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A Study on the Effect of Ni Doping on $Bi-Pb-Sr-Ca-Cu-O$ System

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Abstract

The effect of Ni doping on superconductivity properties of the $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}Ni_x)_3O_y$ system has been investigated by means of x-ray diffraction, ac electrical resistance, ac magnetic susceptibility and critical current measurements. The volume fraction of the 2223 phase decreases with increasing Ni concentration. The zero-resistance temperature and the critical current density are suppressed with Ni substitution.

1. Introduction

In $Bi-Sr-Ca-Cu-O$ (BSCCO) systems, two high temperature superconducting phases are commonly observed, i.e. $Bi_2Sr_2Ca_2Cu_3O_{10}$ (2223) and $Bi_2Sr_2Ca_1Cu_2O_8$ (2212) with transition temperature [1] T_c at 110K and 85K, respectively. The (2212) and (2223) phases have double and triple layers of CuO_2 in the sub-unit cell, respectively, and more CuO_2 planes are believed to be associated with higher values of T_c. It is therefore important to enhance the (2223) 110K phase by using various methods of preparation [2, 3] or by doping. In fact, Ag and Pb doped in Bi-type superconductors [4,5,6] have been reported and the latter member Pb has been found to be a rather good dopant in enhancing the 110K phase. R.N. Kum et al. have shown that doping with Sn [7] and Ge [8] a BPSCCO system results in a decrease of superconductivity volume fraction of the (2223) phase. Therefore, substitution at copper site is an important step in determining the factors influencing the superconducting properties. In this study we have investigated the effect of Ni doping on the $Bi(Pb)-Sr-Ca-Cu-O$ sintered samples.

2. Experimental Procedure

The samples under study were prepared by conventional solid-state rection. $Bi_2O_3(99.99\%), PbO(99.999\%), SrCO_3(99.99\%), CaCO_3(99.9\%), CuO(99.9\%)$ and NiO (99.99 %) powders were used as starting materials. The powders with the molar ratio of $[Bi] : [Pb] : [Sr] : [Ca] : [Cu] : [Ni] = 1.7 : 0.3 : 2 : 2 : 3(1-x) : 3x(x = 0.0 - 0.030)$ were well mixed for 1 h. After mechanically mixing in an agate mortar the final mixture in powder form was calcined twice at $800°C$ for 24 hours. The calcined material was reground and pressed into pellets of 13 mm in diameter. Then the pellets were sintered at $840°C$ for 144 hours with two intermediate grindings and then cooled to room temperature at a rate of 2 $°C.\min^{-1}$. Finally, the pellets were cut into rectangular bars for measurements of the resistance and the critical current and they were shaped cylindrically for the ac magnetic susceptibility measurements.

The ac electrical resistance measurements were made by using a standard four probe method, and contacts were made by using silver paste. The temperature was monitored with an accuracy of $\pm 0.05K$ using a calibrated Pt-thermometer from Lakeshore. The bulk samples for critical current measurements were cut into rectangular bars from the pellets and they were thinned by using abrasive papers. The transport critical current density J_C were resistively measured in zero field cooled (ZFC) condition. The critical currents were determined by a voltage criterion of $1\mu V/cm$. The ac complex susceptibility $(\chi' - i\chi'')$ of the samples were measured by a mutual inductance bridge, using a two-phase model SR530 Lock-in amplifier to pick up in-phase and out-of-phase signals from the secondary coils. The ac magnetic field in the exitation coil was $h(t) = h_O \sin 2\pi ft$ with frequency $f = 1kHz$ and the amplitude of $h_O = 120mOe$. The heating rate in measurements was nearly $0.5K/\min$. In-phase and out-of phase components of the signal were corrected with respect to empty sample holder and were nullified at 120K.

X-ray diffraction patterns of the samples were obtained with Rigaku D.Max.3CXRD by using monochromatic CuK_α radiation in the $2\theta = 20° - 60°$ range.

