# THE INFLUENCE OF PB ,AL DOPED ON THE HIGH-TEMPERATURE SUPERCONDUCTOR (BI2-SR2-CA2-CU3-O10+Δ)

**Dr.widad mahmood faisal**

*Hadramout university of science &Tecchnology- Faculty of Engenering&Petroleum Department of Electronic &Communication*

## Abstract

A ceramic superconductor compounds with the composition $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ were prepared alone or substituted by pb doped($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ ) samples with different values of x =(0.1,0.35,0.4), and Al doped($Bi_2$ $Al_xSr_2Ca_2Cu_3O_{10+\delta}$ ) samples with different values of x=(0.1,0.2,0.3) .The solid state reaction method was used for samples preparation . From the principle routs like Bi-2223,$Ca_2CO_3$ and CuO with high purity 99.999% . Superconducting phase was achieved by sintering (860°C) for (16 hours) .The slow cooling rate (1°C/min).was very important to the formation of superconducting time .The Bi-based system was multi phases . Different measurement such as resistivity measurement , X-ray diffraction(XRD) and Scanning Electron Microscope(SEM) were made to show the improvement in the superconductivity of the high phase. The crystal structures of stable superconducting compound of $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ was orthorhombic with lattice constant about a=5.394°A,b=5.346°A,c=30.753°A for low Tc phase and a=5.394°A,b=5.311°A,c=37.182°A for high phase .The critical temperatures (Tc) of the HTSC $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ were obtained to be Tc value for the un doped sample and for Pb, Al doped systems respectively and Scanning Electron Microscope(SEM) images showed formation plate-like or flake –like texture phases in superconducting samples.

Keywords: (HTSC) High-Temperature Superconductor ($Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$)

## 1-Introduction

Since the discovery of the Bi-Sr-Ca-Cu-O superconducting compounds [1] a great deal of work has been done to prepare the high –Tc phase .The most common method used to synthesize these superconducting oxides is the solid state reaction method[1,2] which consists of the mixing nominal compositions of high purity compounds such as $Bi_2O_3$,$SrCO_3$,$CaCO_3$ and CuO, pelletization of calcined powder , under a pressure of 4-6 tons /cm$^2$ and heating the mixed powders for 10-20 h at 800-820 °C. The formation of single high-Tc Bi-2223 phase however is very difficult due to the appearance of other superconducting phases such as Bi-2212, or non superconducting phases. The most effective methods to enhance the volume fraction of the high-Tc phase was found to be:

1-By starting from composition with surplus Ca and Cu than the possible ideal composition of $Bi_2Sr_2Ca_2Cu_3O_x$ [2]; by prolong the sintering [3] which is necessary to

*The University Researcher Journal, Issue No. (28), pp. 41-58,January- March,, 2012 © The University of Ibb, Yemen, 2012*

form extra layers of Cu-$O_2$ and Ca planes into the layer structure of the low –Tc phase.
2-The annealing in an atmosphere with low oxygen pressure [3,4] which is effective in lowering the reaction temperature , and enhancing the stability of the high – Tc.
3-The substitution of Pb for Bi [5,6] or the addition of Pb in the $Bi_2Sr_2Ca_2Cu_3O_x$ [7] is very effective in increasing the high –Tc phase . It was found that Pb has a catalytic effect on the reaction to from the high –Tc phase, and a certain amount (about 0.3mole) of Pb to the $Bi_2Sr_2Ca_2Cu_3O_x$ compound is necessary for the occurrence of this reaction [6] .Furthermore the Pb has a role in the stabilization of the high-Tc phase[(5].
Uehara et al [7] studied the magnetic measurement of $BiSrCaCu_2O_x$, and they estimated the high- Tc volume fraction to be 21%. Kumakura et al [8]investigated the upper critical field of $BiSrCaCu_2O_x$,with Tc above 100K. They found that at zero fields, the majority of transition occurred above 103K and a zero resistivity was attained below 80K .But as the magnetic field increased, the low temperature portion of transition curve shifts to low temperature ,resulting in abroad transition. Matsuoka et al [9] investigated the effect of In addition on the superconducting behavior of Bi-Sr-Ca-Cu-Pb-O system, and found no effect on the critical temperature ,but is effective in increasing the volume fraction of the $Bi_2Sr_2Ca_2Cu_3O_{10}$ compound.
Wu et al [10] investigated the effect of sintering temperature and duration on the formation of the 110K phase ,in samples of nominal compositions of $(Bi_{3.2}Pb_{0.3})Sr_4Ca_xCu_yO_z$ .They observed that the optimum compositional rang for obtaining large fraction of high –Tc phase (Tc=105K) occurs between PbBi-4457 and PbBi-4468.
The Bi:2223 phase has attracted considerable interest due to its higher Tc and the potential for applications. One of the reasons for the low critical current densities $J_c$ is the granular nature of this sintered HTS compound. It is difficult to prepare single phase material for Bi:2223 phase. The Bi:2212 was frequently observed as a major impurity phase in Bi:2223 samples because of its greater thermodynamic stability with respect to the Bi:2223 phase. The partial substitution of Pb in Bi sites enhances chemical stability and promotes the formation of the 2223 phase [11].
Kovaleva et al [12] presented a systematic study of the c-axis lattice dynamics in $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n}$ (n=1,2,3) cuprate superconductors ( Bi2201,Bi2212,and Bi 2223) based on spectral ellipsometry investigation on single crystal and he compared them with theoretical shell model calculation.
The observation of the multilayer band splitting in the optimally doped trilayer cuprate $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (Bi2223) by angle-resolved photoemission spectroscopy is reported by Ideta et al [13].They obtained values of energy gaps 43( m eV) and 60( m eV) larger than those for the same doping level of the double-layer cuprates, which leads to the large value of Tc in Bi2223.

