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Thermal Properties and Crystallization Kinetic Studies on $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$ Superconducting System

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ABSTRACT

Samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$ compounds with x = 0.1, 0.3 and 0.5 were prepared by solid state reaction method. The values of the kinetic parameters were determined using the kinetic model of Bansal and Doremus .Activation energy of the first crystallization of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ was calculated with a differential scanning calorimeter (DSC) at different rate temperature-time. This value was about 286.431 kJ/mol. Also the loss and gain of mass at temperature range (0-1000)ºC was calculated using thermogravimetric analysis (TG).

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INTRODUCTION

Study of the thermal and structural properties (Kayed, 2004; Aksan, 2008; Tomasi, 2002; Özhanlı, 2002) of BiSrCaCuO (BSCCO) ceramics is important to understand the nucleation and crystal growth mechanism which is essential to obtain high quality material for technological applications.

Khan *et al* (2002) showed that the silver addition process is of considerable potential for production of silver sheathed tapes with confirmed HTSC technological applications. In their research Bi-2223 doped with Sm, Nb, Ag and Gd were prepared by heat treatment of rapidly quenched glass precursors. Activation energies and frequency factors, employing different models were evaluated. It was observed that the reaction rates were dependent on ambient atmosphere.

Dogruer *et al* (2012) studied effect of Mn addition on the superconducting and on the physical properties of $Bi_{1.8}Pb_{0.4}Sr_2Mn_xCa_{2.2}Cu_{3.0}O_y$. The zero resistivity transition temperatures, irreversibility fields, and upper critical fields were deduced from the magnetoresistivity curves. Moreover, thermally activated flux creep model was studied for activation energy values of the samples.

Arslan *et al* (2012) prepared the Sn substituted $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_\delta$ (where x=0, 0.1, 0.3, and 0.5) samples by the melt-quenching method. Crystallization kinetic studies of the samples were conducted using the differential thermal analysis (DTA). The oxidation behavior of the samples was analyzed using the thermogravimetry analysis (TG). The crystallization results were analyzed. Also the activation energy of crystallization process as well as the crystallization mechanisms and the effect of Sn substitution on powder glass ceramic were characterized. The activation energy of crystallization was about 332.8±0.1, 358.0±0.1,353.1±0.1 and 348.9±0.1 kJ/mol for x=0, 0.1, 0.3 and 0.5, respectively. The Avrami parameter (n) values calculated at different Sn ratio were found to be between 1.70±0.01 and 2.57±0.01.These results reflect the growth of small particle with a decreasing nucleation rate.

In a previous work Hermiz *et al* (2014), the effect of Mn content on the critical temperature (T_c),on the structure properties and on the microstructure of the (BPSCCO) compounds were studied. In this search the crystallization kinetics of BSCCO systems have been investigated by differential scanning calorimeter (DSC) and thermogravimetric analysis (TG) methods. Accordingly the crystallization activation energies of the system have been calculated.

Experimental work:

The solid state reaction method was used to prepare samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$ with x=0.1, 0.3 and 0.5.The procedure of samples preparation was described in a previous paper (Hermiz, 2014).

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Thermogravimetric analyzer (model:TGA/DSC1/HT brand: Mettler Toledo) was used to analyze BSCCO samples in the temperature range 30°C – 1000°C. A heating rate of 10°C/min. was used to heat the samples .The crystallization temperature analyzed using Differential Scanning Calorimeter (model: DSC1/700Brand:Mettler Toledo) at operating temperature 300°C - 500°C and different heating rates 10°C/min - 30°C/min was used to determine the crystallization kinetics.

