© 2015 IJSRSET | Volume 1 | Issue 6 | Print ISSN : 2395-1990 | Online ISSN : 2394-4099
Themed Section: Engineering and Technology

Synthesisation of 2223 Superconductors

Rohitash Kumar, H. S. Singh
Department of Physics, Faculty of Science, Jai Narain Vyas University, Jodhpur Rajasthan, India

ABSTRACT

To preparation of superconductors with structure type of $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ (n=1, 2, 3), with formula ($Bi_2M_{1+n}TM_nO_x$) where M= Sr, Ba, Ca transition matel. The composition $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ were prepared by solid state reaction by Ball Mill from the principle routs like Bi-2223, Ca_2CO_3 and CuO with high purity of 99.99%. Different measurement such as resistivity measurement**,** X-Ray Diffraction and FTIR were made to changes in superconductivity. Some transition matel (M) other than Cu, reported belongs to group which contain transition metal such as iron, cobalt, manganese etc. The lattice constant obtained with Fe doped in different amounts. The T_c of the high temperature superconducting cuprate $Bi_2Sr_{4-x}Ca_xFe_{3-y}Cu_yO_z$ were obtained 157K, the resistance decreases nearly zero with decreases temperature.
Keywords: $Bi_2Sr_{4-x}Ca_xFe_{3-y}Cu_yO_z$, ball mill, Fe doping, Electrical Resistivity, XRD, FTIR, MOssbauer spectroscopy.

I. INTRODUCTION

The discovery of the Bi-Sr-Ca-Cu-O superconducting compounds Maeda et al, 1988 [1], a great deal of work has been done to prepare the high critical temperature (T_c) phases.A true superconductor not only shows zero resistance but also excludes a magnetic field completely (the Meissner effect. Muller and Takugi reported the possible existence of percolative superconductivity in $(La_{1-x}Ba_x)_2CuO_{4-y}$ with x= 0.2 to 0.5 in the 30k range and it was concerned by Uchida and Takugi et al [2],[3]. Synthesis and characterization of iron oxidesa with 2223 Bi superconductor in $Bi_2Sr_{4-x}Ca_xFe_{3-y}Cu_yO_z$ with (x=0.0, 1.0…) concerned by H.S.singh and Rohitash kumar in 2013[4]. Superconductivity has been observed in the temperature and 120-130k in Ba-Ca-Sr-Cu-O H.W. Zandbergen et al[5]. The studies of transport properties, such as electrical resistivity, thermoelectric power (S) and thermal conductivity, are important for exploring the conduction mechanisms. The Bi based superconductors offer potential advantages in comparison to the Y-based superconductors. Superconductivity is the phenomenon in which the resistivity of materials falls abruptly to zero around some temperature above the absolute zero called transition temperature (T_c) H.Kamerlingh Onnes et al [6]. The most common method used to synthesize these superconducting oxides is the solid state reaction method Koyoma et al, 1988 [7], which consist of the mixing nominal composition of high purity compounds such as Bi_2O_3, $SrCO_3$, $CaCO_3$ and CuO. The Bi-2223 phase has attracted considerable interest due to its higher Tc and the potential for applications. we can use different methods of characterization of the material as XRD, FTIR, Mossbauer effect and particle analysis etc. Our main aim of this work is to understand the variation in the property like resistivity with the substitution of various cations like Cu, Fe, Sr and Ca with different stoichiometry systematically [8].

II. METHODS AND MATERIAL

2.1 Materials

The samples were prepared by the solid state reaction method using highly pure 99.99% powders of Bi_2O_3, $SrCO_3$, Fe_2O_3 and $CaCO_3$, CuO, with appropriate weights in proportion to their molecular weights through the following chemical reactions type with formula $Bi_2Sr_{4-x}Ca_xFe_{3-y}Cu_yO_z$ [9].

2.2 The Chemical Reactions

The samples were prepared by grinding a appropriate amount of substance through the following chemical reactions.

Bi_2O_3+2$SrCo_3$+2 $CaCo_3$+0.5 Fe_2O_3+2CuO = $Bi_2Sr_2Ca_2FeCu_2O_2$+4Co_2

2.3 Sample Preparation

All the samples were measuring the weight of each reactants with the required amount, using digital electrical sensitive balance machine with (4-digits), type Adair Dutt AD-180. The mixture for each specimen was prepared by homogeneously mixing and grinding of powders into a Ball Mill type Retsch PM-100. The Bi–2223 samples were prepared from four type substance and Fe mixed different atomic weight and grinding this amount of substance in Ball mill 400rpm in 25ml grinding jars with 8 grading balls of 10mm. Total grinding time is 6hour initially. In the grinding process we get the particle size and phase changed in different position and the temperature of the sample in the jars nearly 850^0C-900^0C in 6 hour. The samples were prepared at room temperature 25^0C. The calcinations process was not performed. The resulting powder was pressed into pellets by using cylindrical die set with a stainless steel cylinder of 13 mm diameter and 1.5 to 1.8 mm thickness using manually hydraulic press by technosearch KBr PRESS Model-15**.** All the prepared samples are shown in table-2.1.

