ISSN1027-5495. Functional Materials, 32, No.1 (2025), p. 42-49
doi:http://dx.doi.org/10.15407/fm32.01.42 © 2025 — STC "Institute for Single Crystals"

Double partial substitution effect of silver (Ag) and strontium (Sr) on the structural and electrical properties of high temperature $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ superconductor

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Received December 12, 2024

Superconductor samples with the chemical composition $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ were synthesized using solid-state technology and the effect of partial substitution of Sr (y=0, 0.1, 0.2, 0.3, 0.4) and Ag (x=0, 0.2) on their electrical and structural properties was investigated. X-ray diffraction analysis showed that all samples have the tetragonal crystal structure with the largest lattice constant (c) at x = 0.2 and y = 0.1. Electrical resistance measurements using the four-probe method showed the critical temperatures $T_c(1)$=127.5 K and $T_c(2)$=152.7 K (T_c) for the pure sample (x=0, y=0). When introducing Ag and Sr in the optimal concentration ratio (x=0.2, y=0.1), the T_c values increased significantly reaching $T_c(1)$=139.2 K and $T_c(2)$=170.5 K. This is explained by the regularization of the crystal structure, as evidenced by the increase in the c-axis length and the oxygen percentage (δ). However, as the Sr concentration ratio further increased (from x=0.2, y=0.2 to x=0.2, y=0.4), the T_c values tended to decrease, with the lowest values being $T_c(1)$=134.9 K and $T_c(2)$=163.2 K at (x=0.2, y=0.4). This decrease was attributed to the introduction of Cu-O impurities, which disrupted the crystal structure. Overall, the study identified the concentration ratio (x=0.2, y=0.1) as the most favorable for achieving the highest T_c, confirming its potential for improving the superconducting properties of the material. The samples were classified as type II superconductors.

Keywords: Superconductor, Solid state reaction method, Sintering and annealing process, Structural and Electrical Properties.

Ефект подвійного часткового заміщення срібла (Ag) та стронцію (Sr) на структурні та електричні властивості високотемпературного надпровідника $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$. *Mudatheer M. Al-Slivani, Muhammod A. Hammod, Mazin A. Abed*

Зразки надпровідника з хімічним складом $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ були синтезовані з використанням твердотільної технології, і було досліджено вплив часткового заміщення Sr (y=0, 0,1, 0,2, 0,3, 0,4) і Ag ($_x$). Рентгеноструктурний аналіз показав, що всі зразки мають тетрагональну кристалічну структуру з найбільшої постійної решітки (c) при x=0,2 та y=0,1. Вимірювання електричного опору з використанням чотиризондового методу показали, що критична температура (T_c) чистого зразка (x=0, y=0) була $T_c(1)$=127,5 K і T_c (2)=152,7 K. Коли Ag і Sr були введені при оптимальному співвідношенні концентрацій (x=0,2, y=0,1), значення T_c значно збільшилися, $T_c(1)$=139,2 K і T_c (2)=170,5 K. Це пояснюється регулярністю кристалічної структури, про що свідчить збільшення довжини осі c і відсоткового вмісту

кисню (δ). Однак, у міру подальшого збільшення співвідношення концентрацій Sr (з x=0,2, y=0,2 до x=0,2, y=0,4) значення T_c мають тенденцію до зниження, при цьому найнижчі значення склали T_c (1)=134,9 K і T_c (2)=163,2 K при (x=0,2, y=0,4). Це зниження було пов'язане із введенням домішок Cu-O, які порушили кристалічну структуру. В цілому дослідження визначило співвідношення концентрацій (x=0,2, y=0,1) як найбільш сприятливе для досягнення найвищої T_c, підтвердивши його потенціал для поліпшення надпровідних властивостей матеріалу. Зразки були класифіковані як надпровідники II типу.

