

## Study of the Structural and Electrical Properties of

### $\text{Bi}_{2-x}\text{Ag}_x\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ System

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#### Abstract:

This study included preparing samples of the compound ( $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ ) by the solid state reaction method under a hydrostatic pressure 8 ton/cm<sup>2</sup> and annealing temperature 840°C. X-ray diffraction (XRD), scanning electron microscopy (SEM), dc electrical resistivity measurements by four point probe method were used to investigate the microstructural and superconducting properties of Bi-2223 samples. The X-rays diffraction study for the compound ( $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ ) showed that it has Tetragonal type of crystal structure. The partial replacement of the component Ag in Bi, and Sr component in Ba simultaneously, the compound becomes ( $\text{Bi}_{2-x}\text{Ag}_x\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ ) with (x ,y) values equal to (x= 0.2, y= 0.1, 0.2, 0.3,0.4). The study of the crystal structure test showed that the structure retains on the tetragonal type with a high Tc phases (2223), low Tc phase(2212) and impurity phases, and the critical temperature Tc steps-up from (125K) to (137K) at substitution rate (x= 0.2, y= 0.1). But at increasing the substitution rate for (y) and the stability of (x) rate more than (x= 0.2, y= 0.1), the temperature declines to (108 K). Finally, the microstructural of the samples has been studied and tested by Scanning Electron Microscope for knowing the components' rates in the compound; how the compound partial substitution affect in the components; and specifying the quantitative and qualitative rates of the components in the compound.

**Keywords:** Bi-based, Bi-2223, Partial Substitution, Crystal Structure, Bi-Ba-Ca-Cu-O, LT phase , HT phase

#### Introduction

Bi-based high-temperature superconductor (HTSC) materials discovered by Maeda et al. [1] in 1988 are the most promising materials for potential technological and industrial applications[2] because of their remarkable smaller power losses, high current and magnetic field carrying capacity, optical and electronic properties [3]. The  $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_y$  system obtaining a layered structure has three different phases with regard to its chemical compositions, the Bi-2201 phase (n = 1, Tc ≈ 20 K), the Bi-2212 phase (n = 2, Tc ≈ 85 K) and the (Bi, Pb)-2223 phase (n = 3, Tc ≈ 110 K) [4]. In these series, the (Bi, Pb)-2223 phase is the most attractive owing to the highest critical temperature (Tc) of about 110 K [5]. Since discovery of the Bi-based superconductor ceramics, researchers have tried to improve their structural, mechanical and superconducting properties by using several techniques [6]. Especially, the chemical doping in the superconductor materials plays a major role to improve these properties [7]. The increase in the

critical temperature results in the enhancement of average Cu valency. Therefore, the density of mobile holes in the CuO<sub>2</sub> planes containing the magnetic Cu<sup>2+</sup> ions probably improve the superconductivity [8]. On the other hand, several techniques may occasionally cause a decrease of the number of charge carriers (either holes or electrons) in the materials and hence the superconducting properties might be lowered [9]. In this paper we studied the effect of Sr substitution on the structure and transition temperature of BBCCO system to bring this superconducting material into more useful technical forms. Current application of high temperature superconductors include, medical imaging systems, magnetic shielding devices, infrared sensors and microwave devices[10]. In the present work, we report the influence of partial substitution of Sr doping on the superconducting properties of the superconducting system  $\text{Bi}_2\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ , with (y= 0,0.1,0.2,0.3,0.4).

