Kasetsart J. (Nat. Sci.) 40 : 28 - 34 (2006)

Vanadium-Lead Doped Bi-2223 Superconductor

Supreya Trivijitkasem[1]*, Chirawat Kluenrat[1] and Viboon Pumchuchit[2]

ABSTRACT

The effect of vanadium-lead doped in Bi-Sr-Ca-Cu-O superconducting system was studied. A series of initial nominal composition $Bi_{1.75}Pb_{0.25-x}V_xSr_2Ca_2Cu_3O_y$, x = 0, 0.025, 0.050, 0.075 and 0.1 were fabricated. The influence of the dopant was investigated by x-ray analysis, scanning electron microscopy and electrical resistivity. The results showed both critical temperature and the 2223 phase formations were affected by vanadium and lead concentration. The pure (Bi, Pb) samples showed nearly uniformed homogenous microstructure. More 2223 phase was found in the intermediate ground-pressed sample. The highest critical temperature of 107 K belonged to pure non intermediate ground-pressed (Bi, Pb) sample.

Key words: BSCCO, V-Pb, superconductor

INTRODUCTION

Since the discovery of $Bi_2Sr_2Ca_2Cu_3O_y$ (Bi-2223) superconductor, great progress has been achieved in enhancing the volume fraction of this phase. Much efforts have been directed to produce single phase (110 K) or nearly single phase materials by manipulations of the ceramic processing conditions (Dorris *et al.*, 1993) or by adopting some strategies to optimize the yield of the 110 K phase (Sharma *et al.*, 1995). The mechanism and dynamic model of the formation of Bi-2223 phase was presented and verified by Wang *et al.* (1993). The high-temperature Bi-2223 superconductor is, now, the most promising material for high current applications, tapes and wires. Despite of different preparation methods, partial substitution of Pb for Bi is successfully in producing a highly stabilizing proportion of Bi-2223 phase (Wahlbeck *et al.*, 1995). Ward *et al.* (1992) tried to find out the conditions required for generation of high volume fraction of the 2223 phase. Trivijitkasem and Sratongluan (1999) reported the formation of complex multicomponent oxides which resulted in extremely slow kinetics to form the high Tc phase. Zhang *et al.* (2000) found phase separation in $(Bi_{1.6}Pb_{0.4})$-2223 superconductors under flowing N_2 at 730 °C upon annealing process. Lu *et al.* (2002) synthesized (Bi , Pb)-2223 oxide superconductors by diffusion reaction which facilitates much shorter heat treatment time than the conventional sintering process. Mune *et al.* (2003) introduced mechanical uniaxial pressing method to improve the general transport properties of (Bi , Pb)-2223 superconductor.

However, the synthesis of single-phase Bi-2223 superconductor is still difficult due to a poor understanding of the complex phase diagram (Ikeda *et al.*, 1991), as well as melting and

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[1] Department of Physics, Faculty of Science, Kasetsart University, Bangkok 10900, Thailand.
[2] Wapeepathum school, Mahasarakham 44120, Thailand.
* Corresponding author, e-mail: fscisum@ku.ac.th

solidification behavior. The major limitations of the Bi-2223 superconductor applications are the intergrain weak links and weak flux pinning capability (Salamati and Kameli, 2004). Besides Pb, any other dopants have been investigated in order to enhance the formation and stabilize the high Tc phase. Application of different second phase additives to Bi-2223 superconductors are being attempted for further improvements in mechanical and transport properties of the material (Xin *et al.*, 1990; Nováková *et al.*, 1997). The enhancement effect of low concentration vanadium dopant on the Tc of (Bi,Pb)-2223 superconductor was observed, further increase in vanadium resulted in lowering of Tc (Tepe and Abukay , 1998). Vanadium doping appears to influence considerably Bi(Pb)-2223 processing, V-containing surplus accelerates Bi(Pb)-2223 phase formation and causes Tc droping a few degrees (Kazin *et al.,* 1998). Chanda and Dey (1996) found similar features of mixed state Seebeck voltage for pure and vanadium substituted Bi(Pb)-2223 samples. Mishra *et al.* (2000) reported a double role on vanadium doping with (Bi,Pb)-2223 superconductors : as a sintering agent and as a substituent element which has typical transition element magnetic properties due to the presence of an empty d-band. In order to investigate the role of a second dopant, in enhancement effect on the Tc of (Bi-Pb)SrCaCuO system, vanadium, a 3d element, with variable valency of its cation, comparable cation radius and electronegativity with bismuth was chosen in present work.

