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Structural and Superconducting Properties of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$. System

[1]Ghazala Y. Hermiz, [2]Amal K. Jassim, [3]Ebtisam K. Alwan

[1]Ghazala Yalda Hermiz, Professor, Physics Department , Science College, Baghdad University, Baghdad, Iraq.
[2]Amal Kadhim Jassim, Assist. Professor, Physics Department, Science College, Baghdad University, Baghdad, Iraq.
[3] Ebtisam Khalil Alwan, Ph.D. Student, Physics Department , Science College, Baghdad University, Baghdad, Iraq.

Address For Correspondence:
Amal Kadhim Jassim, Baghdad University, Physics Department, Science College, Karrada Road, Baghdad, Iraq.

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ABSTRACT

Superconductivity is a unique and powerful phenomenon of nature. it is a characterized by the disappearance of electrical resistance in various metals, alloys and compounds when they are cooled below a certain temperature (critical temperature, T_c). The effects of vanadium addition on the properties of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ bulk and thin film samples for ($0 \leq x \leq 0.4$) were studied. The XRD analyses for all superconductor samples as bulk and thin films showed an orthorhombic structure with

The critical temperature for the most films is less than that .two superconducting phases of the bulk samples except for pure film. The surface morphology of bulk $Bi_{1.7}Pb_{0.3}V_x Sr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ and thin films before and after annealing obtained by AFM in three dimensions views. It was observed that the average roughness was higher at x=0.2 (179.66nm) with a particle size of 219 nm.

INTRODUCTION

The discovery of superconductivity brings a tremendous change in the field of science and engineering from twentieth century onwards. Research is being carried out to develop superconductors for high temperatures.Bismuth and thallium-based superconductors were discovered in 1988, which became superconducting at 110K and 125K, respectively (Maeda *et al*.1988; Verma *et al*.2012). Physical properties of these compounds are very different from the properties of conventional superconductors.

(Trivijitkasem *et al*. 2006)studied the effect of vanadium-lead doped in Bi-Sr-Ca-Cu-O superconducting system. Their results showed both critical temperature and the 2223 phase formations that affected by vanadium and lead concentration.

(Jannah *et al*.2009) studied the properties of BSCCO thin film deposited by PLD. Their results showed an improvement of the superconducting properties of the film by annealing with zero resistivity temperature at about 60 K (Elsabawy and Newhya. 2010) synthesized and processed $Bi_2SrV_{2-x}Ti_xO_9$, where (x= 0.05, 0.1, 0.2 mole) by using freeze dry route depending on solution precursors and sintering temperature at 1000 ºC for 8 hrs. XRD-analysis of the prepared samples proved that Ti dopant can substitute successfully within narrow range $0.0 \leq x \leq 0.2$

(Mua *et al*. 2014) investigated the electrical transport properties of $Bi_2\ Sr_2\ Ca_2\ Cu_3\ O_{10+\delta}$ superconducting thin films that fabricated by Pulsed -Laser deposition on SrTi O_3 substrate. A critical temperature as high as 110K and critical current density of $6.2x10^6$ A/m^2 at 20 K were obtained at a substrate temperature of 760 ºC.

(Darsono *et al*. 2015) prepared $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_7$ superconducting system by a solid-state reaction. The samples showed good superconducting properties with T_c~ 100 K. (Hermiz *et al*. 2016) studied the

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addition effect of V2O5 on the high-superconducting materials of the Bi-Pb-Sr-Ca-Cu-O system (2223 phase). Their results showed that both critical temperature and the 2223 phase formations were affected by vanadium concentration and the highest Tc at 118 K was obtained for the sample with V =0.2.

This work is aimed to investigate the effect of the V substitution on the properties of superconducting $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ bulk and thin film samples.

MATERIAL AND METHOD

The system of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ bulk samples were prepared by a solid state method using appropriate weight of high purity powders (99.9) of material Bi_2O_3, PbO, Sr $(NO_3)_2$, CaO, CuO, TiO_2 and V_2O_4 that proportional to their molecular weight. The powder was calcined in air at 800◦C for 24 h. Then it was pressed into disk-shaped pellets 13 mm in diameter and 1–2 mm in thickness using a manual hydraulic press type (SPECAC) under pressure 0.7 GPa. The pellets were sintered in air at 835◦C for 140 h. The PLD of thin films of BPSCCO was carried out by using laser Nd:YaG .The laser had a wavelength of (532 nm) and the beam energy was focused onto the target to obtain an flounce 0.6-0.7J/cm^2, the ablated frequency at (6 Hz) by second harmonic with Q-switched Nd :YaG pulse laser. All thin film samples were grown on n-type silicon wafers (Si) substrate (111), size (10 ×10) mm^2 . The structure of the prepared samples was obtained by using X-ray diffraction (XRD) (Philips) with CuKα source. The four point probe method was used to measure the resistivity and to determine the critical temperature (Tc). The morphologies of the bulk and films were analyzed by atomic force microscopy (AFM) type origin USA model AA300-CSPM .

