Effect of Magnesium on the Bi-based (2212) superconductors

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Abstract— A series of Mg-doped bismuth (2212) superconducting materials with nominal composition Bi\textsubscript{1-x}Pb\textsubscript{x}Sr\textsubscript{2}Mg\textsubscript{x}Ca\textsubscript{1-x}Cu\textsubscript{2}O\textsubscript{y} and Bi\textsubscript{2}Sr\textsubscript{2}Mg\textsubscript{x}Ca\textsubscript{1-x}Cu\textsubscript{2}O\textsubscript{y-δ} (x = 0, 0.05) were synthesized by simple solid-state reaction route. The effect of magnesium on structural properties and superconducting behaviour has been investigated by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), electrical resistivity X-ray diffraction experiments and SEM observations revealed the degree of texture in the superconductor. SEM photographs show that the addition of MgO affects the mechanism of the grains growth. In all samples, the a-lattice parameter remains the same, but the c-parameter decreases slightly with the content of MgO. All the samples appear to have a multiphase character as shown by AC magnetic susceptibility and XRD data. Mg was found to be effective in the formation of the high-Tc phase at 5\% of doping in this system. The onset temperature Tc (onset) of all the samples remains within the temperature range 80–86 K.

Keywords—Bi-based high-Tc superconductors; Scanning electron microscopy; X-ray diffraction.

I. INTRODUCTION

The bismuth-based family of high-Tc superconductors (HTS) has been extensively studied in the past 22 years, both from the point of view of its fundamental physical properties and from that of the thermodynamical stability of its different components. In fact, it is well known that the BSCCO material consists of at least three phases: Bi\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{δ} (Bi-2212), Bi\textsubscript{2}Sr\textsubscript{2}CaCu\textsubscript{2}O\textsubscript{6+δ} (Bi-2201) and Bi\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{2}O\textsubscript{6} (Bi-2223). The thermodynamical stability limits between them have been investigated in polycrystalline samples of macroscopic sizes [1] and this information has been exploited in order to design the most suitable processes for the synthesis of pure samples in the different phases. The critical current density (J\textsubscript{c}) and critical temperature (T\textsubscript{c}) are the parameters of primary importance for potential applications of HTS and the Jc value is primarily limited due to the insufficient flux pinning properties. Therefore numerous efforts have been made to enhance the flux pinning properties of Bi-based systems, and hence the Jc value, by introducing effective pinning sites, several alloying elements, such as Ag, MgO, BaSO\textsubscript{4}, SrSO\textsubscript{4}, etc., can be added to improve both the electrical properties and mechanical integrity of polycrystalline 2212, and the addition of MgO was reported to have beneficial effects on the grain connectivity and grain alignment.

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In order to improve transition temperature and critical current densities in Bi-2212 high temperature Tc, introduction of effective pinning centres into the superconductors is needed [5]. In this article, we discuss the effect of Mg doping on the crystal structures, the electrical properties of superconducting BiSrCaCuO, BiPbSrCaCuO ceramics.

II. EXPERIMENTAL PROCEDURE

The Mg doped (BSCCO; BPSCCO) ceramics were prepared by the conventional ceramic method. Predetermined amounts of high purity starting chemicals (Bi\textsubscript{2}O\textsubscript{3}; SrCO\textsubscript{3}, CaCO\textsubscript{3}, CuO, PbO) were used for the preparation of the ceramics of fixed nominal composition Bi\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{δ} \textsubscript{(Bi-2212)}, Bi\textsubscript{2}Sr\textsubscript{2}Pb\textsubscript{x}Sr\textsubscript{2}Mg\textsubscript{x}Ca\textsubscript{1-x}Cu\textsubscript{2}O\textsubscript{6-δ}. These powders were well mixed and ground by using an agate mortar and pestle and were further calcined at 800°C for 30 and 820°C for 30 hours in a furnace. Calcined powders were ground again to form a fine powder. Pellets 13 mm in diameter and 1 mm in thicknesses were pressed by uniaxial compaction in a die at 5 ton/cm\textsuperscript{2}. These pellets were sintered at 860°C for 60 hours in air and then furnace cooled to room temperature. The obtained Bi-2212, Bi(Pb)-2212 powders were mixed with MgO particles. The additional amount of MgO in this case varying from (x = 0- 5\%) of the total mass of the sample. The mixed powders were pressed into pellets under 5 ton/cm\textsuperscript{2} then sintered at 860°C for 60 hours. Phase identification was carried out with an X-ray diffractometer (DRX). The cell parameters were calculated from XRD patterns using Dicvol 06. Resistance-temperature data obtained by using four point probe AC method.

Keywords—Bi-based high-Tc superconductors; Scanning electron microscopy; X-ray diffraction.
III. RESULTS

All the samples appear to have a multiphase structure which indicates the difficulty in obtaining a single phase material by the conventional ceramic method. X-ray powder diffraction patterns of samples with 0% and 5% are shown in Fig. 1. The peak positions and intensities show that samples are essentially a mixture of Bi-2201, Bi-2212. Few small peaks may be indexed to CuO and MgO. Our synthesizing process described above produces, without MgO, but the reflection of MgO can not be detected by XRD. Bi-2212 with a minor amount of secondary phases. The peaks of the added Bi(Pb)2212 sample show dominancy of (00l) plans indicating a preferential orientation parallel to ab plans which implies that a single phase Bi-2212 is obtained.

