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Preparation of the Compound ($Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta}$) Superconductor and Research of the Impact of Partial Substitution on the Structural Properties

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RESEARCH ARTICLE

Preparation of the Compound $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ Superconductor and Research of the Impact of Partial Substitution on the Structural Properties

Ekhlas Hassan^{1,2,*}, Haider M. J. Haider²¹ Material Department, Faculty of Engineering, University of Kufa, Najaf, Iraq² Physics Department, College of Education for Girls, University of Kufa, Najaf, Iraq**ABSTRACT**

The purpose of this study is to find out the optimal substitution ratio for lead with yttrium through the structural properties of the prepared compounds. The superconductor compound $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ was prepared by the solid state reaction (SSR) method. The effect of partial substitution for the lead on the compound's structural characteristics has been studied. The variation of (x) is (0, 0.05, 0.1, 0.15, 0.20, 0.25). With a pressure of 7 tons per square centimeter, the combined powder was formed into a disc with a thickness of (about 0.2 cm). The samples were sintered for a while (24 hours) at a rate that changed from room temperature (27°C) to (850°C). Based on the X-ray diffraction (XRD) study. All samples were discovered to contain an orthorhombic structure. It was established that variations in the lead concentrations in each of our samples result in variations in parameters for a lattice (a, b, c, c/a), dm, Tc, and energy gap. Its best values were recorded at a concentration (x = 0.1), where the values of the lattice constants in angstrom units (a, b, c) were (3.807346, 3.886549, and 11.70871) respectively, the highest value for (Tc) was (112.1). The density value (dm) was (6.42604 gm/cm³), the highest percentage was (c/a = 3.075295) and the highest energy gap was (Eg = 0.03384 eV). So, it can be considered the optimal replacement ratio under the conditions of this work. Scanning electron microscopy (SEM) was used to analyze microscopic pictures of the compounds prepared with different substitution ratios in addition to the pure sample at very high magnifications (SEM) at a degree of magnification (5 μm, 200 nm).

Keywords: Partial substitution, Scanning electron microscope (SEM), Solid state reaction (SSR) method, Superconductor, X-ray diffraction (XRD)

Introduction

Bednorz and Müller's discovery of lanthanum barium copper oxide (LBCO), with a critical temperature of (Tc = 35 K), marked the beginning of the High-Tc superconductor (HTS) epoch. By substituting Y³⁺ ions with La³⁺ ions, YBa₂Cu₃O₇ (Y123) is formed, raising HTS's transition temperature to about (92 K). The first substance with a superconducting transition temperature discovered above the temperature

of liquid nitrogen is Y123. Family of compounds Y-Ba-Cu-O, including Y123, YBa₂Cu₄O₇ (Y247), (Tc = 40 K) and YBa₂Cu₄O₈ (Y124, Tc = 80 K); have undergone extensive research since their discovery.¹ The qualities of the bulk sample, such as fracture and bending toughness, will be significantly improved by the partial addition or substitution of certain chemical elements, especially those of metallic quality, such as cadmium, nickel, silver, lead, and copper in the crystalline structure.² Typically, it took hundreds

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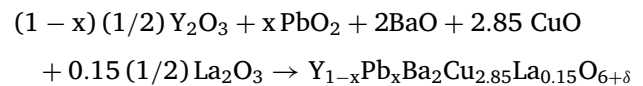
of hours to achieve a virtually pure phase material (this includes several sintering stages).³ In addition to assisting in the discovery of new uses for superconductivity, research on the doping or replacement of additional elements into cuprate superconductors aids in knowing the pairing strategy in HTSs. In (Y123), the chemically linked planes between copper and oxygen atoms are of utmost importance. Directional effective electrical conductivity is a result of the unique properties of the chemical interaction between copper and oxygen. The presence (of Cu-O) chains and (CuO₂) planes in cuprate superconductors demonstrate the significance of Cu atoms in these materials. Thus, a key factor in defining the properties of novel superconductors, which include both the dopant element's valence number and ionic radius. Doping (Y123) with different substances is done primarily for two reasons. The first one is modifying the microstructure to get fundamental knowledge about potential processes, while the second involves enhancing its physical attributes to enhance Y123's capacity for superconductivity.¹ These materials' physical characteristics might be modifiable by partial or whole atomic replacements.⁴ Perovskite compound YBa₂Cu₃O_{6+δ} is regarded as a ceramic substance.⁵ CuO₂ mediates the layering of the perovskite structure of (YBCO), in addition to that the (CuO) levels are mediated by yttrium along the crystal's c-axis in the stacking order (BaO-CuO₂-Y-CuO₂-BaO).⁶