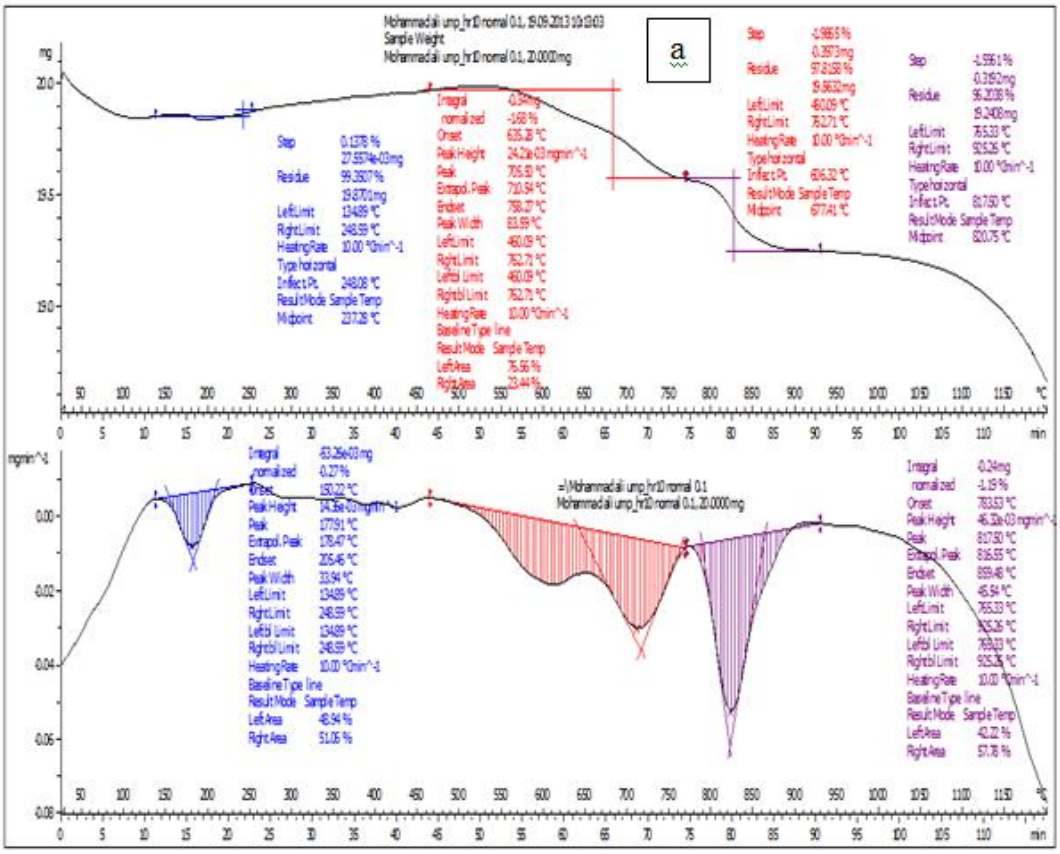
RESULTS AND DISCUSSIONS

a-Thermogravimetric analysis:

Fig.1 (a-c) shows the thermogravimetric (TG) curves taken at the heating rate of 10°C/min for different Mn concentrations (x=0.1,0.3 and 0.5). The mass gain of the samples was obtained by TG analysis (Table (1)). All samples exhibited the same TG behavior. It can be observed from Fig.(1a) that the mass gain started around 134.89 °C and finished around 535±0.5°C. Hence, the oxidation process was completed around 535±0.5 °C. Another feature can be observed from Fig.(1)is that the temperature at which the oxidation process was finished has shifted towards high temperature as Mn concentration is increased from x=0.1 to 0.3.

The total mass gain 27.557×10^{-3},55.4848×10^{-3} and 116.5×10^{-3} mg for $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$ of x=0.1, 0.3 and 0.5 respectively was obtained at 10 °C/min heating rate. This means that the mass gain increased with increasing Mn concentration. It is well known that the mass gain in the BSCCO system is due to the oxidization of the material so as to fill the oxygen deficiencies in the unit cell Aksan *et al* (2005).

Mass loss at temperatures below 300°C is associated with the removal of moisture and decomposition of hydrates. The peaks in the range from 460°C- 925°C, 751°C -919°C and 470°C -943°C for x=0.1,0.3 and 0.5 respectively, observed in Fig.(1) suggest a crystallographic transformation of the BSCCO structure. The percentage weight loss of 1.9865 from 460.09°C -762.71°C and 1.5961 from 765.33°C -925.26°C were observed for x=0.1 at a rate 10°C/min. The TG curve of BSCCO -2223 with x=0.3 shows a percentage weight loss of 1.7915 from 751°C -919.27°C. While the TG curve for the composition of Mn=0.5 indicated that percentage weight loss of 1.9619 from 713.23°C -943.23°C for the same rate, as shown in Fig.(1c)and Table(1).