#### 2. Experimental Technique

The solid state reaction technique was used to prepare the superconducting samples  $\text{Bi}_2\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  from appropriate amounts of the high purity oxides  $\text{Bi}_2\text{O}_3$ ,  $\text{BaO}$ ,  $\text{CaO}$ ,  $\text{CuO}$  and  $\text{SrO}$  as starting materials. The high purity powders were mixed well and loaded in an alumina crucible which was then placed in a furnace. The temperature of the furnace was raised to  $840\text{ }^\circ\text{C}$  at a rate of  $120\text{ }^\circ\text{C/hr}$ . This material was sintered at  $840\text{ }^\circ\text{C}$  for 12 hrs then cooled down at a rate of  $30\text{ }^\circ\text{C/hr}$  in air to room temperature. The same heating process was repeated but with a flow of oxygen. The resulting material was then ground and pressed by hydrostatic pressure ( $8\text{ ton/cm}^2$ ) into 1gm pellets of approximately 12mm diameter and 1.2 mm thickness. Fig.(1) shows the annealing process carried on these pressed pellets in oxygen environment. X-ray diffraction patterns for the  $\text{Bi}_2\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  samples at room temperature were obtained using Shimadzu X-ray diffractometer with  $\text{CuK}\alpha$  source and  $\lambda=1.54060\text{ \AA}$ . From the crystal structure analysis, we have calculated the lattice parameters, c/a ratio, density ( $\rho_m$ ) and volume fraction  $V_{\text{ph-2223}}$  of the samples. The relative proportions of the Bi-2223 phase in the range of ( $2\theta = 20-70$ ) were determined from (0012), (0010), (008), (109), (200), (1011), (0212) and (1115) peaks while the other peaks represent the peak intensity of other phases. Therefore, the volume fraction of the 2223 phase ( $V_{\text{ph-2223}}$ ) in our samples was determined by using the following equation[11]:

$$V_{\text{ph-2223}} = \frac{\sum I_{2223(\text{peaks})}}{\sum I_{2223(\text{peaks})} + \sum I_{\text{other}-(\text{peaks})}} \dots (1)$$

The crystal lattice parameters (a, b and c) from a set of measured and indexed Bragg angles were calculated by using a computational program. The density of the prepared samples ( $\rho_m$ ) were calculated by using the following equation [12]:

$$\rho_m = \frac{W_m}{N_A V} \dots \dots (2)$$

Where  $N_A$  is the Avocado's number in unit (particales /g.mol), V is the volume of unit cell and  $W_m$  is the molecular weigh in unit (amu).

Standard four-probe-method was used to measure the resistivity ( $\rho$ ) versus temperature (T). For each

sample, we plotted resistivity ( $\rho$ ) versus temperature (T) and the critical temperature ( $T_c$ ) was determined. We have used the onset value and the  $T_c$  value for zero resistance to find  $T_c$ . the microstructure, surface morphology and element composition analyses of the samples are investigated by SEM and EDX measurements, respectively. The oxygen content in each sample was measured by using iodometric titration method.

### 3. Results and Discussion

The results can be discus as following:

#### 3.1 Structural Properties of the $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ System

Fig.(2) shows x-ray diffraction pattern of the Bi-2223 phase ( $x=0$ ,  $y=0$ ). It shows the existence of a single tetragonal phase with lattice parameters  $a=b=5.44\text{ \AA}$  and  $c=34.38\text{ \AA}$ .

The X-ray diffraction pattern for Bi-Ba-Ca-Cu-O system exhibited a decrease of the Bi-2212 phase and enhancement of peaks due to Bi-2223 phase and peaks corresponding to Ba compounds for example,  $\text{BaBiO}_3(2\theta=29^\circ)$  and  $\text{BaCuO}_3(2\theta=29.3^\circ \text{ and } 30^\circ)$  were strongly observed in Fig.(2). The  $\text{BaBiO}_3$  might play a role in supplying Bi for the formation of the Bi-2223 phase. Thus, the above results suggest that the growth of the Bi-2223 phase by Ba-exiting ,similar to Pb addition of the Bi-Sr-Ca-Cu-O system .This may be attributed to the ordered growth under partial melting point.

#### 3.2 Structural Properties of the $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ System

The X-ray diffraction patterns of these samples are shown in figures (3-6) for the values:  $x=0, 0.1, 0.2, 0.3, 0.4, 0.5$  respectively. From Fig.3 we can see several peaks (0012), (0010), (008) (109), (200), (1011), (0212) and (1115) for Bi-2223 phase, It is noticed that there was a slight shift in the original peaks associated with the decrease in the c-parameter in addition to appear new peaks. we can see that the peak positions are shifted towards lower  $2\theta$  values, which is evidence to exchange the atomic position within Bi-cuprates. However, this observation is also associated with broadening of some peaks, which could be result of closely spaced or overlapping peaks of LT phase and HT phase. From the crystal structure