RESULTS AND DISCUSSION

Fig. (1) represent the XRD patterns for $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ samples for different values of x which were prepared by two sintered steps at 835°**C** for 140 h. These analysis indicate the dominant phase of Bi2223 together with the amount of low-phase Bi2212 in addition to some unknown phase. The appearance of more than two phases could be related to the displacement of an ion or oxygen defects or to the ordering of cations which leads to the stacking faults along the c-axis then this leads to deform the structure [8]. It appears from the above figure that the peak position are shifting to lower or higher angle with the increasing of x content.

Table (1) shows a reduction of the ***a*** and ***c*** lattice parameters for V addition samples as comparable with the V-free samples .Indeed this behavior agree with (Novakova *et al*.1996), they found a decreasing in the ***c*** lattice parameter from 37.173 Å to 37.079 Å when increases the amount of V from 0 to 0.6 in Bi-based superconductors. It is well known that the lattice parameter ***a*** is controlled by the length of in-plane Cu-O bond (Pandey *et al*.1998). The length may be expanded or contracted with the change of the electrons into antibonding orbital. It is observed a non-systematic variation of the lattice parameters with the increasing of the Vanadium concentration. The deformation in the lattice constant as a result of addition or deficiency of some atoms, adjusts the amount of charge transfer from Bi layer to Cu layer, this will be a driving force to the pairing generation of superconductor.

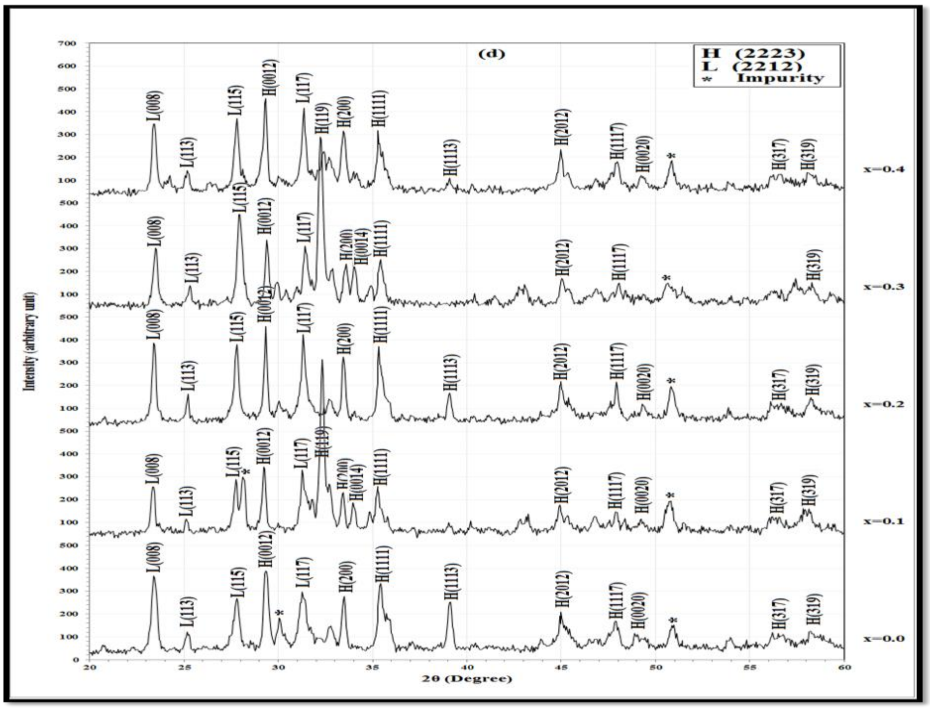

Fig. 1: XRD patterns for $Bi_{1.7}Pb_{0.3}Sr_2V_xCa_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ bulk samples for (x= 0,0.1,0.2,0.3 and 0.4) that sintered at 835 ºC for 140 h.

