

Synthesis and characterization of Bi-2212 superconductor with iron oxides

H.S.Singh, Rohitash Kumar

Abstract— The Bi based 2212 type superconductor is prepared with the formula $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_x$ ($n=1, 2, 3$) and mixing iron oxides. After mixing of iron oxide in the BiSrCaCuO system, then the structure formula $\text{Bi}_2\text{Sr}_{4-x}\text{Ca}_x\text{Fe}_{3-y}\text{Cu}_y\text{O}_z$ and x, y are variables. In this paper we study Bi-2212 type superconductor which is prepared by ballmill and characterized by XRD and FTIR analysis. XRD results shows that crystal size, cell parameter, type of crystal structure. FTIR gives the bond length change and MÖSSBAUER spectroscopy gives the Hyperfine structure of iron.

Index Terms— Ball Mill, XRD, FTIR, MÖSSBAUER spectroscopy.

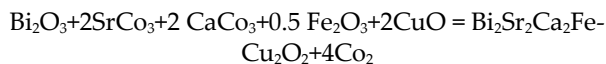
1 INTRODUCTION

IN the BSCCO System, the Bi-2212 phase is thermodynamically stable over a wide temperature range and among the three members of the BSCCO family. The Bi-2212 and Bi-2223 phases have been the most extensively studied to date. In contrast to the Bi-2212 phase, the Bi-2223 phase is stable only in an extremely narrow temperature range and thus it is difficult to prepare single phase Bi-2223 ceramics [1]. The Bi-Sr-Ca-Cu-O system exhibits three different superconducting phases of (Bi-2201), (Bi-2212) and (Bi-2223), which show critical temperatures (T_c) of about 10, 80 and 110 K, respectively [2-4]. Since the discovery of Bi-based superconducting systems, a great amount of work concerning preparation, superconducting properties, and the structure of these compounds has been done [5-7]. The modulated structure is most pronounced for bismuth high temperature superconductors and has been studied in detail for the compound with $n = 2$ or $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$ (Bi-2212) [8].

2 EXPERIMENTAL METHOD

2.1 Material

The samples were prepared by the solid state reaction method using highly pure 99.99% powders of Bi_2O_3 , SrCO_3 and CaCO_3 , Fe_2O_3 , CuO , with appropriate weights in proportion to their molecular weights through the following chemical reaction. All the compounds were commercially available and used as provided without further purification.



2.2 Sample Preparation

The samples were prepared by homogeneously mixing and grinding prescribed amounts of powders into planetary ball mill PM-100. The mixture rounded 5h initially with 400rpm by 25ml grinding jars and used 10mm grinding balls. The composition of compound is shown below in table -1.

3 CHARACTERIZATION

3.1 XRD Analysis

The structure of the samples was studied by X-ray diffraction (XRD) type BRUKER D8 ADVANCE. A computational program has been used to find the lattice parameter of the unit cell from the pattern of XRD by $\text{CuK}\alpha$ (1.5406 Å) radiation. X-ray diffraction (XRD) pattern of the sample with nominal composition $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Fe}_{2.8}\text{Cu}_2\text{O}_z$. XRD pattern was taken using fitting limit 10° to 90° measurements were taken at room temperature. The grinding time in the Ball mill is 5 hours and the mol weight is 479.77, 238.09 and 834.45 respectively. The system of the samples after 5 hours grinding is tetragonal lattice (body centered), Orthorhombic and Orthorhombic (base centered) respectively. We obtained full width at half maximum $\text{FWHM}(3\theta) = 0.641^\circ, 0.218^\circ, 0.575^\circ$, Volume (CD) = 444.43, 295.09, 898.18, Number of stapes = 18,16,19 with Asymmetry (constant) = 1, 1.4953, 1.4953 for sample 1, 2 and 3 respectively. All the peaks in the XRD pattern are indexed FIG-1, 2, 3 respectively. The comparable study between Fig-1,2,3 and Fig-4 we get that all the peak of Bi-2212 and Bi-2223 matched with compound $\text{Bi}_2\text{Sr}_4\text{CaFe}_{2.8}\text{Cu}_2\text{O}_z$ which sample was prepared by annealing method and whose XRD pattern in Fig-4 [9].

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350cm⁻¹; FTIR spectra were carried out in the range 4000-500 cm⁻¹, with a resolution of 4cm⁻¹ and a mirror speed of 2 cm⁻¹/sec. The calibration and reproducibility of this apparatus elsewhere [10].The powdered sample was homogenized in spectroscopic grade KBr (1:20) in an agate mortar and pressed in to 3 mm pellets with a hand press. We tried to minimize the grinding time to avoid the deformation of the crystals structure, the ion exchange and the water absorption from atmosphere as suggested by [11].

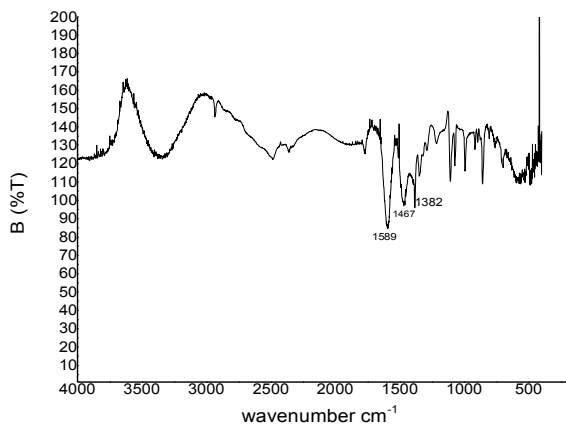


Fig-1

Table -1 sample1. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm ⁻¹	%T
1	1382	104.72
2	1467	99.02
3	1589	85.34

After examination of the data presenting in table-1, results that in the range 1600-1300 cm⁻¹. The most important in the present study for evidence of bonding formation positioned at 1382 cm⁻¹, 1467cm⁻¹,1589cm⁻¹ respectively can be assigned in a tetragonal lattice. These positions are very important for cell parameter. So cell parameter is not symmetrical.