The study aims to prepare the high-temperature superconducting compound (YBa₂Cu_{2.85}La_{0.15}O_{6+δ}) by using pure oxides and by solid-state reaction method (SSR). The impact of partial Pb replacement for the Y site on the superconducting characteristics of Y123 superconductor was examined.

(Y_{1-x}Pb_xBa₂Cu_{2.85}La_{0.15}O_{6+δ}) where (x = 0, 0.05, 0.1, 0.15, 0.2, 0.25), search the structure characteristics of superconducting compounds as well as determine the coefficients of the lattice (a, b, c) and the density of the unit cell. The study also seeks to obtain the highest critical transfer temperature (High-T_c), investigate the optimal substitution ratio for the formation of the high phase (Y123) and its stability by using (XRD) diffraction and images from a scanning electron microscope (SEM).

Materials and methods

The superconducting compound (Y_{1-x}Pb_xBa₂Cu_{2.85}La_{0.15}O_{6+δ}) with (x = 0.00, 0.05, 0.1, 0.15, 0.20, 0.25), was prepared by utilizing appropriate amounts of pure powders of (Y₂O₃, BaO, CuO, La₂O₃, PbO₂) and using method (SSR), depending on:



were mixed and ground together manually using agate mortar for half an hour and electrically for two hours using a Vortex mixer to obtain fineness and optimal homogeneity of the powders, then the resulting powders dried at a temperature of 200°C in a drying oven for a period of 1hr, employing a pressure-hydraulic press (7tons/cm²). For one minute, the samples were pressed into discs with a diameter 1.5 cm and thickness 0.2 cm, then the samples were sintered inside the sintering furnace at a temperature 820 degrees Celsius gradually as shown in Fig. 1.

An (X-ray diffraction) test was performed, through which the structural properties of the compound

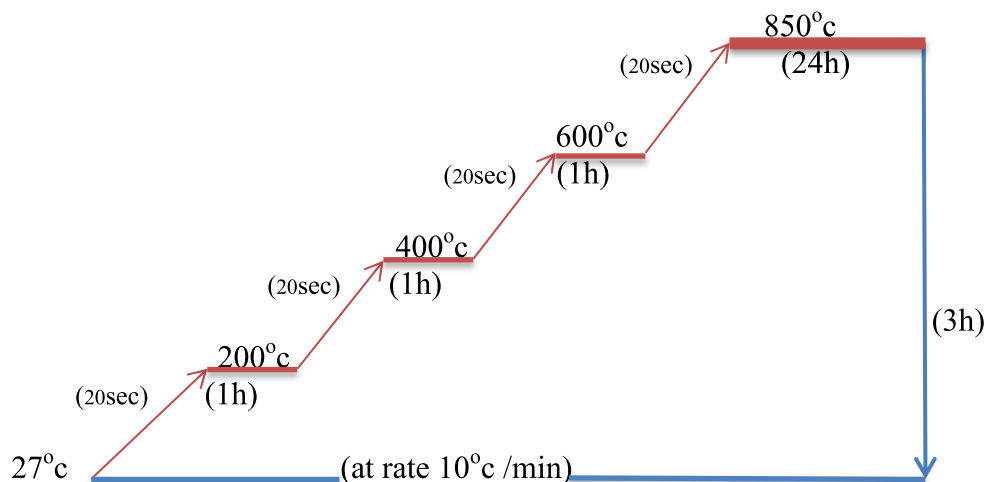


Fig. 1. The diagram shows the rise and fall of the temperature during the sintering process.