MATERIALS AND METHODS

The samples with the initial cation compositions corresponding to $Bi_{1.75}Pb_{0.25-x}V_xSr_2Ca_2Cu_3O_y$, x = 0, 0.025, 0.050, 0.075 and 0.1, were prepared by conventional solid-state reaction method as described by Trivijitkasem and Sratongluan (2000). Bi_2O_3(99.5%), PbO(99.0%), $SrCO_3$(99.5%), $CaCO_3$(99.5%), CuO(99.0%) and V_2O_5(99.0%) powders were used as the starting materials. The last heat treatment of the pellets was performed in air at 845 °C for 100 h, followed by slow cooling to 300 °C in the furnace.

The influence of systematic stoichiometric replacement of lead by vanadium in Bi-2223 superconductors which were subjected to non or an intermediate ground and pressed process (N or A process) was investigated by using various techniques. X-ray diffraction (XRD) study was performed by using Ni-filtered Cukα radiation. Powder diffraction patterns were collected at room temperature at 30 kV, 25 mA on a Philips model PW 3710 wide angle goniometer, by using the scanning mode with a step size $\Delta(2\theta) = 0.05°$ and $3 < 2\theta < 70°$.

Scanning electron microscopy (SEM) was carried out with a SEM, JEOL, model JSM-220A. The microstructures of the samples were observed at room temperature. The electrical resistivity as a function of temperature, $\rho(T)$, was measured by using the standard DC four-probe technique in slabs with typical dimension of 0.5 mm thickness, 2 mm width and 10 mm length. The contacts were made with platinum wire and silver epoxy. The sample was cooled from room temperature (T_{rt}= 297 K) down to 77 K, then a constant current of 1 mA from Keithley current source was applied throughout the measurements, and the specimen was warmed slowly up to room temperature. Using a Keithley 195A digital multimeter and a Lakeshore temperature controller, the temperature and the voltage across the sample were collected as the temperature was raised from 77 K to 220 K.

RESULTS AND DISCUSSION

The amount of vanadium doped in $Bi_{1.75}Pb_{0.25-x}V_xSr_2Ca_2Cu_3O_y$ samples are listed in Table 1, and the powder XRD patterns of the 10 pellet specimens are shown in Figure 1. The XRD spectra of both pure and V-doped (Bi,Pb)-2223 samples showed the presence of both phases : the 110 K-2223 phase and 80 K-2212 phase. A Pb-rich phase with stoichiometry Ca_2PbO_4 was found in all samples. The peaks observed were invariant and were present in all samples. As a result, there was not any formation of a new phase or compound in the V-doped samples. The peaks belonging to the Bi-2223 phase were indexed with respect to an orthorhombic unit cell with lattice constants as shown in Table 1.

There was a consistent variation in the intensity and the position of the peaks which indicated the change in the phase composition and the lattice parameters of the sample. The variation in the intensities of the Bi-2223 peaks was used to reflect the variation of phase composition. Using

