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Partial substitution effect of silver on the structural and electrical properties of high temperature superconductor ($Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$)

Wpływ zawartości srebra na właściwości strukturalne i elektryczne nadprzewodnika wysokotemperaturowego ($Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$)

The solid-state reaction method was used to generate superconductor samples with the chemical composition $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ which were pressed under a load of 7.5 t/cm^2. The samples were prepared using two methods: the sintering method for 24 h (increasing temperature to 800°C at a rate of 120°C/h, fixing it for 10 h, and then cooling to room temperature at a rate of 30°C/h under a pressure of 7.5 t/cm^2) and the annealing method for 68 h (increasing temperature to 600°C at a rate of 120°C/h and fixing it for 10 h, then raising it to 750°C at the same rate and holding it for 20 h, followed by cooling to 600°C at a rate of 30°C/h and holding it for 10 h, and finally cooling to room temperature at the same rate). The impact of partially substituting Ag with x = (0, 0.1, 0.2, 0.3, 0.4) on this compound's electrical and structural characteristics was investigated. All of the samples form a tetragonal crystal structure according to the results of the X-ray diffraction technique used to quantify the lattice characteristics. This method demonstrated that the value of the lattice constant (c) increased as the concentration of Ag increased from x = 0.1 to x = 0.3, and then declined at x = 0.4. The standard four-probe method was used in electrical resistivity measurements, and the measurements showed an improvement in T_c from x = 0.1 to x = 0.3. After this concentration, the T_c value decreased with the substitution of Ag in Bi when x = 0.4. The samples were type II superconductors, which had two critical temperatures, where the critical temperature increased from $T_c(1)$ = 127.5 K, $T_c(2)$ = 152.7 K to $T_c(1)$ = 135.1 K, $T_c(2)$ = 167.7 K with increasing x from 0.1 to 0.3, and decreased after this concentration to $T_c(1)$ = 133.9 K, $T_c(2)$ = 165.1 K. The oxygen content (δ) was observed to increase with increasing T_c.

Keywords: superconductor, solid state reaction method, sintering and annealing process, structural and electrical properties

Metoda reakcji w fazie stałej została wykorzystana do wytworzenia próbek nadprzewodnika o składzie chemicznym $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$, które zostały sprasowane pod obciążeniem 7,5 t/cm². Próbki przygotowano dwiema metodami: metodą spiekania przez 24 h (zwiększano temperaturę do 800°C z szybkością 120°C/h, utrzymywano ją przez 10 h, a następnie obniżano do temperatury pokojowej z szybkością 30°C/h pod ciśnieniem 7,5 t/cm²) oraz metodą wyżarzania przez 68 h (zwiększano temperaturę do 600°C z szybkością 120°C/h i utrzymywano ją przez 10 h, a następnie podnoszono do 750°C w tym samym tempie i utrzymywano przez 20 h, po czym zmniejszano do 600°C z szybkością 30°C/h i utrzymywano przez 10 h, a na koniec obniżano w tym samym tempie do temperatury pokojowej). Zbadano wpływ częściowej substytucji Ag przy x = (0, 0,1, 0,2, 0,3, 0,4) na właściwości elektryczne i strukturalne badanego związku. Wszystkie próbki wykazują tetragonalną strukturę krystaliczną zgodnie z wynikami badań metodą dyfrakcji rentgenowskiej przeprowadzonymi w celu określenia parametrów sieci krystalicznej. Wykazano, że wartość stałej sieciowej (c) wzrastała wraz ze zwiększaniem zawartości Ag od x = 0,1 do x = 0,3, a następnie malała przy x = 0,4. Pomiar oporu elektrycznego przeprowadzono standardową metodą czteropunktową. Uzyskano poprawę wartości T_c w zakresie od x = 0,1 do x = 0,3. Po przekroczeniu tej wartości T_c malała wraz z dalszą substytucją Ag w Bi, gdy x = 0,4. Próbki były nadprzewodnikami typu II, charakteryzującymi się dwoma wartościami temperatury krytycznej. Temperatura krytyczna zwiększała się od $T_c(1)$ = 127,5 K, $T_c(2)$ = 152,7 K do $T_c(1)$ = 135,1 K, $T_c(2)$ = 167,7 K wraz ze wzrostem x od 0,1 do 0,3, a po osiągnięciu tej wartości malała do $T_c(1)$ = 133,9 K, $T_c(2)$ = 165,1 K. Zawartość tlenu (δ) zwiększała się wraz ze wzrostem T_c.

