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Phase Evolution and Superconducting Properties of Boron-doped (Bi,Pb)-2223 HTSs

N G Margiani[1], I R Metskhvarishvili[2], T D Medoidze[1], N A Papunashvili[1], D I Dzanashvili[3] and G A Shurgaia[1]

[1]Department of Coherent Optics and Electronics, Vladimer Chavchanidze Institute of Cybernetics, Georgian Technical University, S. Euli str. 5, Tbilisi 0186, Georgia

[2]Department of Physics, Faculty of Exact and Natural Sciences, Iv. Javakhishvili Tbilisi State University, Chavchavadze Ave. 3, Tbilisi 0128, Georgia

[3]Laboratory of Physicochemical analysis, R. Agladze Institute of Inorganic Chemistry and Electrochemistry, Iv. Javakhishvili Tbilisi State University, Mindeli str. 11, Tbilisi 0186, Georgia

E-mail: nmargiani@gmail.com

Abstract Two series of the nominally pure (control) and boron-doped (Bi,Pb)-2223 HTSs with composition $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ (x=0, 0.05, 0.5, 1.5) were prepared under the different conditions: in an alumina crucibles and on an alumina plates. The influence of boron-doping as well as annealing conditions on the high-T_c 2223 phase evolution was studied using X-ray diffraction (XRD), resistivity and AC susceptibility measurements. Obtained results indicate that boron dopant drastically accelerates the formation of (Bi,Pb)-2223 HTS synthesized in an alumina crucibles. The boron-doped sample with x=0.5 revealed significant improvement in the T_c compared to the control sample (from 72K up to 100K). On the other hand, the additives of boron (x=0.05 and 0.5) have shown to have a beneficial effect on the formation of (Bi,Pb)-2223 HTSs prepared by the heat treatment of precursors on alumina plates, although do not essentially affect the critical temperature T_c=102 K of nominally pure compound.

1 Introduction

Synthesis of a nearly pure single-phase high-T_c 2223 phase (T_c≈110K) is a complicated process due to its extremely narrow stability range, which results in the formation of intergrowth of the multiple phases co-existing with the 2223 phase [1-2]. Partial substitution of Bi by Pb is the most widely used method to enhance the formation of the Bi-2223 phase [3]. However, it usually takes many days to produce an appropriate fraction of Bi-2223 HTS material in the final product. Moreover, it was found that the high-T_c 2223 phase is very sensitive to the partial pressure of oxygen and both O_2 and ambient sintering atmospheres suppress the formation of this compound [4]. The effects of boron doping in Bi-2223 ceramics have been reported in [5-6]. Long sintering time above 200 h in the Ar-7.5% O_2 atmosphere and rapid cooling out of furnace are required to improve slightly the formation of (Bi,Pb)-2223 phase in the boron-doped specimen [5].

How to further promote formation of 2223 phase, i.e. how to establish simpler and less expensive processing routes are very important subjects of study in order to achieve the acceptable levels for

certain technological applications. No comparative studies on the boron-doped (Bi,Pb)-2223 precursors heat treated in alumina crucibles and on alumina plates have been reported yet. The purpose of the present paper is to investigate comparatively the influence of B_2O_3 addition on the formation of (Bi,Pb)-2223 HTSs prepared via annealing of the same precursors under the different conditions: in an alumina crucibles and on an alumina plates.

2 Experimental

For comparison, two series of the nominally pure and boron-doped (Bi,Pb)-2223 HTSs with the same nominal compositions $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ (x=0, 0.05, 0.5, 1.5) were prepared by the solid state reaction method in an alumina crucibles and on an alumina plates. The appropriate mixtures of Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$, CuO and B_2O_3 were thoroughly ground and calcined at 850°C for 40 h in air. The resulting materials were pressed into pellets under hydrostatic pressure at 29 MPa and annealed at 850°C for 15 h in air, then cooled to room temperature in the furnace. The synthesized compounds were characterized by powder X-ray diffraction (XRD) analysis using the Dron-3M diffractometer (CuK_α radiation). The resistivity as a function of temperature, ρ(t), and temperature dependence of the AC susceptibility, χ'(T), were also measured [7].

3 Results and discussion

XRD patterns of the undoped and boron-doped specimens are presented in Figure 1. The dominance of the low-T_c 2212 phase over the high-T_c 2223 phase was observed in the undoped sample heat-treated in an alumina crucible. The intensities of the peaks attributable to the 2223 phase become higher in the same compound prepared on an alumina plate. With the increasing of boron-doping level, the 2223 phase is enhanced in both series and its increase is associated with the decrease of the 2212 phase. Although, the weak peaks of the very low-T_c 2201 phase appear and are intensifying with increasing a doping level. It could be seen from Figure 1, that samples with the highest boron concentration in both series of (Bi,Pb)-2223 HTSs, x=1.5, mainly consist of the 2201 and low-T_c 2212 phases with trace amount of high-T_c 2223 phase in the sample prepared in an alumina crucible.