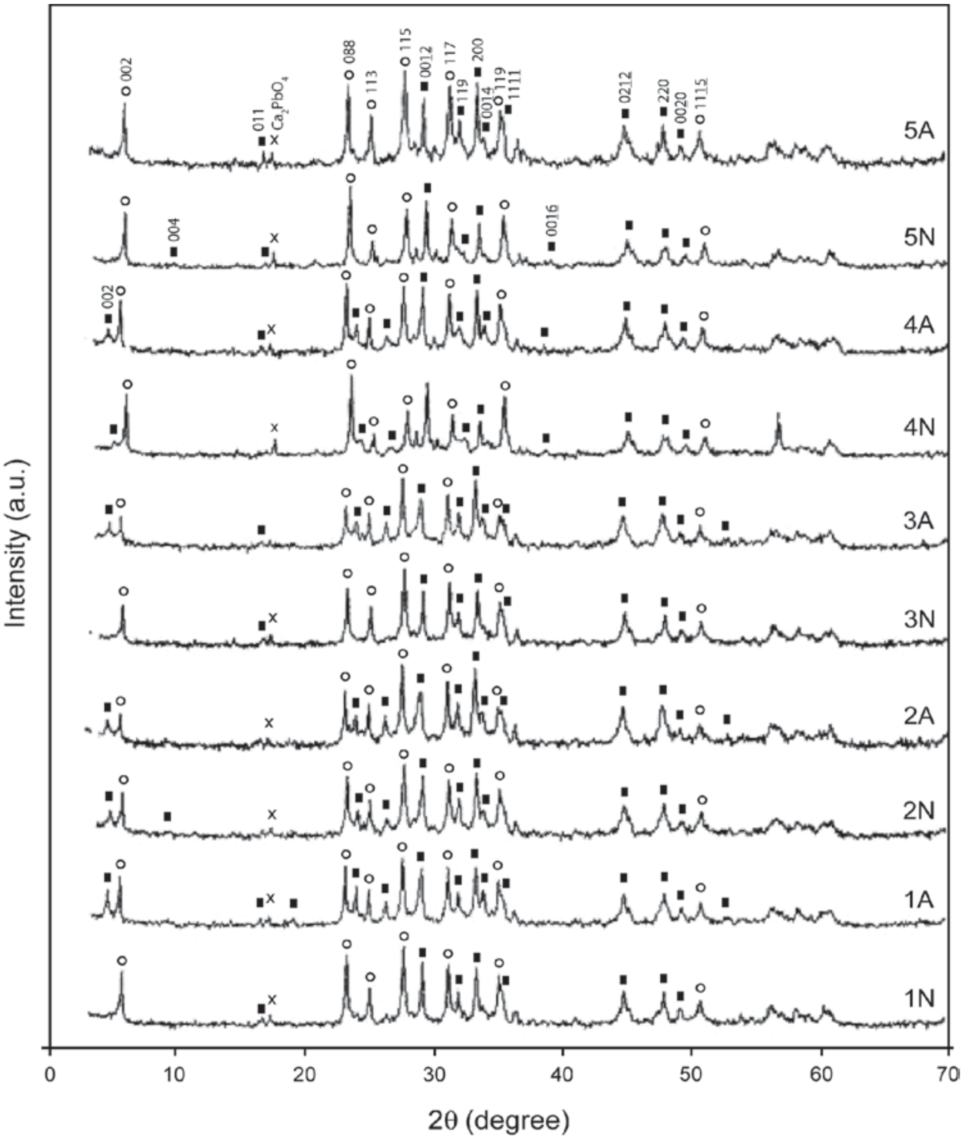

Figure 1 The XRD patterns of the $Bi_{1.75}Pb_{0.25-x}V_xSr_2Ca_2Cu_3O_y$ samples, ■ : Bi-2223, ❍: Bi-2212 phases and × : Ca_2PbO_4.

intensity of all the peaks from the XRD spectra of the samples, the volume content of the 110 K high Tc (2223) and 80 K low Tc (2212) phases were estimated in percent from the following equations:

$$\text{Bi-2223}(\%) = \frac{\Sigma \mathrm{I}(\text{Bi-2223})}{\Sigma \mathrm{I}(\text{Bi-2223}) + \Sigma \mathrm{I}(\text{Bi-2212}) + \Sigma \mathrm{I}(\mathrm{Ca_2PbO_4})} \times 100,$$

$$\text{Bi-2212}(\%) = \frac{\Sigma \mathrm{I}(\text{Bi-2212})}{\Sigma \mathrm{I}(\text{Bi-2223}) + \Sigma \mathrm{I}(\text{Bi-2212}) + \Sigma \mathrm{I}(\mathrm{Ca_2PbO_4})} \times 100,$$

where the voids were ignored. The results are reported in Table 1. The intermediate ground-pressed sample showed more Bi-2223 phase. The percentage of Bi-2223 increased from 36.8% for the pure sample to 45.2% for the V-doped ($x = 0.025$) sample, and then decreased to 30.7% for the V-doped ($x = 0.1$) sample.

