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Structural Investigation of Higher order members of Bismuth System Superconductors

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Abstract: Structural formation of higher order Bismuth superconducting compounds $Bi_{1.65}Pb_{0.35}Sr_2Ca_4Cu_5O_y$ (2245) phase and $Bi_{1.65}Pb_{0.35}Sr_2Ca_8Cu_9O_y$(2289) phase were investigated. The samples were synthesized by solid state reaction technique. Morphological and micro-structural features of the synthesized samples were analysed by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX). The XRD of both the samples revealed the presence of Bismuth – (2212)& (2223) phases of which (2223) phase was found to be the predominant. The average grain size was found to be around 65 nm. Traces of Ca_2PbO_4 was also noticed when the samples were synthesised at 835 ˚C. Superconducting transition temperature (T_C) of the samples measured by self-inductance method, showed a two-step transition, one around 110 K and the other around 90 Kindicating the presence of (2223)&(2212) phases respectively. But the T_C value observed for (2212) phase is about 10 K more than the expected value of 80 K.There was no signature of the formation of (2245) or (2289) phases in this synthesis.

Keywords: X- Ray Diffraction; Phase formation; T_C measurements; SEM; EDAX.

Introduction andExperimental:

Thebismuth system superconductors are generally represented by the formula $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with critical temperature (T_C) values around10 K , 80 K and 110 K for n = 1, 2 and 3members respectively [1-2]. Among the above, n = 2 member namely $Bi_2Sr_2Ca_1Cu_2O_8$ (2212) is found to be the most stable one. Preparation of n = 3 member; $Bi_2Sr_2Ca_2Cu_3O_{10}$ (2223) as a single phase is extremely difficult because of the intergrowth of low T_C (2212) phase with (2223) phase. Many research groups have tried to synthesize (2223)compound as a single phase [3-7]. Addition ofLead to the superconducting Bi-Sr-Ca-Cu-O compounds leading to the formation of (2223)phase has been reported [5]. Another research group [6] have tried to synthesize (2223) phase by heating off stoichiometric starting composition with excess Ca and Cu. The matrix method was adopted to obtain a single phase (2223)compound with a T_C of120 K [7]. Though there were many attempts made to synthesize n = 4 (2234) and n = 5 (2245) members [8-12], no conclusive evidence is available on the structure and T_C values of the above phases. In this paper we report the results of our attempt to synthesize the (2245) and (2289) phases by the solid state reaction method.

The samples $S_1 = Bi_{1.65}Pb_{0.35}Sr_2Ca_4Cu_5O_y$(2245) and $S_2 = Bi_{1.65}Pb_{0.35}Sr_2Ca_8Cu_9O_y$(2289) were prepared by solid state reaction method. High purity (99.9%) Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$ and CuO powders were used as starting materials. The powders in the molar ratio of [Bi]:[Pb]:[Sr]:[Ca]:[Cu]::1.65:0.35:2:4:5 and [Bi]:[Pb]:[Sr]:[Ca]:[Cu]::1.65:0.35:2:8:9 were separately prepared by mixing thoroughly the components for about one hour using an agate mortar and pestle. The final mixtures in powder form were kept in two alumina crucibles and calcined at 810 °C for 16 hours in a tubular furnace. The calcined powders were reground and pressed into pellets of about 10 mm in diameter and 2 to 3 mm in thickness under a pressure of 5 tons/cm^2. These pellets of the samples S_1& S_2 were sintered at 835 °C for 45 hours with two intermediate grindings and repelletisation. One part of the pellet was cut, powdered and used for X-ray diffraction, Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray analysis (EDAX).The other piece of the pellet was used for T_C measurement. The T_C of the sample was determined by self-inductance method. A Colpitt's oscillator and a frequency counter along with a liquid nitrogen cryostat were used for the purpose. The temperature of the sample was recorded using a calibrated chromel–alumel thermocouple with an accuracy of ±1 °C.

Results and discussion:

The X-ray diffraction pattern of the samples S_1-(2245)and S_2- (2289) are shown in Fig.1.Both XRD patternsof the samples indicatemultiphase compound. In sample $S_{1,}$ two phases have been identified namely (2212) phase marked as (L) and(2223) phase marked as (H). Majority of the peaks belong to (2223) phase, which are indexed in an orthorhombic cell with cell parameters a = 5.423 Å, b = 5.407 Å andc = 37.003 Å for the sample (2245). The (2212) phase is indexed in tetragonal cell with cell parameters a ≈ b = 5.400Å and c = 30.877 Å. In the XRD pattern of sample S_2, a similar behaviour is observed. Most of the peaks belonged to the (2223) phase for which thecell parameters werea = 5.413 Å, b = 5.400 Å and c = 37.003 Å. The cell parameters of (2212) phase werea ≈ b = 5.400 Å and c = 30.877 Å. The presence of $Ca_2PbO_4$is also evident inboth the samples from the three peaks appearing at2θ = 17.62°, 31.06° and 32.08° respectively.

