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Effect of Relative Substitution of Nanomaterials (Pb, Mg) on the Structural and Electrical Properties of the Superconductor Compound $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_{2-y}\text{Mg}_y\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$

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Abstract

This study investigates the effect of Pb and Mg substitution on the structural and electrical residences of the Bi-Ba-Ca-Cu-O superconducting machine. Four compositions with different Pb/Mg contents have been investigated: $x = 0, y = 0$; $x = 0.5, y = 0.5$; $x = 0.1, y = 0.3$; and $x = 0.1, y = 0.5$. The structural homes had been tested the usage of X-ray diffraction (XRD), with particular attention to diffraction-peak function, d-spacing, relative depth, complete width at 1/2 most (FWHM), apparent crystallite size, and microstrain. The XRD patterns show that changes in Pb/Mg content are accompanied through shifts inside the major diffraction top and variations in top broadening and relative depth. The strongest diffraction peak was observed at 29.3177° , 29.2985° , 29.4832° , and 29.2225° in 2θ for $x = 0, y = 0$; $x = 0.5, y = 0.5$; $x = 0.1, y = 0.3$; and $x = 0.1, y = 0.5$, respectively. The corresponding d-spacings were 3.04390, 3.04585, 3.02719, and 3.05360 Å. The peak-specific apparent crystallite sizes were 402, 415, 429, and 396 Å, respectively. The profile-fitting results also varied with composition, with Rwp values of 0.06309, 0.05352, 0.05250, and 0.06258, respectively. The surface morphology exhibited noticeable changes in the distribution of elevated regions and valleys with variation in Pb/Mg content. Electrical transition measurements gave midpoint critical-temperature values of 122.00 K, 140.35 K, 96.75 K, and 96.85 K for the four compositions, respectively. The highest calculated $T_c(\text{mid})$ was obtained for $x = 0.5, y = 0.5$. The results indicate that Pb/Mg substitution influences both the structural characteristics and the electrical transition behavior of the Bi-based superconducting system.

Keywords

Bi-Based Superconductor;
Pb Substitution;
Mg Substitution;
XRD;
Crystallite Size;
Micro-Deformation;
Electrical Properties;
Morphology.



1. Introduction

Superconducting materials have attracted significant interest due to their extraordinary electrical and electromagnetic houses. The transition from the everyday carrying out nation to the superconducting nation is strongly affected by the chemical composition and structural traits of the material. Among excessive-temperature superconductors, Bi-primarily based cuprates are particularly exciting due to their layered crystal structure and their ability to accommodate chemical changes.

The residences of Bi-primarily based superconductors are influenced by means of several elements, along with the association of the constituent atoms, oxygen content, segment formation, crystallinity, and grain shape. Chemical substitution is one of the methods used to regulate those characteristics. Introducing special elements into decided on crystallographic websites can produce adjustments in lattice spacing, top role, crystallite length, lattice strain, and grain connectivity.

Pb substitution has been widely investigated in Bi-based superconductors because of its influence on phase formation and structural development. Mg substitution can also modify the local crystal environment and consequently affect the structural and electrical behavior of the ceramic. When Pb and Mg are introduced simultaneously, their effects may depend on their relative concentrations.

In the present compound, Pb is introduced through partial substitution of Ba, while Mg is introduced through partial substitution of Ca. The composition is therefore represented as:



where x represents the Pb substitution level and y represents the Mg substitution level.

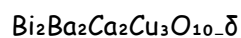
The goal of this work is to investigate the impact of various Pb/Mg substitution ratios on the structural residences of the Bi-based totally superconducting compound. The look at specializes in XRD top characteristics, d -spacing, crystallite length, microstrain, profile-fitting parameters, surface morphology, and crucial-temperature conduct.

2. Materials and Methods

2.1 Preparation of the investigated compositions

The investigated compositions were prepared from the corresponding oxide precursors. The required portions of Bi_2O_3 , PbO , BaO , MgO , CaO , and CuO were determined in step with the nominal composition of every sample.

The four compositions investigated in this work were:



The precursor quantities used for the compositions are given in Tables 1–4.

2.2 Powder Calculations

2.2.1 Powder calculation for $x=0$, $y=0$

Nominal composition: $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$

Table 1. Powder calculation for $x = 0$, $y = 0$.