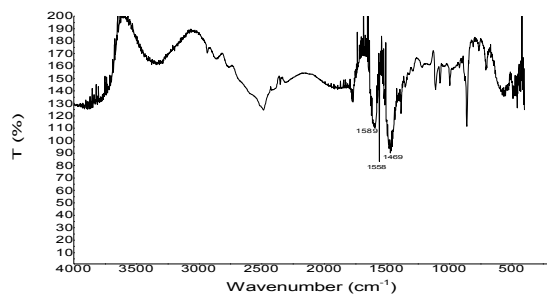


Fig-2

Table -2 sample2. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm ⁻¹	%T
1	1469	97.51

2	1558	83.27
3	1589	111

After investigation of table -2, results that in the range 1600-1400cm⁻¹, the most important in the present study for the evidence of bonding formation in superconductor at positioned 1469cm⁻¹,1558cm⁻¹, 1589cm⁻¹ respectively. These positions are effective to the cell parameters of orthorhombic base centered.

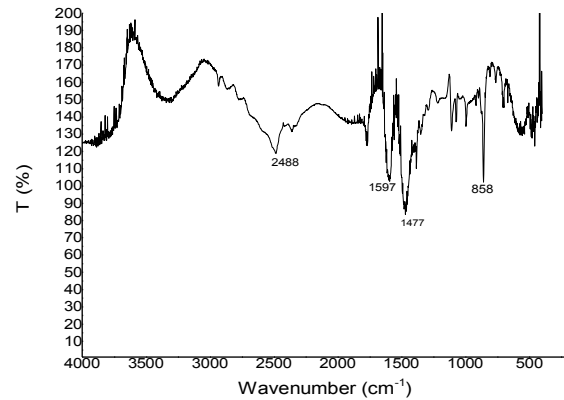


Fig-3

Table -3sample-3. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm ⁻¹	%T
1	858	102
2	1477	89.65
3	1597	103
4	2488	188

After observation of the data presented in table-3, results that in the range 2500-800 cm⁻¹. The most important in the present study for evidence of bond formation positioned at 858 cm⁻¹,1477cm⁻¹1597cm⁻¹2488 cm⁻¹ respectively can be represented to a vibration in superconductor but position 2488 is ineffective for vibration so this is the orthorhombic system and its cell parameter is quietly different.

3.4 MÖSSBAUER SPECTROSCOPY

Mössbauer spectroscopy is the most useful tool to study the behavior of crystals when iron used as a metal dopant at Cu sites. Mössbauer spectra were recorded of the samples with Mössbauer spectrometer using γ -ray source, but these samples do not show any Mössbauer spectrum at room temperature. So there is no internal magnetic field in the superconductor.

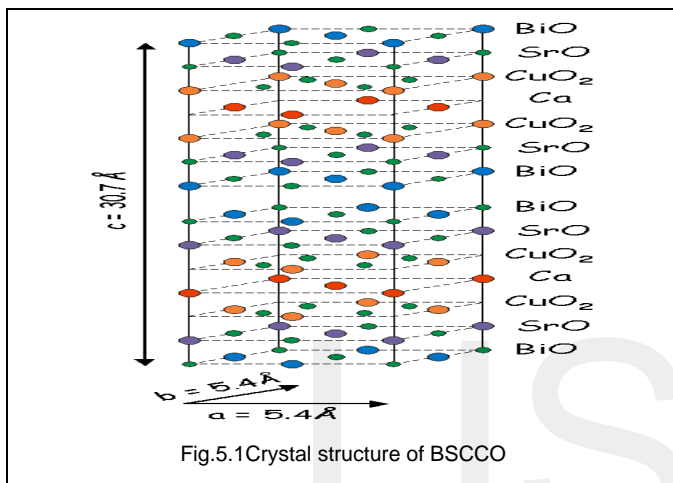
4 RESULT AND DISSCUSSION

X-ray diffraction (XRD) pattern of the sample with nominal composition Bi₂Sr₄CaFe_{2.8}Cu₂O_z, Bi₂Sr₄CaFe_{2.6}Cu₄O_z, Bi₂Sr₄CaFe_{2.4}Cu₆O_z after the final grinding 5h is shown in Fig-1, 2 and 3. XRD pattern was taken using CuK α radiation ($\lambda = 1.5046 \text{ \AA}$) and measurements were taken at room temperature. All the peaks in the XRD pattern are indexed. XRD pattern showed peaks belonging to the high-T_c (2223) phase as a major component along with peaks due to the low-T_c (2212) phase.

The dominant is the high- T_c (2223) phase having orthorhombic structure with lattice constants: $a = 5.4260 \text{ \AA}$, $b = 5.4020 \text{ \AA}$ and $c = 30.643 \text{ \AA}$. But other two samples(1, 2) cell parameter is not identical because bonding vibration is more as shown by FTIR figures and interaction is very weak so superconductor nature is not destroyed. The Mossbauer spectroscopy is not useful at room temperature.

5 CONCLUSION

Crystal structure of superconductors and their compounds involves large unit cells in which points defects, oxygen vacancies, impurity atoms twin planes and inter granular impurity phases are pervasive [12-20].



It is made a comparative analysis of the observed XRD, FTIR, and Mossbauer spectroscopy. These superconductors were prepared by ball mill and reported samples were prepared by annealing method. This study reveals both study gives same results and a complex formation.

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