Słowa kluczowe: nadprzewodnik, metoda reakcji w fazie stałej, proces spiekania i wyżarzania, właściwości strukturalne i elektryczne

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1. Introduction

The phenomenon of superconductivity is characterized by the flawless transmission of electricity through a material that exhibits no electrical resistance whatsoever at remarkably cold temperatures. This attribute is commonly witnessed in metallic substances in severely cold situations [1]. Superconductivity involves a joint pair of simultaneous phenomena. The primary phenomenon is denoted by perfect conductivity, whereas the second is delineated by absolute diamagnetism. This strange manifestation arises in diverse materials when subjected to frigid settings, termed the critical temperature. This pivotal threshold is represented by the symbol T_c. Upon reaching the critical temperature, the material transforms from its standard state to the superconducting state, and the magnitude of the critical temperature demonstrates variance across differing elements. While perplexing at first glance, further examination has illuminated this captivating concept [2].

Superconductors are high-tech materials with wide applications in energy harvesting, magnetic technologies, power generation and transmission as well as maglev transportation, demonstrating zero electrical resistance, perfect conductivity, diamagnetic property and the Meissner effect. The potential applications are what drives this field and the research that goes into superconductors. Given their unusual properties, it is essential that superconductors are scrutinized thoroughly and explored to understand whether they could continue to be used for technologies in all realms. Superconductors remain an evolving area of study and can be classified based on coherence length and critical temperature. They are generally categorized as type I or type II superconductors and as high- or low-critical-temperature superconductors, with some exhibiting crossover properties between these classifications [3].

In 1911, Dutch physicist Heike Kamerlingh Onnes investigated the temperature dependence of electrical resistance in solid mercury at extremely low temperatures using liquid helium as a coolant. He discovered that below 4.2 K – the boiling point of liquid helium – mercury exhibited zero electrical resistance, a phenomenon later termed superconductivity. This groundbreaking discovery revealed a novel solid-state property: electric superconductivity [4].

Further research into this remarkable phenomenon continued, and in 1933, Meissner and Ochsenfeld [5] identified one of its most fundamental characteristics, now known as the Meissner effect. They observed that a superconducting material, in addition to having zero electrical resistance, completely expels magnetic fields, making it an ideal diamagnet.

In the superconducting system Ba–La–Cu–O, which Bednorz and Müller discovered in 1986 [6], there is a new class of superconducting material that is mostly formed of copper oxide. This compound is superconducting up to a critical temperature of T_c = 93 K. The research conducted by Awad [7] involved synthesizing superconducting Ag-doped $(Tl_{0.8}Pb_{0.1}Bi_{0.1})Ba_2Ca_2Cu_3O_{9.8}$ samples with increasing silver concentrations between $0 \le x \le 0.4$ by using high-purity oxides such as Tl_2O_3, PbO, Bi_2O_3, BaO_2, CaO and CuO and metallic Ag. The research team created the samples at 870°C for 4 h before testing electric resistance and magnetoresistance. The test results showed that silver chemicals added to the $(Tl_{0.8}Pb_{0.1}Bi_{0.1})Ba_2Ca_2Cu_3O_{9.8}$ compounds decreased both *a* and *c* lattice parameters. The lattice parameters experienced contraction after silver was added because the structural modifications affected crystal lattice compression. The grain size of the samples showed increasing improvement with increasing silver concentration up to $x = 0.2$ and stabilized thereafter.

Schilling, Cantoni, and Gao [8] achieved T_c-(133 K) in Hg–Ba–Ca–CuO, after which they reported two new phases named Hg-1212 and Hg-1223 for related mercury-based series having a composition of the *n*1*n*23 form 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 a maximal critical temperature of T_c = 95 K, and lattice parameters of $a = 3.876$ Å and $c = 9.515$ Å [9]. Each of the phases has a similar tetragonal structure but differ both in their critical temperature and lattice constants. Hg-1212 has $a = 3.8552$ Å, $c = 12.6651$ Å, and T_c in the range from 124 K to 128 K [10, 11]. At the same time, Hg-1223 has $a = 3.852$ Å, $c = 15.83$ Å, and T_c equal to 135 K [12]. As for Hg-1234, it's $a = 3.8540–3.858$ Å, $c = 19.006–19.011$ Å and T_c up to 125 K [13, 14].