Precursor	Calculation expression	Recorded mass (g)
Bi_2O_3	$2 \times [\frac{1}{2}(2 \times 208.98 \times 2 + 15.9994 \times 3)] \div 1000$	0.4651
BaO	$2 \times [137.33 + 15.9994] \div 1000$	0.3066
CaO	$2 \times [40.078 + 15.9994] \div 1000$	0.1125
CuO	$3 \times [63.546 + 15.9994] \div 1000$	0.2386

2.2.2 Powder calculation for $x=0.5$, $y=0.5$

Nominal composition: $\text{Bi}_{1.5}\text{Pb}_{0.5}\text{Ba}_{1.5}\text{Mg}_{0.5}\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$

Table 2. Powder calculation for $x = 0.5$, $y = 0.5$.

Precursor	Calculation expression	Recorded mass (g)
Bi_2O_3	$1.5 \times [\frac{1}{2}(2 \times 208.98 \times 2 + 15.9994 \times 3)] \div 1000$	0.3494
PbO	$0.5 \times [207.2 \times 2 + 15.9994] \div 1000$	0.1115
BaO	$1.5 \times [137.33 + 15.9994] \div 1000$	0.2299
MgO	$0.5 \times [24.305 + 15.9994] \div 1000$	0.02015
CaO	—	0.1125
CuO	—	0.2386

2.2.3 Powder calculation for $x=0.1$, $y=0.3$

Nominal composition: $\text{Bi}_{1.9}\text{Pb}_{0.1}\text{Ba}_{1.7}\text{Mg}_{0.3}\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$

Table 3. Powder calculation for $x = 0.1$, $y = 0.3$.

Precursor	Calculation expression	Recorded mass (g)
Bi_2O_3	$1.9 \times [\frac{1}{2}(2 \times 208.98 \times 2 + 15.9994 \times 3)] \div 1000$	0.4426
PbO	$0.1 \times [207.2 \times 2 + 15.9994] \div 1000$	0.02231
BaO	$1.7 \times [137.33 + 15.9994] \div 1000$	0.2606
MgO	$0.3 \times [24.305 + 15.9994] \div 1000$	0.01209
CaO	—	0.1125
CuO	—	0.2386

2.2.4 Powder calculation for $x=0.1$, $y=0.5$

Nominal composition: $\text{Bi}_{1.9}\text{Pb}_{0.1}\text{Ba}_{1.5}\text{Mg}_{0.5}\text{Ca}_2\text{Cu}_3\text{O}_{10-\delta}$

Table 4. Powder calculation for $x = 0.1$, $y = 0.5$.

Precursor	Calculation expression	Recorded mass (g)
Bi_2O_3	$1.9 \times [\frac{1}{2}(2 \times 208.98 \times 2 + 15.9994 \times 3)] \div 1000$	0.4426
PbO	$0.1 \times [207.2 \times 2 + 15.9994] \div 1000$	0.02231
BaO	$1.5 \times [137.33 + 15.9994] \div 1000$	0.2299
MgO	$0.5 \times [24.305 + 15.9994] \div 1000$	0.02025
CaO	—	0.1125
CuO	—	0.2386

2.3 X-ray diffraction measurement

X-ray diffraction measurements have been accomplished over the angular variety 8.0054° – 99.9874° in 2θ . The step size was 0.0110° , with a step time of 6.1200 s.

$\text{Cu K}\alpha_1$ radiation with a wavelength of $1.54060 \text{ \AA}$ used. The generator voltage and current have been 40 kV and 8 mA, respectively. Measurements have been accomplished at 25°C with specimen spinning.

The diffraction records have been processed using profile/Rietveld fitting. The resulting peak data protected diffraction perspective, d-spacing, relative intensity, top peak, FWHM, obvious crystallite length, and microstrain.

2.4 Structural analysis

The interplanar spacing turned into determined the use of Bragg's law:

$$n\lambda = 2d \sin\theta$$

wherein λ is the wavelength of the incident radiation, d is the interplanar spacing, θ is the diffraction perspective, and n is the diffraction order.

The FWHM values received from the diffraction peaks have been used within the evaluation of height

broadening and obvious crystallite size. Microstrain values were received from the furnished height-evaluation information.

2.5 Electrical measurements

The electrical transition temperatures have been decided from the recorded transition values. The midpoint temperature was calculated according to:

$$T_c(\text{mid}) = (|T_1| + |T_2|) / 2$$

The resulting values were used to compare the electrical transition behavior of the investigated compositions.

