Manufacturing and studying the effect of partial substitution on the properties of the compound Bi$_{2-x}$Ag$_{3}$Sr$_{1.9}$Ba$_{0.1}$Ca$_{2}$Cu$_{3}$O$_{10+\delta}$ superconductors.

Noor S. Abed$^1$, Abdulsalam S. Baqi$^1$, Bilal A. Omer$^2$, Shatha H. Mahdi$^3$, Kareem A. Jaseem$^3$ and Saja Amer Ahmed$^3$.

1 Director of Education of Kirkuk, Kirkuk, Iraq  
2 Dep. of Physics, College of Sciences, Kirkuk University, Kirkuk, Iraq  
3 Dep. of Physics, College of Education for Pure Science (Ibn-Al-Haitham), University of Baghdad, Iraq

Abstract. The effect of the silver fractional exchange at Bi sites on the structure, electric, morphologic and $\delta$ content for Bi-2223 have been thoughtful. High temperature superconductor composition (Bi$_{2-x}$Ag$_3$Sr$_{1.9}$Ba$_{0.1}$Ca$_{2}$Cu$_{3}$O$_{10+\delta}$) with (x=0.1 - 0.4) which preparing by method of SSR. XRD diffraction technique was used to estimate the structural properties of the specimens which show an orthorhombic crystalline structure for all the specimens with elevated rate of Bi-2223 phase and growing of c-axis lattice parameter with growing Ag rate. By using AFM in 3D the surface morphology has been thoughtful. The results of all samples specimens showed that have homogeneous surface and good crystalline. $T_c$ measured by using 4-probe technique. The highest $T_c$ was noted to be $T_c=138$ K for x=0.4, and oxygen contend was observed increase with increase Ag concentration.

1. Introduction

Ago the detection of the superconducting copper oxides the fractional exchange methods or the doping used to enhance some of applications and properties of matters [1-5]. (Bi-Sr-Ca-Cu-O) system is the important and thoughtful superconducting systems because it has a promising future in high magnetic field applications at liquid nitrogen temperature [6]. The $T_c$s of such system changed between (7-110) K and they are concerning directly for four possible superconducting phases, i.e., Bi$_2$Sr$_2$CuO$_y$, Bi$_2$Sr$_2$CaCu$_2$O$_{y+\delta}$, Bi$_2$Sr$_2$CaCu$_2$O$_{y+\epsilon}$, and Bi$_2$Sr$_2$Ca$_2$Cu$_{3}$O$_{12}$ [2, 7-9]. It is difficult to prepare the phase (Bi-2223) which is recognized by the highest $T_c$ (110K) among the four phases as a single phase. The enhancement in the properties of superconductors can be controlled by adding or substituting an element different in its ionic radius and its bonding properties. There are many research that studied different doping elements in different position of (BSCCO), like rare earth elements [4,10-22], oxides as the Cr$_2$O$_3$ [23], and several transition elements and alkali metals such as (Ba, Hg, Na, Li, Pb, Zn, V,Y, Fe) [1,7,10,22,24-27,28,29,30,31]. Those studies have shown exchanges in crystal structure, electric and morphologic characteristic over a certain dopant content [4, 5, 10-28, 32].

In this research, we study doping of the influence on the electric and morphologic characteristic and the crystal structure of the (BSCCO) system. The specimens which prepare with titular structure Bi$_{1-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+\delta}$ with (x=0.1, 0.2, 0.3 and 0.4).