## 2-Experimental

### 2-1 Materials

The samples were prepared by the solid state reaction method using highly pure 99.999% powders of $Bi_2O_3$,$SrCO_3$, $CaCO_3$,CuO , with appropriate weights in proportion to their molecular weights through the following chemical reaction. Three types of superconductors systems were obtained, namely $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ , $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$,$Bi_{2-x}Al_xSr_2Ca_2Cu_3O_{10+\delta}$,. The preparation of ceramic superconductor by solid state reaction includes a series of stages as shown in the following : Measuring the weight of each reactants with the required amount , using sensitive balance with (4-digits), type (STATON)462AL.

## 2-2 The chemical reactions

The systems were prepared by mixing appropriate amount of the sating_materials through the following chemical reactions:

1-The first system ( $Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$) with 2223 phase was prepared by mixing appropriate amounts of the starting materials through the following reaction Scheme (14 )

$$(BiO)_2\ Co_3+2SrCO_3+2\ CaO+3CuO \rightarrow Bi_2Sr_2Ca_2Cu3O_{10}+2CO_2$$

2-The second system (i.e., $Bi_2$-$Pb_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ ) was prepared by mixing appropriate amounts of the starting materials through the following reaction:

$$(1-\frac{x}{2})\ (BiO)_2\ Co_3+xPbO+2SrCO_3+2\ CaO+3CuO \rightarrow Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10}+(3-0.05)\ CO_2$$

3-The third system ( $Bi_2$-$Al_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ )was prepared by mixing appropriate amounts of the starting materials through the following reaction:

$$(BiO)_2\ Co_3+0.5Al_2O_3+2SrCO_3+2\ CaO+3CuO \rightarrow Bi_2Al_xSr_2Ca_2Cu_3O_{10}+(3-0.05)\ CO_2$$

## 2-3 Sample preparation

The mixture for each specimen was prepared by homogeneously mixing and grinding prescribed amounts of powders into a gate mortar. Appropriate amounts of these powders were mixed with alumna mortar and pestle for (2hours) in 2-propanole and dried. The calcinations process performed at (810°C) for (16 hours) by heating rate of 30°C/h ,then cooled to room temperature ,with a rate of 30°C/h in order to remove the $CO_2$ from the mixture as gas and then crushed into fine powder . The calcinations and grinding procedure were repeated three times at least. That assured the complete evolving of $CO_2$ from the mixture. The resulting powder then was pressed into pellets by using cylindrical and the die has a stainless steel cylinder of (13mm)diameter and (1.5-1.8)mm thick using manually hydraulic press PERKIN-ELMER, under a pressure of (0.5GPa).The mixture with the final step of sample preparation was high temperature treatment that causes particles of the materials to join together and gradually reduce the volume of pore space between them, the powder is compacted into a pellet shape with a certain pressure then powder particles will be in contact with one another at numerous sites, with significant amount of pore space between the particles .

In order to reduce the boundary energy , atoms are diffuseed to the boundaries, permitting the particles to be bound together and eventually causing the pores to shrink . If sintering is carried out for a long time the pores may be eliminated and the material becomes dense.

The programming data for this process include, the rate of heating (60°C/h) up to (860°C) for (16 hours) with the flow of oxygen gas of about (1.25)L/min and then with slow rate of cooling (1°C/h)down to room temperature reached . Resintering the pellets for up to (860°C) for (16 hours) using the same method as above .

## 2-4 Doping process

The samples of ($Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ ) , ( $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ for x=0.1,0.35,0.4). and ( $Bi_2Al_xSr_2Ca_2Cu_3O_{10+\delta}$ ,for x=0.1,0.2,0.8).At the end of the weight of the powder was measured .The mixture of the powder was grinded in agate mortar for 45 min with the presence of a suitable amount of 2- propanol .Then it was dried in the oven for an hour at temperature of 60°C, after the powder was pressed into pellets as explained above and sintered at a temperature of 860°C for 16 hours.

## 2-5 Sample testing

The critical temperature (Tc) of the superconducting sample has been measured by using the resistivity measurement carried out by four – probe technique, which is considered as a good method for studying the electrical behavior of superconducting materials and a good tool for determining the critical temperature, although the last sample was measured roughly by Meissner effect .In this method, a small current is passed through a sample and the voltage drop across it. The terminals distinct from those used for passing the main part of the current through the specimen, where voltage drop in both leads and contacts are the electrical contact to the sample were made with fine copper wires, adhered with silver paste. The cryostat system was used for the measurement of critical resistivity of the sample, with the presentence of liquid Nitrogen. The cryostat was joined to a rotary pump to get a pressure of ($\sim10^{-2}$ ) mbar inside the cryostat. The oxygen content was determined by using a simple chemical method called" Iodometric titration " . The structure of the samples was obtained by using X-ray for grinded pieces of the samples adhered to glass substance .The x-ray diffraction(XRD) type PHELIPS has been used with the following features (Source CuKα , Cu current 20mA, Voltage 40kV, wavelength 1.5405A° Scanning speed 2θ/min).A computational program has been used to find the lattice parameter of the unit cell from the pattern of XRD .Fine powder were obtained by grinding the pieces of the samples, then adhered to glass substrate and examined by the X-ray diffract meter. Scanning electron microscope (SEM) type JEOL JSM 6400 , has been used to study the surface morphology and grain size of the samples for the composition ($Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$, $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ ,$Bi_2Al_xSr_2Ca_2Cu_3O_{10+\delta}$) .

## 3 -Results and Discussion

X-ray diffraction patterns of $Bi_2Sr_2Ca_2Cu_3O_{10}$ compounds in figure (1) show that two phases with lattice parameter constant a= 5.394 °A, b=5.3462 °A and c=30.753 °A for low phase (90°K) whereas a= 5.394 °A, b=5.311 °A and c=37.182 °A for high phase (110°K) . This is in agreement with the results of Sequeira (15) .