The scanning electron micrographs of the specimens are displayed in Figure 2. The pure (Bi-Pb) samples showed nearly uniform homogeneous microstructure which comprised of platelets dark gray grain of 110 K phase, pellet white grain of 80 K phase, and spherical white Ca_2PbO_4 contrasts which distributed inhomogeneously; while the V-doped (Bi-Pb) specimens showed greater and more pellet grains. More porosity was found in the high V-content specimens. The grain size of the intermediate ground-pressed specimens were smaller, and oriented more dense than the non-intermediate ground-pressed specimens.

Relative electrical resistivity $\rho(T)/\rho_{rt}$ versus temperature between 80-220 K of the pure and V-doped 2223 samples are shown in Figure 3, where ρ_{rt} is the resistivity at room temperature T_{rt} = 297 K. The curves exhibited a almost linear dependence of $\rho(T)/\rho_{rt}$ at high temperature and a transition to the superconducting state at the onset critical temperature, T_c(onset) ~ 113-117 K, where $\rho(T)$ started to deviate from the linear behavior at high temperature. This temperature was nearly constant for all samples, and was related to the transition of the isolated grains to the superconducting state, hence the grain in these samples might have similar stoichiometry. The resistivity ρ_{rt} at room temperature and the critical temperature T_c ($\rho = 0$) of the samples are given in Table 1, the resistivity was higher and T_c was lower when more vanadium content was added ($x \geq 0.075$).

These results were in agreement with those data obtained from the XRD spectra of the samples described above, which confirmed the decreasing volume content of the Bi-2223 phase with increasing amounts of vanadium. The T_c for the A-process is slightly lower than the N-process, The sample 1N with the lowest porosity and

Table 1 The amount of vanadium doped(x) and the calculated data from the observed XRD spectra and resistivity measurements of the $Bi_{1.75}Pb_{0.25-x}V_xSr_2Ca_2Cu_3O_y$ pellet samples.

Sample	x	Lattice constant (Å)			c/a	Bi-2223	Bi-2212	ρ_{rt}	T_C(onset)	T_C
		a	b	c		%	%	(mΩ·cm)	(K)	(K)
1N	0.000	5.510	5.358	36.936	6.70	36.8	59.0	2.82	117	107
1A	0.000	5.401	5.350	37.130	6.88	49.5	49.2	2.89	115	103
2N	0.025	5.402	5.385	37.496	6.94	45.2	54.2	2.71	116	106
2A	0.025	5.412	5.368	37.133	6.86	52.5	47.1	2.26	114	102
3N	0.050	5.403	5.594	37.100	6.87	44.5	53.9	2.04	115	107
3A	0.050	5.405	5.475	37.021	6.85	54.6	43.9	2.41	115	105
4N	0.075	5.399	5.387	36.953	6.84	32.6	67.6	6.83	113	105
4A	0.075	5.430	5.374	37.367	6.88	44.7	54.4	4.60	114	102
5N	0.100	5.403	5.407	36.784	6.81	30.7	65.7	6.46	115	105
5A	0.100	5.408	5.376	37.163	6.87	41.5	57.8	4.49	113	104