2. Materials and methods

The specimens are prepared by the solid state reaction using a specific weight of highly purified oxides powder of (Bi$_2$O$_3$, SrO, CaO, CuO, AgO). The high $T_c$ superconducting samples with nominal composition of Bi$_{1-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_{3}$O$_{10+\delta}$ (where x=0.1, 0.2, 0.3 and 0.4) ceramic superconductor were prepared from powders of Bi$_2$O$_3$, SrO, CaO, CuO, BaO and AgO with 99.9% purity. The
Powders were weighted by digital balance according to their stoichiometric ratio. Powders mixing and grinding in a gate mortar for 60 minutes together to get the best state of homogeneity. Isopropanol is added through the grinding operation to avert the waste parts of powders through the grinding operation. After grinding the powders place inside an electric furnace at temperature (120°C) for (1.5 h) to take the isopropanol alcohol away from the specimens. After that the results powders compressed to disc shaped pellets with diameter 1.5 cm and thickness 0.3 cm, under pressure (7 ton/cm²) by using hydraulic press for one minute. High Tc phase BSCCO superconductor form by sintering the pellet for 140 hours at 850°C to form a at heating rate (5C˚/min) after that cool to R.T with similar heating ratio. The heat treatments timetable for Bi-2223 pellets in programmable temperature controlled silicon carbide (SiC) furnace. The structure characteristics of 2223-phase was examine by XRD technique with diffraction angle range (20˚–60˚). By using (hkl) of the XRD and d-values lattice constants (a, b and c) are evaluate employ standard card (ICDD-042-0743) for Bi-2223, (ICDD-079-2183) for Bi-2212 and (ICDD-39-0283) for Bi-2201. Volume fraction (V-ph) evaluate by using the relation (1) for any phase:

\[ V_{ph} = \frac{\Sigma I'}{\Sigma I' + \Sigma I1 + \Sigma I2 + \Sigma I_n} \times 100\% \]  

I' is peak intensity for the evaluate phase, I1, I2, In intensity for any peaks of all XRD. Mass density (dm) evaluated by employ the relation:

\[ dm = \frac{M_{wt}}{N_A \times V} \]  

Where \( M_{wt} \) is the molecular weight, NA is Avogadro no. (6.022*1023 mol⁻¹), and V is unit cell volume.

The oxygen content evaluated by used the chemical method which called iodometric titration [33]. 4-probe technique was employ to measure the electrical resistivity as function of temperature with range (80–300) K employ the relationship:

\[ \rho = \frac{RA}{L} \]  

(LCR) meter in range of [50 Hz–10 MHz] is used to measurement dielectric properties at the R.T with range of frequency between [50 Hz – 1MHz]. The dielectric loss factor (\( \varepsilon'' \)), the alternating electrical conductivity (\( \sigma_{A,C} \)), and dielectric constant (\( \varepsilon'_r \)), evaluated employ the following relationships [34, 35]:

\[ \varepsilon'' = \varepsilon'_r \times D \]  
\[ \sigma_{A,C} = 2 \times \pi \times f \times \varepsilon'' \]  
\[ \varepsilon'_r = \frac{C}{A \times \varepsilon} \]

D dissipation factor, A effective area and t pellet thickness. For the morphology characterization of the surface, it was used atomic force microscopes (AFM).

3. Results and Discussion

3.1. X-Ray Diffraction Results:

XRD analysis showed orthorhombic polycrystalline structure for all samples that have been composed and observed that all the specimens contain a high ratio of the high temperature phase (Bi-2223) and a few peaks of low temperature phases (Bi-2212) and (Bi-2201) that has a low temperature, with appearance of some unknown impurity phases which are agreement with reference [36, 37]. The aspect of two or three phases could be related to the stacking mistake along the c-axis or/and attributed to the exchange of Ag in Bi position which leads to deform the structure. It was noted that growing the Ag concentration lead to growing the intensity of the peaks as shown in Figures 1. The maximum intense peak pattern of specimens belongs to the high (Tc) phase (Bi-2223) which also point out an increase in the volume fraction of the high-Tc phase. All specimens exhibited an enhancement of high Tc (Bi-2223) peaks with increasing Ag concentration as listed in Table 1. These results pointed that the exchange by Ag enhance the crystalline arrangement degree [38], and (v-ph)
and stability of the Bi-2223 phase the formulation can be modified by replacement or addition of elements with different ionic radius.

The parameters (a, b, c), V, c/a and dm were also calculated from the XRD analysis as shown in Table 2. From estimation of lattice parameters (a, b, c) the results show an orthorhombic crystalline structure type for all specimens. The change in (c) axis can be due to many causes, the first was the increment in (O$_2$) concentration that resulted from the partial substitution of (AgO) this increment will be taken by the double (Bi-O) layers which cause a stronger link because of the High equivalence of (Ag) that lead to increase ion bonding forces formed in the (Bi-O) layers and thus increase the lattice parameter c with increase Ag content. It can be understand that the volume of (Bi-2223) phase change systematically with c-parameter. This change in the lattice parameters affect the volume of the unit cell and then cause an increase in the density. The rate c/a increase with (Ag) content as shown in figure 2. The second is that the difference in ionic radius can cause deformities in the lattice parameters [39, 40].Which can be explained by substitution of (Ag) (1.26 Å) larger ionic radius instead of (Bi) (1.03Å) ionic radius. Which lead to generate the coupling of superconductor holes and the main carriers which are the forming bosons.