Table-2.1

S. No.	Compound	Weight in (gm)				
		Bi_2O_3	$SrCO_3$	$CaCO_3$	Fe_2O_3	CuO
1	X=0, Y=0.2	0.3909	0.4934	0.0836	0.1868	0.0132
2	X=0, Y=0.4	0.3862	0.4876	0.0826	0.1714	0.0262
3	X=0, Y=0.6	0.3817	0.4819	0.0816	0.1563	0.0389
4	X=0, Y=0.8	0.3773	0.4763	0.0807	0.1416	0.0513
5	X=0, Y=1	0.3730	0.4709	0.0798	0.0636	0.0634
6	X=1, Y=0	0.3517	0.3330	0.0752	0.1801	0.0598
7	X=1, Y=0.2	0.4459	0.4222	0.0952	0.2130	0.0151
8	X=1, Y=0.4	0.4399	0.4165	0.0941	0.1952	0.0299
9	X=1, Y=0.6	0.4340	0.4109	0.0928	0.1778	0.0442
10	X=1, Y=0.8	0.4283	0.4050	0.0916	0.1608	0.0582

Figure 2.1: Operating Ball Mills Type PM100

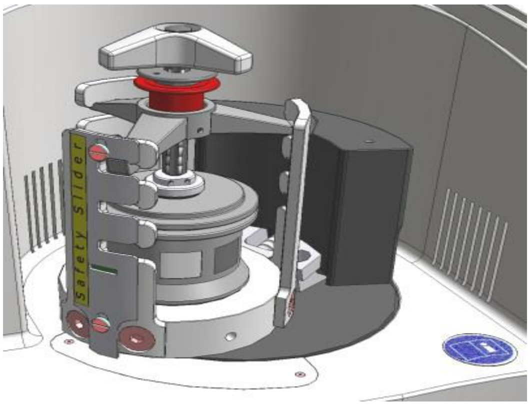

Figure 2.2

III. RESULTS AND DISCUSSION

3. SAMPLE TESTING

3.1 X-Ray Diffraction

According to table-2.1, the first five samples XRD study has been investigated by Rohitash kumar and H.S. Singh [10]. In this paper we are doing study for (x=1…..,y=0….) next five samples (6-10) are show in table-3.1. The prepared samples were characterized by X-Ray diffraction machine type PANalytical Xpert-PRO in MNIT jaipur. A automatic computational setup has been used to investigated the lattice parameter and planes of unit cell. The XRD pattern were find by CuKα *(*1.5406 Å) radiation with fitting limit 10^0 to 90^0. All the collected data is shown in table-3.1 and get nearly results of Bi-2223 superconductors and XRD pattern show in Fig-3.6, 3.7, 3.8, 3.9, 3.10.