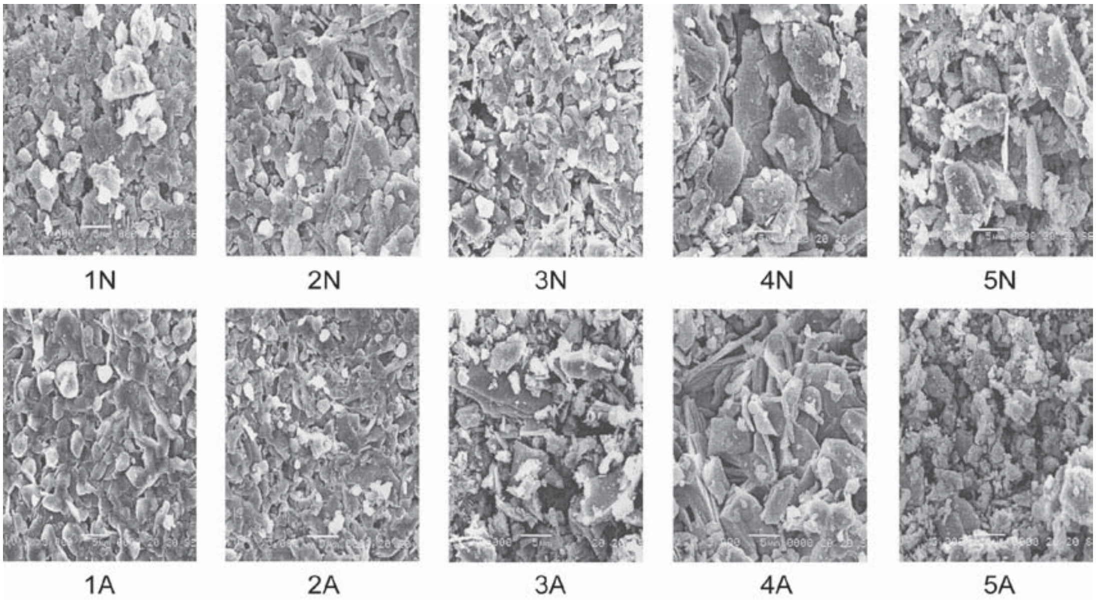

Figure 2 SEM microstructure of the specimens.

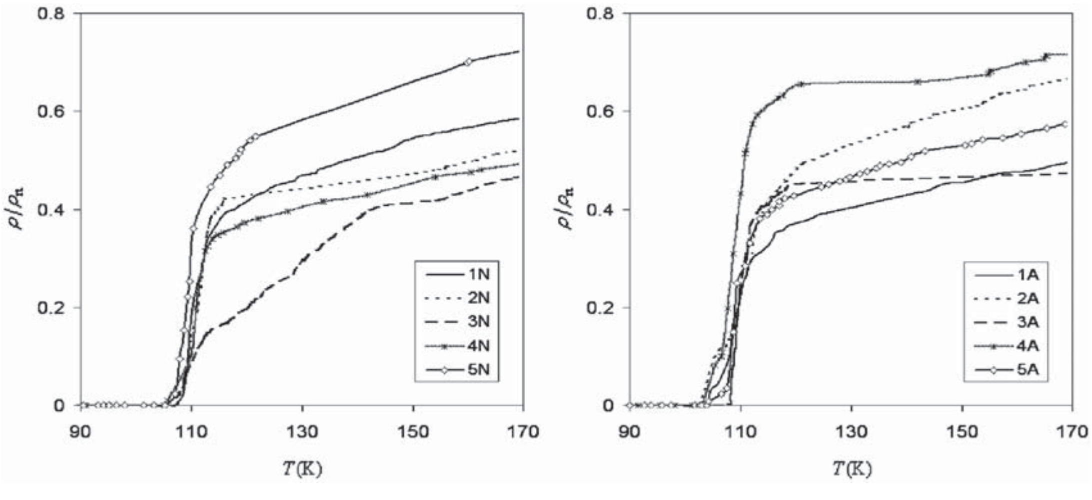

Figure 3 Temperature dependence of the relative electrical resistivity $\rho(T)/\rho_{rt}$ of the samples.

moderate content of the Bi-2223 phase as inferred from XRD analysis exhibited the highest value of T_c.

CONCLUSION

A substitution of vanadium for lead for a constant content of Bi in the Bi-Sr-Ca-Cu-O ceramic superconductors was studied. The result led to bad superconducting properties when more vanadium content was added.

From XRD analysis, the lattice constant ratio, c/a, of the pure (Bi,Pb) sample without intermediate ground-pressed, (1N), was the lowest.

More 2223 phase was appeared in the intermediate ground-pressed sample. The percentage of Bi-2223 for the pure (Bi,Pb) sample, (1N), was 36.8% and decreased to 30.7% for the vanadium-doped ($x = 0.1$) sample.

From scanning electron micrographs, the pure (Bi,Pb) sample revealed nearly uniform homogeneous microstructure, while the V-doped samples revealed more porosity inhomogeneous microstructure.

From electrical resistivity measurements, the resistivity at room temperature of the pure (Bi,Pb) sample, (1N), was 2.82 mΩ·cm, and the resistivity was increased to 6.83 mΩ·cm when more vanadium content was added. The onset critical temperature was 117 K and the critical temperature was decreased from 107 K for the pure (Bi,Pb) sample, (1N), to 102 K for the vanadium-doped samples.

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