3. Results and Discussion: Structural Properties

The XRD styles of the four compositions are proven together in Figure 1. The styles showcase numerous commonplace reflections, confirming the similarity of the primary crystalline framework. At the equal time, changes in top function, intensity, and width are seen between the extraordinary compositions.

The fundamental diffraction mirrored image happens near 29° in 2θ for all compositions. Its actual position modifications with the Pb/Mg ratio, indicating that the interplanar spacing is affected by the substitution.

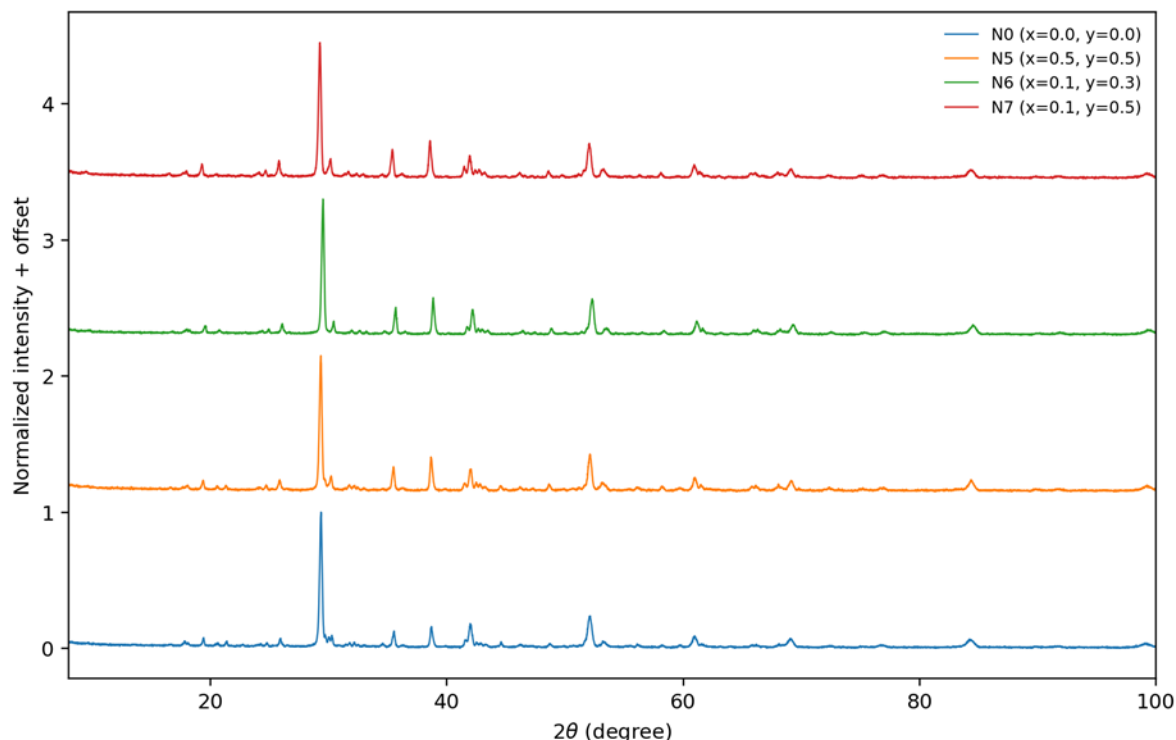


Figure 1. Combined XRD patterns of the four investigated compositions, normalized to the strongest intensity in each dataset.

3.1 Representative diffraction peaks

Table 5. Strongest diffraction peak in each dataset.

x, y	2θ ($^{\circ}$)	d ($\text{\AA}$)	Height (cts)	Rel. Int. (%)	FWHM ($^{\circ}$)	Crystallite size ($\text{\AA}$)
$x=0, y=0$	29.3177	3.04390	4971.72	100.00	0.2273	402
$x=0.5, y=0.5$	29.2985	3.04585	4391.73	100.00	0.2206	415
$x=0.1, y=0.3$	29.4832	3.02719	4962.57	100.00	0.2278	429
$x=0.1, y=0.5$	29.2225	3.05360	4380.83	100.00	0.2385	396

3.2 Effect for $x=0, y=0$

For $x=0, y=0$, the strongest recorded mirrored image is positioned at $2\theta=29.3177^{\circ}$ with $d=3.04390 \text{ \AA}$ and relative intensity of one 100%. The corresponding FWHM is 0.2273° , even as the peak-precise apparent crystallite length is $402 \text{ \AA}$. Across the whole top list, the pronounced obvious crystallite-size values make bigger from 58 to $28096 \text{ \AA}$ and the microstrain values amplify from 0.002% to 1.539%. These values imply that the substitution state is observed via adjustments in top broadening and lattice imperfection parameters.