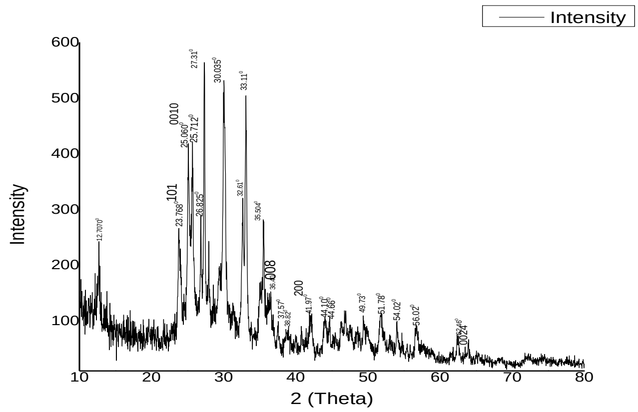

Figure 3.6 - Indexed X-ray pattern of $Bi_2Sr_3CaFe_3CuO$ Superconductor

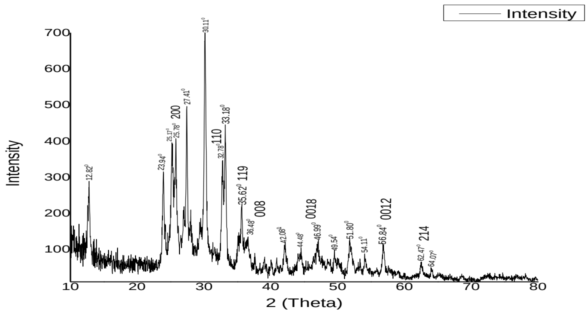

Figure 3.7 Indexed X-ray pattern of $Bi_2Sr3CaFe_{2.8}Cu_{.2}O_z$ Superconductor

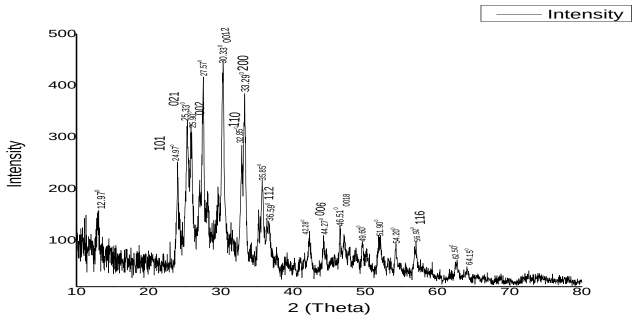

Figure 3.9 Indexed X-ray pattern of $Bi_2Sr_3CaFe_{2.4}Cu_{.6}O_z$ Superconductor

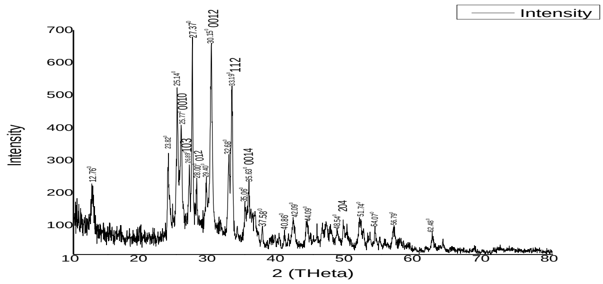

Figure 3.8 Indexed X-ray pattern of $Bi_2Sr_3CaFe_{2.6}Cu_{.4}O_z$ Superconductor

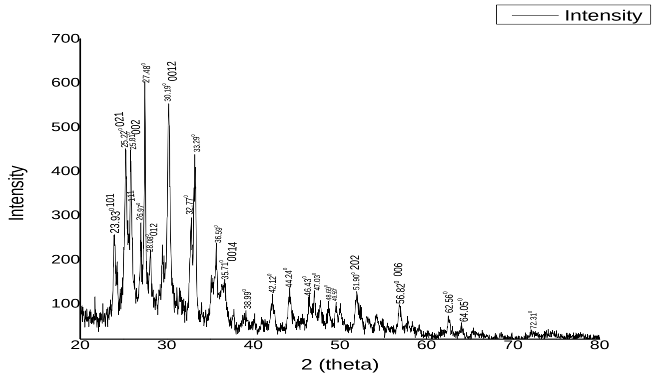

Figure 3.10 Indexed X-ray pattern of $Bi_2Sr_3CaFe_{2.2}Cu_{.8}O_z$ Superconductor

Table-3.1

S .N O.	Compound	Bi-2212 & Bi-2223			Grinding time	System	Cell Parameter
		$2\theta^0$	**d(A)**	**(h, k, l)**			A^0
6	$Bi_2Sr_3CaFe_3CuO_z$	23.76 25.06 36.40 41.97 63.96	3.743 3.552 2.467 2.152 1.455	(101) (0010) (008) (2 0 0) (0024)	6h	Tetragonal Lattice-(body centred)	a=3.810 c=30.60
7	$Bi_2Sr3CaFe_{2.8}Cu_{.2}O_z$	25.78 32..7 8 35.62 36.48 46.99 56.84 62.47	3.455 2.731 2.520 2.463 1.933 1.619 1.468	(200) (110) (119) (008) (0018) (0012) (214)	6h	Orthorhombic	a=6.008 b=8.376 c=5.863
8	$Bi_2Sr_3CaFe_{2.6}Cu_{.4}O_z$	25.77 26.82 28.00 30.15 33.19 35.63 48.54	3.456 3.314 3.186 2.963 2.699 2.519 1.875	(0010) (103) (012) (0012) (112) (0014) (204)	6h	Orthorhombic (Base centred)	a=5.426 b=5.402 c=30.64
	$Bi_2Sr_3CaFe_{2.4}Cu_{.6}O_z$	24.97 25.33 25.90 30.33	3.706 3.515 3.439 2.946	(101) (021) (002) (0012)	6h	Orthorhombic	a=5.114 b=5.420 c=37.29

9		32.85 33.29 36.59 44.27 46.51 56.92	2.725 2.690 2.455 2.045 1.952 1.617	(110) (200) (112) (006) (0018) (116)			
10	$Bi_2Sr_3CaFe_{2.2}Cu_{.8}O_z$	23.93 25.22 25.81 26.97 28.08 30.19 35.71 49.56 56.82	3.718 3.530 3.451 3.304 3.177 2.960 2.513 1.87 1.620	(101) (021) (002) (111) (012) (0012) (0014) (202) (006)	6h	Orthorhombic	a=6.010 b=8.380 c=5.870