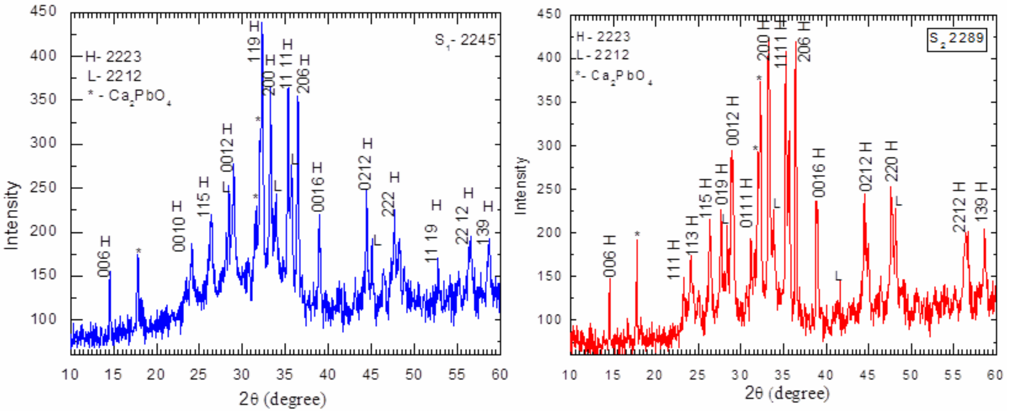
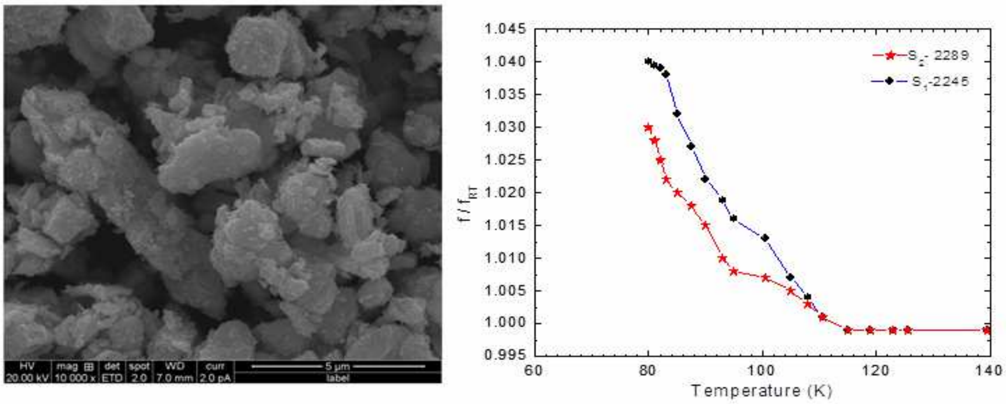


Fig.1. X-ray diffractogram of samples S_1– 2245 and S_2–2289

The SEM image ofsample S_1 is represented in Fig. 2(A). The EDAX spectrum is in conformity with the atomic weight percentage of constituent elements present in the starting composition. As evident, SEM images reveal a granular and porous surface. The average grain size calculated from XRD data using Scherrer formula is found to be about 65 nm.



(A) **(B)**

Fig. 2. (A) SEMimage of the sampleS_1- (2245),
(B) Normalised frequencies versus temperature curvesof S_1 and S_2

The normalised frequency of Colpitt’s oscillator versus temperature plots of the samples are represented in Fig. 2B. Boththe samples S_1& S_2sintered for 45 hours at 835 °C shows a two-step transition with T_C on set at 115K for (2223) phase and 95 K for (2212) phase respectively. The T_C values of the above samples were around 110 K for (2223) phase and 90 K for(2212) phase, which is around 10 K more than the reportedvalue of 80 K for (2212) phase [2]. This may be due to the intergrowth of the (2212)&(2223) phases. The T_C value of cuprate superconductors is known to vary with oxygen stoichiometry and is found to be a maximum for an optimum value of oxygen content. This can be accounted in terms of partial change of the valence state of copper from Cu^{2+} to Cu^{3+} in the Bi system superconductors [10-12].

The results of the attempt made to synthesize $Bi_{1.65}Pb_{0.35}Sr_2Ca_4Cu_5O_y$ and $Bi_{1.65}Pb_{0.35}Sr_2Ca_8Cu_9O_y$ superconductors by solid state reaction method have not yielded a single phase compound. The X-ray diffraction analysis of the prepared samples revealed the presence of the (2223) phase as the major component along with (2212) phase. Presence of Ca_2PbO_4 is also noticed in both the samples. The superconducting transition temperature (T_C) of the (2212) phase is found to be 10 K more than the reported value of 80 K.No traces of formation of (2245) or (2289) phases were noticed under the above preparation conditions.

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