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Research articles

Bi-2212 high-T_c superconductor nanoparticles synthesized via wet-mixing method and its properties

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Abstract

We have synthesized a type of Bi-based high-T_c cuprate superconductors, $Bi_2Sr_2CaCu_2O_8$ and $Bi_{1.6}Pb_{,Pb}Sr_2CaCu_2O_8$, henceforth called Bi-2212 and (Bi,Pb)-2212, respectively, using wet-mixing method. Here we report the investigation of structure, superconductivity and magnetism on Bi-2212 and (Bi,Pb)-2212 nanoparticles. XRD result confirmed the presence of Bi-2212 as a dominant phase with the orthorhombic structure. SQUID measurement confirmed the presence of superconductivity of both samples by the observation of T_c onset at the temperature around 75 K and 78 K and displayed that (Bi,Pb)-2212 has higher superconducting volume fraction compared to that of Bi-2212. The difference between T_{irr} and T_{onset} is attributed to the difference flux pinning in both Bi-2212 and (Bi,Pb)-2212. Furthermore, nanoparticles of superconducting Bi-2212 exhibit ferromagnetism at room temperature.

1. Introduction

High-T_c cuprate superconductor is now still being studied since the wide application of this material, especially if the room-temperature superconductor could be found one day. Among high-T_c cuprate superconductor, the Bi-Sr-Ca-Cu-O or BSCCO is considered to be the most attractive superconducting system because of its high critical current density (J_c) and superconducting critical temperature (T_c) [1]. There are three different phases of BSCCO system that can be distinguished from the number of Cu-O layers (n), named $Bi_2Sr_2CuO_6$ (Bi-2201, n = 1), $Bi_2Sr_2CaCu_2O_8$ (Bi-2212, n = 2) and $Bi_2Sr_2Ca_2Cu_3O_{10}$ (Bi-2223, n = 3), with the T_c of 20 K, 80 K and 110 K, respectively [2, 3]. The superconducting properties of BSCCO phase can be controlled and improved by chemical substitution [4–9].

Among three phases of BSCCO system, Bi-2212 is the most stable and exhibits layered structure with strong anisotropy [10]. Therefore, the properties of Bi-2212, which extensively studied for various practical applications is fascinating to be explored [11–13]. As reported in some references, the substitution of Pb to Bi-site in Bi-2212, henceforth called $Bi_{2-x}Pb_xSr_2CaCu_2O_8$ or (Bi,Pb)-2212, was found to exhibit dramatically enhanced critical current density and flux pinning properties, with the optimal Pb substitution is situated near x = 0.4 [1, 14–18].

The quality and performance of superconducting materials strongly depend on the preparation procedure or the synthesis process. Synthesis results in Bi-2212 using various kinds of synthesis method display the presence of secondary phases indicating the difficulties to obtain the Bi-2212 single phase [10, 19–21]. Here in this letter, we report the study of structure, superconductivity and magnetism of nanoparticles Bi-2212 and (Bi,Pb)-2212 synthesized by wet-mixing method. The wet-mixing method ensures the homogenization on atomic scale and high possibly produces the growth of particles in nanometer size [22]. Besides, the nanoparticles of superconducting material exhibit ferromagnetism at room temperature as observed in other types of high-T_c cuprate superconductor [22, 23].

2. Materials and Method

The starting powders used in this study were Bi_2O_3, $SrCO_3$, PbO, $CaCO_3$, and CuO (all from Sigma-Aldrich Chemicals with a purity >99%). A nominal stoichiometry of Bi-2212 sample was chosen for the preparation of the precursors using wet-method. The same technique was also applied to synthesize Pb-doped Bi-2212 material ($Bi_{2-x}Pb_xSr_2CaCu_2O_8$, with x = 0.4), named (Bi,Pb)-2212. Each starting powder was dissolved into nitric acid (HNO_3). Further, the solutions from all dissolved starting powders were mixed then stirrer and heated continuously at ~80 °C and finally converted into precursor in the powder state. The drying process at 120 °C was also performed to remove the water content. The precursor powder was calcined at the temperature of 790 °C within 4 hours to remove organic impurities. Then, the calcined powder was sintered at the temperature of 840 °C. The sintering process is essential for the nucleation process and for reducing the size and number of pores.