Figure (2)shows that the samples doped with Pb oxide with no change on the structure behavior . However , 2θ shifted to lower angle was observed . Besides an increase in peak intensity indicated that structure was stabilized but crystallization phase is more favored ,.Like other high Tc superconductors , the Bi-Sr-Ca-Cu-O system has a layer structure but with multiple planes . The structure are similar , differing only in the number of $CuO_2$-Ca-$CuO_2$ slabs packed along the c-axis . The crystal structure of 2223-phase consists of three Cu-O layers with double Bi-O layers. The two Sr cations between the Cu-O and Bi-O below and above layers , and two Ca cations between adjacent Cu-O layer which form a corner –shared square based $CuO_5$ pyramids of opposite vertices and the third layer (square planar)that is sandwiched between the first two.

The 2223-$Pb_{0.35}$ system has a different behavior that Tc(onset)unchanged (120°K)but have more sharp drop with transition width (10°K) as in figure (3),But the lattice parameter is unchanged due to the influence of deformation that is because in doped sample with Pb it is assume that Bi-O layers are more flat due to substitution of Pb that leads to gain strength against the deformation while the increase in Tc(zero)by(10°K)is due to the increase in the crystallization exhibited by an increase in intensity as in figure(2),

figure (4,5) show the system doped with Al , it states different influence with higher doping rate . At a higher doping rate (0.8Al)for 2223- Bi system that are deformed leads to the disappearance of the superconducting state and also the transition to semiconductor and the x-ray diffraction pattern shows decreased in c-axis to 33.379 °A . An explanation of the changes to the semiconductor –like may result from the localization of charge carries because of distortion in crystal structure formed by deformation. For low Al doping $Br_2$-$Ca_2$-$Cu_3$-$O_{10}$ , there is a decrease in the Tc( zero) from (80°K) to (60°K) and then there are sharper drops as in figure (8) which is indicated by an increase in peak intensities XRD pattern. In this work , this case is explained by the tendency for localization of carries , which are stimulated by disorder introduced as a result of deformation . This static defect is predominant in Cu-O and Bi-O layers because these layers are sensitive to producing the superconducting properties . The presence of Pb in the structure of Bi-2223 compound has a direct influence on increasing the high-$T_c$ phase ,which can be seen clearly in the X-ray diffraction pattern in figures 2and 3. In the first one the high- $T_c$ phase reflection (008,115,113,117)are created and the intensities of the reflection (0012,119,0011) are increased by the addition of Pb .At the same time low- $T_c$ phase reflections (008,113,115,117 and 0012) are reduced .,compared to the same reflections in figure (3) we conclude that $Pb^{+2}$ ions my occupy the Bi-positions[16] or the Ca positions[17] and the presence of Pb in Bi positions enhances the structure stability of the superconducting phases[18,19] . Our experiments also reveals that the addition of Pb lowers the optimum sintering temperature ,required to form the high – $T_c$ phase to about 848-850°C .This could be attributed to the presence of the $CaPbO_4$ phase in the system[20] ,which changes the kinetic process of the formation of the Bi-2223 high-$T_c$ phase .$CaPbO_4$ melts at 822°C° and thus induces a liquid phase below 850 °C and increases drastically , through a dissolution process , the diffusion of the reactive species ($Ca^{2+}$,$Cu^{2+}$) . The resistivity of the sample was measured as a function of temperature using standard four probe technique. For the preparation $Bi_2Sr_2Ca_2Cu_3O_{10}$ sample, the onset Tc is at 110 °K and the resistivity drops to zero at 90 °K.

Table ( 1) shows the resistivity(ρ) measurements vs temperature. In 2223-$Pb_{0.4}$ system , it is found that the influence of deformation on the Tc(onset) not on Tc (zero) which decreases Tc (onset)from (125°K) to (120°K). That refers to un stability in this phase with this rate of doping as in figure( 6 ). A computer program was used as a helpful tool for the determination of the cell parameters. X-ray diffraction patterns for different values of x . It can be noticed that the samples are a mixture of a major high -$T_c$ phase and minor low –$T_c$ phase. The intensity of the high-$T_c$ phase reflections is greater and the peaks are sharper than those of the low-$T_c$ phase .

Mizuno et al[18] reported that effective methods to increase the volume fraction of high-$T_c$ phase for Pb-Bi-Sr-Ca-Cu-O system are staring from nominal compositions with more Ca and Cu than in Bi-2223,and the addition of Pb to Bi-Sr-Ca-Cu-O system ,and annealing at 870°C under higher oxygen pressure than 0.2 atm. Chavira et al [20] confirmed that the volume fraction of high –$T_c$ phase is rapidly increased , when a small amount of Pb is incorporated in the Bi-Sr-Ca-Cu-O system .They showed that the XRD patterns reveals the possibility for Pb to occupy Ca or Bi sites. Syono etal[21] showed that the XRD patterns and scanning electron microscope images , of $Bi_3Sr_3Ca_2Cu_4O_y$ compound reveal a structure derived from the $Bi_4Ti_3O_{12}$ type in which double bismuth layers along the c-axis . The orthorhombic unit cell dimensions showed an approximate relation of a=√2ap, b=√2ap ,and c=8ap where ap =3.82A the parameter of a cubic perovskite and their appearance are similar to samples n=3 . The oxygen content was determined by idometric titration which is found in all samples for $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ , ($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ for x=0.1, 0.35, 0.4) and ($Bi_2$-$Al_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ for x=0.1, 0.2, 0.8) . Table (1) shows the oxygen content for all samples .the excess oxygen in (2212) phase may reside in the double Bi-O layers in the lattice and by inserting a Cu-$O_2$ la yer and Ca layer into(2212)phase for high Ts and for long sintering time.The (2223) phase is formed which is a high Tc- phase .The double Bi-O layers in the (2212,2223) phases may play the role of reservoir for holes for the Cu-$O_2$ layers similar to the role played by the Cu-O chains in the $YBa_2Cu_3O_{6.5+\delta}$(22). Finally , it was observed for the (2224)phase that Tc is increased with increasing oxygen content .For (2212)phase an opposite effect for the oxygen content was observed (22). Figure ( 7,8 ) shows the resistivity measurements vs temperature and values of Tc =112K,120K,125K and for the Pb- doped for x=0.1,0.35,0.4), and for the Al- doped for x=0.1,0.2,0.8).HTSC samples respectively. Surface Morphology, to compare the microstructure of pure and Pb, Al, doped-( $Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$)samples, micrographs were taken using JOEL JSM 4600 scanning electron microscope (SEM) operating at 15 kV. For better micro structural observations, the samples were polished, etched using a dilute HCl solution then coated with metal. Figure (9) shows the microstructure of pure $Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ sample and Figure (10) (1,2,3) show the microstructure of ($Bi_{1-x}Pb_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$) samples (0. 1, 0.35,0. 4)the samples doped Pb sintered 860 °C showed needle –like structure between the flake-like grains that happened when (2223-Bi) and Figures (11,12,13) show the microstructure of ($Bi_2$-$Al_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$) samples (0. 1,0,2,0. 8),slowly cooled in $O_2$ atmosphere after sintering at 860°C.The micrographs clearly show that the size of the plates shaped-particles are in the range

