

THE EFFECT OF PARTIAL SUBSTITUTION OF LEAD (Pb) ON THE STRUCTURAL AND ELECTRICAL PROPERTIES OF HIGH TEMPERATURE SUPERCONDUCTIVITY SYSTEM (BSCCO)

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Abstract

The partial substitution of Pb was carried out in the superconducting system BSCCO with the formula $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ where ($x=0.1,0.2,0.3,0.4,0.5$), the samples were prepared by solid-state reaction with annealing temperature of 800 °C and at a pressure of 7 ton/cm², four probes method was used to study the electrical properties and determine the critical temperature and the ratio of the high and low phase and the lattice constants of the samples were determined using X-ray diffraction (XRD), for the purpose of studying the structural properties, atomic force microscopy (AFM) was used, as well as to know the surface roughness ratio and the average grain diameters. It was found that the best substitution ratio is $x = 0.4$, where a critical temperature of 120 K was obtained, as well as the best surface and structural properties.

Keywords: Superconductivity , Critical Temperature , X-ray diffraction , AFM , BSCCO

Introduction

Electrical resistance is lost when some materials are cooled to a low temperature, this phenomenon is known as superconductivity, as an electric current passes through it without the presence of electrical resistance [1]. In general, superconducting materials can be divided into two types, the first type is called (low-temperature superconducting materials), which need very low cooling using liquid helium (4.2K), and the second type is called (high-temperature superconducting materials). which was discovered in 1987 [2] ,The superconducting system BSCCO, which consists of (bismuth oxide, strontium oxide, calcium oxide, and copper oxide), is one of the most important

superconducting systems at high temperatures, as it has a high critical transition temperature, especially at the (Bi-2223) phase [3], this system is characterized by being environmentally friendly as well as water-resistant, the purity of its elements, its tolerance to high magnetic fields, and the ease of using crystal growth techniques in its preparation. The general formula of the BSCCO system is $\text{Bi}_2\text{Sr}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{2n+6}$ where n is a positive integer ($n=0,1,2,3$) and this system has layers of CuO_2 and each atom is separated by a calcium atom Ca and CuO_2 atoms are linked by layers other separators of BiO and SrO and as shown in Figure 1 [4,5]. In view of the possibility of improving the properties of this system and controlling it by adding or substituting elements that differ from its main elements in the ionic radius, so lead was chosen for partial Substitution instead of bismuth.

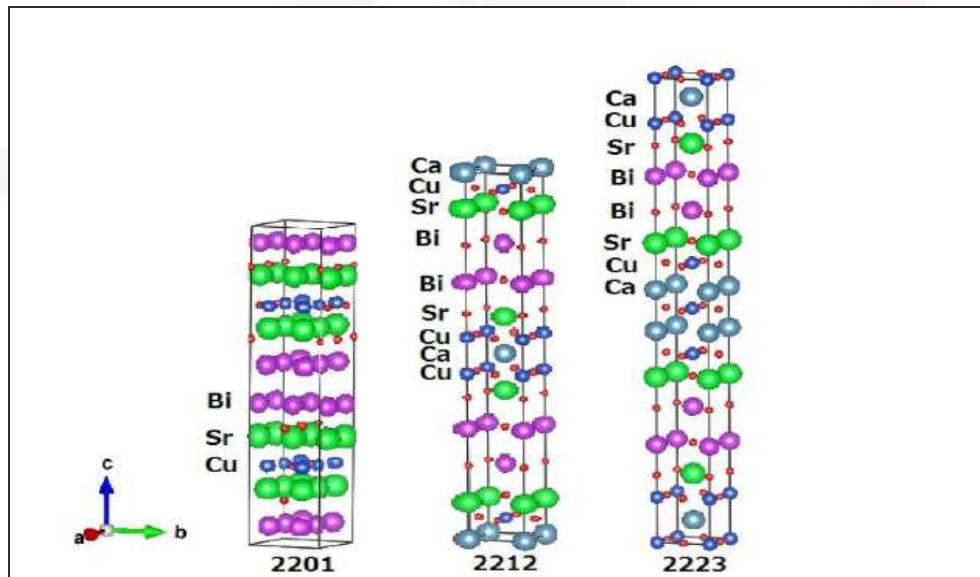


Figure 1 crystal structure of the three phases of the BSCCO system [5].