3.3 Effect for $x=0.5, y=0.5$

For $x=0.5, y=0.5$, the strongest recorded reflection is placed at $2\theta=29.2985^{\circ}$ with $d=3.04585 \text{ \AA}$ and relative depth of 100.00%. The corresponding FWHM is 0.2206° , even as the peak-unique apparent crystallite size is $415 \text{ \AA}$. Across the complete peak list, the pronounced obvious crystallite-length values amplify from 142 to $2170 \text{ \AA}$ and the microstrain values expand from 0.052% to 1.464%. These values imply that the substitution country is observed by using changes in peak broadening and lattice imperfection parameters.

3.4 Effect for $x=0.1$, $y=0.3$

For $x=0.1$, $y=0.3$, the most powerful recorded mirrored image is located at $2\theta=29.4832^\circ$ with $d=3.02719 \text{ \AA}$ and relative depth of 100.00%. The corresponding FWHM is 0.2278° , even as the height-precise apparent crystallite size is $429 \text{ \AA}$. Across the complete height listing, the mentioned apparent crystallite-size values extend from 117 to $2870 \text{ \AA}$ and the microstrain values expand from 0.031% to 3.930%. These values imply that the substitution country is observed by way of changes in height broadening and lattice imperfection parameters.

3.5 Effect for $x=0.1$, $y=0.5$

For $x=0.1$, $y=0.5$, the strongest recorded reflection is located at $2\theta=29.2225^\circ$ with $d=3.05360 \text{ \AA}$ and relative intensity of 100.00%. The corresponding FWHM is 0.2385° , while the peak-specific apparent crystallite size is $396 \text{ \AA}$. Across the complete peak list, the reported apparent crystallite-size values extend from 97 to $8705 \text{ \AA}$ and the microstrain values extend from 0.012% to 4.840%. These values indicate that the substitution state is accompanied by changes in peak broadening and lattice imperfection parameters.

For $x=0.1$, $y=0.5$, the strongest recorded mirrored image is placed at $2\theta=29.2225^\circ$ with $d=3.05360 \text{ \AA}$ and relative intensity of a 100.00%. The corresponding FWHM is 0.2385° , while the peak-particular apparent crystallite size is $396 \text{ \AA}$. Across the complete top listing, the suggested obvious crystallite-length values enlarge from 97 to $8705 \text{ \AA}$ and the microstrain values increase from 0.012% to 4.840%. These values indicate that the substitution kingdom is observed by using modifications in peak broadening and lattice imperfection parameters.

3.6 Comparative structural effect of Pb/Mg substitution

The XRD consequences suggest that the Pb/Mg substitution ratio influences the structural characteristics of the investigated compound. The principal diffraction peak shifts from 29.3177° for $x = 0$, $y = 0$ to 29.2985° , 29.4832° , and 29.2225° for the substituted compositions.

The corresponding d-spacings also exchange from $3.04390 \text{ \AA}$ to 3.04585 , 3.02719 , and $3.05360 \text{ \AA}$. This behavior suggests that the interplanar spacing responds to the specific Pb/Mg ratio.

Changes are also discovered in top width and apparent crystallite size. The most powerful-mirrored image crystallite length varies from 396 to $429 \text{ \AA}$ most of the investigated compositions, at the same time as the whole height records show wider variations in each crystallite length and microstrain.

The electrical results show a comparable dependence on composition. The highest calculated $T_c(\text{mid})$, 140.35 K is received for $x = 0.5$, $y = 0.5$. The compositions with $x = 0.1$ have midpoint temperatures near ninety-seven K.

The structural and electrical effects consequently reveal that the effect of Pb/Mg substitution depends at the relative amount of every substituting element. The structural becoming parameters and the calculated transition temperature do no longer display the equal trend, indicating that the electrical response can't be defined entirely through the numerical satisfactory of the XRD profile in shape.