4. RESISTIVITY MEASUREMENTS

All the measurement were carried out between 298K to157K. All the experimental work wsa carried out at IIT Roorki Uttrakhand. The Tc of the superconducting sample has been measured by using the resistivity measurement carried out by four-probe technique, which is considered as a good method for studying the electrical behavior of superconducting materials and a good tool for determining the Tc [11]. In this method, a small current is passed through a sample and the voltage drop across it. The terminals distinct from those used for passing the main part of the current through the specimen and the electrical contacts of the sample were made of fine copper wires and adhered with silver paste. The system was used for the measurement of critical resistance of the sample, with the presence of liquid nitrogen. The experimental results obtained for the different compounds are show in figure below 4.1 to 4.10. It can be seen from the variation of the resistance the sample exhibit superconducting behavior [12].

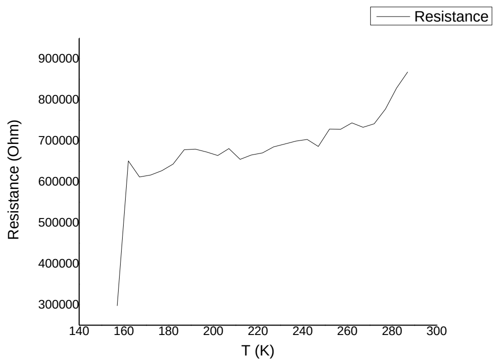

Figure 4.1 The resistance versus temperature for HTSC $Bi_2Sr_4CaFe_{2.8}Cu_{.2}O_z$ Tc, 157K

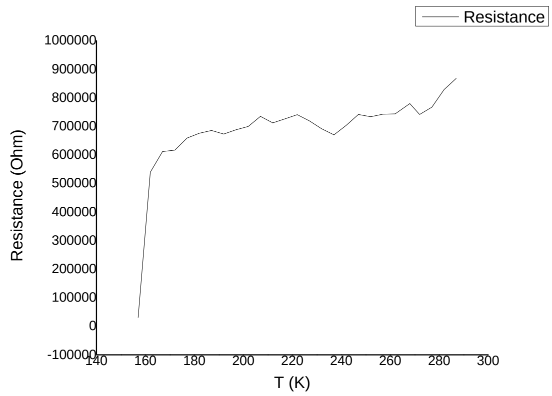

Figure 4.2 The resistance versus temperature for HTSC $Bi_2Sr_4CaFe_{2.6}Cu_{.4}O_z$ Tc, 157K

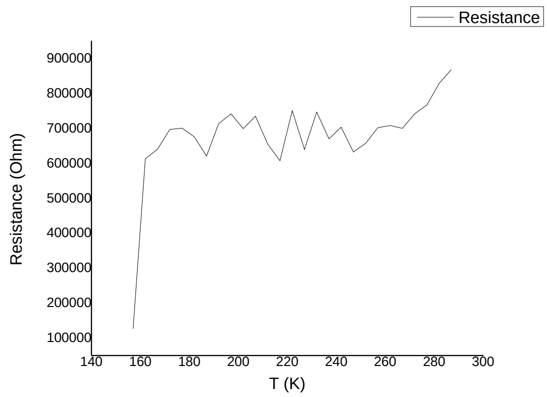

Figure 4.3 The resistance versus temperature for HTSC $Bi_2Sr_4CaFe_{2.4}Cu_{.6}O_z$ Tc, 157K

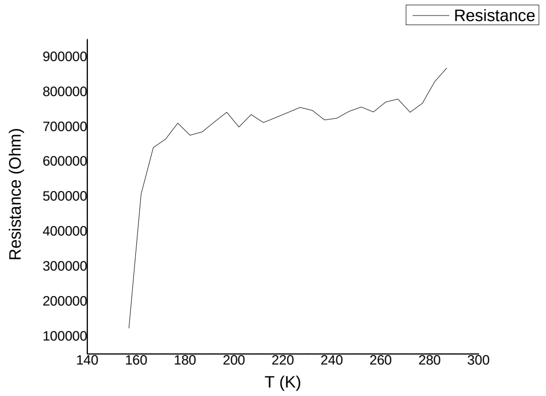

Figure 4.4 The resistance versus temperature for HTSC $Bi_2Sr_4CaFe_{2.2}Cu_{.8}O_z$ Tc, 157K