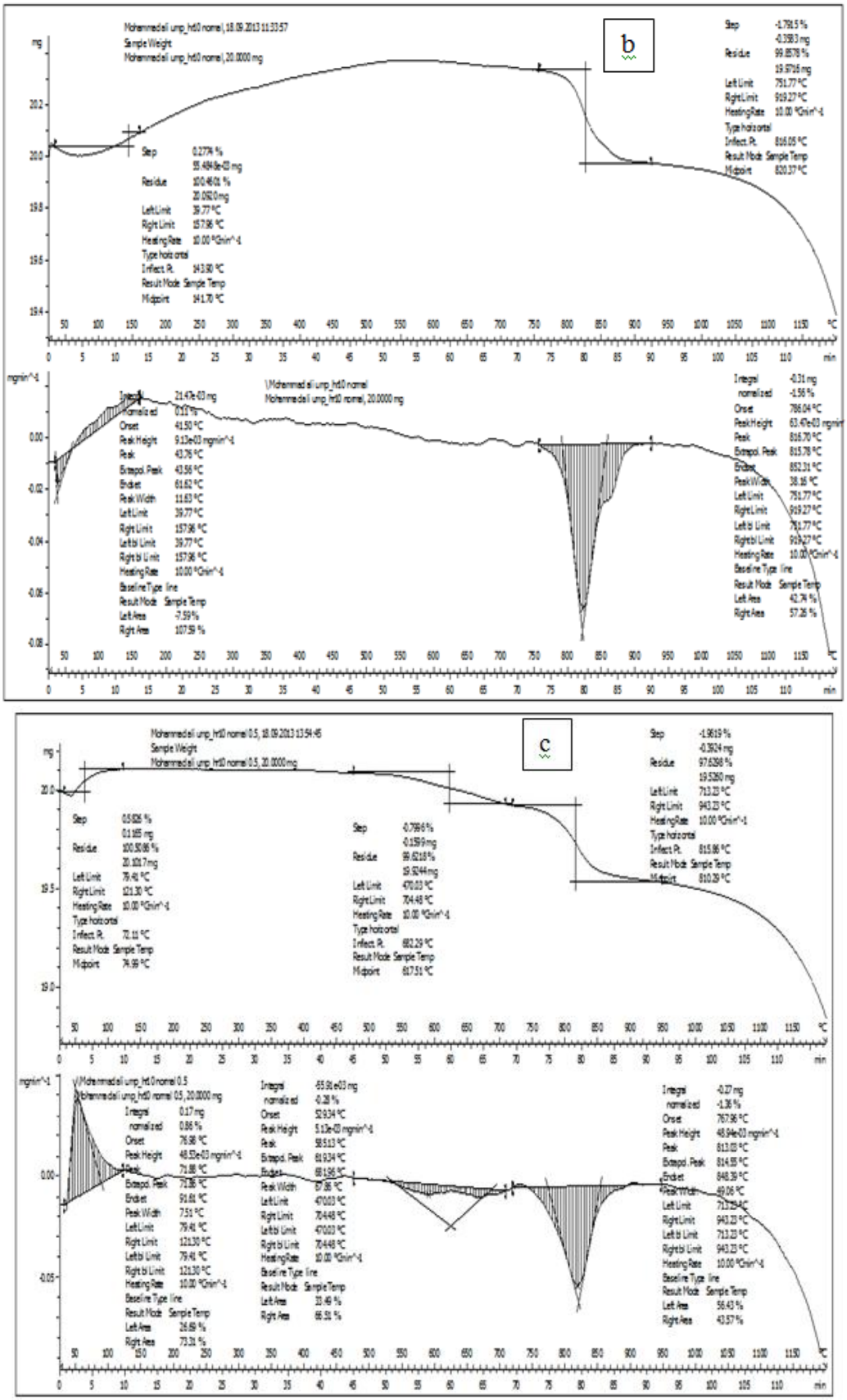


Fig. 1: TG and DTG plot of :(a)$Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ (b)$Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.7}Mn_{0.3}Cu_3O_{10+\delta}$ (c)$Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.5}Mn_{0.5}Cu_3O_{10+\delta}$ samples at 10 ^oC/min heating rate.

Table 1: The values of mass gain and loss for $Bi_{1.7}Pb_{0.3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$ with (x=0.1, 0.3 and 0.5).

x	Total mass gain(mg)*10^{-3}	ΔT(K)	Total mass losses(mg)*10^{-3}	ΔT(K)
0.1	27.357	134.89 - 248.59	340	460.09 - 762.71
			240	765.33 - 925.26
0.3	55.4848	39.77 - 157.96	310	751.77 - 919.27
0.5	116.5	79.41 - 121.3	55.91	470.30 - 704.48
			270	713.23 - 943.23

b- Differential scanning calorimeter analysis:

The variable heating rate (α) from 10 to 30 ^oC/min of different scanning calorimeter (DSC) method was used to evaluate the crystallization kinetic parameters.