In 2010, Twessan [15] examined the effect of partial substitution of the element Tl on the physical properties of the superconducting compound $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ at high temperatures, reaching a critical temperature of 136 K with a substitution ratio $x = 0.2$ by partial substitution of Tl with Bi, with the application of a hydrostatic pressure of 8 t/cm^2 and an annealing temperature of 1123 K.

2. Materials and methods

2.1. Materials used

The following materials were used in the study:

- silver nitrate ($AgNO_3$), calcium carbonate ($CaCO_3$), bismuth oxide (Bi_2O_3), copper oxide (CuO), barium carbonate ($BaCO_3$), strontium nitrate ($Sr(NO_3)_2$), and other chemicals of Indian origin in powder form with a purity of 99%;
- isopropanol alcohol (C_3H_8O);
- oxygen gas (O_2) – was used in the annealing process in order to provide an atmosphere saturated with oxygen inside a convection oven.

2.2. Devices and tools

The study utilized the following tools and devices (Photo 1):

- a sensitive type M314Ai Japanese balance with an accuracy of ±0.0001 to measure the weight of the powders;
- a Chinese ceramic crucible in which the sample is placed and which can withstand temperatures up to 1200°C;
- a small Japanese agate mortar for grinding and mixing powders;
- a British Elite electric furnace for sintering and annealing the samples, located at the University of Mosul, College of Science, Department of Physics;
- hydraulic press (IR) for pressing the samples, located at the University of Mosul, College of Science, Department of Chemistry;
- a mold for pressing the samples, made locally;
- a Japanese oxygen regulator, used to pump oxygen gas into the electric furnace at a rate of 2 l/h.

2.3. Methods

2.3.1. Sample preparation

By using a combination of the previously mentioned devices, the samples $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ at $x = 0, 0.1, 0.2, 0.3, 0.4$ were created using the solid state reaction method. A sensitive balance was used to weigh the stoichiometric amounts of these powders. The powders were then combined and ground using an agate mortar. We added

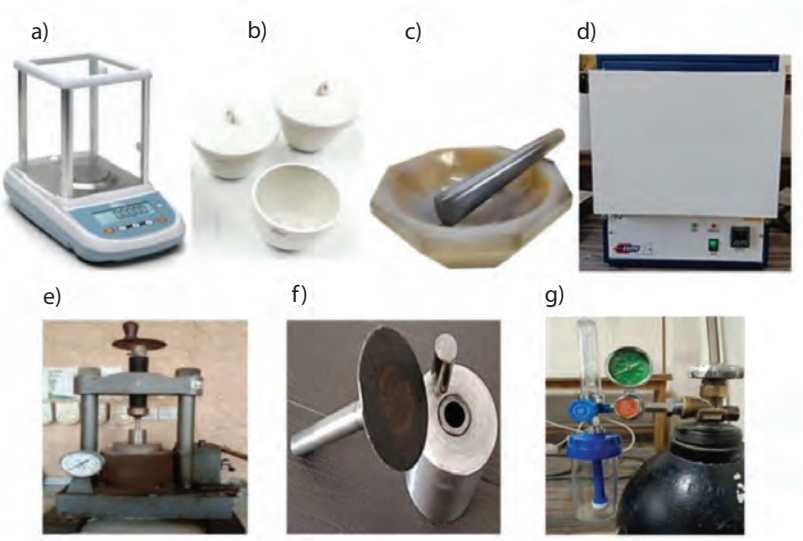

Photo 1. Devices and tools used: a) sensitive balance, b) ceramic crucible, c) agate mortar, d) electric furnace, e) hydraulic press, f) mold for pressing the samples, g) system for pumping oxygen into the furnace

Fot. 1. Używane urządzenia i przyrządy: a) czuła waga, b) porcelanowy tygiel, c) moździerz agatowy, d) piec elektryczny, e) prasa hydrauliczna, f) forma do prasowania próbek, g) system do pompowania tlenu do pieca