1. Introduction

The phenomenon of superconductivity is characterized by the flawless transmission of electricity through a material that exhibits no electrical resistance whatsoever at extremely low temperatures. This property is usually observed in metallic substances under conditions of severe freezing [1]. Superconductivity involves a joined pair of simultaneous phenomena. The first phenomenon is ideal conductivity, the second is absolute diamagnetism. This strange phenomenon occurs in various materials when they are exposed to low temperatures, called critical temperature. This main threshold is designated by the symbol T_c . Upon reaching the critical temperature, the material transforms from its standard state to the superconducting state, and the critical temperature value varies for different elements. Although this may seem strange at first, further study shed light on this fascinating concept [2].

Superconductors are high-tech materials with wide applications in energy collection and transmission, magnetic technologies, power generation and transmission as well as maglev transportation. They exhibit zero electrical resistance, perfect conductivity, diamagnetic properties, and the Meissner effect. The potential applications are what drives this field and superconductor research. Given their unusual properties, it is imperative that superconductors be thoroughly studied and understood to see if they can be further used in technology across all fields. Superconductors are a field in a state of rapid evolution; they can be classified according to coherence length and critical temperature into different classes: Type I and Type II superconductors, as well as high- or low-critical-temperature superconductors with crossover properties between each other [3].

In the superconducting system Ba-La-Cu-O, which Bednorz and Müller discovered in 1986, there is a new class of superconducting materials that are mostly formed of copper oxide. This compound is superconducting up to a critical temperature of (T_c=93 K) [4]. Drolet et al. and Schilling subsequently [5] achieved (T_c=133 K) in (Hg-Ba-Ca-CuO), then they reported two new phases named Hg-1212 and Hg-1223 for related mercury-based series having composition of the form n1n23, so that there is no agreement on formula or structure. The n =1 one member of this series, Hg-1201 with a tetragonal crystal structure has the maximal critical temperature (T_c=95 K) and lattice parameters (a=3.876 Å and c =9.515 Å) [6]. Each of the phases has a similar tetragonal structure but differs both in the critical temperature and lattice constants: Hg-1212 with a=3.8552 Å; c=12.6651 Å and T_c in the range of 124 to 128 K [7,8], while Hg-1223 has a=3.852 Å; c=15.83 Å and T_c = 135 K [9]. Lamia Khdhair Abbas studied the influence of Ag and In doping on the critical temperature (T_c) and structural characteristics of the $Hg_{1-X}(Ag,In)_XBa_{2-y}$ and $Sr_yCaCu_3O_{8+\delta}$ superconductors. The study revealed that the critical temperature increased from 116 K to 117.5 K, while the c-axis length increased from 15.7779 Å to 15.8143 Å with the increase in the concentration ratio of Sr from 0 to 0.1 when x was set to 0, thereby determining that the optimal partial substitution ratio for Sr in Ba is 0.1. Additionally, it was observed that the partial substitution of Ag resulted in an enhancement of T_c from 117.5 K to 118 K, subsequently reaching 127 K, accompanied by an increase in the c-axis length from 15.8143 Å to 15.8519 Å and 15.9238 Å as the concentration ratio of Ag in Hg increased escalated x=0 to 0.1 and 0.2 with y=0.1. The author noted that the observed increase in the c-axis length can be attributed to variations in ionic diameters, since the ionic diameter of Ag exceeds the ionic diameter of Hg. Conversely, it was concluded that an increase in the concentration ratio of In in Hg from 0 to 0.1, 0.2, and 0.3 at y = 0.1 results in a decrease in the critical temperature from 117.5 K to 116 K, which then rises to 122 K and subsequently to 125 K. Furthermore, the length of the c axis decreased from 15.8143 Å to 15.8091 Å and 15.7602 Å, before increasing to 15.8318 Å; the author noted that the reduction in the c-axis length is attributable to differences in ionic diameters of In and Hg [10]. Researchers Bilal A. Omar and Sabah J. Fathi investigated how zinc affected the electrical

and structural characteristics of a high-temperature $HgBa_2Ca_2Cu3O_{8+\delta}$ superconductor. The optimal ratio of Zn concentrations in Ca was determined to be 0.05, which led to an increase in the critical temperature from 120 K to 132 K. The samples were found to have a tetragonal crystalline structure both before and after partial substitution of Zn in Ca [11].