The peak corresponding to the temperature (T_p) at which the rate of transformation of viscous liquid into crystal becomes maximum. Fig.(2) and Table(2) show a shifting of T_p to higher temperatures with increasing the heating rate. This is attributed to the increase of nucleation sites.

Fig.(3) shows a linear relation between (T_p^2/α) and ($1000/T_p$) for $Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ powder samples. From this graph the value of the activation energy was deduced from the slope of the line and its value is 286.431 kJ/mol. This value is in good agreement with previous studies (Arslan, 2012).

The values of kinetic parameters were determined using the kinetic model of Bansal and Doremus Bansal *et al* (1984).The relation is:

$$\ln(T_p^{\,2}/\alpha) = \ln(\frac{E}{R}) - \ln\nu + \frac{E}{RT_p}$$

where T_P is the peak maximum temperature, α the heating rate, E the activation energy, R the gas constant and ν the frequency factor. The kinetic parameters (E and ν) are related to the reaction rate constant K by Arrhenius equation:

$$K = \nu \exp(-\frac{E}{RT})$$

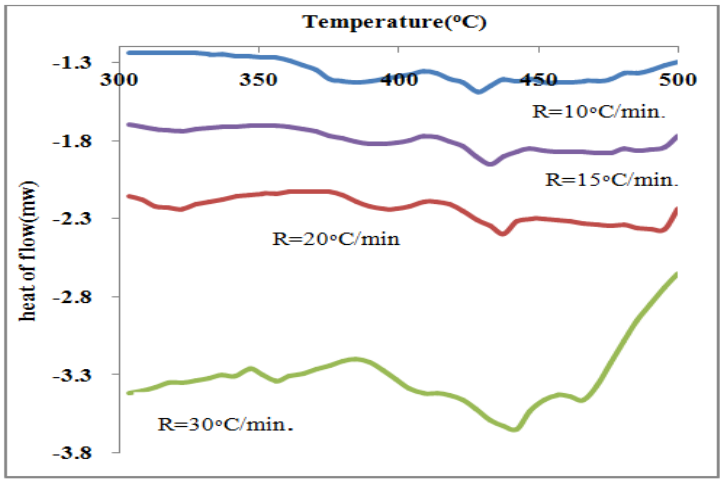


Fig. 2: DSC of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ for different heating rate.

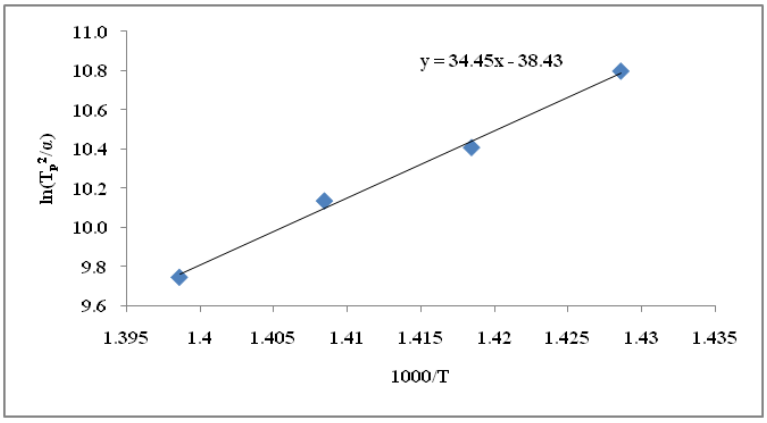


Fig. 3: A plot of ln [T_p^2/α] versus $1000/T_p$ for $Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ powder sample.

Table 2: The values of heating rate, peak temperature, activation energy, frequency factor and reaction rate constant.

α (^{o}C/min)	T_p (^{o}C)	E_a (kJ/mol)	ν	K
10	427	286.431	$7.59*10^{15}$	0.155
15	432			
20	437			
30	442			

Conclusions:

All samples exhibited the same TG behavior. The total mass gain increased with increasing Mn content. The activation energy of the first crystallization of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{1.9}Mn_{0.1}Cu_3O_{10+\delta}$ was 286.431kJ/mol. The oxidation finished temperature of the samples has shifted toward higher temperatures with increasing the Mn concentration from x=0.1 to 0.3.

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