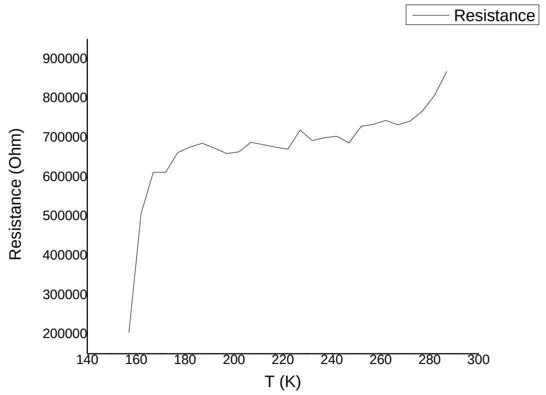

Figure 4.5 The resistance versus temperature for HTSC $Bi_2Sr_4CaFe_2CuO_z$ Tc, 157K

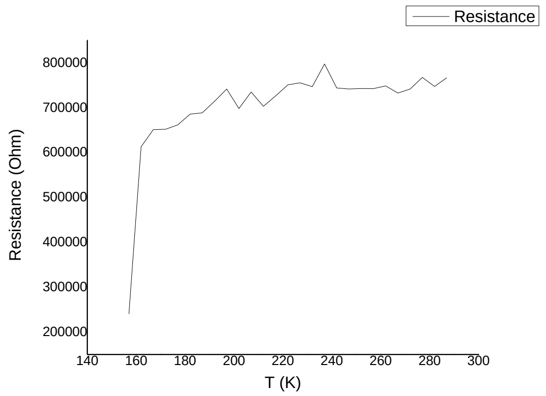

Figure 4.6 The resistance versus temperature for HTSC $Bi_2Sr_3CaFe_3CuO$ Tc, 157K

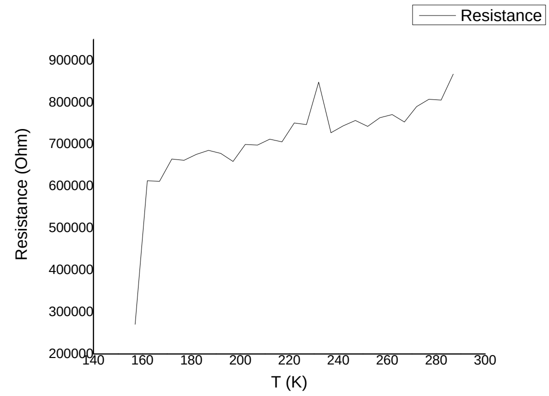

Figure 4.7 The resistance versus temperature for HTSC $Bi_2Sr3CaFe_{2.8}Cu_{.2}O_z$ Tc, 157K

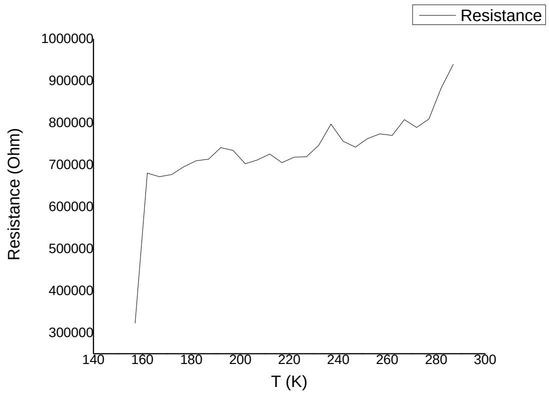

Figure 4.8 The resistance versus temperature for HTSC $Bi_2Sr_3CaFe_{2.6}Cu_{.4}O_z$ Tc, 157K

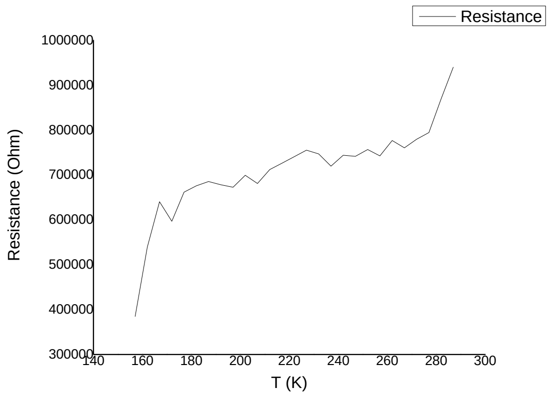

Figure 4.9 The resistance versus temperature for HTSC $Bi_2Sr_3CaFe_{2.4}Cu_{.6}O_z$ Tc, 157K

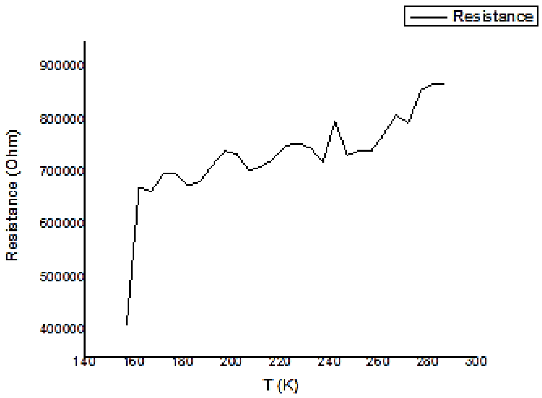

Figure 4.10 The resistance versus temperature for HTSC $Bi_2Sr_3CaFe_{2.2}Cu_{.8}O_z$ Tc, 157K