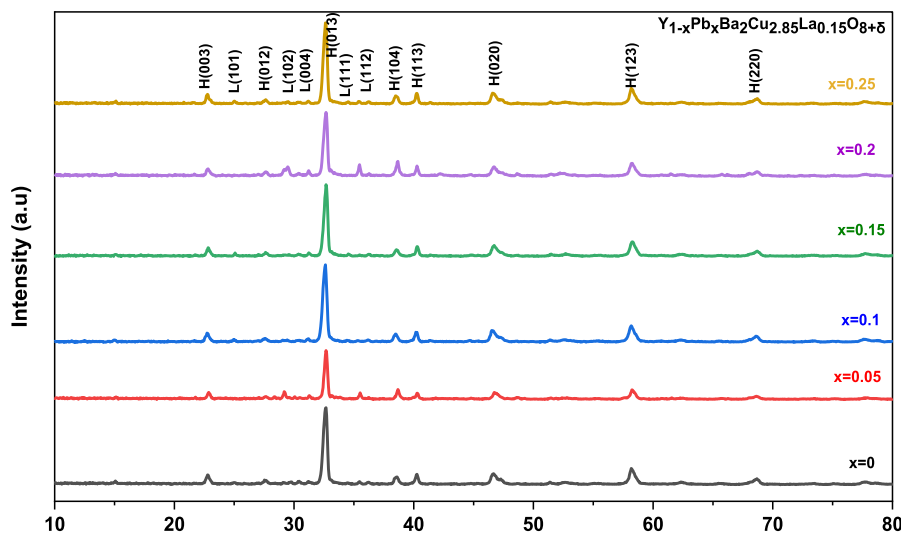


Fig. 2. X-ray diffraction for $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ bulk polycrystalline samples where $x = 0, 0.05, 0.1, 0.15, 0.2$ and 0.25 .

were studied. Mathematically, the sample's lattice constants were computed by finding the (2θ) and Miller's coefficients (hkl) for each vertex and applying Eq. (1)⁶:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (1)$$

The formula below was used Eq. (2) to get the volume fraction of phase⁷:

$$(V_{ph})\% = \frac{\sum I_0}{\sum I_1 + \sum I_2 + \sum I_{other(peaks)}} \times 100\% \quad (2)$$

Where I represent the intensity of each phase's peaks. The density of a unit cell was determined using the Eq. (3) shown below⁸:

$$D_m = \frac{W_m}{N_A V} \quad (3)$$

Where W_m : the molecular weight (amu), N_A : Avocado's number (particles/gm.mol), and V : the volume of a unit cell (cm^3). Following that, there determined a gap in the samples' energy (E_g) using Eq. (4)⁶:

$$E_g = 3.53K_B T_c \quad (4)$$

Where K_B represents the Boltzmann constant, T_C critical transition temperature, then used the four probes technique with the presence of liquid nitrogen to determine the critical temperature of the transition as a function of temperature. SEM was used to view microscopic pictures of the materials at extremely high magnifications (SEM).

Results and discussion

XRD was examined for $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ bulk polycrystalline samples where $(x = 0, 0.05, 0.1, 0.15, 0.2, 0.25)$ for a deflection angle ranging between $(20^\circ-80^\circ)$. The X-ray diffraction patterns are shown in Fig. 2. The findings reveal polycrystalline structures with orthorhombic structures for all samples. The phase (Y-123) and certain impurity phases may be seen in the XRD pattern. The rate of higher phase development was impacted by the increase in the substitution ratio.

By substitution of Pb concentration, the substitution process will lead to a very small shift in the XRD chart angle and peak intensities as shown in Table 1. The lengths of the lattice constants can expand or contract with the change of electrons in the orbitals, and the behavior of the samples prepared by different

Table 1. Values of the parameters a,b, and c with different substitutions.

X=	a(A°)	b(A°)	c(A°)	c/a	V(A°) ³	dm (gm/cm ³)	Vph (H)	Vph (L)
0	3.873335	3.881677	11.61	2.997417	2.997417	6.378262	85.7	14.3
0.05	3.861861	3.874241	11.65287	3.017424	3.017424	6.38591	84.6	15.4
0.1	3.807346	3.886549	11.70871	3.075295	3.075295	6.42604	86.7	13.3
0.15	3.824839	3.879866	11.66987	3.051074	3.051074	6.428994	85.7	14.3
0.2	3.879688	3.881003	11.682	3.011067	3.011067	6.329667	71.4	28.6
0.25	3.899964	3.877052	11.65313	2.988011	2.988011	6.318792	75	25

