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Effect of Aluminum Oxide (Al_2O_3) Addition on the Structural and Electrical Properties of Bi-2223 High-Temperature Superconductors ($\text{Bi}_2\text{Ca}_2\text{Ba}_2$) $_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$

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Abstract: This research aims to study the effect of adding aluminum oxide (Al_2O_3) on the structural and electrical properties of the compound ($\text{Bi}_2\text{Ca}_2\text{Ba}_2$) $_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$. The samples were prepared using the solid-state reaction technique with varying ratios of Al_2O_3 , followed by sintering and annealing processes under controlled conditions. The structure was analyzed using X-ray diffraction (XRD) to determine the phases and lattice constants, in addition to electrical resistivity measurements to determine the superconducting transition temperature (T_c). The results showed that adding Al_2O_3 affects the phase purity and improves the magnetic flux pinning centers, with a slight modification in the lattice constants, while the optimal concentrations contributed to improving the transition sharpness without a significant reduction in T_c . These results confirm the importance of precise control of the structural composition to improve the functional performance of high-temperature superconductors.

Keywords: High-temperature superconductivity, Aluminum oxide (Al_2O_3), Structural Properties, Electrical Properties, X-ray Diffraction (XRD), Critical temperature (T_c)

1. Introduction

Superconductivity is considered one of the most important physical discoveries that has garnered global attention since its discovery in 1911 by Heike Kamerlingh Onnes when he cooled mercury to 4.2 Kelvin [1]. After decades of research, a significant breakthrough was achieved in 1986 with the discovery of the La-Ba-Cu-O compound, which exhibited superconductivity at temperatures above 30 Kelvin [2], leading to the emergence of a new Among the most prominent of these compounds are the cuprates, especially the compound $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$, known as Bi-2223, which is characterized by a high critical temperature ($T_c \approx 110$ K) and a complex layered crystal structure comprising three CuO_2 layers separated by layers of Bi-O, Sr-O, and Ca [3], [4]. These properties make it ideal for advanced applications such as ultra-high-power electrical transmission, magnetic levitation systems, magnetic resonance imaging (MRI) devices, and superconducting quantum interference devices (SQUIDS) [3], [4]. The critical properties of these materials, including the critical temperature (T_c), critical current (J_c), and upper critical magnetic field (H_{c2}), are significantly affected by precise control of the ideal chemical composition, oxygen content level, and structural modifications [5].

In the context of improving the functional performance of these vehicles, numerous studies have sought to introduce non-superconducting additives to control the microstructure and enhance conductivity. The addition of aluminum oxide (Al_2O_3) has proven effective in enhancing magnetic flux pinning centers and improving the crystallinity and purity of the crystalline phases [6], [7]. However, the results of these additions vary depending on the addition ratios, preparation techniques, and sintering

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conditions, which necessitates precise systematic studies to understand the relationship between structural modifications and electrical performance.

In this research, we conduct a systematic study on the effect of adding different proportions of Al_2O_3 on the structural and electrical properties of the $\text{Bi}_2\text{Ca}_2\text{Ba}_2\text{Cu}_3\text{O}_{10+\delta}$ compound prepared by the solid-state reaction method followed by controlled sintering and annealing processes. The study relied on X-ray diffraction (XRD) analysis to determine the structure, lattice constants, and phase ratios, in addition to electrical measurements to monitor changes in resistivity and the superconducting transition temperature [7].

2. Materials and Methods

Sample Preparation Model

In this study, a series of samples with the chemical formula $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ were prepared to investigate the effect of gradually adding aluminum oxide (Al_2O_3) on the structural and electrical properties. The solid-state reaction method was adopted as the primary preparation method due to its simplicity, efficiency in achieving homogeneity, and precision in controlling the ratio of chemical components.

Sample Preparation Steps the samples were prepared according to the following steps:

1. Weight:

The high-purity raw materials, Bi_2O_3 , CaCO_3 , BaCO_3 , CuO , and Al_2O_3 , were weighed in precise stoichiometric ratios using an electronic balance with a sensitivity of ± 0.1 mg, according to the general formula $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$, with varying Al_2O_3 ratios in the prepared compositions.