The thermal analysis (TGA) was carried out to determine the decomposition temperatures. The EDX measurement was conducted to confirm the chemical compositions. The x-ray diffraction measurement was also performed to check the sample quality. TEM image of Bi-2212 sampe was collected to investigate the morphology and particle size. Further, we use Rietveld method using MAUD software to analyze the phase, structure, lattice parameter and particle size [24]. Finally, magnetic susceptibility $M(T)$ and $M(H)$ measurement was also carried out to check the magnetic and superconducting properties of the synthesized sample Bi-2212 and (Bi,Pb)-2212.

3. Results and Discussion

Figure 1 displays thermogravimetric analysis (TGA) of the precursor of Bi-2212, which is useful to determine the decomposition temperatures. The weight loss in the first stage is about 25 wt%, within the temperature range 20 °C to 250 °C, that can be attributed to the removal of adsorbed water. The next stage (15% weight loss) from 250 °C to 500 °C can be ascribed to the decomposition of mixed carbonates and to the formation of Bi-2201 phase. The third stage (8% weight loss) is from 500 °C to 580 °C and stays constant up to 1100 °C can be assigned to the crystallization and the grain growth of the prepared Bi-2212. It was argued that the Bi-2212 phase may be formed via the Bi-2201 phase by inserting CaO and Cu-O layers, making Bi-2212 is crystallography and thermodynamically more stable than Bi-2212 phases

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[11], there is a possibility of the decomposition to another phase. After several trials, the best temperature to obtain the optimum Bi-2212 phase was using calcination temperature of 790 °C and sintering temperature of 840 °C.

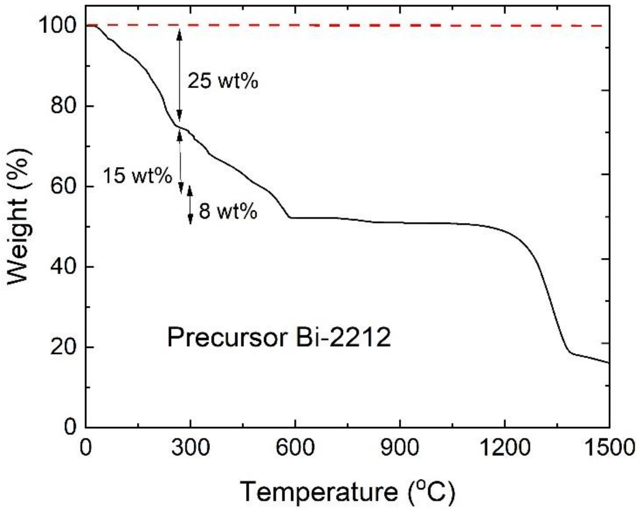

Fig. 1. Thermogravimetric analysis curve for Bi-2212. Temperature was measured from room temperature to 1500°C.

Figure 2 exhibits the EDX spectra of the powders Bi-2212 and (Bi,Pb)-2212 to confirm the chemical compositions of the synthesized samples. As shown in Fig. 2(a), Bi, Sr, Ca, Cu and O peaks are presented in Bi-2212 sample, and also in Fig. 2(b), Bi, Pb, Sr, Ca, Cu and O peaks are presented in (Bi,Pb)-2212 sample. The three highest peaks in the energy range of ~2, ~2.5, and ~3.8 keV belong to Sr, Bi, and Ca atom, respectively. The result of EDX image displays that there is no unwanted element. It implies that there is no contamination during synthesis.