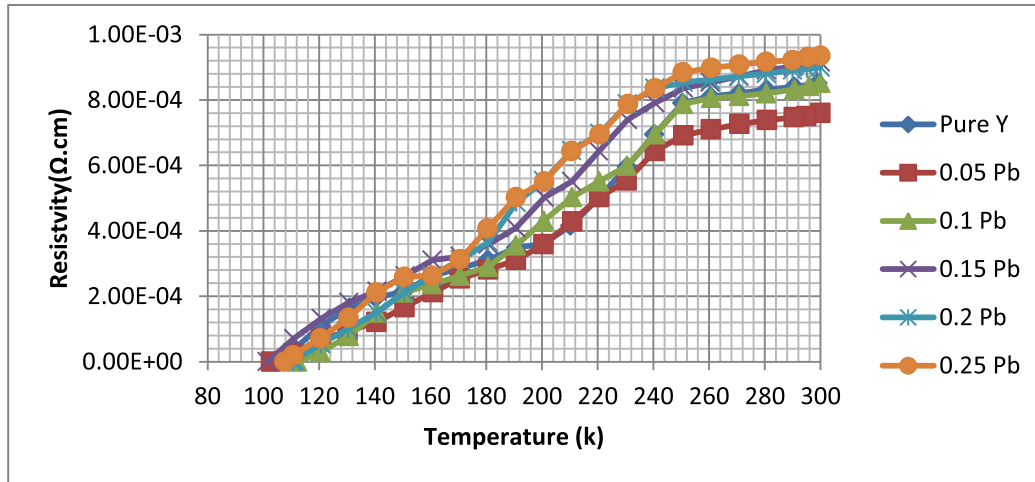


Fig. 3. The relationship between temperature and electrical resistivity of $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ for $(x = 0, 0.05, 0.1, 0.15, 0.2$ and $0.25)$.

concentrations affected structural attributes. This is demonstrated by the variation of density, the ratio c/a , and constants. These samples' altered lattice constants may be an indication that oxygen has been added, which might be brought about by the environment's impact on the preparation's circumstances, and can have a substantial effect on the oxygen concentration. Additionally, it could be caused by the ionic radius difference, which changes the phases and lattice coefficients of superconducting compounds.⁹

When compared to alternative substitutions, the one with the value of $(x = 0.1)$ has the best structural features. The properties of the obtained samples may be significantly impacted by the preparation procedures (sintering time, pressure, the powder's granularity, sintering temperature, mixing technique, and grinding). The sintering process' temperature and length are crucial factors in the creation of high-frequency samples because they do not only have the potential to cause microscopic cracks in the sample but also to bring the material closer to a state of solubility, which encourages the growth of low phases and impurities at the expense of the high phase and its formation. According to studies, the temperature just below the melting point is ideal for sintering.¹⁰

Fig. 3, illustrates the relationship between temperature and electrical resistivity, where we see that before the $(T_c\text{-offset})$, which causes the material to enter the superconducting state, all the samples displayed metallic behavior. The likelihood that more impurities and secondary phases might contribute in additional energy levels to the (CuO) layers, which correspond with the (CuO) level and result in a bond between the two levels, is what causes the abrupt reduction in resistivity.⁶ Additionally, the critical temperature for this compound varied from that recorded for the samples; the maximum critical temperature for substitution $(x = 0.1)$ is 112.1 K, as shown in **Table 2**.

Regarding the transition width (T_c) , we observe that it had low values, which suggests that the sample **was homogeneous. The samples' low phases and impurities**, which are present in different amounts, are the cause of this behavior. The substitution of lead for yttrium, which tends to modify the concentration of charge carriers and conductive layers in this system, maybe the cause.^{6,11,12}

The SEM was used to conduct the electronic survey $(Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta})$ in various substitutions. At different magnification levels $5 \mu m$,

Table 2. Shows the ratios of phases, critical temperature, energy gap, and concentration of gaps for the samples.