Practical part (preparation of materials and method of work):

The compound $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ was prepared by solid-state reaction using pure oxides and carbonates, which are (Bi_2O_3 , SrO , CaCO_3 , CuO , PbO), the samples were crushed and mixed using a mortar made of agate, during the grinding process, isopropanol $\text{C}_3\text{H}_8\text{O}$ was added to prevent loss and volatilization of the powder, then it was placed in an oven at a temperature of 70°C to get rid of the remaining isopropanol. The second stage includes the sintering process, where the process begins after drying the powder of each sample, then the samples are placed inside a ceramic boat, then the boats is placed in a thermal oven, then the temperature is raised to 800°C at a rate of $5^\circ\text{C} / \text{min}$ for 24 hours,

after which the temperature is gradually reduced and at the same rate 5 °C/min until it reaches room temperature, as shown in Figure 2.

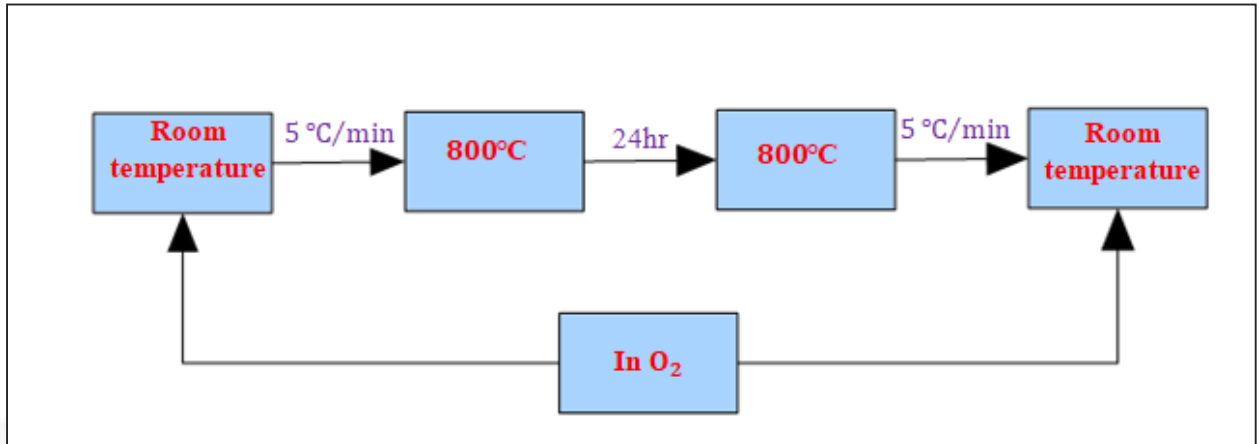


Figure 2. sintering process of samples

The third stage included the annealing process, the hardened samples are ground using a mortar with the addition of isopropanol alcohol and then placed in an electric oven for half an hour at a temperature of 70 °C, then pressed under pressure of 7 ton/cm² and then placed in a thermal oven and the temperature is raised to 600 °C at a rate of 5 °C / min and remains for 12 hours, after which the temperature is raised again to 800 °C at a rate of 5 °C/min and the samples remain for 24 hours, then the temperature is reduced to 600 °C at a rate of 5 °C/min for 12 hours and as shown in Figure 3, the samples were coded according to substitution ratios (S1,S2,S3,S4,S5) Then , X-ray diffraction and atomic force microscopy (AFM) tests were performed, and the four probes method was used to study the electrical properties.

