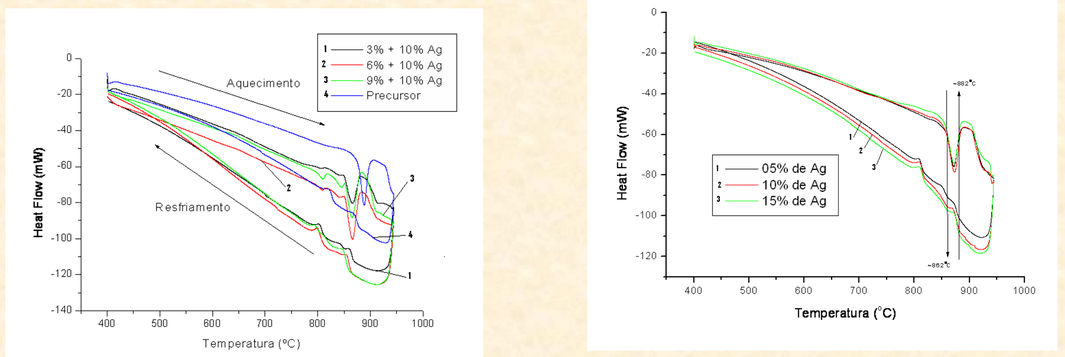

Figure 03: Comparative DTAs - show the behavior during the cooling and heating of Ag and PbO additives combined with their change.

CONCLUSIONS

By analyzing the recrystallization behavior it was possible to plot the evolution of the percentage growth of the phase (Bi, Pb) -2223. As shown in Figures 04 and 05, it was possible to show through this study, on the recrystallization of the phase (Bi, Pb) -2223, that it is possible to find a thermodynamic equilibrium window for the phase, and this is found through the following Heat treatment: heating rate 5ºC / min, peritoneal decomposition level, with total absence of the phase (Bi, Pb) -2223 at the end of 2h at temperatures of 884ºC (without additives), 872ºC (with addition of 5% Ag And / or 6% PbO by mass), then with a cooling rate of 0.1 °C/min, annealing between 850° C-855 °C/10h, 20h, 40h, and extending to higher values of time.

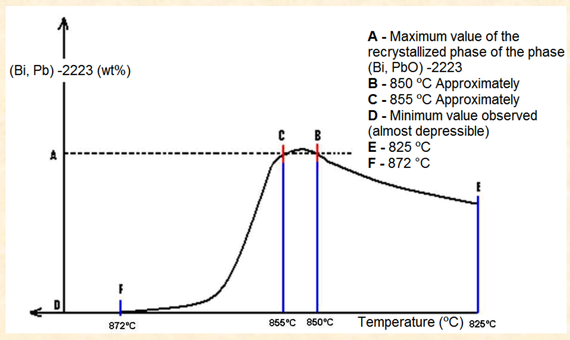

Figure 04: Hypothesis on the behavior of phase recrystallization (Bi,Pb)-2223 from the experimental results.

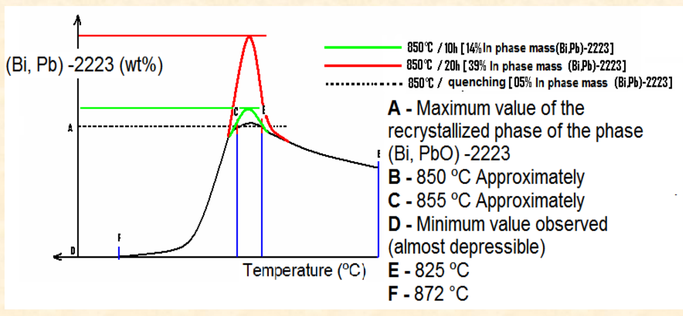

Figure 05: Hypothetical representation of the phase recrystallization behavior (Bi, Pb) -2223 with respect to the annealing temperature variation.

***Acknowledgments**: UFRRJ, UNISUAM and CEPEL for financial support. To Drs. A. R. Bigansolli, A. Polasek and F. Rizzo for valuable discussions in the initial studies. To P&D ANEEL D712:SUPERCABO Project for funding the upgrade in LMDS/UFRRJ* facilities.

References

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