X=	Tc (K)	Tc (off) (K)	Tc (on) (K)	ΔT (K)	T (mid) (K)	Eg (eV)
0	104.5	104.5	130.9775	26.47752	117.7388	0.031546
0.05	102.8	102.8	130.2629	27.46294	116.5315	0.031033
0.1	112.1	112.1	130.7591	18.65911	121.4296	0.03384
0.15	101.5	101.5	130.7986	29.29856	116.1493	0.03064
0.2	110.5	110.5	130.4891	19.98909	120.4945	0.033357
0.25	107.7	107.7	130.6225	22.92254	119.1613	0.032512

200 nm, we noted the material's regular inhomogeneity as seen from the presence of dark and bright zones. We noticed that the proportion of bright regions is greater than the proportion of dark areas and is homogenous. As seen in many images with various substitutions the material's compound homogeneity and the appearance of big grains; these granule sizes match the (XRD) results in terms of the occurrence of sharp peaks. The tops of the sharp show an increase in granule size and the reduction in peak breadth implies an increase in granule size (based on Schiller's law). The area of the granular border decreases as granule size increases, which in turn prevents superconducting currents from moving as seen in Fig. 4. This sample's granules are made up of a tiny number of granules and granules with a plate type. Additionally, we see that the granule size distribution is largely uniform as well as the growth of tiny grains and black plate particles. According to Lopera *et al.*, the high phase is thought to be the cause of the black platelet morphology.¹⁰

Conclusion

The superconducting compound ($Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta}$) with ($x = 0.00, 0.05, 0.1, 0.15, 0.20, 0.25$) was successfully prepared as part of the current investigation utilizing the (SSR) method. All of the samples were found to exhibit metallic behavior in that they changed their electrical resistance as the temperature fell before entering the superconducting state. The sample with $x = 0.1$ has the best structural characteristics, as shown by the high phase height and the decline of the low phase (Y-123) and by looking at the microscope images. Thus, when compared to other samples, may be regarded as the best replacement ratio for substituting lead instead of yttrium.

The XRD diffraction test findings indicated that the substance was polycrystalline and had an orthorhombic structure. The highest critical temperature was at the concentration ($x = 0.1$), where ($T_c(\text{offset}) = 112.1$ K). It also showed how crucial the prolonged sintering