5. FTIR ANALYSIS

The FTIR study is obtain by SHIMADZU IRAFFinity-1S Spectrophotometer. One of the great advantages of infrared spectroscopy is that virtually any sample in virtually any state may be studied. Liquids, solutions, pastes, powders, films, fibres, gases and surfaces can all be examined with a judicious choice of sampling technique[13]. 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 range of spectrophotometer is 7800 to 350cm–1. The interactions of infrared radiation with matter may be understood in terms of changes in molecular dipoles associated with vibrations and rotations [14]. In order to begin with a basic model, a molecule can be looked upon as a system of masses joined by bonds with spring-like properties. Taking first the simple case of diatomic molecules, such molecules have three degrees of translational freedom and two degrees of rotational freedom. The atoms in the molecules can also move relative to one other, that is, bond lengths can vary or one atom can move out of its present plane. This is a description of stretching and bending movements that are collectively referred to as vibrations.

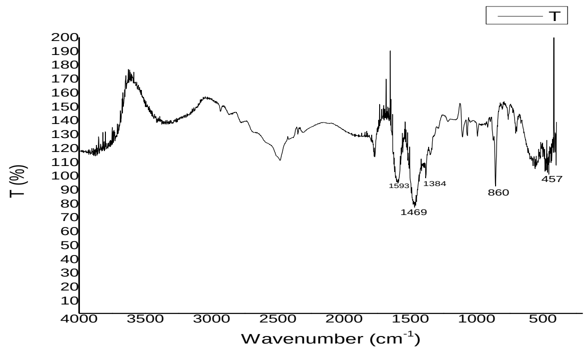

Figure 5.6

Table –5.1 sample6. FTIR spectrum of Bi-2223 superconductor

Number of effective peaks	Wave number cm^{-1}	%T
1	1593	95.15
2	1469	77.49
3	1384	98.70
4	860	94.96

After examination of the data presenting in table-5.1, results that in the range 1593-860 cm-1. The most important in the present study for evidence of bonding formation positioned at 1593 cm-1 , 1469cm-1,1384cm-1 and 860cm-1 respectively can be assigned in a tetragonal lattice. These positions are very important for cell parameter. So cell parameter is not symmetrical.

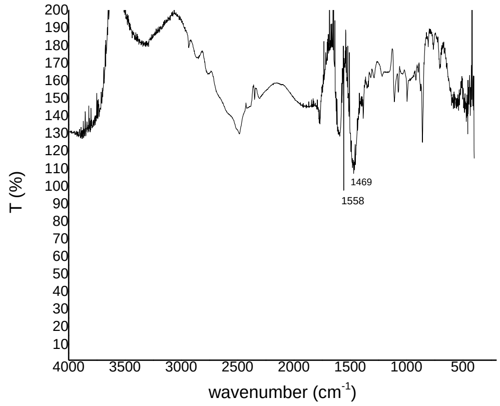

Figure 7

Table –5.2 sample-7. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm^{-1}	%T
1	1558	97.51
2	1469	107.12

After investigation of table-5.2, results that in the range 1558-1469cm^{-1}, the most important in the present study for the evidence of bonding formation in superconductor at positioned 1558cm^{-1}, 1469cm^{-1} respectively. These positions are effective to the cell parameters of orthorhombic base centered. This represented to a vibration in superconductor but position 1469 is ineffective for vibration so this is the orthorhombic system and its cell parameter is quietly different.

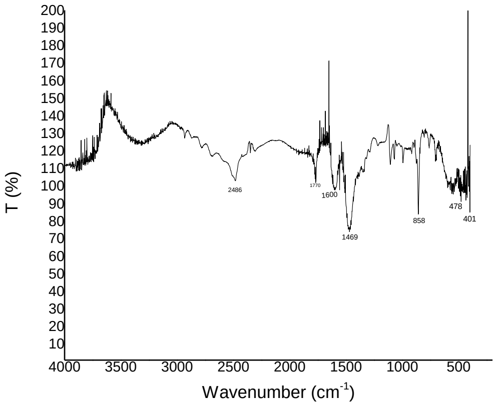

Figure 8 :

Table 5. 3 - sample-8. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm^{-1}	%T
1	401	85.13
2	478	91.25
3	858	84.07
4	1469	74.12
5	1600	97.74

In the tablel-5.3, the results that in the range 401-1600 cm^{-1}. The most important in the present study for evidence of bond formation positioned at 401 cm^{-1},487cm^{-1}, 858cm^{-1}, 1469cm^{-1}, 1600cm^{-1} respectably can be represented to a vibration in superconductor.