2. Mixing and Initial Grinding:

After weighing, the materials were mixed manually using a porcelain mortar and pestle. Wet grinding was performed after adding an appropriate amount of alcohol (as a temporary solvent) to reduce the volatilization of fine particles and improve the homogeneity of the mixture. Grinding continued for approximately an hour to ensure a homogeneous distribution of the ingredients.

3. Pre-sintering (Calcination):

The prepared mixtures were transferred to a programmed electric furnace, where the temperature was gradually increased to 750°C and held for 24 hours to activate the reaction between the oxides and form the initial phase of the compound.

4. Grinding After Sintering:

After cooling to room temperature, the resulting materials were ground again to ensure a homogeneous distribution of the components and to eliminate any agglomerates formed during sintering.

5. Pelletizing:

The prepared powders were pressed into tablet form using a hydraulic press under 7 tons of pressure, and the tablets were formed into regular dimensions, preparing them for the final sintering process.

6. Sintering:

The pressed pellets were subjected to an annealing process at 850°C for 48 hours in a programmable tube furnace to improve the crystalline structure and enhance the formation of the high-phase (2223).

After the duration ended, the samples were gradually cooled inside the furnace to room temperature to avoid any internal thermal cracks.

3. Results and Discussion

This section analyzes the effect of adding Al_2O_3 on the structural and electrical properties of a compound. The XRD technique was used to determine the crystal phase and lattice constants, along with resistance measurements to determine the critical temperature T_c . The results are discussed in light of previous literature to understand the relationship between structural addition and high conductivity performance.

X-ray Diffraction (XRD)

Samples of $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ with varying Al_2O_3 ratios were analyzed using XRD to assess the effect of the addition on the crystal structure. The reference sample

clearly showed the formation of the Bi-2223 high-phase, while the addition of a moderate amount of Al_2O_3 showed an improvement in phase purity and crystallinity. With increasing Al_2O_3 content, a deterioration in phase purity and the appearance of secondary phases were observed, indicating a maximum addition limit that can be exceeded without damaging the crystal structure. These results showed that the crystal system is tetragonal.

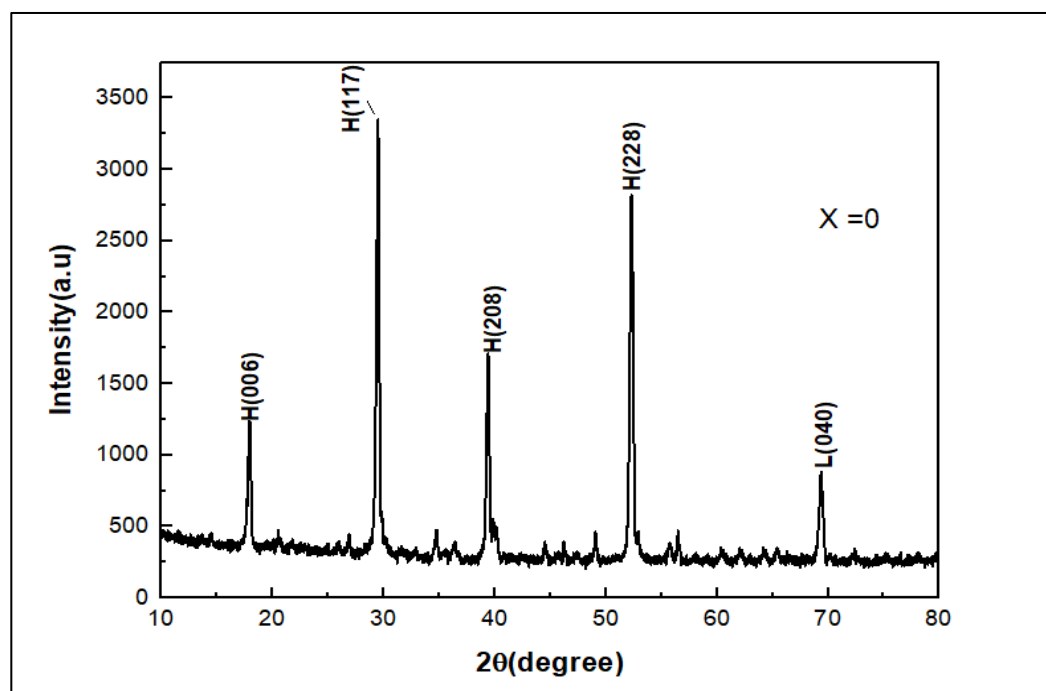


Figure 1. X-ray diffraction pattern of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ at $x=0$.