analysis to the substitution of Ag and Sr together (see Table(1)), we saw an improvement in structure properties of Bi-2223 cuprates, as shown in Figs.(3,4) for ( $x=0.2, y=0.1, 0.2$ ), it can be noticed that the high rate of phase (2223), indicating the dominant phase of Bi-2223 together with small amount of Bi-(2212) phase and unidentified impurities which may be attributed to emerge of secondary peaks, it is noticed that increasing the volume of the lattice cell, which could be caused by growth of a larger number of Cu-O layers in the unit cell, but further substituting of Sr caused an overlapping of some peaks. This case appeared more clearly with increasing y content ( $y=0.3, 0.4$ ) see Figs. (5,6), which may be attributed to that the substitution of Ag and Sr cause an antisymmetric in crystalline structure. Moreover such results may also be due to the difference between the ionic radii for both of Sr and Ba, where the ionic radii of  $Sr^{+2}$  (1.13 Å) is shorter than of  $Ba^{+3}$  (1.35 Å) makes c-parameter shorter or gets deformed and this decreasing in c-axis can affect  $T_c$ . Similar behavior were obtained by other authors [13-15] for different compounds.

### 3.3 Electrical Properties of the $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ System

The resistivity versus temperature for sample with nominal composition  $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$  with different values of y(0.1- 0.4) are shown in Figure(7). shows a clear drop in the critical temperature with increasing the Sr except for  $y=0.4$ , where our apparatus could not help us to obtain the value of  $T_c$ (off) because it is less than the liquid nitrogen temperature.

The values of the critical temperatures,  $T_c$ , determined from the Figure(7) are summarized in Table (2). It was observed that the critical temperature was enhanced drastically for small amounts of Ag and Sr and decreased significantly with increasing Sr content. The decrease in  $T_c$  values could be attributed to the change in structure from tetragonal to orthorhombic phase. The situation was much improved with samples substituted with Ag, which is help inducing or enhancing superconductivity in a sample. Since the substitution of Ag to 0.2 promotes the high- $T_c$  phase formation of BBCCO. The increase

in  $T_c$  could mean that there was an increase in the mobility of electrons in the b-direction of Cu-O plane, can lead to an increase in  $T_c$ [16- 18].

### 3.4 Results of (EDX) and (SEM)

The  $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$  compound (with  $y=0.1, 0.2, 0.3$  and 0.4) was studied by using energy dispersive x-ray spectroscopy (EDX).are shown in Figs.(8). The EDXS plot reveals no extra peaks related to elements other than the constituents. All the samples show the exact match for standard peak position for Bi, Ag, Ba, Sr, Ca, Cu and Oxygen. This reveals that the elemental composition of all the samples does not contain any foreign elements.

The spectrum illustrated in Figs.(8a,b) show the elemental distribution in the sample .EDX analysis indicated that Ag and Sr ions have partially replaced Bi and Ba ions respectively in the system while Ca or Cu have not been replaced. We can see from Figs. ( 8c,d) that such data exhibiting the losses of element specially Bi, Ca and Cu with increasing both of Ag and Sr concentration.

Surface area images of  $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$  samples, show the formation of randomly plate-like grains. The occurrence of grains with plate-like structure is a signature of the (2223) phase formation<sup>[19]</sup>. The SEM images of the samples (a), (b), (c), and (d) are shown in Fig. (9). It is seen that the microstructure consists of large chaotically distributed crystals of plate-like form grains with porous regions between them. It is clear from the figure that the grain boundaries are in touch as to form weak bonds between each other.

The difference in the grain size and the distribution of grains on the surface of the samples indicate the influence of substitution of  $Ag^{+1}$  and  $Sr^{+1}$  together for  $Bi^{+3}$  and  $Ba^{+1}$  respectively on the morphology of the samples. The SEM image of low concentration of dopant specimen shows very small size grains with thin plate like structures, the plate-like grains were aligned to make more dense and conductive sample.