The aim of this study is to prepare differential conductivity compounds of the Bi-2223 type with new electrical and structural properties and to study the effect of partial substitution of silver (Ag) and strontium (Sr) on bismuth (Bi) and barium (Ba) in different proportions in order to achieve a high critical temperature. This research is of great importance by achieving a new critical temperature of 140 K in Bi-2223 phase compounds, which is a significant improvement compared to previous studies, enhancing the practical application of these materials. Modifying the preparation conditions, such as sintering, annealing, and oxygen flow ratio, improved the stability of the superconducting phase and increased the *c*-axis length, which enhanced the overlapping of Cu-O layers and contributed to raising the critical temperature. In addition, doping the compound with calculated proportions of silver and strontium improved the crystal structure and increased the electrical current transmission efficiency, which enhances the overall performance of the material. The practical importance of these results lies in their potential use in various industrial fields, such as the manufacture of superconducting power cables, powerful magnets, and renewable energy systems, as this reduces the need for expensive cooling using liquid helium. In addition, the research contributes to providing a deeper understanding of the relationship between the crystal structure and superconducting properties of Bi-2223 compounds, paving the way for further development and innovation in the field of superconducting materials.

2. Experimental

2.1 Materials Used

99% pure powdered chemicals of Indian origin, including strontium nitrate ($Sr(NO_3)_2$), calcium carbonate ($CaCO_3$), bismuth oxide (Bi_2O_3), copper oxide (CuO), barium carbonate ($BaCO_3$), and silver nitrate ($AgNO_3$) and alcohol isopropanol (C_3H_8O) were used. During the annealing process, oxygen gas (O_2) was used to create an oxygen-saturated environment inside the convection oven.

2.2 Devices and Tools Used

A delicate balance of the M314Ai type (Japan) that can measure powder weights with a precision of ±0.0001 g was used. A Chinese ceramic pot was used to hold the sample and endure temperatures as high as 1200°C. A tiny mortar was made from the Japanese mortar for mixing and grinding the powders. A British type electric furnace (ELITE) housed in the Physics Department of the College of Sciences, University of Mosul was used for sintering and annealing the samples. Samples were pressed using a hydraulic press (I.R. type) at the University of Mosul's Chemistry Department, College of Science. A locally produced sample pressing mold was used. The oxygen regulator, developed in Japan, was designed to supply oxygen to the electric furnace at a rate of two liters per hour. The resistivity measurement system used a set of instruments to ensure the accuracy and reliability of measurements. A vacuum system employed to provide a steady atmosphere for the measurements was complemented by a digital power supply to accurately regulate the electric current supplied to the samples during the assessment. A digital voltmeter was employed to measure voltage with great precision, and low-temperature measuring equipment was used to record temperatures during the experiment. A nitrogen cylinder was used to cool the samples to low temperatures during the experiments.

2.3 Methods

Using a combination of the above devices, the samples $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$) at y=0.1, 0.2, 0.3, 0.4, x=0.2 were created using the solid state reaction method. A sensitive balance was used to weigh the stoichiometric amounts of the powders. The powders were then mixed and ground in an agate mortar. We added enough isopropanol to the grinding process to create a homogeneous mixture and the grinding process continued for about 30 minutes. After drying the mixture for approximately 30 minutes at temperatures between 40 and 50°C to eliminate residual isopropanol solution, it was compressed using a hydraulic press to form disc-shaped samples of diameter 1.2 cm and thickness 0.3 cm under a pressure of (7.5 tons/cm^2). A programmable furnace sintered the pellets at 800 °C for 24 hr at a rate of 120 °C/hr, and then heated them down to room temperature at a rate of 30 °C per hour. The sintering process was carried out in air, as shown in Fig.1(A). The samples were annealed