3.2. Oxygen Content and T$_c$ Results

The temperature dependence on the electrical resistivity of (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+\delta}$) with $x$= (0.1, 0.2, 0.3 and 0.4) are shown in Figure 3. It is noticed that the pellets shows a metallic conduct, then follow by a superconductivity transmission with a transition temperature equal to 125,128,130 and 138 K for the samples with $x$= ( 0.1, 0.2, 0.3 and 0.4) respectively. From Table 3. it was found that the oxygen content ($\delta$) of the specimens growing with growing of Ag content and the enhance of $\delta$ is going to enhance the hole content in the CuO$_2$ layers then the T$_c$ will enhance from 125 K to 138 K respectively with growing Ag content as shown in Figure 4 . These refer to that the increase in the T$_c$ and the $\delta$ values will lead to the increase of oxygen absorption through crystallization process of the superconductors. From the results we can understand that the $\delta$ equal 0.3491 is optimum value.

3.3. Dielectric Results

The dielectric loss factor and dielectric constant lowering with growing the frequency from (50Hz-1MHz) at R.T. with Ag doping. This is because of the ionic radius of Ag which exchange with Bi which is longer than Ag. The real-part affords the value of energy which stocked inside the material when electrical field is utilized to the material. Inter the grains are the extreme expected place which this energy may be stocked and $\varepsilon'_r$ refer to the attenuation and absorption of the energy. The $\sigma_{AC}$ value are growing with growing of the frequency, and transport of electrons no. is little at low frequencies, because of the quantity of kinetic energy in which the electrons have to hold to pass the crystal is little ,which means $\sigma_{AC}$ is little and $\varepsilon''_r$ is increase. The quantity of kinetic energy at high frequencies is growing in which electrons should hold to pass the crystalline, thus the electrons no. transport is more, so $\sigma_{AC}$ growing and $\varepsilon''_r$ decline. These results were nearly matching to the reported in references [41].

3.4. AFM Results

All samples of (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+\delta}$) with $x$= (0.1, 0.2, 0.3, 0.4) are imaged after prepared in SSR method by using an atomic force microscope as shown in Figure 8. The elements of compound are modify in to a tiny dimensions. Table 4 shown the surface grossness and average diameter for all specimens. The values of average size range are found to be change with the Ag content with (80, 65, 50 and 35) nm respectively. These images show that these specimens are homogeneous, uniformly distributed features with no pinholes or island structure.
4. Conclusions

In this paper, we have studied (Bi$_2$Ag$_x$Sr$_{1.3}$Ba$_{0.7}$Cu$_2$O$_{8+δ}$), from superconductor compounds with ratio (x= 0.1 - 0.4) which prepared by SSR method. X-ray pattern of Bi-2223 superconductor-phase analyses have exhibit orthorhombic structure and the c-axis lattice parameter increase with increasing Ag concentration. The optimum value of x is that the optimum rate for Ag in the compound (Bi$_{2.3}$Ag$_x$Sr$_{1.3}$Ba$_{0.7}$Cu$_2$O$_{8+δ}$), is at x = 0.4 where a high ratio of Bi-2223 phase appears. The substitute of Ag in the compounds (Bi$_{2.3}$Ag$_x$Sr$_{1.3}$Ba$_{0.7}$Cu$_2$O$_{8+δ}$), has showed a maximum value of Tc (138 K) at x=0.4 in addition, oxygen content δ have been found increases with increasing Ag concentration because the exchange created a local pressure, variation electronic state, hole carrier concentration and its deal. The atomic force microscopy results showed up that the values of the average diameter and surface roughness at ratio x =0.4 because the samples have homogeneous surface, v. good crystalline with best value of nano size (35 nm).

