Fe Doping effect on Structural Properties of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$CaCu$_{2-x}$Fe$_x$O$_{8+\delta}$

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Abstract

Since the discovery of the high temperature superconductivity in the Bi-Sr-Ca-Cu-O system [1], many investigations have focused on it to understand its properties and to improve them. Belonging to this system, the Bi$_2$Sr$_2$CaCu$_2$O$_{8+d}$ (Bi-2212) phase has been widely studied in the recent years in order to understand, by various kinds of substitution, the relation between the structure and the superconductivity. Most of these substitutions have concerned the Cu site in the superconducting layer CuO$_2$. Among these, as showed by G. Ilonca et al [2], the substitution by Fe kills the superconductivity at a lower concentration than Ni. At our knowledge, nothing has been made when there is in the same time a substitution by lead (Pb) on the Bi site. For that purpose samples of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$CaCu$_{2-x}$Fe$_x$O$_{8+\delta}$ with $x = 0-0.06$, have been prepared using the solid state reaction technique. The samples have been characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Structural analysis shows the influence of the Fe doping on the crystalline lattice structure of the samples.

Keywords: Bi-Pb-Sr-Ca-Cu-O system, Bi(Pb)-2212 phase, Pb and Fe substitution, high-$T_c$ superconductors.

1. Introduction

Doping in high temperature superconductors has been found to be of considerable importance. The properties of Bi-Sr-Ca-Cu-O (BSCCO) materials can be changed by substitution or addition of elements having different ionic radii and different bonding characters [3]. Belonging to this system, the Bi$_2$Sr$_2$CaCu$_2$O$_{8+d}$ (Bi-2212) phase has been widely studied in the recent years in order to understand, by various kinds of substitution, the relation between the structure and superconductivity. The effect of different dopant with different valence states has been reported to clarify whether any improvement of the temperature of transition ($T_c$) is possible [4]. Substitutions in BSCCO concerned mainly the Bi$_2$Sr$_2$CaCu$_3$O$_{10+d}$ (Bi-2223) phase and the Bi site with the aim of an easier preparation and

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an improved stability of the phase [5]. The partial replacement of bismuth by lead (Pb) allows synthesizing compound with stable superconductive properties and higher $T_C$. In the Bi-2212 phase, the Bi substitution by Pb results in a remarkable improvement of the superconductive properties. Moreover, if oxygen stoichiometry is well controlled, a great improvement of the density of critical current is possible up to 77 K [6-8]. Substitution by rare earth elements or magnetic or non magnetic impurities like Pr or Ba [9] in the Sr site was made by other studies. Substitution in the Ca site is, in general, carried out with an aim of preparing under doped samples. The substitution of Ca$^{2+}$ by Y$^{3+}$ results in a reduction of the concentration of holes in the CuO$_2$ planes [10]. Calcium can also be substituted by Pr as it is shown on Bi$_2$Sr$_2$Ca$_{1-x}$Pr$_x$CuO$_y$ by X. F. Sun et al [11]. The effect of the substitution on the Cu site is much stronger because it directly affects the superconductive properties by changes in the CuO$_2$ planes whereas substitution on Ca affects mainly the reservoir of charge block. A reduction in $T_c$ is observed by Y. K. Kuo et al [12] with a substitution as well by a magnetic impurity, such as nickel, or a nonmagnetic, one such as zinc. Doping by Ni affects also the physical properties in the normal state as shown by H. L. Liu et al [13] with a study of the optical properties in the ab plan. Compared to the substitution by Ni, substitution by Fe kills the superconductivity at lower concentration. There are, at our knowledge, few works dealing with substitution by lead (Pb) on the Bi site and in the same time a substitution on another site. Among these, a work about the Bi(Pb)-2212 phase doped with Ca$^{2+}$ [14] shows that the ferromagnetic and superconductive phases are not separated but constitute a unique phase. Substitution of Cu by Co or Zn in the Bi(Pb)-2212 phase results in a reduction of $T_c$ as it is shown by the work of M. K. Yu and J. P. Franck [15]. The purpose of this work is to study the effect of substituting Cu by Fe in the Bi(Pb)-2212 phase. Samples of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_{2-x}$Fe$_x$O$_{8+}\gamma$ with $0 \leq y \leq 0.05$ were prepared using the solid state reaction technique. The samples have been characterized by XRD, SEM and AC magnetic susceptibility measurements. Structural analysis shows the influence of the Fe doping on the crystalline lattice structure.