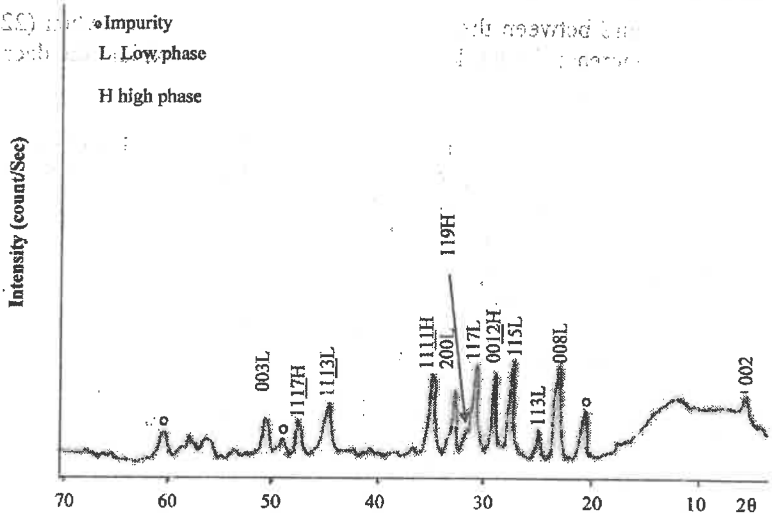
of 5-11 μ m. It was found that the grain growth of plate like-shaped increased rapidly with increasing Pb and Al substitution, reaching a maximum size at the interior region of the fracture surface. The samples doped Pb sintered 860 °C showed needle –like structure between the flake-like grains that happened when (2223-Bi) doped with Pb to increase the week link between grains that explain the decrease in resistivity of such samples.

## 4-Conclusions

In this paper a systematic study on the ($Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ , ($Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ for x=0.1, 0.35, 0.4) and ($Bi_2$-$Al_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ for x=0.1, 0.2, 0.8)) HTSC prepared by solid state reaction method is presented . The Bi-Sr-Ca-Cu-$O_y$ system consist of two phase(low Tc and high Tc phases)related to 2212 and 2223 phases .The Pb substitution of Bi-Sr-Ca-Cu-$O_y$ system increases the volume fraction of high Tc phase(2223) and we seen the increase of Pb concentration increases the value of Tc, but when doped Al with the (Bi-Sr-Ca-Cu-O) the best value of Tc when the x=0.8 but smallest value when x=0.2. The X-ray diffraction analysis of all samples showed orthorhombic structure. An increase in b-axis from 5.346 °A to 5.679°A play an important role to increase Tc of 2223-Bi system. Appearance of texture in SEM images for all samples under study such as plate–like or flake–like accompanies the formation of superconducting phase.

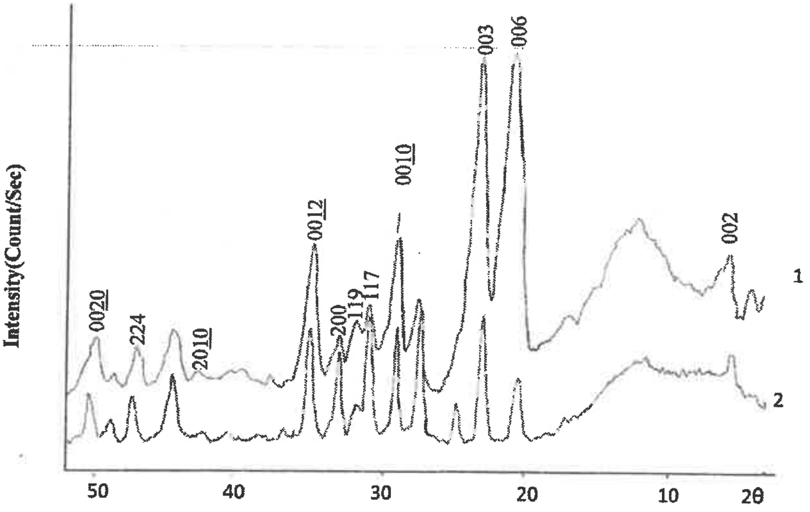
Table (1) the result of titration process as a Tc dependence

| Samples | δ (Oxygen Content) | Tc( °K)onset | Tc(°K)Offset |
|---|---|---|---|
| $Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.518 | 110-90 | 80 |
| $Bi_{1.9}$-$Pb_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.683 | 112 | 100 |
| $Bi_{1.65}$-$Pb_{0.35}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.511 | 120 | 100 |
| $Bi_{1.6}$-$Pb_{0.4}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.781 | 125 | 105 |
| $Bi_2$-$Al_{0.2}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.192 | 90 | 50 |
| $Bi_2$-$Al_{0.8}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ | 0.412 | 120 | 80 |
| $Bi_2$-$Al_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{8+\delta}$ | 0.329 | 115 | 60 |