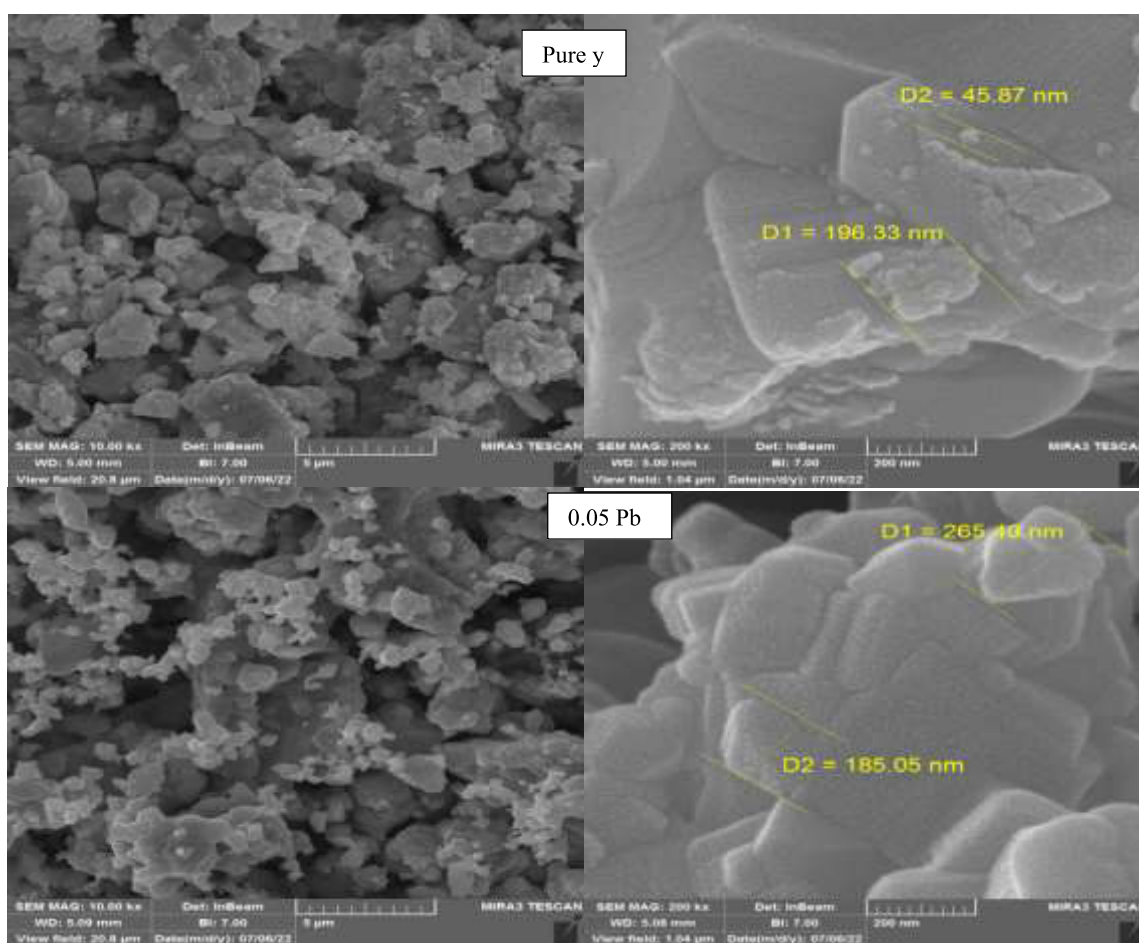


Fig. 4. Shows the SEM of ($Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta}$) for ($x = 0, 0.05, 0.1, 0.15, 0.2$ and 0.25) at degree of magnification ($5 \mu m, 200 nm$). (Continued on next page)