The XRD analysis results for the reference sample free of Al_2O_3 showed a clear formation of the high-phase Bi-2223, with a notable absence of secondary phases, indicating good phase purity. The lattice constants were calculated using a structural analysis program, with values of $a = b = 5.4 \text{ \AA}$, while the vertical axis $c = 30.8 \text{ \AA}$, reflecting a regular layered structure consistent with the ideal composition of the Bi-2223 phase. The c/a ratio was approximately 5.7, which falls within the acceptable range for this compound and indicates stability in the layered structure. The equivalent cubic cell volume (V^3) was also found to be approximately 898.63 \AA^3 , a value that supports good crystallization and efficient high-phase growth. These results indicate that the compound prepared with the composition $\text{Bi}_2\text{Ca}_2\text{Ba}_2\text{Cu}_3\text{O}_{10-\delta}$ and without external additives exhibits clear structural stability and forms a suitable basis for studying the effect of different additives such as Al_2O_3 on its structural properties.

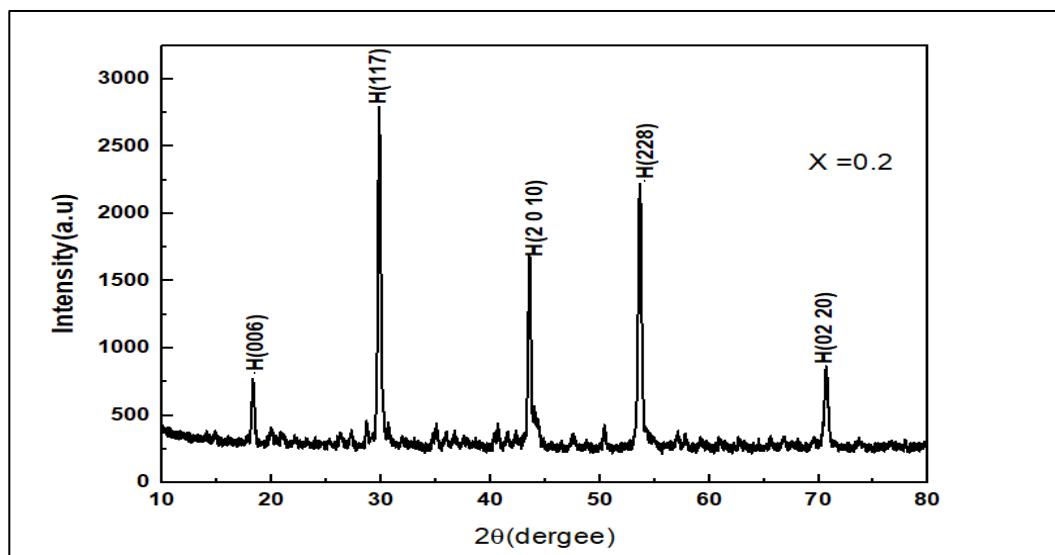


Figure 2. X-ray diffraction pattern of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ $x=0.2$.

XRD analysis of the sample with 0.2% added Al_2O_3 showed a slight improvement in the regularity of the crystalline structure compared to the reference sample. An increase in the c-axis length was observed, reaching 31.5 Å compared to 30.8 Å in the additive-free sample, while the a and b axis constants remained almost unchanged (5.403 Å). This indicates that the additive affected the vertical direction of the layered structure of the compound.