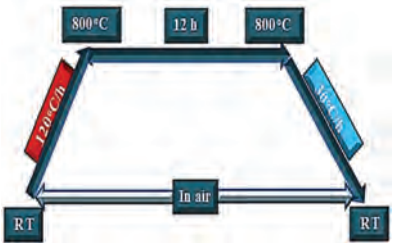

Fig. 1. Sintering process

Rys. 1. Proces spiekania

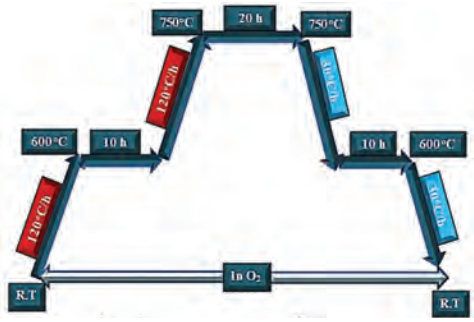

Fig. 2. Annealing process

Rys. 2. Proces wyżarzania

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

Tabela 1. Charakterystyka aparatury do dyfrakcji rentgenowskiej

Source	Cu target
Current	40 mA
Voltage	40 kV
Scan range	10–90°
Scan mode	Bragg-Brentano (θ–2θ) reflection geometry
Wave length	1.15406o
Preset time	1–100 s
Scan speed	up to 10°/min

Photo 2. Four probe DC method

Fot. 2. Metoda czteropunktowa DC

Photo 3. X-ray diffraction device

Fot. 3. Urządzenie do dyfrakcji rentgenowskiej

enough iso-propanol to the grinding process to create a homogenized mixture, and the grinding process continued for about 30 min. After the mixture was dried for approximately 30 min at temperatures between 40°C and 50°C to eliminate the additional isopropanol solution, it was compressed using a hydraulic press to form 1.2 cm diameter disc-shaped pellets at a pressure of 7.5 t/cm^2. The programmable furnace sintered the pellets at 800°C for 24 h at a rate of 120°C/h and then heated them down to room temperature at a rate of 30°C/h. The sintering process was carried out in air, as seen in Fig. 1. The samples are annealed in the second step, as illustrated in Fig. 2, where the pellets are ground and then pressed in the same manner as before. They were then placed in the furnace, where the temperature was raised from room temperature to 600°C with a heating rate of 120°C/h and remained at this temperature for approximately 10 h. Afterwards, the furnace heat was raised to 750°C with a heating rate of 120°C/h and remained at this temperature for approximately 20 h. The samples were then cooled to 600°C at a rate of 30°C/h and remained at this temperature for approximately 10 h, after which they were finally cooled to room temperature at a rate of 30°C/h. This process was carried out using saturated oxygen at a feed rate of 2 l/h.

2.3.2. X-ray diffraction (XRD)

The structure and crystal structure was verified by the PANalytical Aeris device characterized by the parameters shown in Table 1.

The following relation was used to compute the relative volume fraction of any phase:

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

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

2.3.3. Four probe DC method

The critical temperature (T_c) and resistivity (ρ) were measured using the four probe DC method. This relation can be used to determine the critical temperature:

$$\rho = \frac{(R \times A)}{L}\ldots, \quad (2)$$

where L is the length of the specimens, A is the area of the specimens, and R is their electrical resistance. The oxygen content was determined using the iodometric titration method.

Table 2. Results of the compositional examination of the compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ at $x = 0, 0.1, 0.2, 0.3, 0.4$

Tabela 2. Wyniki badania składu związku $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ przy $x = 0, 0{,}1, 0{,}2, 0{,}3, 0{,}4$

Sample	*a* [Å]	*b* [Å]	*c* [Å]	*c*/*a*	*V* [Å]^3	V_{Ph}
$x = 0$	4.20	4.20	**24.21**	5.764	427.06	0.542
$x = 0.1$	4.205	4.205	**24.25**	5.767	428.79	0.596
$x = 0.2$	4.21	4.21	**24.29**	5.778	430.52	0.627
$x = 0.3$	4.24	4.24	**24.53**	5.785	440.99	0.713
$x = 0.4$	4.225	4.225	**24.45**	5.780	436.45	0.648