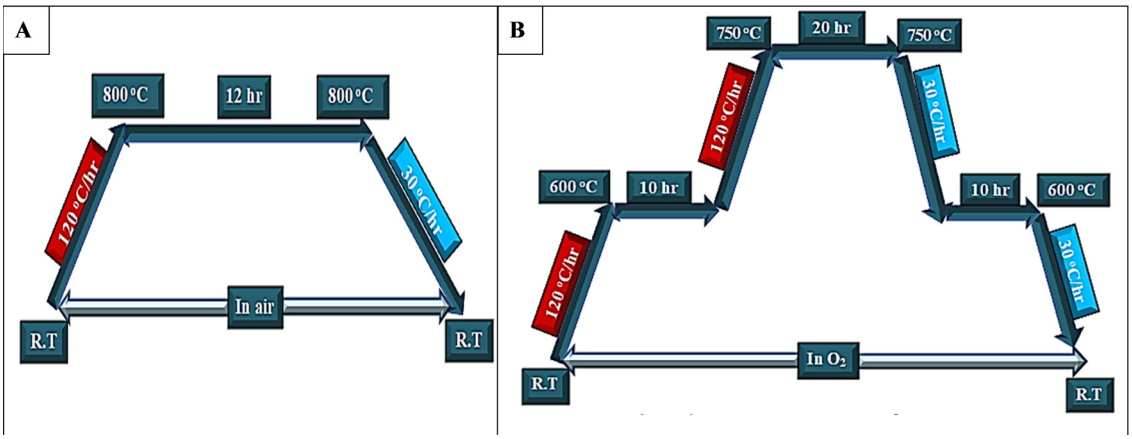

Fig. 1 : (A) sintering process and (B) annealing process.

in the second step, as illustrated in Fig.1(B), where the pellets are ground and then pressed in the same manner as before. They were then placed in a furnace where the temperature was raised from room temperature to 600 °C at a heating rate of 120 °C/hour and kept at this temperature for about 10 hours. After that, the furnace temperature was raised to 750 °C with a heating rate of 120 °C per hour and the pellets were kept at this temperature for approximately 20 hr. The samples were then cooled to 600 °C at a rate of 30 °C/hr and kept at this temperature for approximately 10 hr, and finally cooled to room temperature at a rate of 30 °C/hr. This process was carried out in a stream of saturated oxygen at a rate of 2°C per hour.

X-ray diffraction XRD. Table.1 shows the characteristics of the PANalytical Aeris machine, which was used to verify the crystal structure.

To compute the relative volume fraction of any phase, the following relation was used [12]:

$$V_{\text{ph}} = \frac{\Sigma I_0}{(\Sigma I_0 + \Sigma I_1 + \Sigma I_2 + \Sigma I_n)} \times 100, . \quad (1)$$

where I_1, I_2,...I_n are the intensities of all XRD peaks, and I_0 is the XRD peak intensity of the phase that was determined.

The distance between the crystal planes d_{hkl} was found based on Miller indices and the lengths of the crystal axes (a,b,c) through the following relationship [13]:

$$d_{hkl} = \frac{1}{\sqrt{\left(\frac{h^2}{a^2}\right) + \left(\frac{k^2}{b^2}\right) + \left(\frac{l^2}{c^2}\right)}} . \quad (2)$$

Table.1 : Characteristics of the X-ray diffraction machine used

Source	Cu Target
Current	40 mA
Voltage	40 k.V
scan Range	10-90 deg
scan mode	Bragg-Brentano (θ-2θ) reflection geometry
Wave length	1.15406Å
preset time	1 to 100 seconds
Scan speed	Up to 10 deg / min