The c/a ratio reached approximately 5.83, which is higher than its counterpart in the first sample, indicating a slight expansion in the layered structure. This could be attributed to the effect of Al_2O_3 on the interlayer spacing. The equivalent cubic cell volume (V^3) also increased to 921.86 Å³, an indication of expansion in the overall structure. This change indicates that the moderate addition of Al_2O_3 has contributed to improving the structural homogeneity of the compound without causing significant distortions, which could positively impact the electrical properties, particularly concerning magnetic flux pinning.

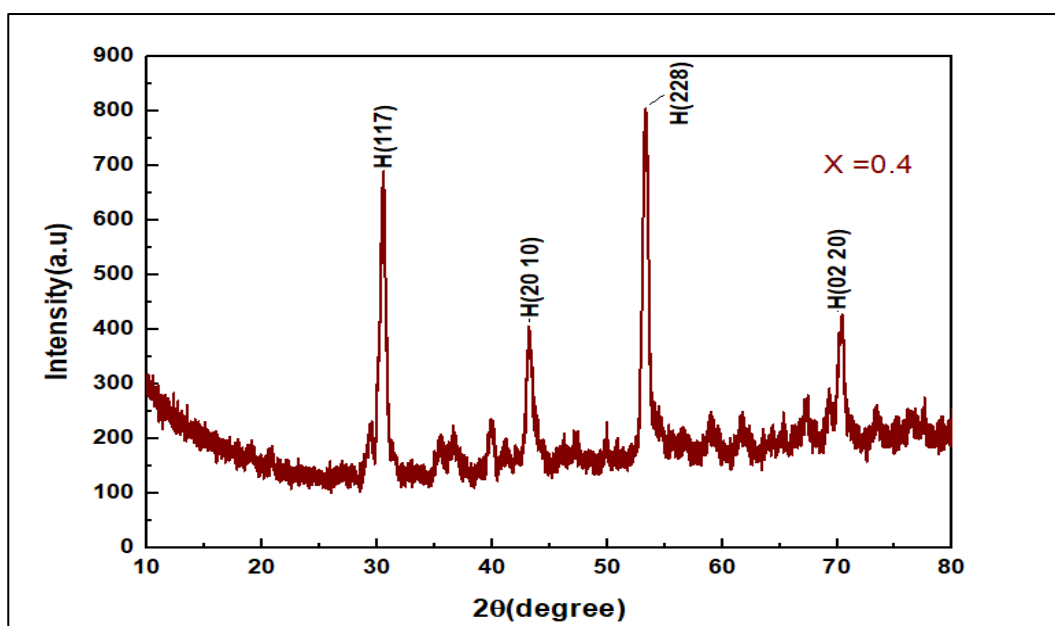


Figure 3. X-ray diffraction pattern of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ at $x=0.4$.

X-ray diffraction (XRD) analysis of the sample with a high Al_2O_3 addition ($x = 0.4$) showed the continued presence of the high-phase Bi-2223, but the intensity of the phase peaks began to decrease, with secondary peaks emerging indicating the formation of undesirable phases. This suggests that increasing the Al_2O_3 content beyond a certain limit may negatively affect the purity of the high-phase.

A significant increase was observed in the value of the c-axis ($c = 32.8 \text{ \AA}$) compared to the reference sample ($c = 30.8 \text{ \AA}$), indicating a clear expansion of the crystal lattice due to the increase in Al_2O_3 content. The c/a ratio also reached approximately 6.07, which is higher than the previous two samples, indicating an increase in crystal distortion in the vertical direction.

The equivalent cell volume reached $V^3 = 957.72 \text{ \AA}^3$, which is the highest among the three samples, reflecting a larger expansion in the crystal lattice, which may be related to the incorporation of aluminum at different sites within the structure. These results indicate that excessive addition of Al_2O_3 leads to disruption of the crystal structure and the appearance of secondary phases, which could negatively affect the superconducting properties.

Table 1. Lattice Constants for the Compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ at $x=0, 0.1, 0.4$.