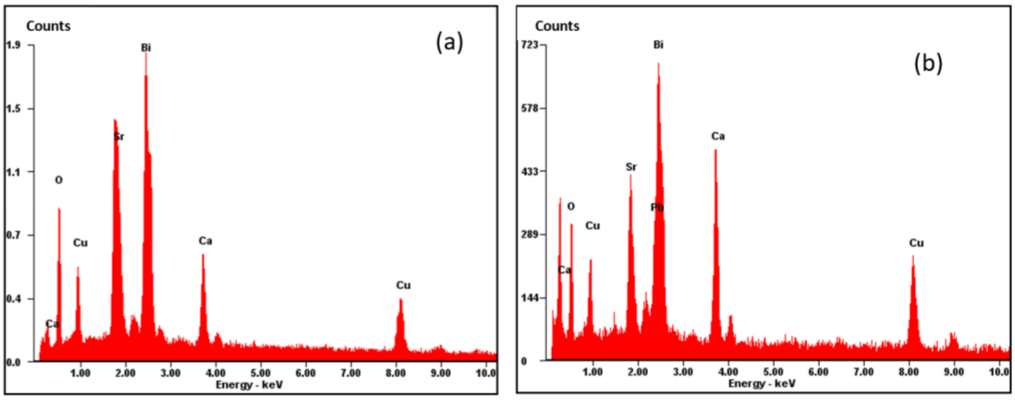

Fig. 2. EDX spectra of the powders (a) Bi-2212 (b) (Bi,Pb)-2212

Figure 3 displays the XRD pattern of (a) Bi-2212 and (Bi,Pb)-2212 samples measured at room temperature 300 K. In our samples, the major phase formed is Bi-2212 (~85%) with few amounts of Bi-2201 (~10%) and impurities that consists of Ca_2CuO_3 and CuO (~5%). The lattice symmetry at room-temperature was confirmed by MAUD software to be orthorhombic with the *Amaa* symmetry as has been already reported [25, 26]. The goodness of fit (GoF) for the room-temperature structure was converged to be ~2% indicating a good fit to the experimental data. The obtained lattice constant for Bi-2212 were a = 5.3968(6) Å, b = 5.3984(6) Å and c = 30.7461(18), while for (Bi,Pb)-2212 were a = 5.3940(6) Å, b = 5.3939(6) Å and c = 30.7456(10). The Pb is successfully inserted to Bi-site indicating by the shifting of the peak to the right-side following the Bragg's Law, since the atomic radius of Pb is smaller than that of Bi as displayed in the inset in Fig.3.

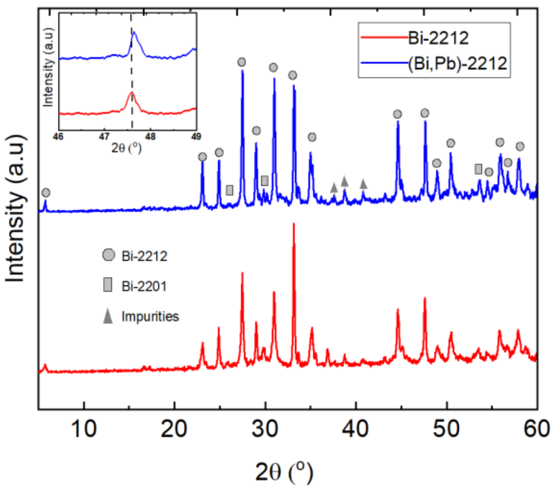

Fig. 3. XRD pattern of sample Bi-2212 and (Bi,Pb)-2212. (Inset) The substitution of Pb to Bi-site results in the peak shifting in XRD pattern

The TEM result, showed in Fig. 4 (a), confirmed the nanoparticles of Bi-2212. The length of the red line in Fig. 4 (b) which is estimated about 1.5 nm supposed to correspond to a half-unit cell of Bi-2212 phase, while the yellow line could be attributed to the presence of Bi-2201 phase as also shown in XRD result. As depicted in Fig. 5, the distribution of particle size in Bi-2212 sample, analyzed using MAUD software, is in the range of nanometer (~100 nm). In average, Bi-2212 tends to has smaller particle size than (Bi,Pb)-2212. The usage of doping elements, like Pb, is significant technological step that governs the growth rate of the high-T_c superconducting phases and their particle size [27, 28].