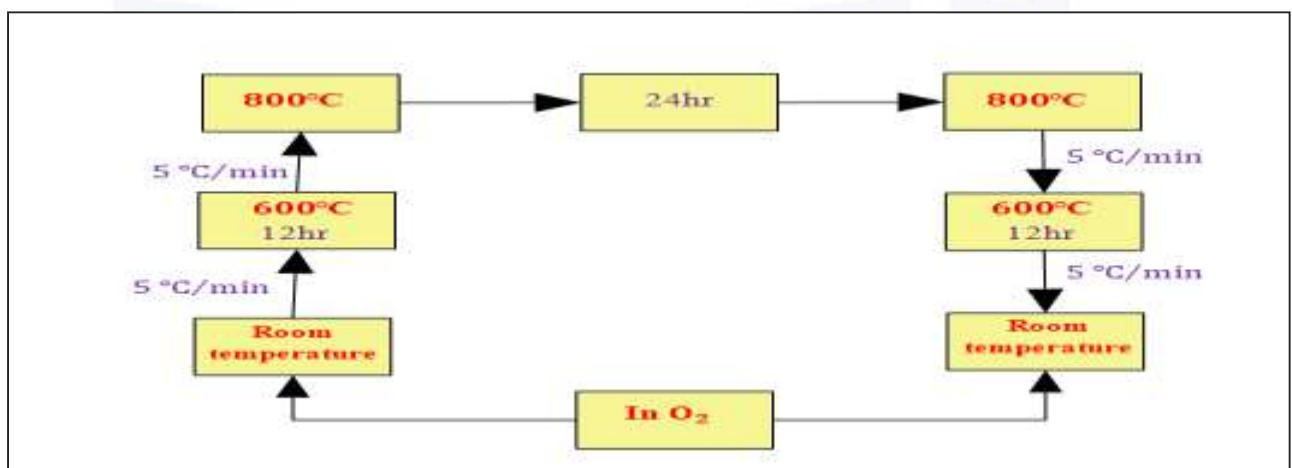


Figure 3 . Samples annealing process



Results and Discussion

1- Results of Electrical Tests

Figure 4 shows the behaviour of the resistivity as a function of temperature with different values of x , it is noted that all the models behaved like a metal, meaning that their electrical resistance decreases with a decrease in their temperature in the region preceding $T_{C(\text{onset})}$ where material changes from the normal state to the state of superconductivity, also an increase in the critical transition temperature $T_{C(\text{offset})}$ was observed with an increase in the percentage of lead for samples (S1,S2,S3,S4), and the reason for this is that the lead compensation had a positive effect in forming the high phase, which made the critical temperature be higher than the sample (S) while its low value is noted for the sample (S5), which may be attributed to the fact that the replacement ratio was not ideal, which caused distortions in the composition, which in turn facilitated the formation of impurities, which led to a decrease in the critical transmission degree for it [6,7]. From Table 1, we notice an increase in the transition width (ΔT), and this can be explained by the increase in the high phase Bi-2223 and that the process of replacing the lead worked to increase the efficiency of the high phase and increase the nucleation sites, and the increase in (ΔT) may be attributed to the fact that the partial replacement of lead with Bismuth may create additional gaps in the copper oxide layers, and these gaps have a role in the survival and continuity of the electron pair, and it can also be said that the ideal sintering process has a role in increasing the contact area between the grains and forming a state of strong bonding, and as a result a decrease in porosity [8]. The critical temperature of the pure sample was 114.5 K, and this result is close to what was found by both researchers (Sedky, Ali) [9,10], As for the sample S3, the critical temperature was 119 K, i.e. for the compound with the formula $(\text{Bi}_{1.7}\text{Pb}_{0.3})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ and this result is consistent with what was stated by the researcher (Amna Salah) and others [11]. The highest value of the critical temperature was obtained at the compensation ratio $x = 0.4$, which amounted to 120.4 K. Figure 5 shows the critical temperature change with different lead concentrations.