Al_2O_3 content (x)	Lattice constant a (Angstroms)	Lattice constant b (Angstroms)	Lattice constant c (Angstroms)	Ratio c/a	Cell volume V^3 (cubic Angstroms)
0.0	5.400	5.400	30.8	5.70	898.63
0.2	5.403	5.403	31.5	5.83	921.86
0.4	5.404	5.404	32.8	6.07	957.72

These results align well with the research of [8] in terms of overall behavior and lattice constants. They showed that substituting aluminum in the compound leads to a gradual increase in the c-axis length and the c/a ratio as the substitution percentage increases up to $x = 0.4$, followed by a clear decrease in these values at $x = 0.5$. This behavior supports the hypothesis that the aluminum element contributes to the expansion of the vertical axis of the crystal structure due to its incorporation into the lattice. However, this effect is limited to a certain degree of compensation, followed by structural degradation resulting from exceeding the crystal lattice's capacity to accommodate this element.

Study of the effect of substitution on the electrical properties of the superconducting compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_x\text{Cu}_3\text{O}_{10+\delta}$:

The electrical properties of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_2\text{O}_3\text{Cu}_3\text{O}_{10+\delta}$ were studied at different substitution ratios (x) of the aluminum element, with the aim of exploring the effect of cationic substitution on the superconducting properties and crystal structure. The samples were prepared using the solid-state reaction method by mixing high-purity oxides in a precisely studied stoichiometric ratio. This was followed by intensive mechanical grinding to achieve complete homogeneity of the powders, then pressing the samples under a hydrostatic pressure of 7 ton/cm^2 . Subsequently, a sintering process was performed at a temperature of $850 \text{ }^\circ\text{C}$ in an air environment with precise control over the heating and cooling rates to ensure the stability of the oxygen state in the crystal lattice and the optimal formation of the Bi-2223 superconducting phase.

Electrical measurements using resistivity-temperature (R-T) curves for the pure, undoped sample ($x=0$), which is completely free of any aluminum oxide (Al_2O_3) content, showed that the critical temperature T_c reached approximately 106 K . This result reflects the highly efficient formation of the high-phase Bi-2223 and indicates good order in the arrangement of the CuO layers, which play a crucial role in forming the Cooper pairs responsible for superconductivity. This excellent performance is also attributed to improved sintering processes and reduced structural defects, which decrease electron scattering and enhance the critical current density (J_c) as shown in the figure 4.

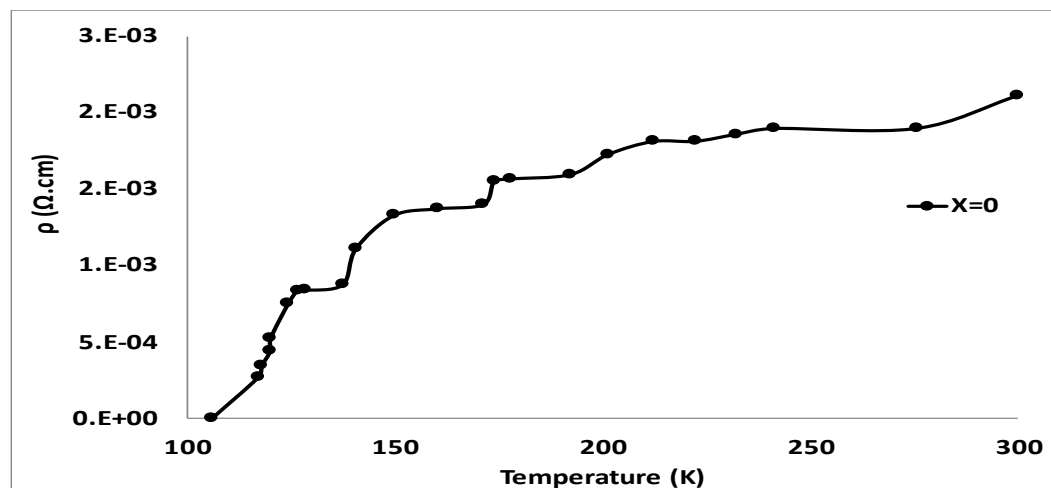


Figure 4. Electrical resistivity of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_x\text{Cu}_3\text{O}_{10+\delta}$ at $x=0$.