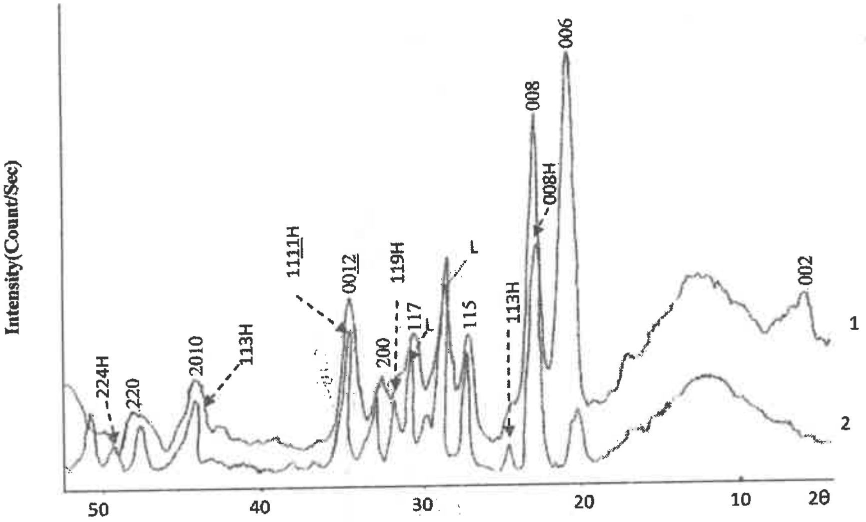
Figure(1) : the x-ray diffraction for the compound

($Bi_2$-$Sr_2$ –$Ca_2$-$Cu_3O_{10+\delta}$)



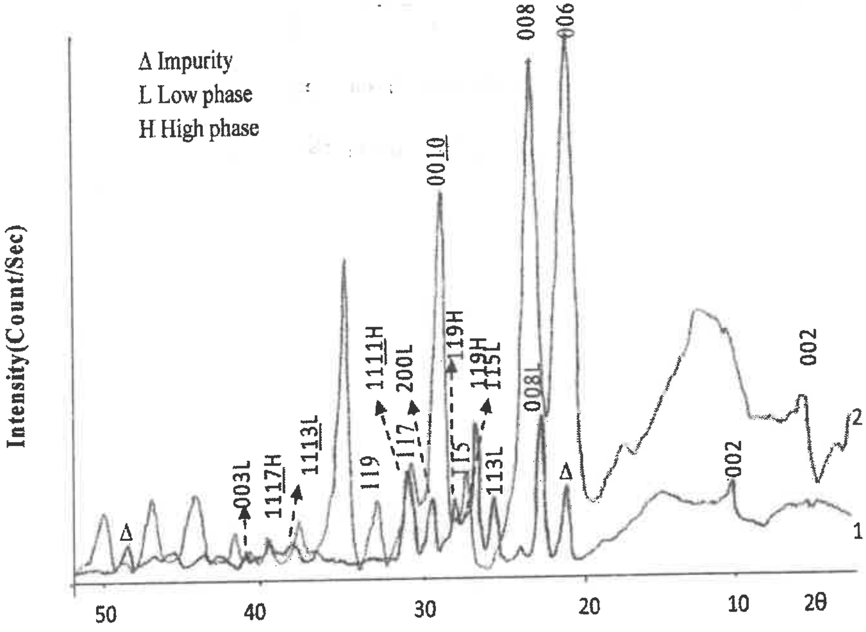
Figure(2):X-ray diffraction pattern for the compound

1-($Bi_{1.9}$-$Pb_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ ), 2-($Bi_2$- $Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ )



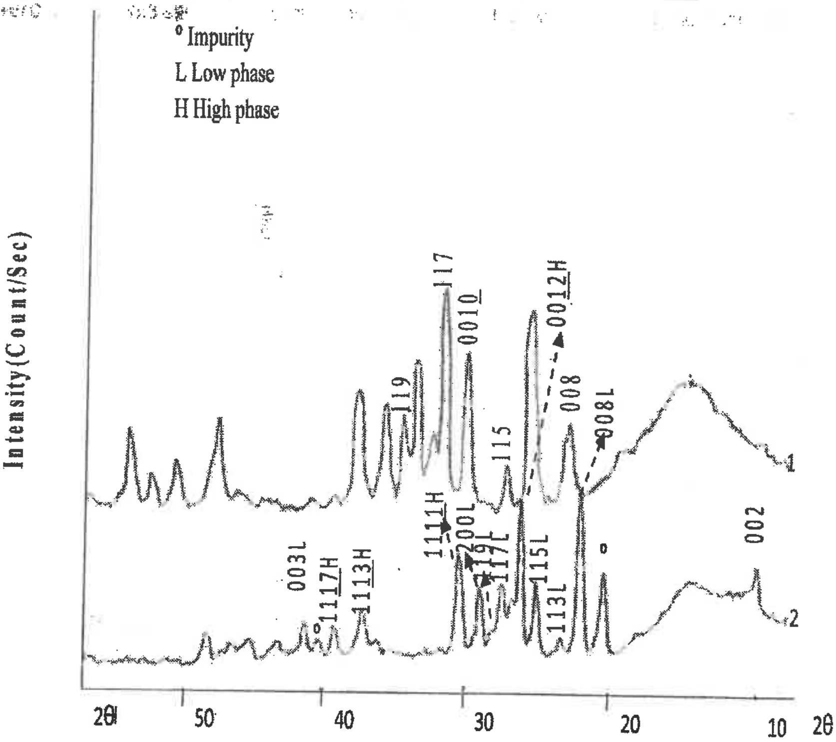
Figure(3):X-ray diffraction pattern for the compound

1-($Bi_{1.65}$-$Pb_{0.35}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ ), 2-( $Bi_{1.6}$-$Pb_{0.4}$- $Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ )