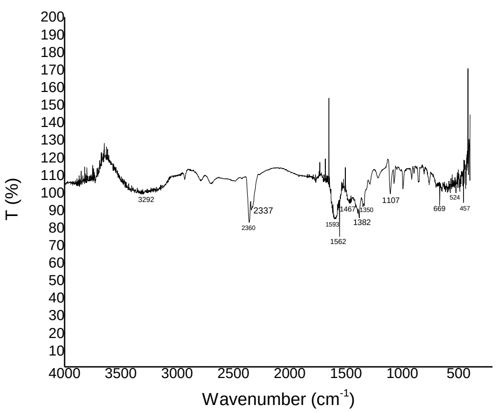

Figure 9

Table –5.4 sample-9. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm^{-1}	%T
1	457	94.46
2	524	100.0
3	669	93.02
4	1107	99.48
5	1350	92.13
6	1382	85.86
7	1467	94.07
8	1562	93.13
9	1593	85.25
10	2337	91.21
11	2360	83.25

After observation of the data presented in tablel-5.4, results that in the range 457-2360cm^{-1}. The most important in the present study for evidence of bond formation positioned at 457 cm^{-1},524cm^{-1} , 669cm^{-1}, 1107cm^{-1}, 1350cm^{-1}, 1382cm^{-1}, 1467cm^{-1}, 1562cm^{-1}, 1593cm^{-1}, 2337cm^{-1} and 2360cm^{-1} respectably can be represented to a vibration in superconductor.

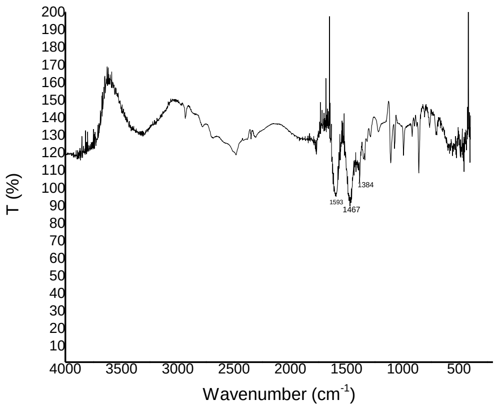

Figure 10

Table –5.5 sample-10. FTIR spectrum of Bi-2212 superconductor

Number of effective peaks	Wave number cm^{-1}	%T
1	1593	96.32
2	1467	92.35
3	1384	103.4

After measuring the data is show in tablel-5.5, the effective results in the range 1593-1384 cm^{-1}. The most important in the present study for evidence of bond formation positioned at 1593cm^{-1}, 1467cm^{-1}, respectably can be represented to a vibration in superconductor but position 1384 is ineffective for vibration so this is the orthorhombic system and its cell parameter is quietly different.

6. MÖSSBAUE SPECTROSCOPY

In 1961 the work of Rudolph L. Mössbauer (see Brown 1963), on resonant absorption of nuclear gamma rays in solids was recognized with the Nobel Prize. In this experiment, at the energy levels in an atomic system of the order of tens of micro-electron-volts using nuclear gamma rays from that system in the energy range of 14 kilo-electronvolts[15]. MÖssbauer spectroscopy is the most useful tool to study the behavior of crystals when iron used as a metal dopant at Cu sites. In the method used here, a spectrum is obtained of the number of counts per second, as a function of the velocity of the source, of 14.4 keV gamma rays that have passed through the absorber into a detector. The detector is a proportional counter which is efficient to the detection of this energy of gamma ray and is inefficient to the detection of higher energy gamma rays. This produces a series of folded count versus time spectra from which counts versus velocity can be deduced [16]. 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.

IV. CONCLUSION

According to the table-2.1, last six composition of samples are analyzing at room temperature. The XRD pattern after final grinding 6h is show in fig3.6 to 3.10. All the peaks are indexed in table3.1. XRD pattern showed peaks belonging to the high-*Tc* (2223) phase as a major component along with peaks due to the low-*Tc* (2212) phase. The dominant is the high-*Tc* (2223) phase having orthorhombic structure with lattice constants: a = 5.11 Å, b = 5.420 Å and c = 37.2 Å. 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 resistance decreases with temperature decreases according to the graphs resistance versus temperature Fig-4.1 to 4.10, this show that the resistivity is decreases with the temperature and conductivity will increases with decreases temperature. So the sample behaves likes 2223 superconductors at 157K temperature. The Mossbauer spectroscopy is not useful at room temperature. 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 It is made a comparative analysis of the observed XRD, Resistivity, FTIR, and Mossbauer spectroscopy[16-23]. These superconductors were prepared by ball mill and reported samples were prepared by annealing method. This study revels both study gives same results and a complex formation.