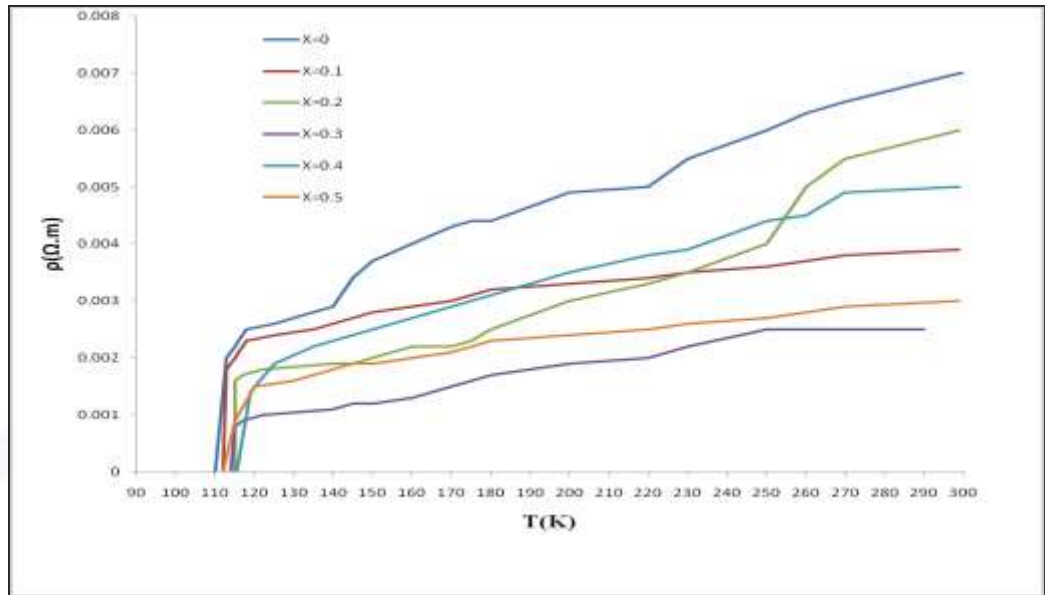


Figure 4 Resistivity change as a function of critical temperature of $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ samples.

Table 1 The critical temperature change of the samples and the different substitution ratios of the samples.

| Symbol | x | $T_{C(\text{onset})}$ K | $T_{C(\text{offset})}$ K | ΔT K | T_c K |
|--------|-----|----------------------------|-----------------------------|-----------------|------------|
| S | 0 | 118.2 | 110.9 | 7.3 | 114.55 |
| S1 | 0.1 | 119.7 | 112 | 7.7 | 115.85 |
| S2 | 0.2 | 122.3 | 114.1 | 8.2 | 118.2 |
| S3 | 0.3 | 123.5 | 114.7 | 8.8 | 119.1 |
| S4 | 0.4 | 125.3 | 115.5 | 9.8 | 120.4 |
| S5 | 0.5 | 120 | 112 | 8 | 116 |

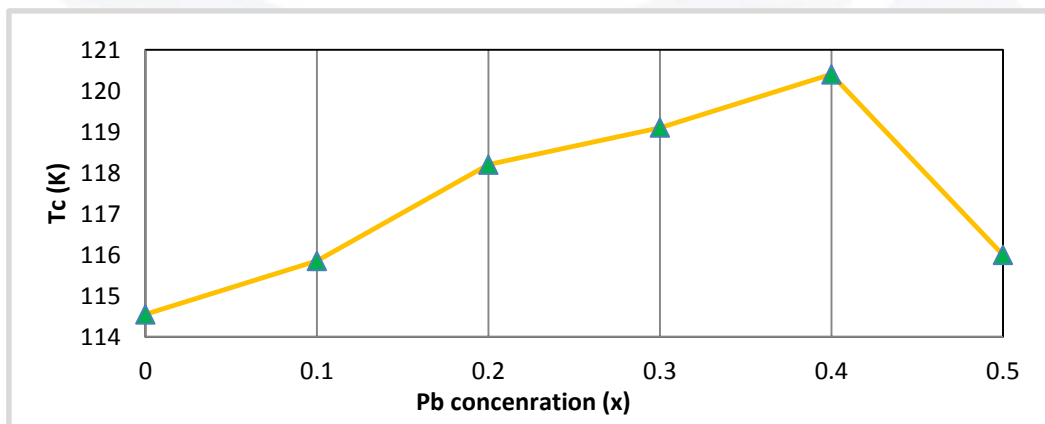


Figure 5 Behavior of critical temperature as a function of lead-compensated ratios of $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$