### 3.5 Results of Iodometric Titration

The  $Ag^{+1}$  and  $Sr^{+1}$  substitution effect on the excess oxygen was also investigated, to determine the optimum value of substitution ratio required to obtain the high –  $T_c$  phase in BBCCO system. Table (2)

shows these results. This Table also shows an increase of  $T_c$ . We can attribute these result to the substitution of monovalent  $Ag^{1+}$  for trivalent  $Bi^{3+}$  in  $CuO_2$  planes with mobile holes then change in lattice parameters that effect on the volume of unit cell and then causes an increase of the density. The deformation in the c-axis adjusts the amount of charge transfer from Bi-o layer to Cu-o layer sheet

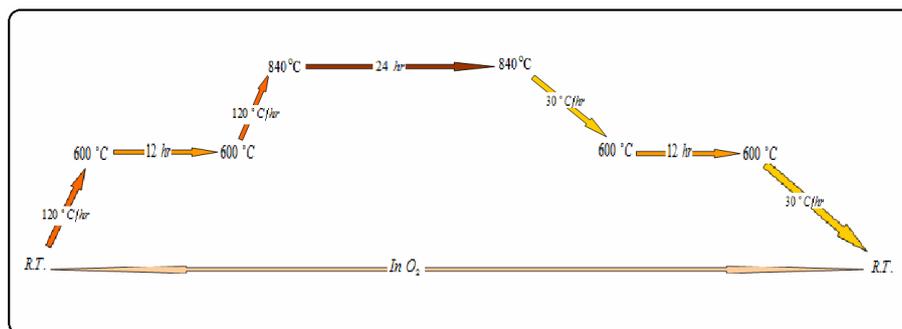
will tend to improve the critical temperature<sup>[20]</sup>, and produce superconductivity in  $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$  with maximum  $T_c$  of 137K at  $x= 0.2$  with  $y= 0.1$ . i.e. extra charge are transferred into the  $CuO_2$  layers leading to the oxidation of some copper atoms into  $Cu^{+3}$ , and a state of mixed copper valence  $Cu^{+2}/Cu^{+3}$  is created resulting in development of superconductivity<sup>[21]</sup>.

**Table 1. Values of lattice parameter, c/a,  $\rho_m$  and  $V_{ph-1223}$  for the samples of  $Bi_2Ba_2Ca_2Cu_3O_{10+\delta}$  and  $Bi_{1.8}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ , at different doping concentrations of Sr.**

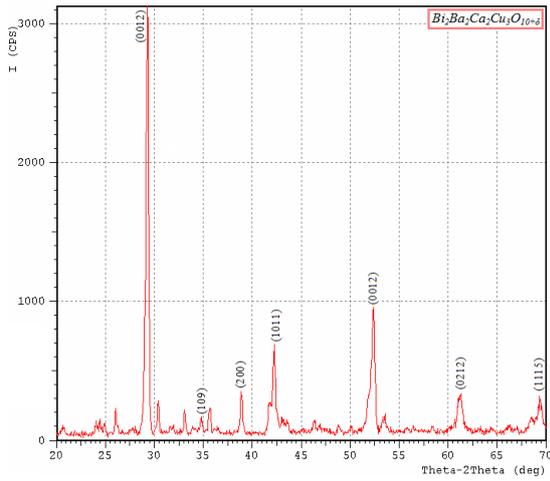
x=	y=	a(Å)	b(Å)	c(Å)	c/a	V(Å) <sup>3</sup>	$\rho_m$ (g/cm <sup>3</sup> )	$V_{ph-2223}$ %
0	0	5.44	5.44	34.38	6.31	1017.42	1.831	64.87
0.2	0.1	5.45	5.45	37.11	6.80	1102.2	1.654	73.68
	0.2	5.43	5.43	34.92	6.43	1029.61	1.763	64.69
	0.3	5.30	5.36	32.11	6.05	912.18	1.98	52.74
	0.4	5.28	5.35	30.81	5.83	870.32	2.066	41.05

**Table 2. Values of ( $\delta$ ) and  $T_c$  for samples of  $Bi_2Ba_2Ca_2Cu_3O_{10+\delta}$  and  $Bi_{1.9}Ag_{0.2}Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$  system for various values of y**