V. REFERENCES

[1] Maeda H, Tanaka Y, Fukutomi M, Asano T (1988). A new high-T_c oxide superconductor without a rare earth element, Jpn. J. Appl. Phys. 27:L209-L210.

[2] S.Uchida et al. Jpn. J.Appl. Phy . 26L1 (1987).

[3] H. Takagi, S. Uchida et al. Jpn. J.Appl. Phy . 26L123 (1987).

[4] H.S. singh and Rohitash kumar, international conference on ceramic, Bikaner, india IJMPCS world scientific publication, Vol. 22 (2013) 35-36.

[5] H.W. Zandbergen et al. Nature 332 620 (1988).

[6] H. Kamerlingh Onnes, research notebooks 56, Kamerlingh oness Archive, Berhaave Museum, Leiden, the Netherlands.

[7] Koyoma S, Endo U, Kawai T (1988). “Formation of high T_c phase of Bi-Pb-Sr-Ca-Cu-O superconductors”, Proceeding of the 1st international symposium on superconductivity, Tokyo Berlin, 88: 833-836.

[8] H.S. singh & Rohitash kumar, international conference on ceramic, Bikaner, india IJMPCS world scientific publication, Vol. 22 (2013) 35-36.

[9] Widad M. Faisal and Salwan K. J. Al-Ani, “Enhanced superconducting properties of the Pb doped trilayer high-temperature ($Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4\delta}$) cuprate superconductor” International Journal of the Physical Sciences Vol. 7(2), pp. 171 - 181, 9 January, 2012.

[10] Rohitash Kumae and H.S.Singh, “Spectroscopic Study on Iron Oxide Based Bi-2223 Superconductors” INTERNATIONAL JOURNAL OF SCIENTIFIC PROGRESS AND RESEARCH (IJSPR), Volume-15, Number - 01, 2015.

[11] K. Iida, S. Haindl, T. Thersle, J. Ha¨nisch, F. Kurth, M. Kidszun, R. Hu¨hne, I. Mo¨nch, L. Schultz, B. Holzapfel, and R. Heller: Appl. Phys. Lett. 97 (2010) 172507.

[12] T. Katase, H. Hiramatsu, V. Matias, C. Sheehan, Y. Ishimaru, T. Kamiya, K. Tanabe, and H. Hosono: Appl. Phys. Lett. 98 (2011) 242510.

[13] K. H. Tan, “Principles of soil chemistry,” Mariel Dekker Inc., New York, USA, 1998.

[14] G. Parthasarathy, “Effect of High-Pressures on the Electrical Resistivity of Natural Zeolites from the

Deccann Trap, Mahrashtra, India," *Journal of Applied Geophysics* Vol. 58, No. 4, pp. 321-329.

[15] H. Frauenfelder, The Mössbauer Effect, W.A. Benjamin, New York, 1962. (QC 490 F7).

[16] R.L. Mössbauer, Z. Physik **151**, 124 (1958). Later review paper: R.L. Mössbauer, Recoilless nuclear resonance absorption, Ann. Rev. Nuclear Sci. **12**, 123 (1962).

[17] R.M. Hazen, L. W. Finger, R. J. Angel, C. T. Prewitt, N.L. Ross, H. K. Mao, C. G. Hadiddiacos, P.H. Hor, R.L. Meng, C.W. Chu, Phys Rev. 835 7137 (1987).

[18] T. Siegrist, S. Sunshine, D.w. Murphy, R.J. Cava, S.M. Zahurak, Phys. Rew. 835 7137 (1987).

[19] J.D. Jorgensen, H.B. Schutter, D.G. Hinks, D.W. Capone ii, K. Zhang, M.B. Brodsky, D.J. Scalapino, Phys. Rew. Lett. 58 1024 (1987).

[20] Hoydoo You, R.K. Mcmillan, J.D. Axe, D.E. Cox J.Z. Liu, G.W. Crabtree and G.J. Lam < Sol. St Com. 64 739 (1987).

[21] P. Boolchand and D. McDaniel, Studies of High Temperature superconductors Ed. A.V. Narlikar Vol. 4, Nova, New York.

[22] H.S. Singh, Rohitash Kumar, MÖssbauer Spectroscopic Study of the Mahadevpur Meteorite Arunachal Pradesh and Assam, J. Pure Appl. & Ind. Phys. Vol.3 (2), 138-141 (2013).

[23] H.S. Singh, Rohitash kumar, "MÖSSBAUER SPECTROSCOTIC STUDY OF CHEMICAL STATE OF IRON IN NATURAL SAMPLES HORNBLENDES FROM UDAIPUR DISTRIC (RAJ)", International Journal of Scientific & Engineering Research, Volume 5, Issue 7,1114-1115 July-2014.