Electrical resistance measurements showed that the critical temperature (T_c) for the sample doped with $x=0.2$ compensation of aluminum oxide (Al_2O_3) increased to approximately 122 K. This represents a clear improvement compared to the undoped, uncompensated pure sample ($x=0$), which had a T_c of approximately 106 K. This improvement reflects the effective role of adding aluminum in enhancing the superconducting properties, as compensation with $x=0.2$ leads to the formation of magnetic flux pinning centers. These centers reduce the movement of magnetic field vortices within the crystal structure, thus maintaining superconductivity when exposed to external conditions such as high magnetic fields.

Choosing a compensation ratio of $x=0.2$ achieves an ideal balance between introducing minor disorder into the crystal lattice and enhancing pinning sites without compromising the regularity of the CuO_2 layers responsible for superconductivity. This leads to an increase in the critical current density (J_c) and an improvement in the superconducting transition quality (ΔT_c), which indicates that this ratio represents the optimal condition for improving the performance of the composite compared to the pure sample, as shown in the figure 5.

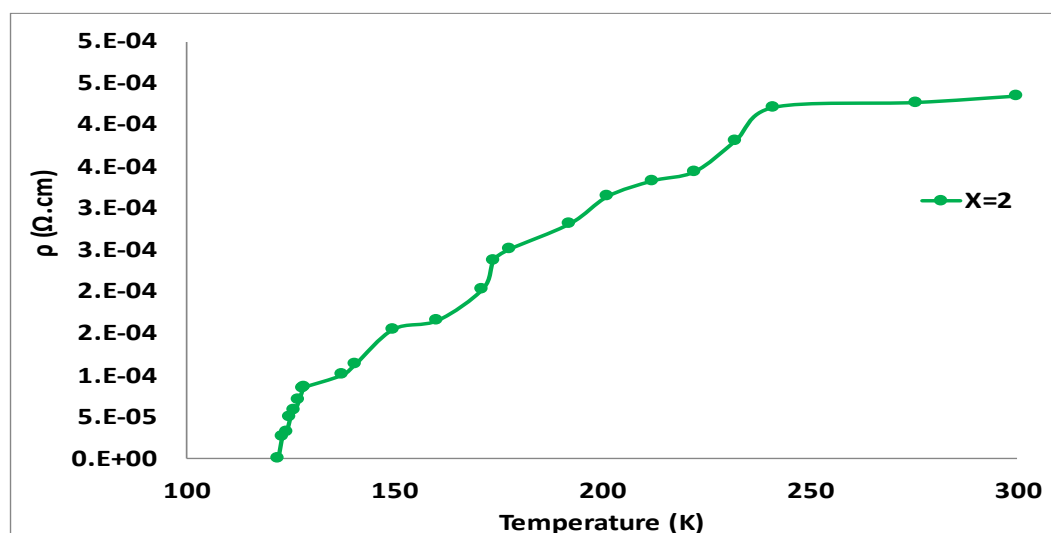


Figure 5. Electrical resistance of the compound $(\text{Bi}_2\text{Ca}_2\text{Ba}_2)_{1-x}\text{Al}_x\text{Cu}_3\text{O}_{10+\delta}$ at $x=0.2$.

Electrical measurements showed that the sample doped with a compensation ratio of $x=0.4$ of aluminum oxide (Al_2O_3) achieved the highest critical temperature, reaching approximately $T_c=130$ K, which is a significant improvement compared to the pure sample ($x=0$) and samples with lower compensation ratios. This increase in T_c is attributed to the

significant improvement in the crystal structure resulting from the introduction of aluminum ions (Al³⁺) into the crystal lattice, which led to a reduction in the density of structural defects and the achievement of better order in the CuO₂ layers responsible for electron superconductivity.

Adding x=0.4% Al₂O₃ also contributed to an increase in flux pinning centers, which are a crucial factor in limiting the movement of magnetic field vortices. This, in turn, improves the stability of the superconducting state and increases the critical current density (J_c). This enhancement in the pinning mechanism allowed Cooper pairs to move more efficiently, which increased the material's ability to maintain superconductivity at higher temperatures and under operational conditions. This makes this sample promising for practical applications requiring superior performance at relatively high temperatures, as shown in Figure 6.