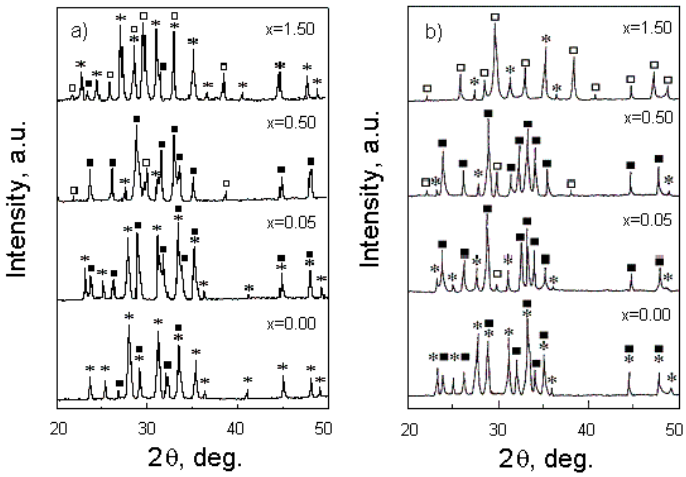

Figure 1. XRD patterns of $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ samples. (a): heat-treated in alumina crucibles; (b): heat-treated on alumina plates. ■ - 2223 phase, ∗ - 2212phase, □ -2201 phase.

Figure 2 represents the temperature dependence of resistivity, ρ(t), for the undoped and B-doped (Bi,Pb)-2223 samples. Onset temperature T_c^{on} of the superconducting transition is about 110 K for all the samples. For the undoped specimen heat-treated in an alumina crucible zero resistivity is reached at T=72K. It is well known that the 2212 compound is able to accommodate relatively high amounts of interstitial oxygen, which are very mobile and contribute to doping the CuO_2 planes with holes [8-9]. The broadened transition with relatively low T_c value indicates the presence of the overdoped 2212

phase in this material which possess a surfeit of oxygen (an excess of holes) due to the sintering in an ambient atmosphere. It is seen that low level boron-doping, x=0.05, drastically enhances the critical temperature by 24 K upto 96K compared to an undoped specimen and T_c value reaches 100K for the next concentration x=0.5. For the undoped specimen heat-treated on an alumina plate zero resistivity is reached at T_c^{off}=101.6 K. This value is by 30 K higher than that for the same composition prepared in an alumina crucible. T_c^{off} remains near 102 K for the low level boron-doping x=0.05, then drops to 99 K at x=0.5. The ρ(t) dependences for x= 0.05 and 0.5 clearly exhibit a second step (a smooth part) below 107 K, reflecting the decrease of the intergrain transition. This behavior may be related to the appearance and increase of the secondary 2201 phase upon increasing boron concentration. Presence of the 2201 phase in these compositions deteriorates a coupling between superconducting grain boundaries and leads to the reduction of the transition of intergrain Josephson junctions. Boron-doping level x=1.5 results in a substantial degradation of the (Bi,Pb)-2223 phase and leads to the sharp increase in the normal state resistivity. Occurrence of 2201 phase as a dominant is believed to be responsible for such behaviour of the resistivity.

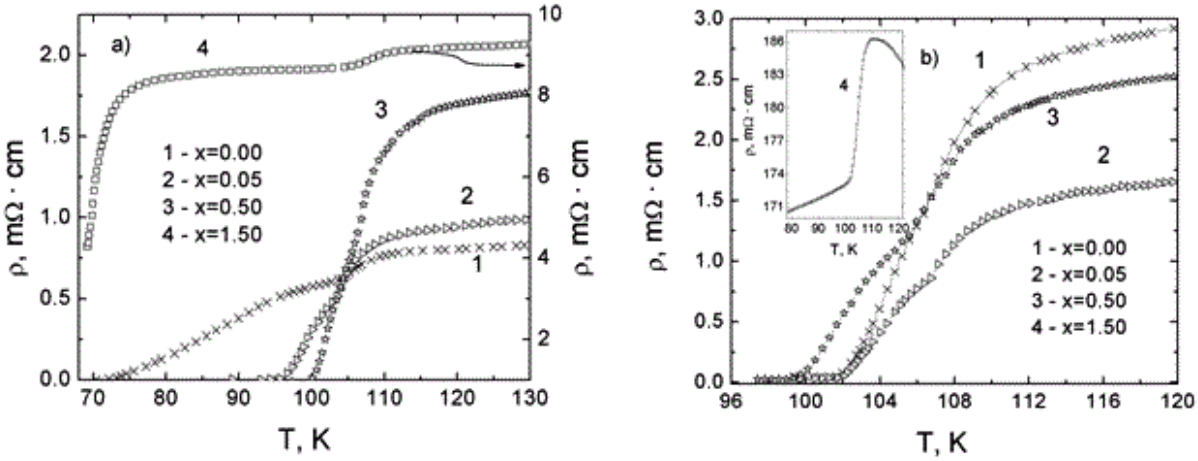

Figure 2. Resistivity versus temperature curves of $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ samples. (a): heat-treated in alumina crucibles; (b): heat-treated on alumina plates.