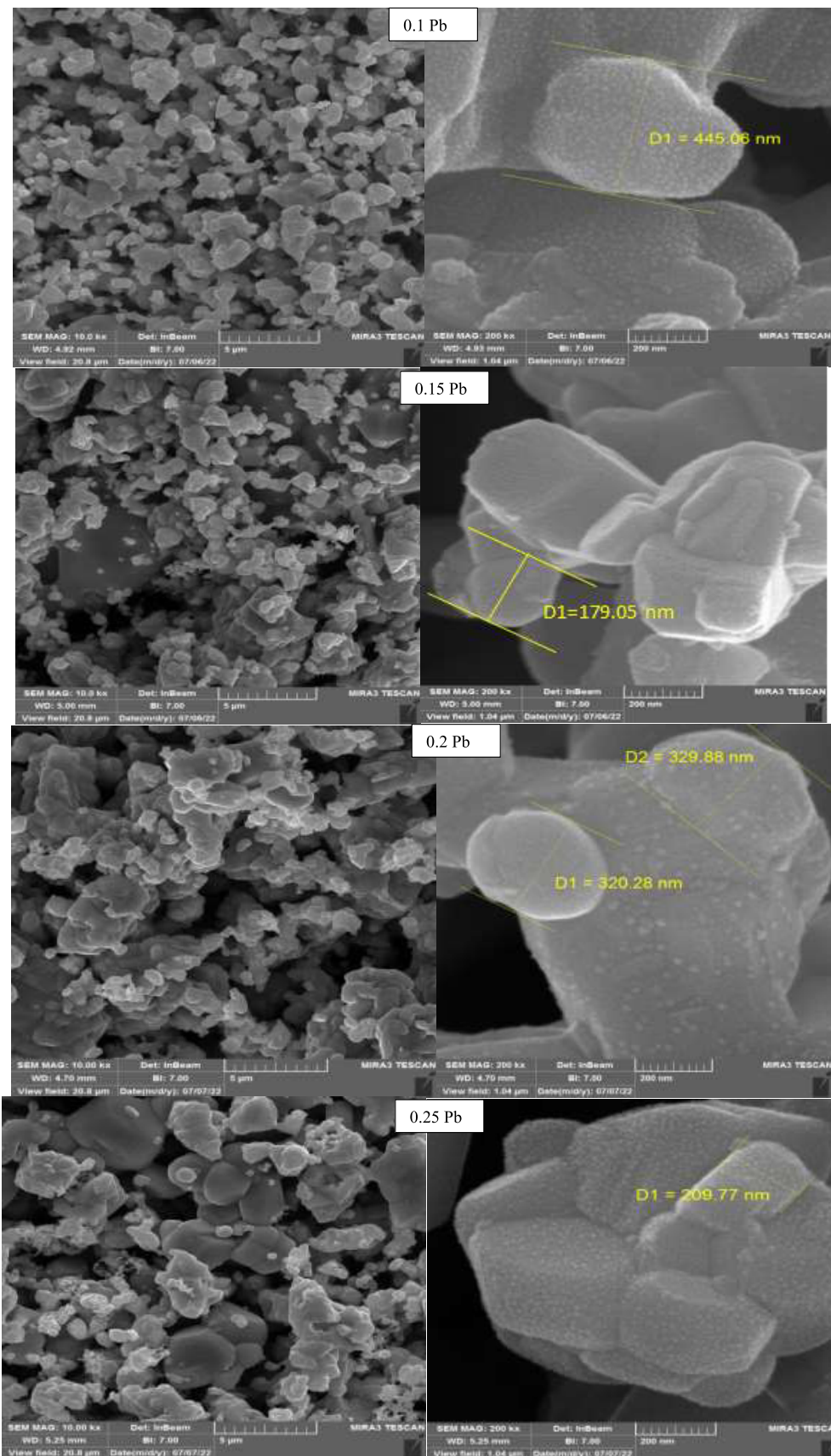


Fig. 4. Continued.

period is to adding more copper oxide (CuO) layers to the composite stratum.

Acknowledgment

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Author's declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Besides, the Figures and Images, which are not ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Kufa.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.

Authors' contribution statement

This work was carried out by the researcher E. H. and under the supervision and follow-up of H. M.J. H. in addition to conducting laboratory tests, analyzing and discussing the results, and reaching the final results of this work.

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تحضير المركب فائق التوصيل ($Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta}$) ودراسة تأثير الاستبدال الجزئي على الخصائص التركيبية

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الخلاصة

الغرض من هذه الدراسة معرفة نسبة الاستبدال المثلى للرصاص مع الايتيريوم من خلال الخصائص التركيبية للمركبات المحضرة. تم تحضير المركب فائق التوصيل ($Y_{1-x}Pb_xBa_2Cu_{2.85}La_{0.15}O_{6+\delta}$) بطريقة تفاعل الحالة الصلبة (SSR). تمت دراسة تأثير الاستبدال الجزئي للرصاص على الخصائص التركيبية للمركب بنسب استبدال ($x = 0, 0.05, 0.1, 0.15, 0.20, 0.25$). بضغط 7 طن لكل سنتيمتر مربع، تم تشكيل المسحوق المتكون على شكل قرص بسمك (حوالي 0.2 سم). تم تلييد العينات لمدة (24 ساعة) بمعدل تغير من درجة حرارة الغرفة (27 درجة مئوية) إلى (850 درجة مئوية). بالإعتماد على تحليل نتائج فحص حيود الأشعة السينية (XRD)، تم اكتشاف أن جميع العينات تحتوي على بنية orthorhombic. ثبت أن التغيير في تراكيز الرصاص في كل عينة من عيناتنا تؤدي إلى تغير في مؤثرات الشبكة، (a, b, c), T_c , d_m , و فجوة الطاقة. حيث تم تسجيل أفضل قيم لها عند التركيز ($x = 0.1$) حيث كانت قيم ثوابت الشبكة ب وحدة الأنجستروم (a, b, c) هي (3.807346, 3.886549, 11.70871) على التوالي، أعلى قيمة ل (T_c) هي (112.1)، قيمة الكثافة (d_m) هي (6.42604 gm/cm^3)، كما كانت أعلى نسبة ل ($c/a = 3.075295$) وأعلى فجوة طاقة هي ($E_g = 0.03384 \text{ eV}$) لذلك يمكننا اعتبارها نسبة الاستبدال الأمثل ضمن ظروف هذا العمل. كما تم استخدام الفحص المجهر الإلكتروني لتحليل الصور المجهرية للمواد بتكبير عالٍ جدًا (SEM) و بقوة تكبير ($5\mu\text{m}, 200\text{nm}$).

الكلمات المفتاحية: الاستبدال الجزئي، الفحص المجهر الإلكتروني (SEM)، طريقة تفاعل الحالة الصلبة (SSR)، فائق التوصيل، حيود الأشعة السينية (XRD).