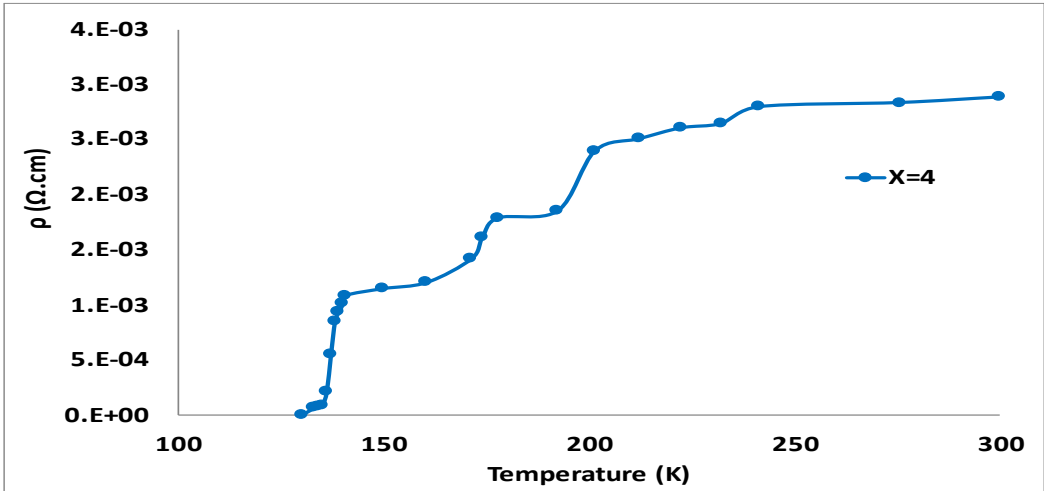


Figure 6. Electrical resistivity of the compound (Bi₂Ca₂Ba₂)_{1-x}Al_xCu₃O_{10+δ} at x=0.4.

Table 2. Calculating the critical temperature of the compound ((Bi₂Ca₂Ba₂)_{1-x}Al_xCu₃O_{10+δ}) Superconductivity with changing oxygen ratio at X = 0, 0.2, 0.4.

Compensation rate	Tc	Δ
0	106	0.12
0.2	122	0.34
0.4	130	0.5

These results align well with the structural properties, as the analyses showed that the high-temperature phase ratio increases with increasing doping concentration. The highest high-temperature phase ratio and critical temperature were recorded at a doping concentration of x=0.4. This behavior is consistent with the findings of the researcher in study [9], indicating a direct relationship between the crystalline phase and the superconducting properties.

4. Conclusion

- The compound (Bi₂Ca₂Ba₂)_{1-x}Al₂O₃Cu₃O_{10+δ} was prepared with different aluminum substitution ratios (x = 0.0, 0.2, 0.4) using the solid-state reaction method, under specific pressing and annealing conditions.
- X-ray diffraction (XRD) analysis showed that the addition of aluminum oxide directly affects the lattice constants, with the vertical axis length (c), the c/a ratio, and the unit cell volume increasing with increasing substitution ratio up to x = 0.4, indicating lattice expansion due to the introduction of aluminum.
- With an increase in the aluminum content to 0.4, a clear improvement in the purity of the high-phase Bi-2223 was observed. However, a further increase could lead to the

- appearance of undesirable secondary phases and a negative impact on the crystal structure.
- d. Measurements of electrical properties showed an increase in the critical temperature (T_c) with increasing compensation ratio, from 106 K for the uncompensated sample to 130 K at $x = 0.4$, indicating an enhancement of the superconducting property.
 - e. The improvement in electrical properties is attributed to the effect of adding Al_2O_3 in pinning magnetic flux vortices, which reduces the movement of these vortices and increases the stability of the superconducting state.
 - f. The results indicate an optimal limit for the aluminum compensation ratio ($x \approx 0.4$) to achieve the best balance between improving the structural and electrical properties of the composite.
 - g. The results are consistent with previous studies, confirming the importance of controlling compensation ratios to improve the performance of high-temperature superconductors.

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