Table 6. Profile/Rietveld fitting indicators reported in the supplied instrument reports.

x, y	χ^2	Rp	Rwp
$x=0, y=0$	1.324720	0.04570	0.06309
$x=0.5, y=0.5$	1.135589	0.04191	0.05352
$x=0.1, y=0.3$	1.120512	0.04105	0.05250
$x=0.1, y=0.5$	1.357566	0.04618	0.06258

4. Morphological Analysis

The supplied morphology files are three-dimensional surface maps. They provide qualitative information approximately surface relief, grain-like capabilities, depressions and the degree of floor uniformity. Because no calibrated grain-length scale or SEM magnification is supplied for these maps, the dialogue is qualitative and does not assign numerical grain sizes from the 3D photos.

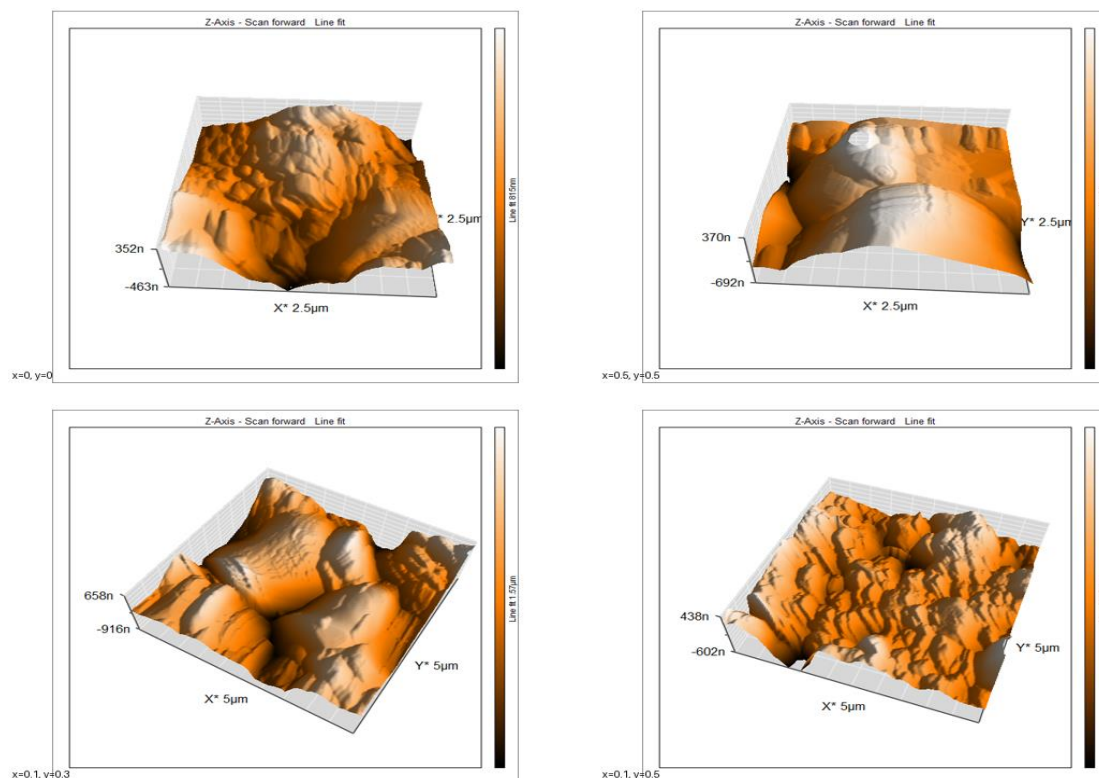


Figure 2. Combined surface-morphology maps for the four investigated compositions.

4.1 $x=0, y=0$

The $x=0, y=0$ surface map shows a relatively broad, continuous relief with said undulations and several valleys. The surface isn't always perfectly flat, indicating a heterogeneous topography at the measured scale.

4.2 $x=0.5, y=0.5$

The $x=0.5, y=0.5$ map famous a strongly developed three-dimensional surface with wonderful raised regions and depressions. The relief seems greater compact and interconnected than a simple flat granular layer, indicating sizable modification of the floor morphology after the very best paired substitution recorded on this dataset.

4.3 $x=0.1, y=0.3$

The $x=0.1, y=0.3$ map shows reported ridges and valleys with a extra segmented grain-like alleviation. The visible topography indicates appreciable floor heterogeneity and a advanced network of obstacles/depressions.