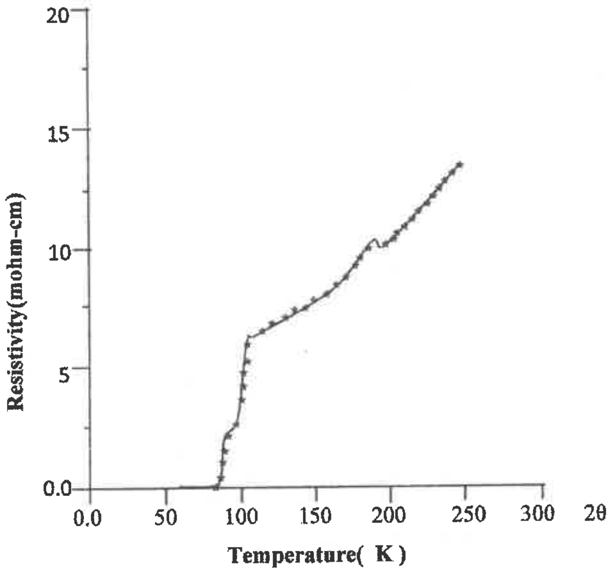
Figure(4 ) : the x-ray diffraction for the compound

1-($Bi_2$- $Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$),2-($Bi_2$-$Al_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$



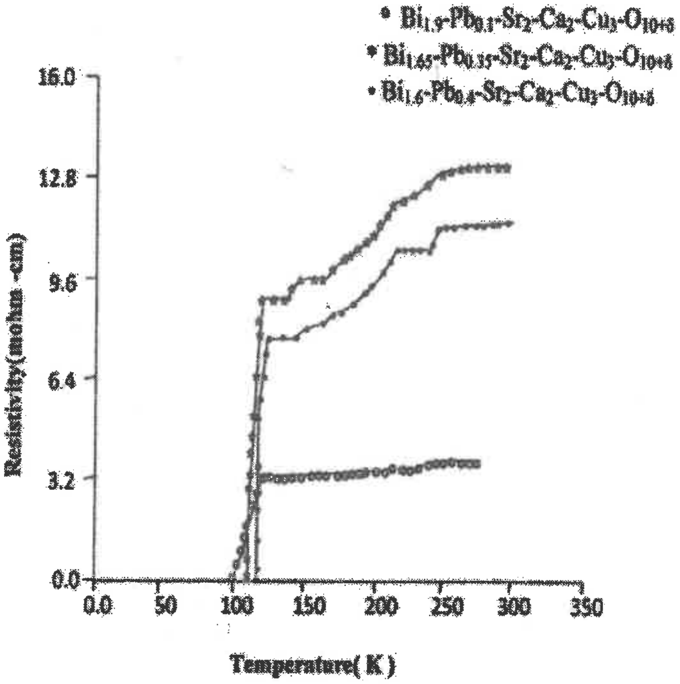
Figure(5) : the x-ray diffraction for the compound

1-($Bi_2$-$Al_{0.2}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$),2-($Bi_2$-$Al_{0.8}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$)



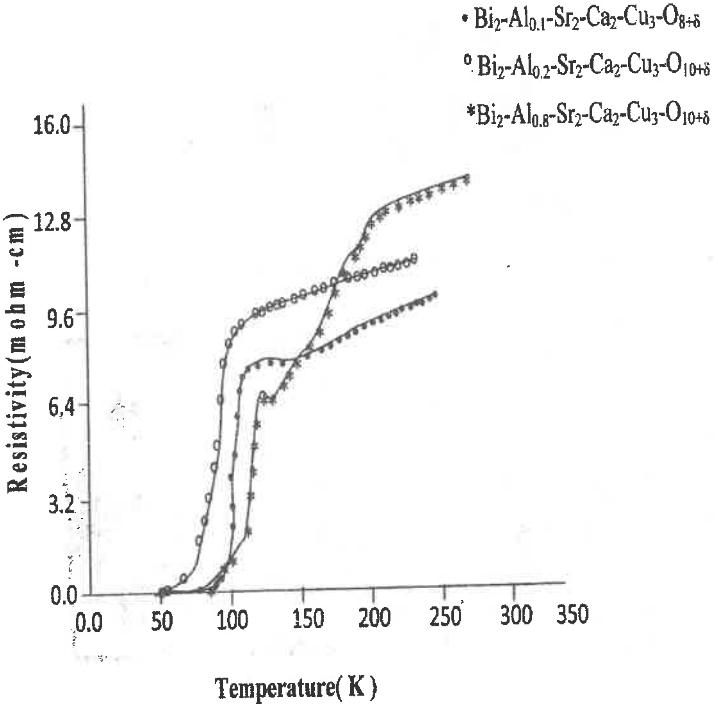
**Figure( 6 ) :The resistivity verus temperature for HTSC**

**$Bi_2$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$**



Figure( 7 ) :The resistivity verus temperature for HTSC

$Bi_{2-x}$-$Pb_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$



Figure( 8 ) :The resistivity verus temperature for HTSC

$Bi_2$ -$Al_x$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$

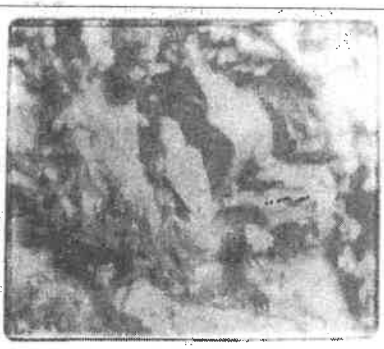
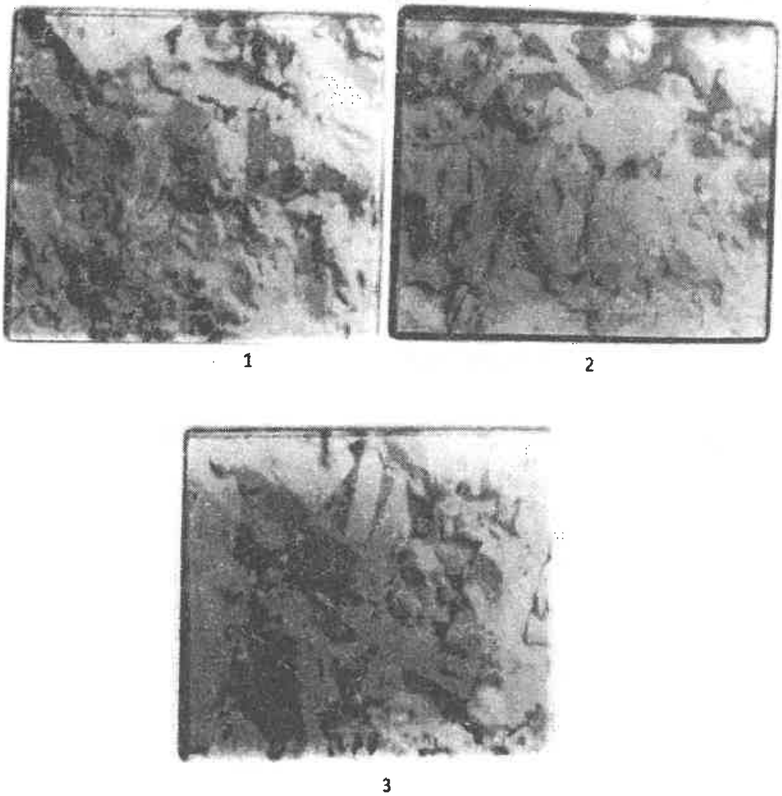