3. Results and Discussion

Resistance versus temperature plots for specimens are shown in Fig. 1. A maximum zero-resistance temperature of 104K is obtained for a pure specimen ($x = 0.00$) with onset T_C at 110K. The Ni concentration dependence of the zero-resistance temperature T_{CO} is shown in Fig. 2. The zero-resistance temperature decreases monotonically with increasing x up to 0.02, and becomes 94K at $x = 0.02$. However, for the sample $x = 0.03$ up to 77K superconducting transition was not observed. Normal state resistance of the sample increases with increasing Ni concentration.

Dong Han Ha et al. [9] found that T_C decreases monotonically from 83K ($x = 0.00$) to 73K ($x = 0.04$) in $Bi_2Sr_2Ca_1Cu_{2-x}Ni_xO_{8+y}$ single crystals, which is different from that for polycrystal $Bi_2Sr_2Ca_1Cu_{2-x}Ni_xO_{8+y}$ prepared by solid state reaction method [10]. Dong Han Ha et al. found that Ni atoms don't form clusters but substitute for Cu

uniformly in CuO_2 plane as atom. It is evident from neutron diffraction [11] and x-ray photoelectron spectroscopy measurements [10] that in the structure Ni atoms substitute for Cu atoms.

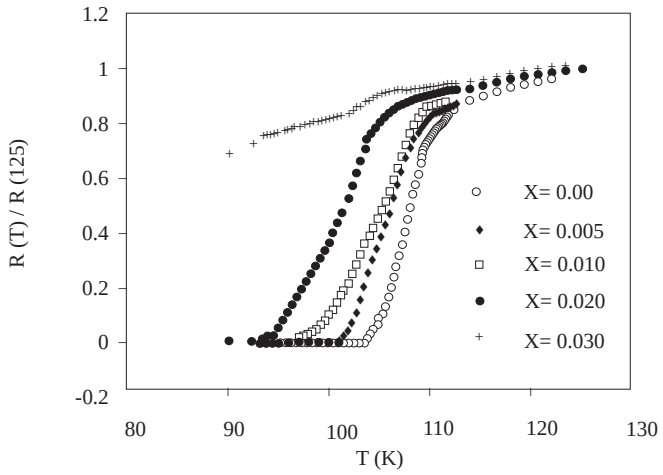

Figure 1. Temperature dependences of resistances of Ni doped $Bi_{1.7}Pb_{0.3}Sr_2Ca_2\ (Cu_{1-x}, Ni_x)_3O_y$ samples with $x = 0.00 - 0.03$

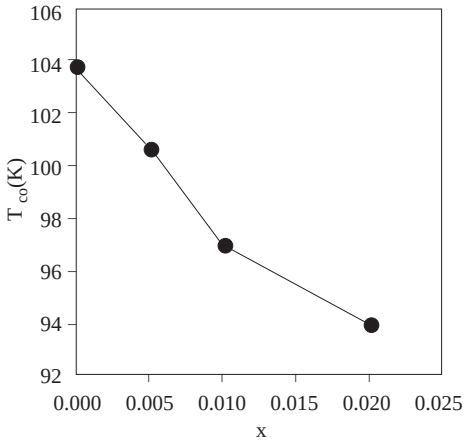

Figure 2. Variation of Tco of Ni doped $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}, Ni_x)_3O_y$ samples with $x = 0.00 - 0.02$

Figure 3 shows the room temperature XRD patterns observed for the undoped and doped samples sintered at $840°C$ and furnace cooled.