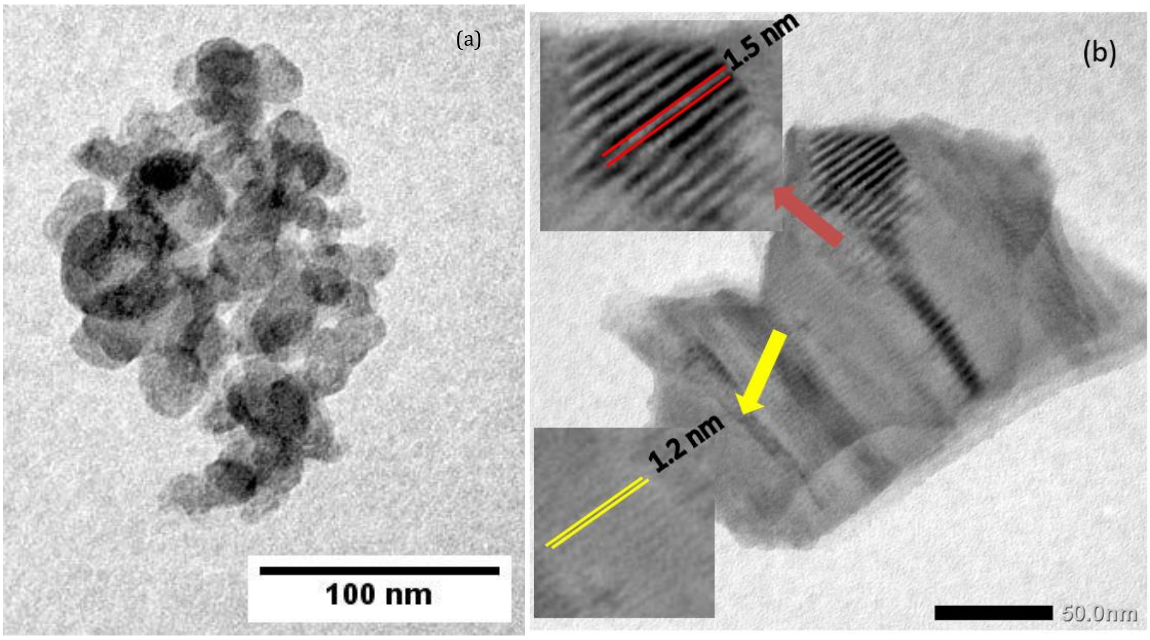

Fig. 4. (a) TEM image confirms the nanoparticle size of Bi-2212 (b) TEM result shows the agreement with a half unit cell of Bi-2212 phase indicated by the red-line, while the yellow-line is the indication of half unit cell of Bi-2201 phase.

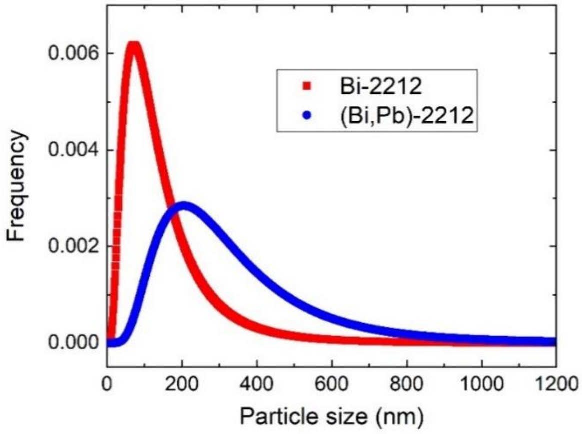

Fig. 5. Distribution of particle size on Bi-2212 and (Bi,Pb)-2212 obtained from MAUD analysis.

The presence of superconductivity can be confirmed from magnetic susceptibility measurement under applied field of 10 Oe as shown in Fig. 6. For the Bi-2212 sample, the T_{onset} is located around 78 K while for (Bi,Pb)-2212 is obtained around 75 K indicating T_C. As shown in the inset in Fig. 6 (a) and (b), the bifurcation of the FC and ZFC curve for Bi-2212 and (Bi,Pb)-2212 exhibited as temperature irreversibility (T_{irr}) starts at around 70.57 K and 70.52 K, respectively. The comparison between T_{irr} and T_{onset} in Bi-2212 and (Bi,Pb)-2212 are 0.90 and 0.94, respectively, which is attributed to the enhancement of flux pinning in (Bi,Pb)-2212.