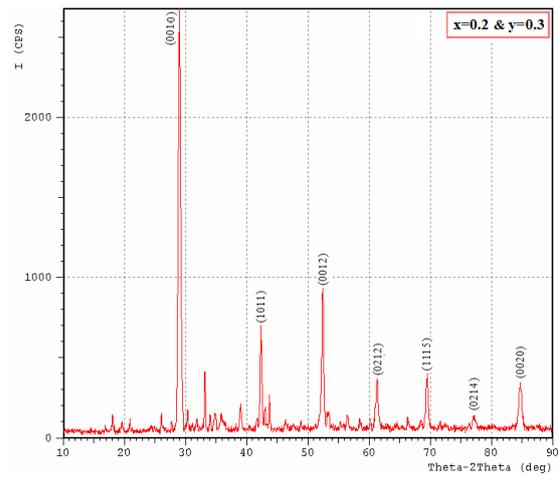
x=	y=	$T_{czero}(K)$	$T_{conset}(K)$	$\Delta T(K)$	$T_c(K)$	$\Delta$
0	0	90	160	70	125	0.18
0.2	0.1	104	170	66	137	0.32
	0.2	106	160	54	133	0.26
	0.3	<b>86</b>	130	44	108	0.21
	0.4		130			0.17



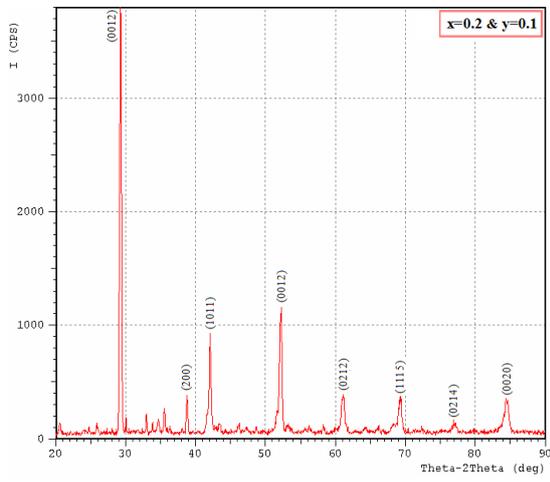
**Figure 1. Annealing Process of Pressed Pellets in O<sub>2</sub>.**



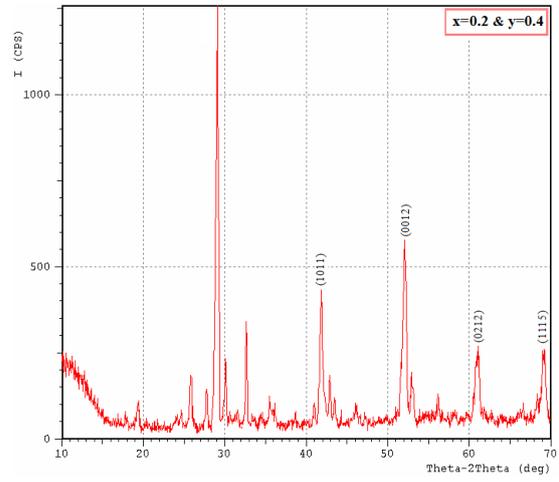
**Fig.2 X-ray Diffraction Pattern of  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  High Temperature Superconductor**



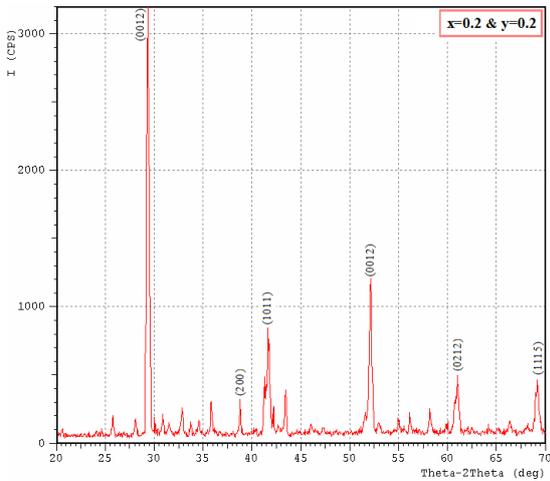
**Fig.5 X-ray Diffraction Pattern of  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{1.7}\text{Sr}_{0.3}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  High Temperature Superconductor**