Figure 3 illustrates the temperature dependence of the AC susceptibility (χ') for control and boron doped samples. The diamagnetic onset temperature is equal to 111K for all the samples, indicating that the high-T_c 2223 phase is present in all the samples. Although due to the insignificant amount of the high-T_c phase, diamagnetic response is not clearly observable in this temperature range for the samples with the highest boron content x=1.5. The insets of Figure 3 (a and b) show χ'(T) curves for x=1.5 in the narrower temperature range.

It could be seen that the χ'(T) curves show a two-step behavior. Superconducting grains first shield the applied magnetic field, whereas the transition at relatively lower temperature is originated by the weak coupling between the grains. Undoped sample prepared in an alumina crucible exhibits a weak diamagnetic signal due to the presence of the small amount of the 2223 phase and absence of bulk diamagnetism in the temperature range 80-100K. Onset of the second transition for samples prepared on an alumina plates decreases gradually with increasing doping level and falls from 105 K to 97 K for x=0.5. From the previous considerations it is possible to state that even for the same compositions high-T_c phase formation kinetics may change considerably as the condition of oxygen incorporation into sample changes. Annealing of nominally pure 2223 on the alumina plate, instead of the alumina crucible allows an uptake of the optimal oxygen content into matrix, which leads to the prominent enhancement of the high-T_c 2223 phase formation and critical temperature.

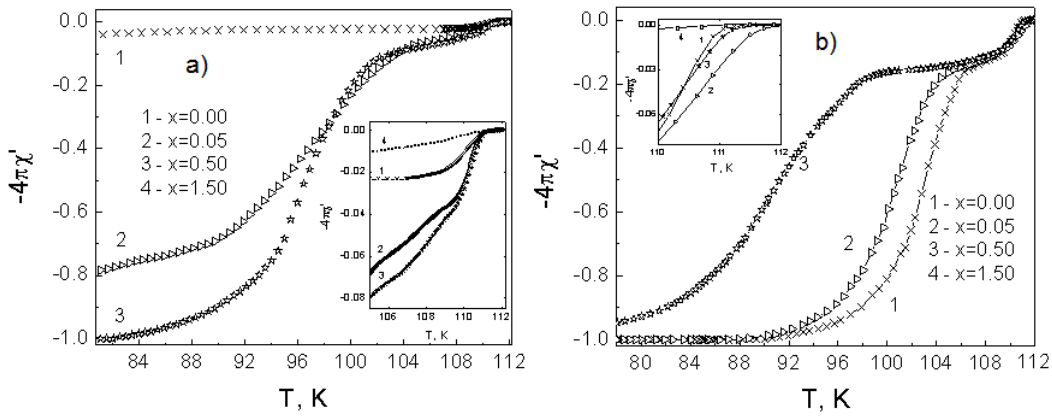

Figure 3. Temperature dependence of real part of the AC susceptibility at 1 Oe, *f*=20 kHz and H=0. (a): heat-treated in alumina crucibles; (b): heat-treated on alumina plates.

As for the boron-doped $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ (x=0.05 and 0.5) precursors that are thermally proceeded on the alumina plates or in the alumina crucibles, additional oxygen enters into the compositions at the boron-doping process. It is also possible that due to its very low melting point, T_m=450 ^oC, B_2O_3 additive acts as a flux during the process of annealing.

4. Conclusion

In this paper we present the preliminary results concerning the superconducting properties of the (Bi,Pb)-2223 HTSs prepared by the heat treatment of $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ (x=0-1.5) precursors in an alumina crucibles and on an alumina plates. Boron-doping promotes the formation of (Bi,Pb)-2223 phase using an alumina crucibles and leads to the drastic increase by about 30 K of the T_c value compared to the undoped sample. Boron-doped (Bi,Pb)-2223 HTSs prepared by the heat treatment of $Bi_{1.7}Pb_{0.3}Ca_2Sr_2Cu_3B_xO_y$ precursors (x=0.05-0.5) on an alumina plates reveal enhancement of 2223 phase formation although B_2O_3 addition leads to the appearance of 2201 impurity phase and marked deterioration of connectivity between the superconducting grains. The higher-level boron doping, x=1.5, leads to the transformation of high-T_c 2223 phase into the low-T_c 2201and T_c 2212 phases.

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