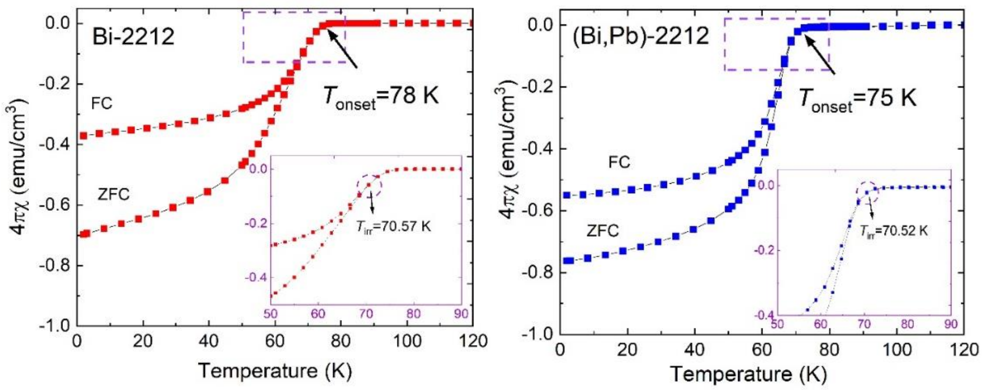

Fig. 6. M-T curve of samples: (a) Bi-2212 (b) (Bi,Pb)-2212 measured under applied field of 10 Oe using magnetic susceptibility measurement

The value of the susceptibility at the lowest temperature provides information about the superconducting volume fraction. The estimation of superconducting volume fraction is 70% for Bi-2212 and 76% for (Bi,Pb)-2212. As shown in Table 1, it is possible to conclude that the synthesized samples in this present work exhibited T_c values which are comparable to other data in the literature.

Table 1. Comparison of superconducting critical temperature in various synthesis procedure in Bi-2212

Sample	Additive	Synthesis Method	T_c (K)	Ref.
Bi-2212	Without	Wet-mixing	78	Present work
Bi-2212	Pb	Wet-mixing	75	Present work
Bi-2212	Without	Solid state	72.3	[20]
Bi-2212	B_2O_3	Solid state	83.2	[10]
Bi-2212	K-Na	Solid state	94.1	[21]

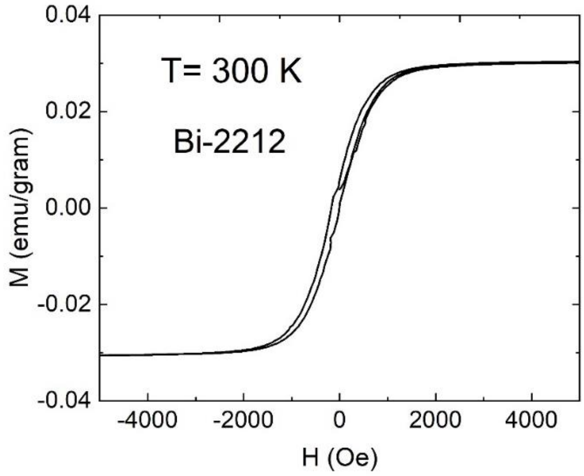

Fig. 7. $M(H)$ data of Bi-2212 nanoparticles at 300 K shows the ferromagnetic behavior

Figure 7 exhibits the $M(H)$ data at 300 K in the field range of -5000 Oe $\leq H \leq$ 5000 Oe of Bi-2212 nanoparticles. Intriguingly, the room-temperature magnetization of Bi-2212 nanoparticles show hysteresis with a weak ferromagnetism. The coercivity, remanence and saturated value of magnetization data are 1.472×10^{-4} Oe, 3.997×10^{-3} Oe and 2.919×10^{-2} Oe, respectively. The origin of ferromagnetism is likely to be due to magnetic moments arising from the oxygen vacancies at the surfaces of Bi-2212 nanoparticles.

4. Conclusions

Nanoparticles of Bi-2212 and (Bi,Pb)-2212 have been successfully synthesized by wet-mixing method. The XRD result confirmed the existence of dominant phase of Bi-2212 with the orthorhombic structure (space group *Amaa*). The dominant phase of Bi-2212 was obtained in this synthesized sample, with the small amount of Bi-2201. It is still difficult to obtain the pure phase of Bi-2212 since the Bi-2212 phase was argued to be formed via the Bi-2201 phase. SQUID measurement exhibits the presence of superconductivity by the observation of T_c at ~78 K for Bi-2212 and 75 K for (Bi,Pb)-2212. Based on this result, the superconducting critical temperature, T_c, is comparable to other literatures. Nanoparticles of superconducting Bi-2212 exhibit weak ferromagnetism at room temperature which is likely to be due to magnetic moments arising from the oxygen vacancies at the surfaces of Bi-2212 nanoparticles.

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