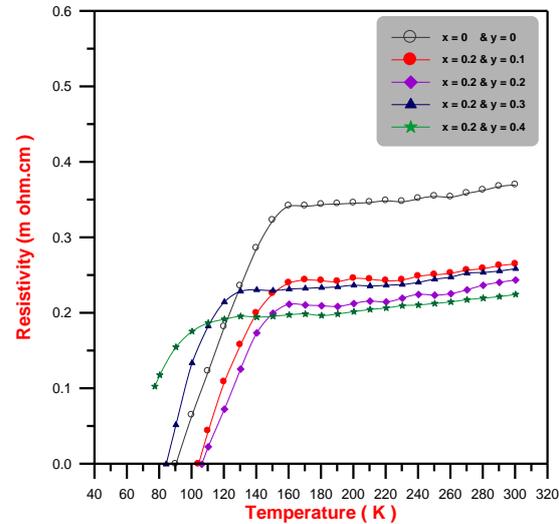
**Fig.3 X-ray Diffraction Pattern of  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{1.9}\text{Sr}_{0.1}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  High Temperature Superconductor**



**Fig.6 X-ray Diffraction Pattern of  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{1.6}\text{Sr}_{0.4}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  High Temperature Superconductor**



**Fig.4 X-ray Diffraction Pattern of  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{1.8}\text{Sr}_{0.2}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  High Temperature Superconductor**



**Fig. 7: Temperature Dependence of Resistivity for  $\text{Bi}_{2-x}\text{Ag}_x\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  System**