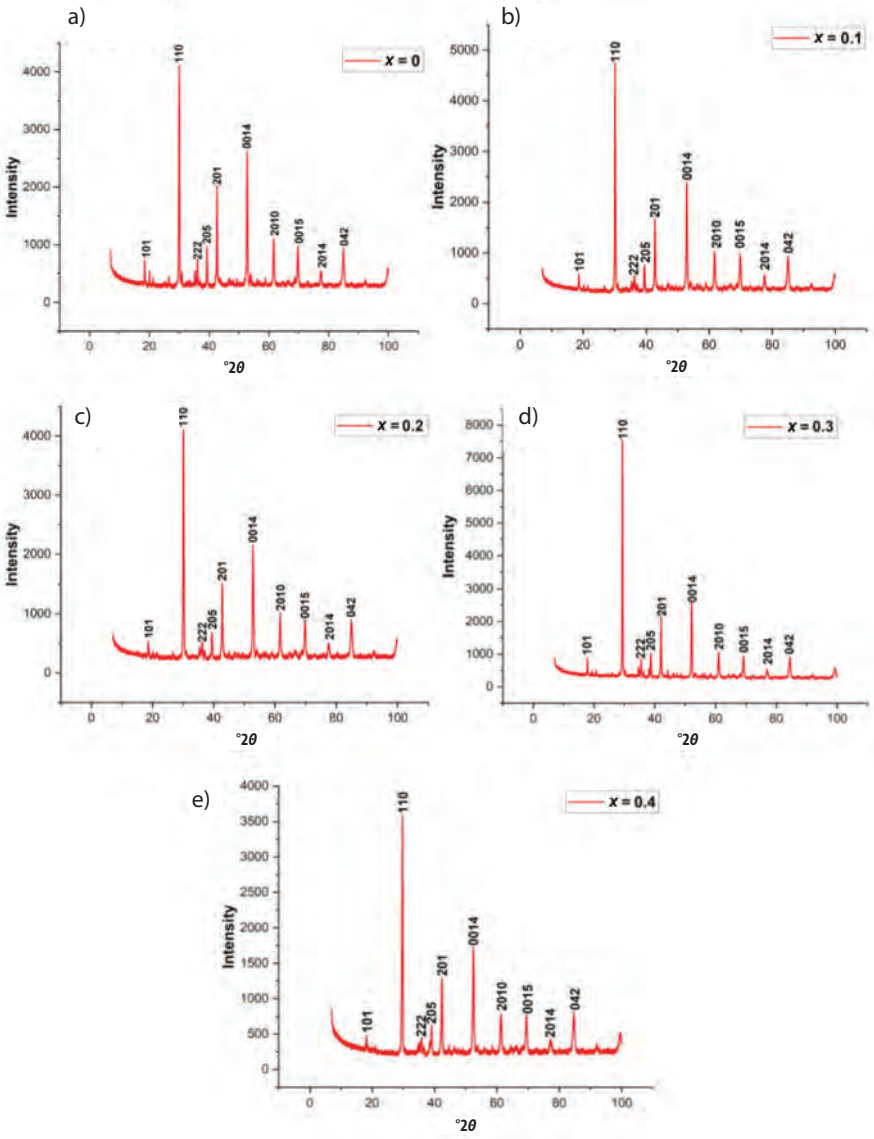

Fig. 3. X-ray diffraction of the compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ at: a) $x = 0$, b) $x = 0.1$, c) $x = 0.2$, d) $x = 0.3$, e) $x = 0.4$

Rys. 3. Dyfrakcja rentgenowska związku $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ przy: a) $x = 0$, b) $x = 0{,}1$, c) $x = 0{,}2$, d) $x = 0{,}3$, e) $x = 0{,}4$

3. Results and discussion

3.1. Study of the structural properties of the superconducting compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$

The structural properties of the compound were studied at an annealing temperature of 750°C and a pressure of 7.5 t/cm^2. Using Bragg's law, the distances between parallel planes (*dhkl*) and Miller coefficients (*hkl*) were calculated based on the reflection angles (2θ) using the X'Pert Highscore program. The dimensions of the unit cell (*a*, *b*, *c*) were determined, which showed that the structure is of a tetragonal type. Table 2 shows the values of the axes *a*, *b*, *c* after the annealing process. The XRD study showed regularity in the crystal structure and the appearance of clear peaks, as shown in Fig. 4a for the first standard sample $x = y = 0$, with the dimensions of the cell being $a = b = 4.20$ Å and $c = 24.21$ Å.