Four probe dc methods. The most common measurement method for determining T_c for superconducting materials is the technique of calculating resistivity ρ as a function of T by four probe methods. The sample is placed in a dedicated cooling system, then the sample is connected to the probes well using silver paste to ensure perfect contact; the outer probes are connected by wires to a constant current source, while the two inner pbes are connected to a voltmeter to calculate the electrical potential difference, then we discharge the liquid nitrogen until the resistance of the sample reaches zero. Then the data is read on a special program in the calculator and then plotted on Microsoft Excel. ρ is found using this relationship to determine the critical temperature T_c [14,15]:

$$\rho = \frac{(R \times A)}{L} . \quad (3)$$

Where L is the length of the specimens, A is the area of the specimens, and R is their electrical resistance.

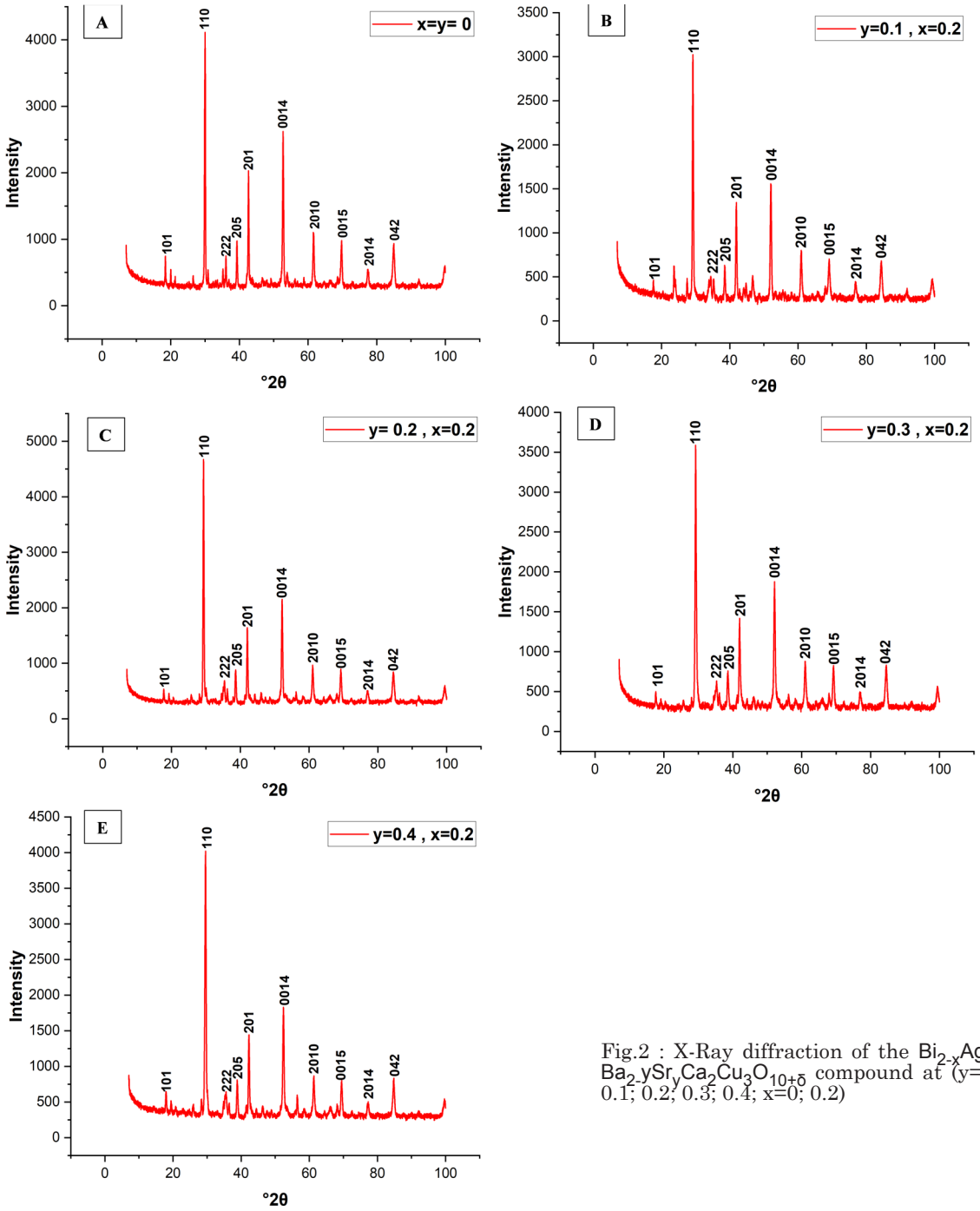

Fig.2 : X-Ray diffraction of the $Bi_{2-x}Ag_x$-$Ba_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ compound at (y=0; 0.1; 0.2; 0.3; 0.4; x=0; 0.2)

3. Results and Discussion

3.1 Study of the structural properties of the superconducting compound $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$

The structural characteristics of the compound were studied at an annealing temperature 750°C and a pressure of 7.5 tons/cm^2, using Bragg's law, the distances between parallel planes (d_{hkl}) and Miller indices were calculated based on the reflection angles (2θ) using the (X'Pert Highscore) program; the determined parameters of the unit cell (*a, b, c*) showed that the structure is of the tetragonal type. Table 2 shows the values of the axes *a, b, c* after the annealing process. The study of the structural characteristics of the compound at different concentration ratios (*x, y*) showed that the crystal structure became more regular while still maintaining its tetragonal type. Clear peaks appeared and became more prominent, and there was also a significant increase