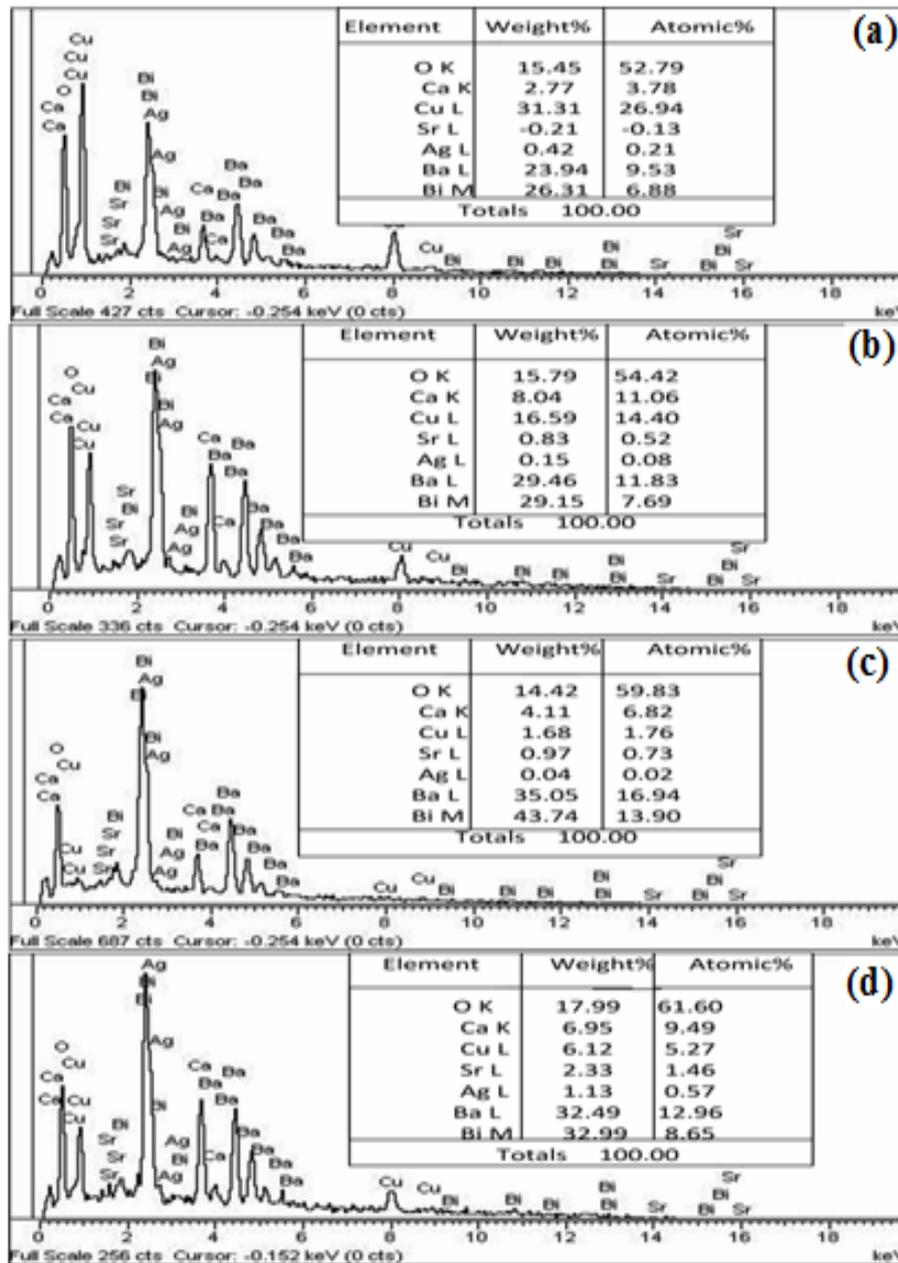
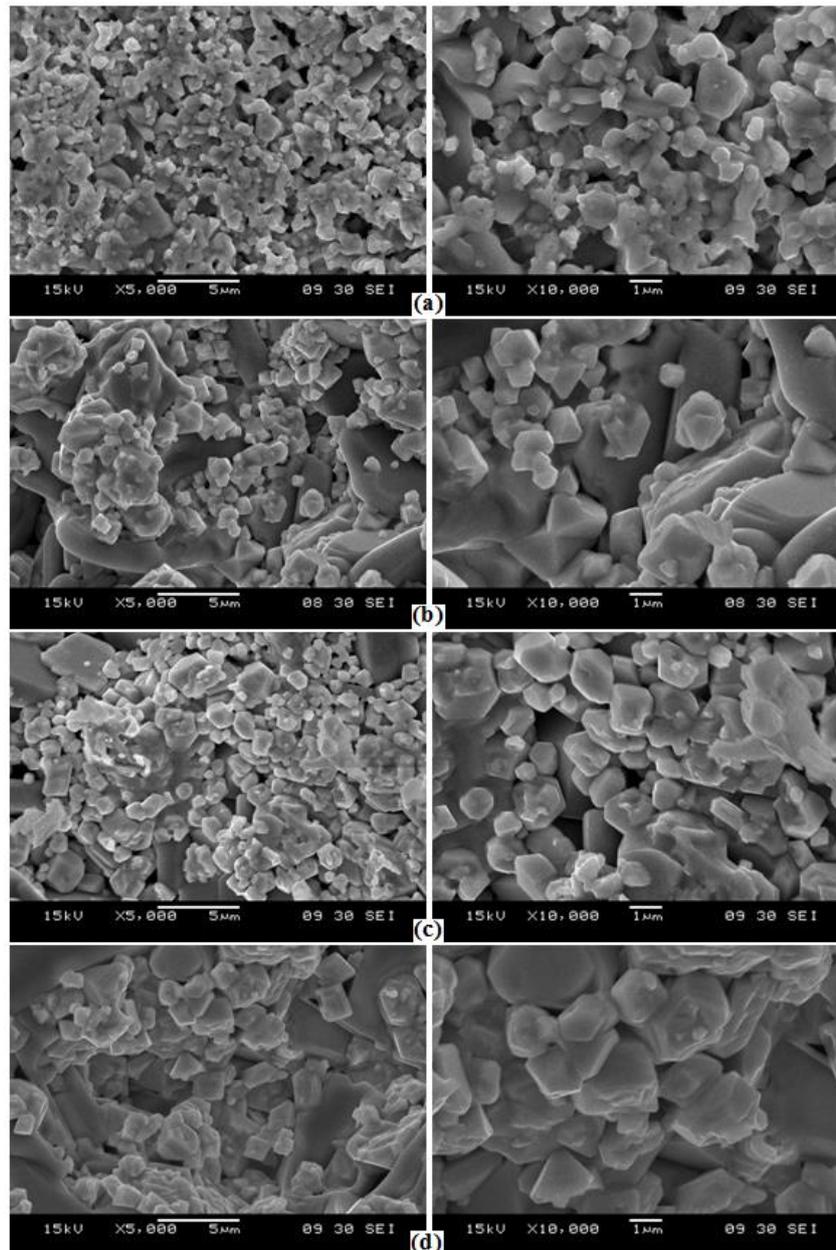