At a compensation ratio of $x = 0.1$, the XRD results showed that the crystal structure remained tetragonal with clear peaks, and the lattice dimensions were $a = b = 4.205$ Å and $c = 24.25$ Å (Fig. 4b). After increasing the compensation ratio to $x = 0.2$, there was an increase in the intensity and regularity of the peaks, while the tetragonal crystal structure remained unchanged, and the length of the *c* axis increased. The lattice dimensions were $a = b = 4.21$ Å and $c = 24.29$ Å, confirming the improvement of the crystal structure of the compound (Fig. 4c), with an increase in the grain size.

At a compensation ratio of $x = 0.3$, the peaks showed greater clarity compared to the previous compensations, with an increase in the length of the *c*-axis, indicating an increase in the crystalline regularity, where the lattice dimensions were $a = b = 4.24$ Å and $c = 24.53$ Å (Fig. 4d). As for the compensation ratio $x = 0.4$, a decrease in the intensity of the peaks was observed (Fig. 4e), indicating a decrease in the previous crystalline regularity as a result of an increase of the compensation ratio, where the lattice dimensions were $a = b = 4.225$ Å and $c = 24.45$ Å, with a clear decrease in the length of the *c*-axis. Through this study, we conclude that the best partial compensation ratio for the Ag element in Bi in the 2223 system is when $x = 0.3$.

3.2. Study of the electrical properties of the superconducting compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$

The electrical properties of the $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ compound were studied when Ag was partially replaced in Bi at different ratios of *x* ($x = 0, 0.1, 0.2, 0.3, 0.4$). The results showed that at the compensation ratio of $x = 0$, the critical temperature was $T_c(1) = 127.5$ K, $T_c(2) = 152.7$ K (Fig. 4a), which is the pure standard sample.

After increasing the compensation ratio to $x = 0.1$, an increase in the critical temperature was observed, reaching $T_c(1) = 131.1$ K, $T_c(2) = 158.9$ K (Fig. 4b). As for the compensation ratio $x = 0.2$, the critical temperature increased to $T_c(1) = 133.5$ K, $T_c(2) = 161.4$ K (Fig. 4c).