2- X-ray Diffraction

The results showed, as shown in Figure $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ 6, the emergence of new peaks with an increase in the value of the compensation x , which is evidence of an increase in the regularity in the crystal structure represented by the partial substitution of Pb instead of Bismuth Bi, and all samples have a tetragonal crystal structure. It is also noted that an increase in the Bi-2223 phase occurred when $x = 0.1$ up to $x = 0.4$, which is evidence of an increase in the rate of formation of the high phase and a decrease in the peaks of other low phases, and consequently a change in the lattice constants and the ratio c/a , as shown in Table 2, as the presence of lead had a direct effect on the formation of the high phase Bi-2223 and this may be attributed to the fact that the ionic diameter of lead (1.19 \AA) Pb is greater than the ionic diameter of bismuth (1.03 \AA), which makes the lattice constant c longer, it is also possible to explain this to the fact that the compound took the optimal composition and the best uniformity, as well as the length of the sintering period required for stability, and these results are close to what was reached by A. Maqsood) (S.M. Khalil) [127,128], and for $x = 0.5$, a deficiency was observed in the high phase and in the length of the constant c . This can be explained by the fact that the lead atoms took interlayer positions instead of being compensated and became among the crystal defects, and therefore the replacement ratio $x = 0.5$ would have a negative impact on the formation of The higher phase and this was clearly reflected in the higher phase ratio shown in Figure 7.

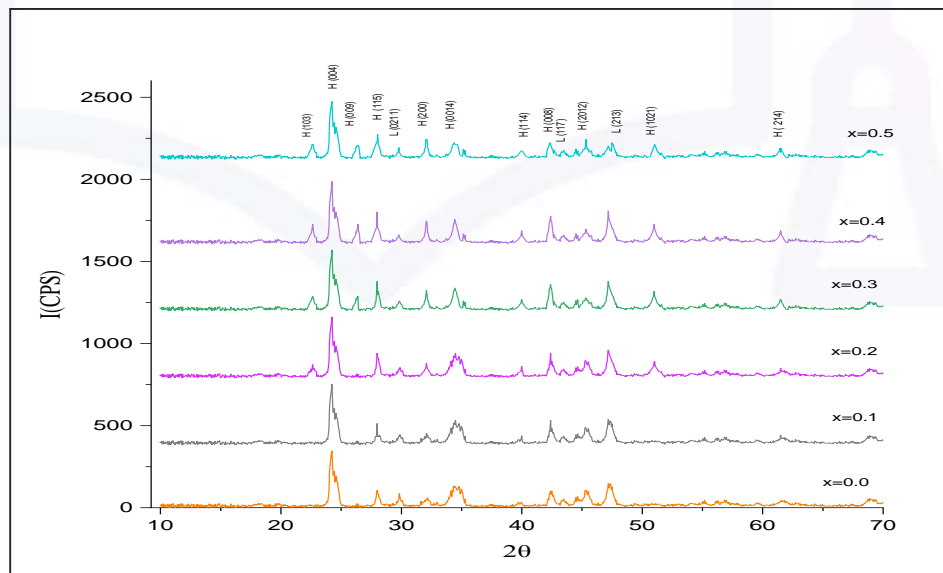


Figure 6 X-ray diffraction diagram of $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$.



Table 2 values of the lattice constants (a,b,c), the ratio of c/a and the ratio of the high phase of the compound $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ with different values of x.

| Symbol | x | a (Å) | b (Å) | c (Å) | c/a | V _{ph(2223)} % | V _{ph(2212)} % |
|--------|-----|--------|--------|---------|--------|-------------------------|-------------------------|
| S | 0 | 5.4206 | 5.4197 | 37.1995 | 6.8626 | 79.725 | 8.2 |
| S1 | 0.1 | 5.4212 | 5.4208 | 37.2031 | 6.8625 | 79.921 | 7.29 |
| S2 | 0.2 | 5.4253 | 5.4247 | 37.2614 | 6.8681 | 81.273 | 7.01 |
| S3 | 0.3 | 5.4260 | 5.4253 | 37.2615 | 6.8672 | 82.107 | 6.82 |
| S4 | 0.4 | 5.4530 | 5.4526 | 37.5701 | 6.8898 | 83.093 | 5.308 |
| S5 | 0.5 | 5.4871 | 5.4866 | 37.5179 | 6.8375 | 80.131 | 6.2 |