Fig. (8): EDX patterns of the  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+y}$  samples for various values of y: (a) y = 0.1, (b) y = 0.2, (c) y = 0.3, (d) y = 0.4.



**Fig.9 SEM Micrographs of the  $\text{Bi}_{1.8}\text{Ag}_{0.2}\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  Samples for Various Values of  $y$ : (a)  $y=0.1$ , (b)  $y=0.2$ , (c)  $y=0.3$  & (d)  $y=0.4$**

#### 4. Conclusions

The partial substitution of Ag and Sr to the  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compounds has a great effect on the enhancement of the higher- $T_c$  phase and enhances the structure stability of the superconducting phases. It could be seen from the spectra that there were two main phases in all samples of the Bi-base systems, high- $T_c$  phase (2223), low- $T_c$  phase (2212) and a small amount of impurity phases of  $\text{Ca}_2\text{CuO}_3$  and CuO. The appearance of more than two phases could be related to the stacking faults along the c-axis. The increasing of Ag and Sr content leads to emerge of

secondary peaks, which corresponded to some impurity phases. This case appeared more with increasing of x and y content, which may be attributed to the substitution of Ag and Sr which cause an antisymmetric in crystalline structure, and conversion of its lattice.

Partial replacement for Ba by Sr decreasing the volume fraction of high- $T_c$ , the small ionic radii of Sr in comparison with Ba resulted in contraction c-axis parameter. Thus, lattice distortion take place with higher substituting and hence decrease its  $T_c$  rapidly.

Moreover we can attribute these results to the similar oxidation, as well as, both of Ba and Sr ions prefer two coordination, this local relaxation about the

oxygen defect would be coupled to the substitution of Sr within Ba–O plane.

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## دراسة الخصائص التركيبية والكهربائية لنظام $(\text{Bi}_{2-x}\text{Ag}_x\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$

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### الملخص

تضمنت هذه الدراسة تحضير عينات لمركب  $(\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$  بطريقة تفاعل الحالة الصلبة وباستخدام طريقة التلدين وتحت ضغط هيدروستاتيكي (8 طن/سم<sup>2</sup>) ودرجة حرارة تلدين (840 درجة مئوية). تم استخدام (XRD) ، (SEM) وقياسات المقاومة الكهربائية المستمرة من خلال طريقة الاقطاب الاربعه لغرض دراسة خصائص النماذج المحضرة .

أظهرت دراسة حيود الاشعة السينية للمركب  $(\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$  بأنه ذو تركيب بلوري من النوع الرباعي القائم وعند التعويض الجزئي للعنصر (Ag) محل (Bi) والعنصر (Sr) محل (Ba) في أن واحد ليصبح المركب بالصيغة التالية  $(\text{Bi}_{2-x}\text{Ag}_x\text{Ba}_{2-y}\text{Sr}_y\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$  وبنسب ل (X,y) مساوية ل (0.2 , 0.1 , 0.2 , 0.3 , 0.4) أظهرت دراسة فحص التركيب البلوري بأن التركيب يبقى محافظاً على صيغة الرباعي القائم بينما ازدادت قيمة درجة الحرارة الحرجة ( $T_c$ ) من (125 K) إلى (137 K) وذلك عن نسبة تعويض

(x=0.2 , y=0.1) ولكن عند زيادة نسبة التعويض ل (y) مع ثبوت قيمة (X) انخفضت درجة الحرارة الحرجة إلى (108K) . تم دراسة وفحص البنية المجهرية للعينات بواسطة المجهر الالكتروني الماسح لمعرفة نسب العناصر بالمركب ومدى تأثير التعويض الجزئي للعناصر في المركب ولتحديد نسبة التركيز الكمي للعناصر في المركب .