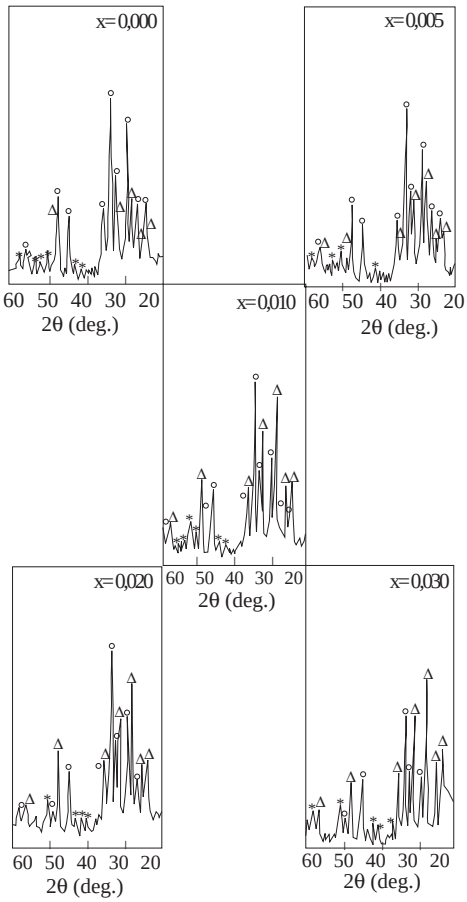

Figure 3. X-ray diffraction patterns of $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}, Ni_x)_3O_y$ o 2223 peaks, Δ 2212 peaks, * unknown peaks

The pure specimen ($x = 0.00$) shows a majority of 2223 phase with 2212 as a minor phase. The intensity of 2212 peaks increases with x. The $x = 0.03$ sample shows a nearly single 2212 phase. The XRD patterns show that the doping of Ni decreases the superconducting volume fraction of the 2223 phase.

The measurements of ac magnetic susceptibility, $\chi = \chi' - i\chi''$, have been widely used to characterize high T_C superconductors. These materials have been modelled as an array of Josephson-coupled strongly superconducting grains in which one distinguishes between intergrain and intragrain components, where the real part χ' represents the superconducting shielding, and a non zero imaginary part χ'' represents the energy losses in the material. The maxima on the $\chi''(T)$ curves originates from the hystresis of the material. The maxima on the $\chi''(T)$ curves originates from the hysteresis loss as the ac

field moves in and out of the superconductor. According to the Bean model, this loss becomes maximum when the superconductor volume is just penetrated by the applied magnetic field, that is, when the shielding current is equal to critical current. The average grain size, r_g, plays an important role in determining the sharpness of the $\chi'(T)$ and $\chi''(T)$ curves. The curves generally become sharp for very large grains ($r_g >> \lambda$, where λ is the penetration depth) and for very low values of h_O. Broadening of the superconducting transitions as measured by χ' is observed with decreasing grain size and also due to the poor connectivity between the grains [13, 14]. In particular, χ'' has been widely used to probe the nature of weak links and to understand intergranular and intragranular coupling.

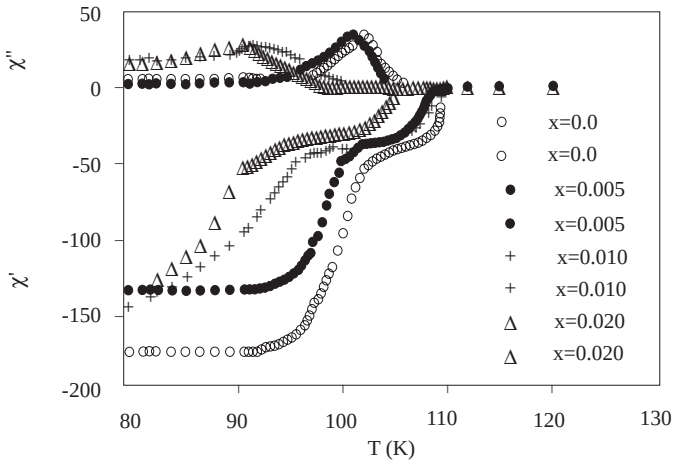

Figure 4. Temperature dependent ac magnetic susceptibility of Ni doped $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}, Ni_x)_3O_y$ samples with $x = 0.00 - 0.020$