Table 2. Results of the compositional examination of the $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ compound at (y=0.1;0.2;0.3;0.4 ; x=0.2).

No. Samples	A (Å)	b(Å)	c(Å)	c/a	V(Å)3	V_{Ph}
y=0 , x=0	4.20	4.20	24.21	5.764	427.06	0.542
y=0.1 x=0.2	4.33	4.33	24.625	5.687	461.69	0.615
y=0.2 x=0.2	4.32	4.32	24.56	5.685	458.35	0.551
y=0.3 x=0.2	4.326	4.326	24.60	5.686	460.37	0.582
y=0.4 x=0.2	4.31	4.31	24.49	5.682	454.92	0.543

in the length of the *c*-axis, indicating an improvement in the crystal structure. For the first standard sample (*x*=*y*=0), the X-ray diffraction results showed regular crystal structure and clear peaks, as shown in Fig.2 (A), and the cell parameters were *a*=*b*=4.20 Å and *c*=24.21 Å. At the ratio of *y*=0.1; *x*=0.2, the lattice parameters (*a*=*b*=4.33Å and *c*=24.625Å) showed a significant increase as shown in Fig.2 (B). At the ratio *y*=0.2; *x*=0.2, a decrease in the length of the *c*-axis was observed compared to the concentration ratio of *x*=0.2; *y*=0.1; and the values of the lattice parameters became *a*=*b*=4.32Å and c=24.56 Å, as shown in Fig.2 (C). Fig.2 (D) shows that when the concentration ratio was *y*=0.3, *x*=0.2, an increase in the intensity of the peaks and an increase in the length of the *c*-axis were observed, indicating that the crystal structure became more regular, the values of the lattice parameters were *a*=*b*=4.326 Å and *c*=24.60 Å. As for Fig.2 (E), when the concentration ratio was at *y*=0.4; *x*=0.2, a decrease in the intensity of the peaks was observed due to a decrease in the length of the *c*-axis, and the values of the lattice parameters were *a*=*b*=4.31 Å and c=24.49 Å. We conclude that the best concentration ratio in this group is *y*=0.1; *x*=0.2, as this concentration ratio led to a significant improvement in the crystal structure. It is the best concentration ratio for all samples.