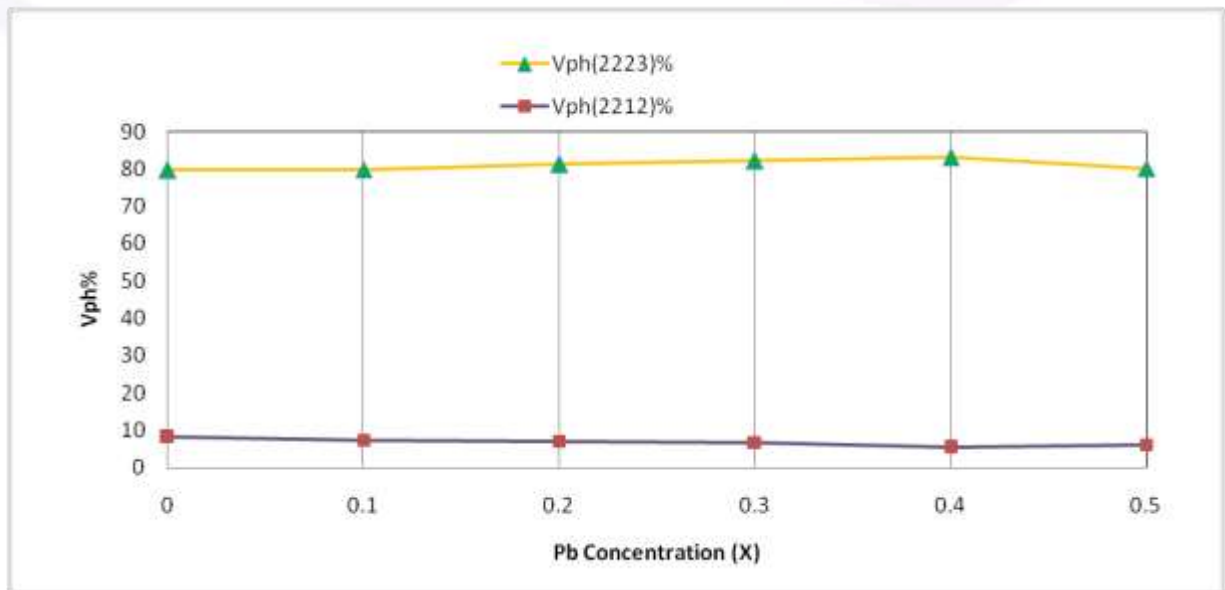


Figure 7 Ratio of the high and low phases of $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ with different x values.

3- Atomic Force Microscopy(AFM) Results:

The surface topography of the samples was studied using atomic force microscopy, where it was observed that the roughness and radius of the grains depended on the concentrations of lead replaced by bismuth, as shown in Figure 8. . Table 3 shows the average roughness of the samples, which indicates that the samples have crystal regularity, and the smallness of these values indicates the smoothness of the surface, which corresponds to the smallness of the grains,

because their smallness means homogeneity with high density. The results match the results of X-ray diffraction and electrical properties.