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Table 1. The (v-ph) of the Bi-2223, Bi-2212 and Bi-2201 phases for all prepared specimens.

| Phase       | Ag (0.1) | Ag (0.2) | Ag (0.3) | Ag (0.4) |
|-------------|----------|----------|----------|----------|
| Bi - 2223 (%) | 67       | 71       | 73       | 77       |
| Bi - 2212 (%) | 19       | 17       | 17       | 14       |
| Bi - 2201 (%) | 14       | 12       | 10       | 9        |

Table 2. Lattice parameters of (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$) with $x= (0.1, 0.2, 0.3, 0.4)$.

| Specimens       | x     | a   | b   | c   | V(A$^3$) | dm   | c/a |
|-----------------|-------|-----|-----|-----|----------|------|-----|
| Bi$_{1.9}$Ag$_{0.1}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.1   | 5.4875 | 5.4279 | 37.1989 | 1108.009 | 2.1887 | 6.778 |
| Bi$_{1.8}$Ag$_{0.2}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.2   | 5.4596 | 5.4729 | 37.3221 | 1115.193 | 2.1237 | 6.836 |
| Bi$_{1.7}$Ag$_{0.3}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.3   | 5.4640 | 5.4011 | 37.5969 | 1109.559 | 2.0833 | 6.880 |
| Bi$_{1.6}$Ag$_{0.4}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.4   | 5.4583 | 5.4458 | 37.7407 | 1121.857 | 2.0098 | 6.914 |

Table 3. Oxygen content and critical temperature ($T_c$) of (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$) with $x= (0.1, 0.2, 0.3, 0.4)$.

| specimens       | $\delta$ | $T_c$ |
|-----------------|----------|-------|
| Bi$_{1.9}$Ag$_{0.1}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.2835 | 125   |
| Bi$_{1.8}$Ag$_{0.2}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.2876 | 128   |
| Bi$_{1.7}$Ag$_{0.3}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.2889 | 130   |
| Bi$_{1.6}$Ag$_{0.4}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 0.3491 | 138   |

Table 4. Surface roughness and average diameter for (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$) With $x= (0.1, 0.2, 0.3, 0.4)$

| specimens       | Average diameter | Surface roughness |
|-----------------|------------------|-------------------|
| Bi$_{1.9}$Ag$_{0.1}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 80 nm            | 0.266 nm          |
| Bi$_{1.8}$Ag$_{0.2}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 65 nm            | 0.202 nm          |
| Bi$_{1.7}$Ag$_{0.3}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 50 nm            | 0.84 nm           |
| Bi$_{1.6}$Ag$_{0.4}$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+d}$ | 35 nm            | 0.62 nm           |
Figures 1. XRD of (Bi$_{2-x}$Ag$_x$Sr$_1.9$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+\delta}$) with $x=0.1, 0.2, 0.3, 0.4$. 
Figure 2. The rate of the lattice parameter \((c/a)\) as a function of \((Ag)\) content. D of \((Bi_{2-x}Ag_xSr_{1.9}Ba_{0.1}Ca_2Cu_3O_{10+\delta})\) with \(x=\) (0.1, 0.2, 0.3, 0.4).

Figure 3. The electrical resistivity vs. temperature behavior of \((Bi_{2-x}Ag_xSr_{1.9}Ba_{0.1}Ca_2Cu_3O_{10+\delta})\) with \(x=\) (0.1, 0.2, 0.3, 0.4).

Figure 4. The critical temperature as a function of \((Ag)\) concentration.
Figure 5. Showed dielectric constant as function of frequency of $(\text{Bi}_{2-x}\text{Ag}_x\text{Sr}_{1.9}\text{Ba}_{0.1}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$ with $x=(0.1-0.4)$.

Figure 6. Showed dielectric loss factor as function of frequency of $(\text{Bi}_{2-x}\text{Ag}_x\text{Sr}_{1.9}\text{Ba}_{0.1}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$ with $x=(0.1-0.4)$.

Figure 7. Showed $\sigma_{AC}$ as function of frequency of $(\text{Bi}_{2-x}\text{Ag}_x\text{Sr}_{1.9}\text{Ba}_{0.1}\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})$ with $x=(0.1-0.4)$. 

Figure 8. 3D AFM images of (Bi$_{2-x}$Ag$_x$Sr$_{1.9}$Ba$_{0.1}$Ca$_2$Cu$_3$O$_{10+\delta}$) with x= (0.1, 0.2, 0.3, 0.4)