3.2 Study of the electrical properties of the $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ *superconducting compound*

The electrical properties of the compound were studied after partial replacement of Bi with Ag and Ba with Sr, as shown in Table 3.The concentrations ratios used are: (*x*=0; *y*=0), (*x*=0.2; *y*=0.1), (*x*=0.2; *y*=0.2), (*x*=0.2; *y*=0.3) and (*x*=0.2; *y*=0.4) The results showed that double substitutions were generally superior to single substitutions, resulting in higher critical temperatures (T_c) and oxygen percentages (δ).

The results showed that for the pure standard sample at the concentration ratio (*x*=*y*=0), the critical temperatures were (T_c(1) =127.5 K; T_c(2) =152.7 K), as shown in Fig.3 (A). With the concentration ratio of (*x*=0.2; *y*=0.1), the critical temperature increased significantly to T_c(1)=139.2 K; T_c(2)=170.5 K, as shown in Fig.3 (B). This ratio is the best among all the concentration ratios, and this can be explained by the fact that the crystal structure of the compound became more regular due to an increase in the length of the *c*-axis and a significant increase in the oxygen percentage (δ). When the concentration ratio was (*x*=0.2; *y*=0.2), the critical temperatures decreased to T_c(1)=136.6 K and T_c(2)=167.3 K, as shown in Fig.3 (C), but it was still better than the samples with single substitutions. With the ratio of (*x*=0.2; *y*=0.3), the crit-

Table 3. Relationships between the concentration ratio (x, y), the critical temperature (T_c) and the percentage of oxygen (δ) in the $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ compound. Effect of changing Sr concentration on the critical temperature of the compound and the percentage of oxygen (δ) present in the crystal structure

No. Samples	T_c(1), K	T_c(2), K	ΔT_c, K	T_c(mid), K	δ
x=0, y=0	127.5	152.7	25.2	140.1	0.288
x=0.2 y=0.1	139.2	170.5	31.3	154.85	0.475
x=0.2 y=0.2	136.6	167.3	30.7	151.95	0.395
x=0.2 y=0.3	137.8	168.6	30.8	153.20	0.422
x=0.2 y=0.4	134.9	163.2	28.3	149.05	0.383