4.4 $x=0.1, y=0.5$

The $x=0.1, y=0.5$ map suggests said elevated domains separated by deep valleys. The surface comfort is strongly advanced, and the larger continuous raised regions endorse coalesced surface features on the dimension scale.

Overall, the morphology changes with the Pb/Mg ratio. The differences in surface remedy are regular with the structural modifications discovered in the XRD effects.

5. Electrical Properties and Critical Temperature

The electrical transition behavior of the investigated compositions turned into evaluated the usage of the

recorded T_1 and T_2 values. The midpoint crucial temperature was calculated from the 2 transition values.

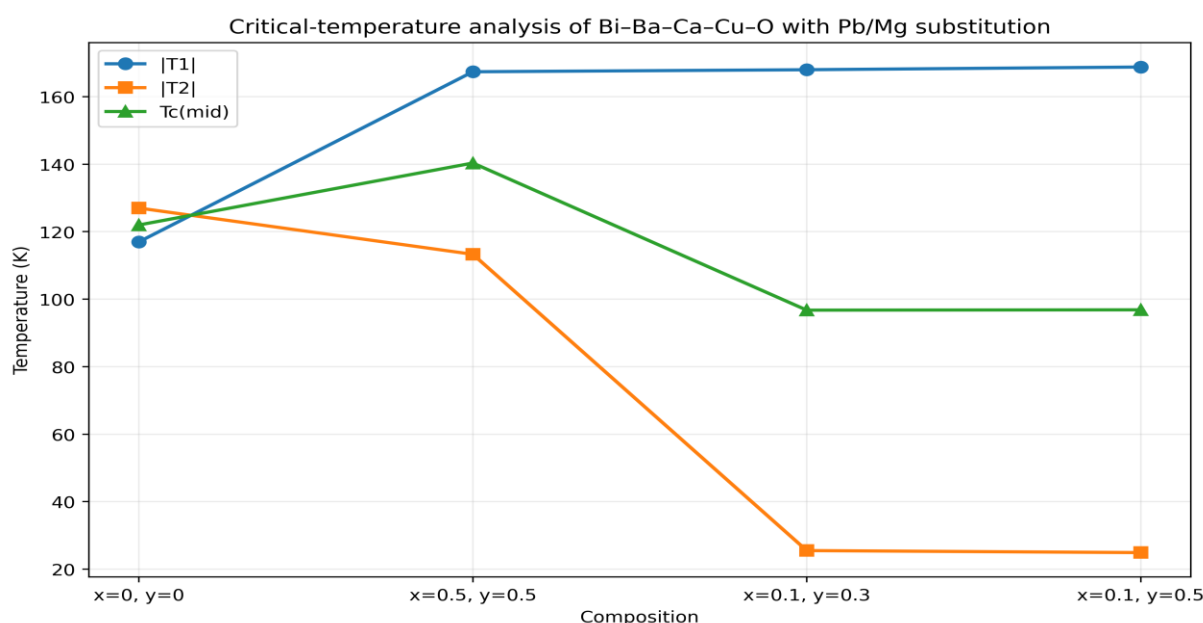


Fig. 3. Temperature characteristics (T_1 , T_2 and calculated $T_c(\text{mid})$) of the investigated Bi-Ba-Ca-Cu-O samples with Pb/Mg substitution.

The calculated $T_c(\text{mid})$ does now not follow a monotonic trend with increasing substitution. The $x = 0.5$, $y = 0.5$ composition shows the best calculated midpoint temperature, while the 2 compositions with $x = 0.1$ show considerably decrease midpoint values.

Table 7. Critical-temperature values calculated in the same format as the supplied reference table.

x	y	Tc offset / T1 (K)	Tc onset / T2 (K)	Tc mid = (T1 + T2)/2 (K)
0.0	0.0	117.0	127.0	122.00
0.5	0.5	167.4	113.3	140.35
0.1	0.3	168.0	25.5	96.75
0.1	0.5	168.8	24.9	96.85

The $x = 0$, $y = 0$ composition offers a midpoint temperature of 122.00 K. When the composition changes to $x = 0.5$, $y = 0.5$, the calculated midpoint increases to to 140.35 K.