Figure ( 9 )morphology of fracture surface of compound $Bi_2$-$Sr_2$-$Ca_2Cu_3O_{10+\delta}$



1

2

3

Figure (10 )morphology of fracture surface of compounds ( 1- ($Bi_{1.9}$-$Pb_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$), 2-( $Bi_{1.65}$-$Pb_{0.35}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$ ),3-( $Bi_{1.6}$-$Pb_{0.4}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$))

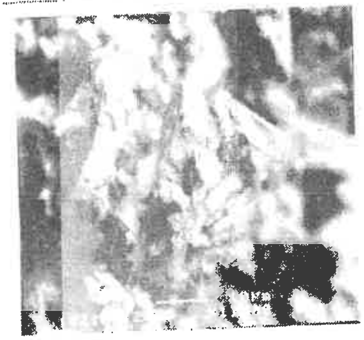


**Figure (11 )morpholog of fracture surface of compound $Bi_2$-$Al_{0.2}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{8+\delta}$**

**Figure( 12 )morpholog of facture surface of compound $Bi_2$-$Al_{0.1}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{8+\delta}$**

**Figure (13 )morphology of fracture surface of compound**

**$Bi_2$-$Al_{0.8}$-$Sr_2$-$Ca_2$-$Cu_3$-$O_{10+\delta}$**

## References

1.Hiroshi Maeda, Yoshiaki Tanaka, Masao Fukutomi and Toshihisa Asano(1988) A New High-$T_c$ Oxide Superconductor without a Rare Earth Element, Jpn. J. Appl. Phys: 27 pp. L209-L210.

2-Koyoma S, Endo U , Kawai T( 1988).Format ion of high Tc phase of Bi-Pb-Sr-Ca-Cu-O superconductors", Proceedings of the 1st international symposium on superconductivity (Iss88),August ,Tokyo Berlin :833-836

3- Hitoshi Nobumasa , Kazuharu Shimizu, Yukishige Kitano and Tomoji Kawai, High $T_c$ Phase of Bi-Sr-Ca-Cu-O Superconductor Jpn. J. Appl. Phys. 27 (1988) ,pp. L846-L848

4-Mizuno M, Endo H,Tsuchiya J, Kijima N, Sumiyama A , Oguri Y, (1988).Synthetic conditions and structural properties of high –Tc phase in the superconducting Bi-Sr-Ca-Cu-Pb-O, Proceedings of the 1st international symposium on superconductivity (Iss88),August ,Tokyo, Berlin :839-841.

5- Mikio Takano, Jun Takada, Kiichi Oda, Hitoshi Kitaguchi , Yoshinari Miura, Yasunori Ikeda, Yoichi Tomii and Hiromasa Mazaki ,(1988) , High-$T_c$ Phase Promoted and Stabilized in the Bi, Pb-Sr-Ca-Cu-O System, Jap, J. Of . Appl. Phys. Vol.(27),no 6,p.L1041-L1043.

6.Usai T, Sadakata N, Ikeno Y, Kohno O, and Osanai H (1988). Preparation of High Tc Bi-Sr-Ca-Cu-O and T1- Ba-Ca-Cu-O Superconductors International symposium on superconductivity (Iss88),august 28-31 Nagoya springier –verlay ,Tokyo, Berlin, Heidelberg, New York, London, Paris.

7. Mitsuru Uehara, Yuji Asada, Hiroshi Maeda and Keiichi Ogawa (1988). Magnetic Properties of BiSrCa$Cu_2O_x$ Superconductors ,Jap .J .Of Appl .Phys. Vol.(27), no.4,p,L665-L667.

8. Kumakura H, Shimizu H, Takashi K, Togano K, and Maeda H, (1988) ( Upper Critical Field of New Oxide Superconductor Bi-Sr-Ca-Cu-O ) ,Jap .J . Of Appl . Phys . Vol.(27),no.4, p(L668-L669).

9. Matsuoka D , Okada M , Murakami T, Cross K, and Homma M, (1990). Superconducting Behavior in the Bi-In-Sr-Ca-Cu-Pb-O System, Materials Transactions,JIM,31, No.9 ,P755-758.

10. Qianzhang Wu , Zhengmin Fu , Aizhen Zhang , Jiashan Huang , Disheng Tang , Pei Yao , Shaoyan Chu ,Sunsheng Yi, Xishen Rong, Anzhi Zhang, and Xianan Cheng, 1992 ,J .Appl .Phys ,71 (6) ,15 . Synthesis and crystal growth of the high□Tc phase in Bi–Pb–Sr–Ca–Cu–O system with variation of excess concentrations of CaO and CuO
J. Appl. Phys. 71, 2772 (1992).