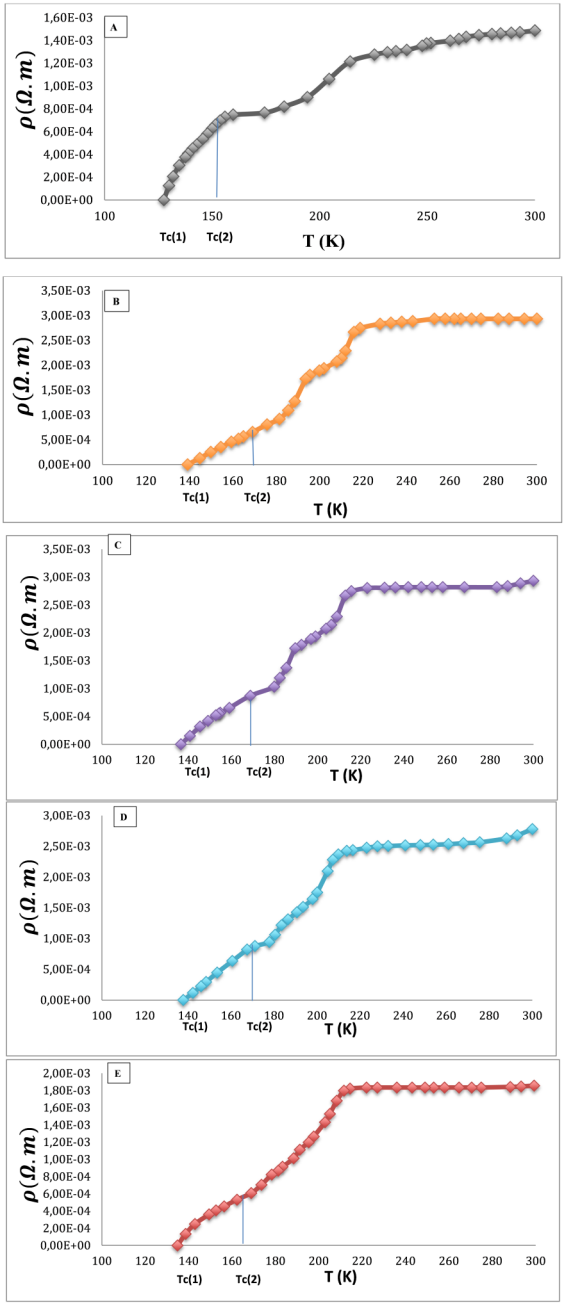

Fig.3. Relationship between the resistivity and the critical temperature of the compound $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ at various concentration ratios: (A) (x=0; y=0); (B) (x=0.2; y=0.1); (C) (x=0.2; y=0.2); (D) (x=0.2; y=0.3) and (E) (x=0.2; y=0.4). Changes in the resistivity with decreasing temperature until the critical temperature is reached

ical temperatures increased to $T_c(1)$=137.8 and $T_c(2)$=168.6 K, as shown in Fig.3 (D). With the ratio of (x=0.2; y=0.4), the critical temperatures decreased to $T_c(1)$=134.9 K and $T_c(2)$=163.2 K), as shown in Fig.3 (E). The introduction of copper oxide (Cu-O) impurities resulted in reduced crystal regularity, which led to a reduction in the length of the c-axis in the compound. Finally, it can be concluded that the best concentration ratio was (x=0.2; y=0.1).

The results obtained are based on optimized preparation conditions that differ from previous studies. We modified the sintering and annealing parameters, as well as adjusted the oxygen gas flow rate during the heat treatment, which improved the structure of the composite. Moreover, Bi2223 was doped with different ratios of silver and strontium, which contributed to improving the structural properties and increasing the stability of the superconducting phase. An increase in the c-axis length indicated an expansion of the interlayer distance, thus improving the overlapping of Cu-O layers, which is a key factor in enhancing the superconducting transition temperature.

4. Conclusion

In this study we considered superconductor compounds $Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$ created using the solid state reaction for ($0 \le y \le 0.4$) and (x=0; 0.2). A tetragonal structure with a high ratio of the Bi-2223 superconductor phase was identified by the XRD pattern analysis. Furthermore, the sample doped with Ag and Sr at x=0.2, y=0.1 displayed the largest rise in the axial lattice constant (c). The ideal replacement ratio for Ag in Bi and Sr in Ba in the complex $Bi_{2-x}Ag_xBa_{2-y}SryCa_2Cu_3O_{10+\delta}$ is (x=0.1; y=0.2). When Ag and Sr are substituted for Bi and Ba, at the concentration ratio (x=0.2; y=0.1), the ($Bi_{2-x}Ag_xBa_{2-y}Sr_yCa_2Cu_3O_{10+\delta}$) compounds have demonstrated the maximum values of critical temperature ($T_c(1)$=139.2 K; $T_c(2)$=170.5 K). In addition, it was found that the critical temperature (T_c) increases with increasing oxygen content (δ) because the substitution leads to local pressure, an increase in the hole charge carrier concentration, a change in the electronic state and its distribution. In the course of the study, Bi-2223 materials with improved electrical and structural characteristics were synthesized due to partial replacement of bismuth (Bi) and barium (Ba) with silver (Ag) and strontium (Sr). This caused a critical temperature increase up to 140 K. Modifications to laboratory processes involving temperature treatment and oxygen management have resulted in both increased phase stability and increased c-axis length, as well as improved superconducting properties of thin samples, leading to higher critical temperatures. Controlling the doping process improved both the development of the crystal

structure as well as electrical current transfer, resulting in improved material performance. The important results obtained from this research will have applications in superconducting power cables, high-performance magnets and renewable energy systems.

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