2. Experimental Procedure

Samples of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_{2-x}$Fe$_x$O$_{8+}\gamma$ with $0 \leq y \leq 0.05$ were prepared by a conventional solid-state reaction method. Powders of Bi$_2$O$_3$, PbO, SrCO$_3$, CaCO$_3$, CuO and Fe$_2$O$_3$, weighed in appropriate ratio, were mixed and grinded in an agate mortar. The obtained mixtures were calcined in a two steps procedure: the first at 800°C for 10h and the second at 810°C for 30h. The prepared powders were successively grinded, pressed in pellet shape under a pressure of 225 MPa and sintered twice at 840°C during 50h. The heating rate was 5°C/min. Structural properties and phase purity were characterized by XRD using CuKα radiation of copper ($\lambda_{CuKα}$=1.5418Å) in a Siemens D8-Advance type diffractometer operating in Bragg-Brentano geometry. Identification of the existing phases was carried out with help of JCPD-ICDD data file (International Center for Diffraction Data) [16]. The lattice parameters of the Bi(Pb)-2212 and Bi(Pb, Fe)-2212 phases were identified using Dicvol 04 software [17]. Microphotographies were taken using a Philips XL 30 Scanning Electron Microscope (SEM). AC susceptibility measurements were carried out with a commercial SQUID magnetometer operating at a frequency of 100 Hz and an applied magnetic field of about 0.6 G.

3. Results and Discussion

3.1. XRD analysis

The XRD patterns of the Bi-2212, Bi(Pb)-2212 and Fe doped samples ($y = 0.02, 0.04$) are shown in fig.1. Fig. 1a shows that the undoped sample consists mostly of the Bi-2212 phase with a parasitic phase detected for $2\theta=17.68^\circ$C and Bi-2201 parasitic phase detected for $2\theta=29.71^\circ$C and $31.39^\circ$C. Fig.1.b shows that the sample consists mostly of the Bi(Pb)-2212 phase with a Ca$_2$PbO$_4$ parasitic peak detected for $2\theta=17.68^\circ$C. The Bi-2201 phase is not observed in this pattern. XRD patterns of Fe doped samples (Fig. 1.c, Fig. 1.d) indicate that:
- When y increases the intensities of main picks began to increase then decrease for the last value;
- The 20 positions of the same peaks change;
- The peaks with small intensity disappear;
- The parasitic phase Bi-2201 disappears when the content y of Fe is equal 0.04.
- Alignment of all the grains according to the same orientation \((0 \ 0 \ l)\).

3.2. SEM Analysis

Fig. 2 shows SEM photographs of two samples, one without Fe \((y = 0)\) and the other doped with Fe \((y = 0.02)\). The SEM photograph of the undoped sample (Fig. 2.a) shows the layered structure which characterizes the growth of Bi-2212 grains. Small white nodules, due to some precipitate of the starting powders, can be noticed. The grains appear rather dense and well connected. The grains of the sample without iron have a flattened form and are aligned in the same direction. Their maximum size is lower than 5 μm. It is clear from the figure that the grains are connected with each other.

3.3. Susceptibility

Susceptibility measurement was done for all the samples and almost the same behaviour of susceptibility versus temperature curves was obtained. Fig. 3 shows that initially two phases Bi-2212 and Bi-2201 are present in the undoped sample Bi-2212. In the lead and iron doped samples there is only the Bi-2212 phase. The critical temperature of transition \(T_C\) estimated from the beginning of the diamagnetic signal is about 65 K and 63K in Bi(Pb)-2212 and Bi(Pb, Fe)-2212 respectively. Doping with Fe not only lowers the \(T_C\) but also broaden the transition width. This effect is accompanied by an enhancement of the purity of the phase.
4. Conclusion

In summary, Fe doping of Bi(Pb)2212 was possible for low concentration. The presence of lead allows substitution of Cu by Fe in a ratio forbidden in the free lead compound. This substitution is indicated by the change in the structural properties deduced from the XRD and SEM analysis. The doping level goes, together with Tc, through a maximum when the concentration of Fe increases. The results confirm that the superconductivity in Bi(Pb)2212 depends not only on the distance between the CuO2 planes but also on the doping level.

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