For $x = 0.1$, $y = 0.3$, the calculated midpoint is 96.75 K, while $x = 0.1$, $y = 0.5$ gives 96.85 K. while while $x = 0.1$, $y = 0.5$ gives 96.85 K. The difference between these two compositions is only 0.10 K.

Relationship Between Structural and Electrical Properties

The XRD outcomes show that Pb/Mg substitution modifications the diffraction response of the Bi-primarily based compound. The most important reflection occurs at 29.2985° for $x=0.5$, $y=0.5$, 29.4832° for $x=0.1$, $y=0.3$, and 29.2225° for $x=0.1$, $y=0.5$, with corresponding d-spacings of 3.04585, 3.02719 and 3.05360 Å, respectively. These shifts display that converting the substitution ratio modifies the nearby lattice surroundings and interplanar spacing. The top-profile information also shows composition-structured broadening, crystallite-size and microstrain versions.

The electric effects offer a 2nd indication that the substitution ratio impacts the cloth reaction. The $x=0.5$, $y=0.5$ composition gives the very best calculated $T_c(\text{mid})$, 140.35 K, while its XRD profile also has a enormously low Rwp of 0.05352. The $x=0.1$, $y=0.3$ composition has the lowest supplied Rwp and χ^2 values, but its calculated $T_c(\text{mid})$ is 96.75 K. Thus, the structural becoming criterion and the electrical midpoint criterion do now not pick the same composition. This is important: a low Rwp or χ^2 suggests agreement between the measured and outfitted diffraction profiles; it is not itself a measure of superconducting overall performance.

The literature helps the overall observation that chemical substitution and processing can adjust section formation, crystallinity, grain connectivity and superconducting transition behavior in Bi-based superconductors. For example, research of (Bi,Pb)-2223 report that Pb substitution can affect the formation of the 2223 phase and that optimized composition and heat remedy can produce essential temperatures across the 108-112 K variety. Other studies also show that dopant awareness could have an choicest instead of a easy monotonic impact on T_c .

7. Best composition

Based on the calculated midpoint critical temperature, the composition $x = 0.5$, $y = 0.5$ gives the highest $T_c(\text{mid})$ value, reaching 140.35 K.

This composition also shows a main diffraction peak at 29.2985° , a d-spacing of 3.04585 Å, an apparent crystallite size of 415 Å, and an Rwp value of 0.05352.

From the XRD profile-becoming parameters, the $x = 0.1$, $y = 0.3$ composition offers the lowest χ^2 and Rwp values, with $\chi^2 = 1.120512$ and Rwp = 0.05250.

Accordingly, the $x = 0.5$, $y = 0.5$ composition is outstanding through the highest calculated midpoint transition temperature, even as $x = 0.1$, $y = 0.3$ shows the lowest profile-becoming parameters.

8. Conclusions

The structural and electrical residences of Bi-Ba-Ca-Cu-O have been investigated for different Pb/Mg substitution ratios.

The XRD styles display clean modifications in diffraction-top role, d-spacing, height width, crystallite size, and microstrain as the composition adjustments.

The strongest diffraction peaks occur at 29.3177° , 29.2985° , 29.4832° , and 29.2225° for $x = 0$, $y = 0$; $x = 0.5$, $y = 0.5$; $x = 0.1$, $y = 0.3$; and $x = 0.1$, $y = 0.5$, respectively.

The corresponding d-spacings are 3.04390, three.04585, 3.02719, and 3.05360 Å. The associated obvious crystallite sizes are 402, 415, 429, and 396 Å.

The XRD profile-becoming parameters also vary among the compositions. The lowest χ^2 and Rwp values are received for $x = 0.1$, $y = 0.3$, with values of 1.120512 and 0.05250, respectively.

The three-dimensional morphology maps display modifications in surface remedy, including versions in increased regions and valleys, indicating that the Pb/Mg ratio influences the floor structure.

The calculated midpoint transition temperatures are 122.00 K, 140.35 K, 96.75 K, and 96.85 K for $x = 0$, $y = 0$; $x = 0.5$, $y = 0.5$; $x = 0.1$, $y = 0.3$; and $x = 0.1$, $y = 0.5$, respectively

The maximum calculated $T_c(\text{mid})$ is obtained for $x = 0.5$, $y = 0.5$, with a value of 140.35 K. The consequences exhibit that the structural and electrical responses depend on the relative Pb/Mg substitution ratio and that the adjustments are not described through a unmarried monotonic trend.

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