This figure 1 shows also that the peak at 2θ = 29.08° for the Bi (Pb)-2212 increases with addition. This shows a remarkable improvement of the high-Tc phase fraction. A nearly single phase material could be synthesized in this way. Some Bi-2201 phase peaks [2θ = 21.2°, 30°, 36.6°] were obtained in the Mg-added composition. This is due to the fact that Mg particles added to the prepared Bi-80 K phase superconductor did not react with superconducting grains. The other secondary phase attributed to Ca2PbO4 phase is denoted by (*) symbol. It is detected at 17.5°.

The main X-ray peaks in Fig 2 may be indexed assuming a tetragonal cell with \( a = b = 5, 40079 \text{ Å} \), \( c = 30,82036 \text{ Å} \) as expected for the Bi-2212 structure. It can be found that the content of Bi2212 increases with increasing MgO concentration, indicating MgO addition in the samples accelerate the reaction rate of Bi2212. The lattice parameters of the Bi(Pb)2212 phase of sample can be compared with those of the other samples prepared by adding MgO. Calcinations of the samples at a temperature higher than the melting point of Bi 2O3 (818°C) causes loss of some of the Bi atoms, which results in the formation of the off-stoichiometric compound. For this reason, we preferred to add MgO rather than substituting it to the Bi site. There is a good possibility of vacant Bi sites being occupied by these atoms, thus offering which has serious implications for conductivity within the Bi2212/MgO.

Figure 3. shows SEM photographs of two samples, without magnesium (y = 0) and doped with magnesium (y = 0.05). The SEM photograph of the undoped sample shows the layered structure which characterizes the growth of Bi2212 grains. The grains appear rather dense and well connected. The average size of the grains is about 4 μm. The grain samples containing lead have a larger size and aspect very different. Their orientation seems less random and porosity
even smaller. Platelets have a strong cohesion. The addition of MgO appears to increase the porosity which is manifested in larger spaces between the grains. MgO particles cause agglomeration in heat treatment and lead to weak links at grain boundaries [8, 9].

The electrical resistivity was measured with a standard AC four-probe method (Fig.4.). Electrical leads were attached to the samples by silver paint and heat treated at 300 °C in air for half an hour, which gave contact resistances of order 1– 2Ω. The transition temperatures Tc with zero resistance for 5% Bi2212/MgO and 5% Bi(Pb)2212/MgO. changed due to the fact that Mg atoms in the samples with 5 wt% Mg were substituted for the elements that compose the Bi-2212 single-phase superconductor[6]. This is confirmed by the XRD patterns of figure 1, which do not exhibit an impurity phase but only a few Bi-2201 phase peaks.

The temperature dependence of the electrical resistivity of the Bi2Mg0.05Sr2Ca1Cu2Oy, Bi1.8Pb0.4Sr20.05Ca1.4Cu2+δOy ceramics addition by x=0.05 Mg is given in Figure 3. The onset temperature for two samples is approximately 80 K, 82 K. The resistivity-temperature curve is linear up to the onset temperature, in accordance with the metallic character of the samples. We can observe the resistance at normal state at room temperature is higher for pure samples than for added one. The higher resistivity is due to new scattering centers, which are introduced in the system by the dopant [7].

IV. CONCLUSION

We devoted our work, on one hand, to the synthesis and the characterization of superconducting ceramics of Bi2Sr2CaCu2+δOy, Bi1.8Pb0.4Sr2Ca1.4Cu2+δOy (y = 0.05) and, on the other hand, to the study of the effect of substitution of Cu by Zn. The samples were prepared with the usual method of solid state reaction. The XRD results show the obtaining with a majority fraction, of the Bi(Pb)2212 phase accompanied by Bi2201 and Ca PbO parasitic phases. By adding magnesium, the proportion of Bi2201 parasitic phase seems to initially decrease then to increase slightly. The indexing by Dicvol04 software confirms the tetragonal structure for the various samples. SEM microphotographs highlight the lamellar structure characterizing the grains of Bi(Pb)2212.
Linear dependence of the electrical resistivity above the onset temperature of superconductivity $T_c$ (onset) is showing the good quality of sample composition $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{Mg}_0.05\text{O}_{6+\delta}\$. A $\text{Bi}_{1.8}\text{Pb}_0.4\text{Sr}_2\text{Ca}_1.1\text{Cu}_2.1\text{O}_{8+\delta}$ superconductor was sintered by the solid state reaction method with Mg concentrations varying $x=0$ to $0.05$. It was found that the addition of MgO enhances the high $T_c$ $\text{Bi}(\text{Pb})2212$ formation phase and transport properties. The normal state of resistivity of addition sample with 5% Mg is enhanced. An increase of the resistivity from the overdoped regime to the underdoped doped one is observed. Their measurements showed that there was a large difference in the carrier concentration between the inner and outer $\text{CuO}_2$ planes due to the doping. The addition of MgO particles may possibly affect the oxygenation, the distribution of oxygen deficient regions in the sample [10].

REFERENCES


Fig.4. Temperature dependence of Resistivity for two phases: (a) $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, (b)$\text{Bi}_{1.8}\text{Pb}_0.4\text{Sr}_2\text{Ca}_1.1\text{Cu}_2.1\text{O}_{8+\delta}$.  

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