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QUANTITATIVE ANALYSIS OF PHASES PRESENT IN PARTIALLY MELT PROCESSED (Bi, Pb) - 2223 SUPERCONDUCTOR

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Abstract – The study of formation of the superconducting phase (Bi, Pb)-2223 by partial melting and recrystallization aims at improving the density and microstructure of this material. In the present work, X-Ray diffraction was used for phases characterization and the Rietveld Method was employed for refinement of the structures and quantification of the phases present in melt-processed samples. We analyzed the precursor powder, which contained $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (90% of the sample). The precursor powder was heat-treated at 820-890°C with addition of 0-20% of Ag and PbO. Partial recovery of Bi-2223 was attained in melt-processed samples. The Bi-2212, Bi-2201, $(Sr,Ca)_2CuO_3$, Ca_2PbO_4 and Ag phases were also found.

Since the discovery of high-temperature ceramic superconductors, numerous researches have been carried out in order to characterize the properties of these materials. High-temperature superconductors of the Bi-Sr-Ca-Cu-O (BSCCO) system have received special attention because of their relatively high transition temperatures and high critical current densities.

In the present work, the Rietveld Method was used for structure refinement and phases quantification in melt-processed $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ ((Bi,Pb)-2223) samples. The melt-processing consisted of the peritectic decomposition of (Bi,Pb)-2223 into liquid and solid phases, followed by further slow cooling down to the temperature range of formation of (Bi,Pb)-2223. The precursor powder was prepared by solid-state reaction. High purity powders of Bi_2O_3, $SrCO_3$, PbO, $CaCO_3$ and CuO were thoroughly mixed in suitable compositions, being then calcined at 700 - 800°C for 12 - 60h in air and sintered at 830 - 870°C in air for 100 - 200h [1]. The sintered precursor was mixed with silver and lead oxide, being further melt-processed at 820 - 900°C in air. Partially melted samples were quenched and investigated by X-ray difraction (XRD). The Rietveld Method was employed for refining the XRD patterns. The precursor powder showed high fraction of (Bi,Pb)-2223 (above 90%), besides $Bi_2Sr_2CaCu_2O_y$ (Bi-2212) and $Bi_2Sr_2CuO_z$ (Bi-2201) (Figure 1). Magnetic Susceptibility Analysis showed T_c = 108K confirming the presence of superconducting Bi-2223 in the precursor (Figure 2). Partial recovery of (Bi,Pb)-2223 (up to 30-50%) was attained by melt-processing. The Bi-2212 (7-70%); Bi-2201 (2-15%), $(Sr,Ca)_2CuO_3$ (10-30%), Ca_2PbO_4 (4-15%), and Ag (5-20%) phases were also found in partially melted samples.The addition of Ag lowers the decomposition temperature, but the addition of Pb increases the formation of Ca_2PbO_4 between 830-840ºC and between 850-860ºC. The Bi-2212 phase shows the same behavior in these temperature ranges. The $(Sr,Ca)_2CuO_3$ content increases at temperatures above 860ºC. The optimization of the melt-processing requires a better knowledge of the Bi-2223 / melt equilibrium.

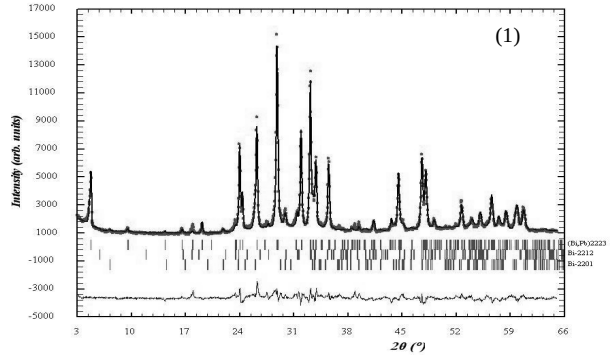

Figure 1: Rietveld analyses of (Bi,Pb)2223 precursor

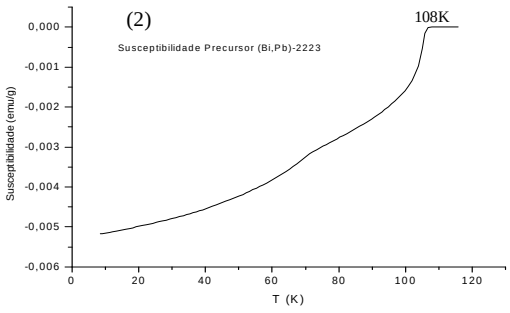

Figure 2: Temperature dependence of AC susceptibility of the precursor (Tc = 108K).

References

[1] T. Hatano, K. Aota, S. Ideda, K. Nakamura and K. Ogawa Japanese Journal of Applied Physics 27 (1988), pp 2055-2058