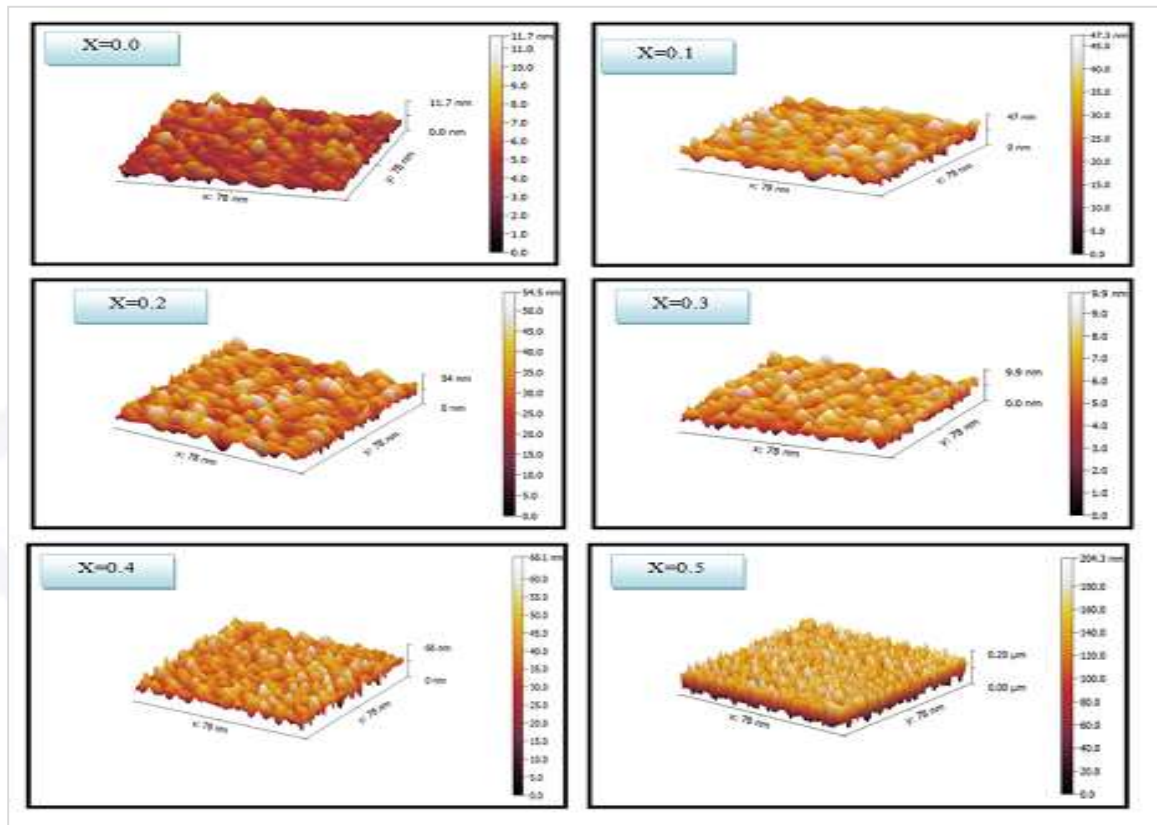


Figure 8 Atomic force microscopy (AFM) images of $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ with different x values

Table 3 The change in the roughness rate S_a and the grain diameter rate for the compound $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ with different values of x

| Symbol | x | S_a (nm) | Avg. diameter (nm) |
|--------|-----|------------|--------------------|
| S | 0 | 1.07 | 100.20 |
| S1 | 0.1 | 1.18 | 97.24 |
| S2 | 0.2 | 1.26 | 88.40 |
| S3 | 0.3 | 0.27 | 81.01 |
| S4 | 0.4 | 0.54 | 76.19 |
| S5 | 0.5 | 0.92 | 78.09 |

Conclusions

The process of preparing the compound $\text{Bi}_{2-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ was carried out with the ratios $x=0.1,0.2,0.3,0.4,0.5$, the critical temperature was calculated using the four point probe method, and it was found that the highest temperature was 120.4K At the compensation ratio $x = 0.4$ and the minimum critical

temperature was 116 at the compensation ratio $x = 0.5$. The results of X-ray diffraction showed that all the samples that were prepared have tetragonal crystal structure and contain high percentages of the high phase Bi-2223 with few percentages of the low phases Bi -2201 and some impurities and the highest percentage of high phase Bi-2223 amounted to 83.093% . The results of the atomic force microscope (AFM) showed the change in the diameters of the grains and the roughness rate with the change in the percentage of impurities and that all samples were highly homogeneous.

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