11. Pop AV, Marconi D, Pop V, Pop M, J. 2006,Optoelectronics and Advanced Materials, 8, No. 2, April p. 476 – 479. Intergranular dissipation processes induced by Nano defects in (Bi,Pb):2223 HTS superconductor.

12.( Kovaleva, N. N., Boris, A. V., Holden, T., Ulrich, C., Liang, B., Lin, C. T., Keimer, B., Bernhard, C., Tallon , J. L ., Munzar, D . and Stoneham , A . M.) , 2004, (*c*-axis lattice dynamics in Bi-based cuprate superconductors ), Phys . Rev . B 69 , 054511 -1 -054511 -16.

13. Ideta S, Takashima K, Hashimoto M, Yoshida T, Fujimori A, Anzai H, Fujita T, Nakashima Y, Ino A, Arita M, Namatame H, Tanniguchi M, Ono K, Kubota M, Lud HY, Shen ZX, Kojima MK, and Uchida S, (2010), Phys.Rev.Lett.104,No 22.

14. Grivel JC, Jeremie A, Hensel B, and Flukigev R, supercond .Sci . Technol.,6 (1993),P.725 printed in UK. Bi,Pb(2212) and Bi(2223) formation in the Bi-Pb-Sr-Ca-Cu-O system. Superconductor Science and Technology Volume 6 Number 10.

15. D. Vasumathi, C.S. Sundar, A. Bharathi, A.K. Sood, Y. Hariharan , physica C167 (1990) , p.149-156 . A positron annihilation study of the decomposition of $Y_1Ba_2Cu_3O_{7-x}$.

16. Sequeira A, Yakhmi J V, Rajagopal and Sastry PVSS, (1990). Physica C167 :149.

17. Akio Oota, Akihiro Kirihigashi, Yoshinobu Sasaki and Kentaro Ohba

,(1988),( The Effect of Pb Addition on Superconductivity in Bi-Sr-Ca-Cu-O ) , Jap , J. Of . Appl. Phys.Vol.(27),no 12,p.L2289-L2292.

18. Mizuno M, Endo H, Tsuchiya J, Kijima N, Sumiyama A, Oguri Y , "Synthetic conditions and structural properties of the high – Tc phase in the superconducting Bi -Sr-Ca-Cu-Pb-O system ," Phys.Rev.Lett.104,No 22 (P839-841).

19. Cloots R, Romain AC, Rulmont A, Diko P, Duvigneaud PH, Hannay C, Gillet F, Godelain PA, and Ausloos M, supercond , Study of the crystallization process in $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10-y}$glass systems: optical polarized light microscopy, electrical and magnetic properties Superconductor Science and Technology, Volume 6, Issue 12, pp. 850-857 (1993).

20. Chavira E, Escudero R, Rios D, Jara and Leon LM, (1988) ,(Influence of lead on the formation of the 110-K superconducting phase in the Bi-Sr-Ca-Cu-O compounds) , Phys Review B ,Vol.(38) ,13,p.(9272-9275.

21. Yasuhiko Syono, Kenji Hiraga, Norio Kobayashi, Masae Kikuchi, Keiji Kusaba, Tsuyoshi Kajitani, Daisuke Shindo, Syoichi Hosoya, Ayako Tokiwa, Shoji Terada and Yoshio Muto , ( An X-Ray Diffraction and Electron Microscopic Study of a New High-$T_c$ Superconductor based on the Bi-Ca-Sr-Cu-O System), (1988) Jap , J. Of . Appl. Phys. Vol.(27),no 4p.L569-L572.

22-J.Zhao.M.Wu,W.Abdul-Razzaq and M.S. Seehra,(1990),Physica C, 165,135-138.

# جامعة حضرموت للعلوم والتكنولوجيا-كلية الهندسة والبترول-
# قسم الهندسة الالكترونية والاتصالات- اليمن

## الخلاصة

تم تحضير مركبات من ( Bi-Sr-Ca-Cu-O ) ذات التوصيلية الفائقة استخدمت طريقة تفاعل الحالة الصلبة في تحضير النماذج ، ثم طعمت هذه المركبات بالرصاص( Pb ) للمركبات ( $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_{10+\delta}$ ) عند قيم ( $x=0.1,0.35,0.4$ ) وكذلك طعمت بالألمنيوم ( Al )للمركبات( $Bi_2\ Al_xSr_2Ca_2Cu_3O_{10+\delta}$ ) عند قيم ( $x=0.1,0.2,0.8$ ) . لقد تم الحصول على طور التوصيل الفائق باستخدام درجة حرارة تلدين 860 درجة مئوية ولمدة16 ساعة بالتعاقب وان معدل التبريد البطيء البالغ ( $1^{o}C/min$ ) تلعب دورا مهما في عملية تشكيل طور التوصيل الفائق لمركبات البزموث متعددة الأطوار. إن التركيب البلوري لمركبات البزموث الفائق التوصيل المستقرة حراريا تكون معينيه قائمة(مستطيلي) ( Orthorhombic) . ذات ثوابت شبيكة ( $a=5.394A^{o},b=5.346A^{o},c=30.753A^{o}$ )للطور الواطئ في حين قيمتها ( $a=5.394^{o}A,b=5.311^{o}A,c=37.182^{o}A$ ) للطور العالي. اعتمدت طريقة قياس المقاومة الكهربائية لإيجاد قيم درجات الحرارة الحرجة ($T_c$) لهذه النماذج وذلك باستخدام منظومة تبريد تعمل بالنيتروجين السائل وكانت أعلى قيمة عند ( $x=0.4$ )للنماذج المطعمة بالرصاص حيث كانت قيمة درجة الحرارة الحرجة تزداد بازدياد قيم ( $x$ )، بينما كانت أعلى قيمة لدرجات الحرارة الحرجة ( $T_c$ )للنماذج المطعمة بالألمنيوم عند قيمة ( $x=0.8$ ). تم فحص النماذج باستخدام المجهر الالكتروني الماسح ( SEM ) وأظهرت الصور تكوين طور صفائحي والطور الرقائقي( flake ) في نماذج التوصيل الفائق.