The ac magnetic susceptibility measurements have been carried out at fixed amplitude of 120 mOe at a frequency of 1 kHz. Figure 4 shows the real and imaginary parts of the ac magnetic susceptibility against temperature for $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Ni_{3x}Cu_{3(1-x)}O_y$ (where x = 0.00, 0.005, 0.010, 0.020, and 0.030). The samples x = 0.00 and 0.005 show a first drop on the inductive transition curves corresponding to intragranular diamagnetic shielding at 110K. The inductive transitions of specimens x = 0.010 and 0.020 were observed 109K and 105.5K respectively. On the real part of the complex susceptibility curves of the samples with x = 0.010 and x = 0.020 a saturation following the first drop and corresponding to the intergranular region was not observed down to temperatures as low as 80K. The broadening at lower part of these curves may be due to the presence of other low T_C phase and non-superconducting inclusions.

The non-zero value of χ'' corresponds to losses occuring in the intergranular regions of the material. The intragranular component χ''_g was not apparent at the fixed magnetic

field amplitude (120 mOe). Figure 4 shows the change of the peak temperature (T_p) of χ'' due to Ni doping. The peak temperature decreases with increasing Ni content. The shift to lower temperature of loss peaks with increasing doping level indicates that the amount of magnetic flux which penetrates the superconductor increases with increasing Ni doping.

This behaviour indicates that the pinning of the magnetic flux in intergranular regions in the doped BPSCCO (2223) system is very weak. The Ni doping gives rise to a degradation of the intergranular current paths, seen as a shift on the lower part of the transition curves.

From the point of view of applications of high-T_C superconductors, one of the most important parameters is the critical current density. Bulk ceramic high T_C materials have the critical current densities limited to relatively low values by the grain boundaries.

Figure 5 shows the critical current density $J_C(T = 77K, H = 0)$ versus Ni concentrations. There is a decrease in J_C values of the samples with Ni doping. This results a weakening in superconductivity in the intergranular region of the sample. Doping with Ni might also cause a reduction in the pinning strength of the pinning centers which are specific to undopped BPSCCO.

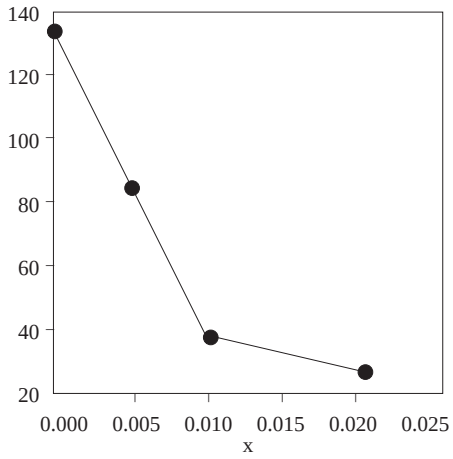

Figure 5. Variation of critical current of Ni doped $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}, Ni_x)_3O_y$ samples with Ni content

4. Conclusions

In summary, the effect of Ni doping on superconductivity properties of the $Bi_{1.7}Pb_{0.3}Sr_2Ca_2(Cu_{1-x}, Ni_x)_3O_y$ system has been investigated by means of x-ray diffraction, ac electrical resistance, ac magnetic susceptibility and critical current measurements. All of the measurements show that the doping of Ni decreases the superconducting

volume fraction of the 2223 phase. The zero resistance temperature T_{CO} decreased from 104K to 91K as a result of increasing Ni content. The $XRD, R(T), \chi(T)$ and $J_C(H = 0, 77K)$ results for the $Bi-Pb-Sr-Ca-Cu-O$ materials are found to be in a good unison. The substitution of Ni for Cu in the $Bi-Pb-Sr-Ca-Cu-O$ system has a negative effect on the phase composition and superconductivity of the samples. Our results are in good agreement with the other works [10, 15] in supporting this view.

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