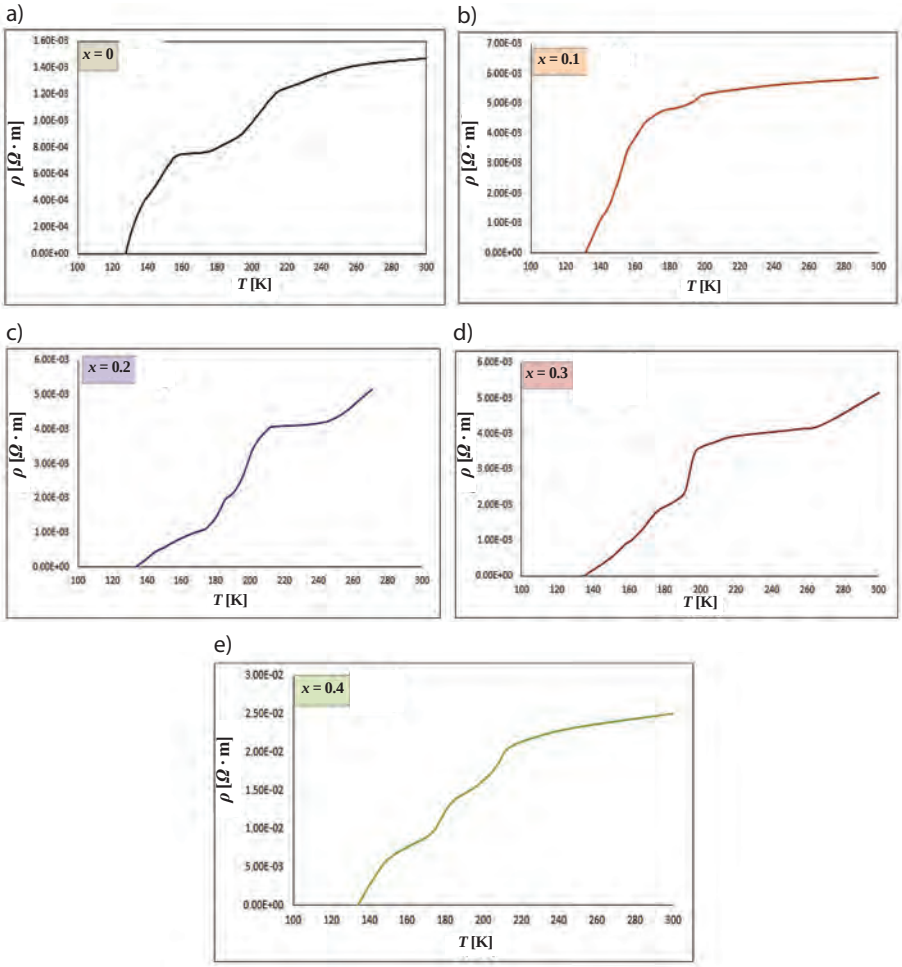

Fig. 4. The relationship between the resistivity and the critical temperature of the compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ at a compensation ratio of: a) $x = 0$, b) $x = 0.1$, c) $x = 0.2$, d) $x = 0.3$, e) $x = 0.4$

Rys. 4. Zależność między opornością a temperaturą krytyczną związku $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ przy współczynniku kompensacji: a) $x = 0$, b) $x = 0{,}1$, c) $x = 0{,}2$, d) $x = 0{,}3$, e) $x = 0{,}4$

At a compensation ratio of $x = 0.3$, the critical temperature increased to $T_c(1) = 135.1$ K, $T_c(2) = 167.7$ K, which can be explained by the fact that the compound became more regular in the crystal structure with the increase in the oxygen ratio, which makes this ratio the best among the ones studied (Fig. 4d).

As for the compensation ratio of $x = 0.4$, the critical temperature increased to $T_c(1) = 133.9$ K, $T_c(2) = 165$ (Fig. 4e). The decrease in the critical temperature can be explained by the appearance of impurities of copper oxides (Cu–O) in the compound, which led to a decrease in the crystal regularity and a decrease in the critical temperature due to the shortening of the *c*-axis. Table 3 shows the values of the critical temperature and the oxygen ratio for each compensation ratio.

4. Conclusion

In this work, we have examined $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ superconductor compounds synthesized by the solid state reaction technique for $0 \le x \le 0.4$. The XRD pattern analysis revealed a tetragonal structure with a high ratio of the Bi-2223 superconductor phase. Additionally, the Ag-doped sample at $x = 0.3$ showed the highest increase in the axial lattice constant *c*.

In the complex $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$, the optimal substitution ratio for Ag is found at $x = 0.3$, where a significant proportion of phase Bi-2223 is seen. This is the optimal value for *x*. Un-doped Bi-2223 had a critical temperature of $T_c(1) = 127.5$ K, $T_c(2) = 152.7$ K.

Table 3. The relationship between the compensation ratio (*x*), the critical temperature (T_c), and the percentage of oxygen (δ) in the compound $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$

Tabela 3. Zależność między współczynnikiem kompensacji (*x*), temperaturą krytyczną (T_c) a procentową zawartością tlenu (δ) w związku $Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$

Sample	$T_c(1)$ [K]	$T_c(2)$ [K]	ΔT_c [K]	T_c(mid) [K]	δ
$x = 0$	127.5	152.7	25.2	140.1	0.288
$x = 0.1$	131.2	158.9	27.7	145.05	0.325
$x = 0.2$	133.5	161.4	27.9	147.45	0.347
$x = 0.3$	135.1	167.7	32.6	151.4	0.392
$x = 0.4$	133.9	165.1	31.2	149.5	0.334

$Bi_{2-x}Ag_xBa_2Ca_2CO_2Cu_3O_{10+\delta}$ compounds have shown a maximum value of critical temperature, $T_c(1) = 135.1$ K, $T_c(2) = 167.7$ K, at $x = 0.3$ Ag substitution ratio.

In addition, the oxygen content was found to increase with the critical temperature, as the substitution induced local pressure, affected hole carrier concentration, and altered the electronic state and its distribution.

CRediT authorship contribution statement

Mudatheer M. Al-Slivani: Investigation, Data analysis, Writing – original draft, Writing – review & editing.

Muhammod A. Hammod: Investigation, Data analysis.

Mazin A. Abed: Data analysis, Writing – original draft, Writing – review & editing.

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