Table 1: Values of lattice parameters (a, b, c) and c/a for different composition of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$, which is sintered at 835 º C for 140 h

X	a Å	b Å	c Å	c/a
0.0	5.7916	5.2358	37.7116	6.5114
0.1	5.4411	5.53587	37.0597	6.8110
0.2	5.4679	5.4317	37.4203	6.8363
0.3	5.4219	5.4205	37.2201	6.8647
0.4	5.3859	5.4581	37.3995	6.9439

The XRD patterns of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ thin films for different values of x = 0 ,0.1,0.2,0.3 and 0.4 that deposited on Si substrate and annealed at 800 ºC for 2h under flow of oxygen are shown in Fig.(2). It can be seen from the patterns that a polycrystalline orthorhombic structure of all the films and most of them has two main phases: high-T_c phase (2223), low-T_c phase (2212). Beside of these phases, there are unknown phases which leads to deform the structure and delay onset of longer phase coherence to appear at transition temperature to stay cooper pairs longer distance . From Table (2) we can notice the variation of lattice parameters (***a, b, c***) have the same behavior for the bulk sample

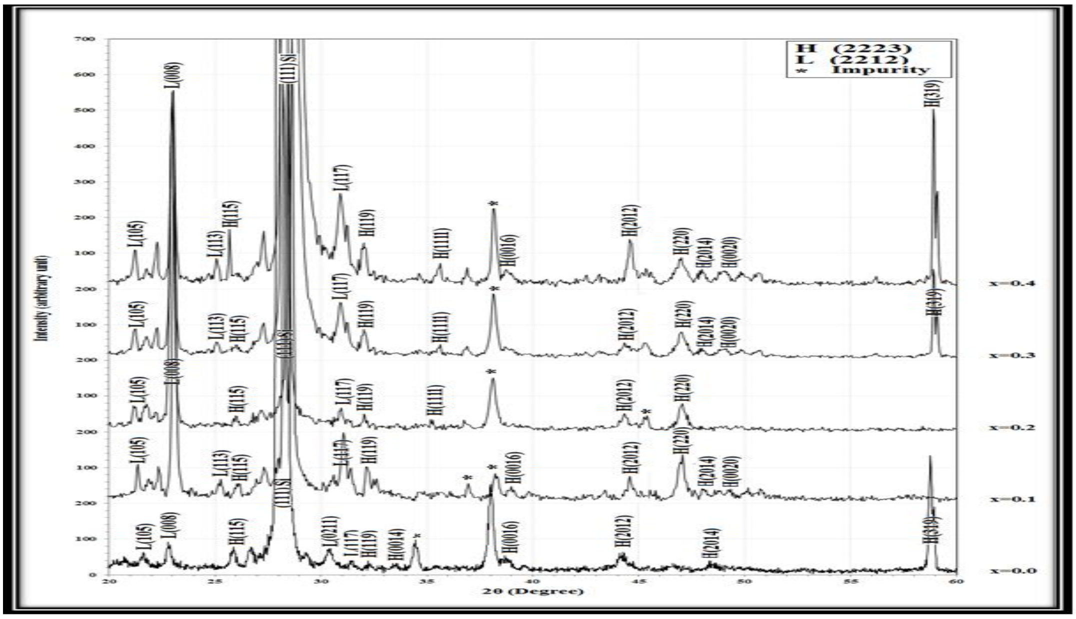

Fig. 2: XRD patterns for the deposited $Bi_{1.7}Pb_{0.3}V_x Sr_2Ca_2Cu_{2.7} Ti_{0.3}O_{10\pm\delta}$. thin film on Si substrate and annealed at 800 °C for 2h for different x =0 , 0.1,0.2,0.3 and 0.4.

Table 2: The Values of lattice parameters for deposited $Bi_{1.7}Pb_{0.3}Sr_2V_xCa_2Cu_{2.7}Ti_{0.3}O_{10\pm\delta}$.

X	*a* Å	*b*Å	*c* Å	*c/a*
0.0	5.3653	5.3688	37.7631	7.0384
0.1	5.1677	5.1591	37.0843	7.1762
0.2	5.5299	5.3901	36.399	6.5822
0.3	5.3863	5.3504	36.8854	6.8480
0.4	5.3547	5.344	37.3488	6.9749

Temperature dependence of resistivity for samples of $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ with (0-0.4) as x ranges are shown in Fig. (3). Values of the critical temperature are listed in Table (3). It was found from this figure that the critical transition temperature depends on the concentration of V. In general, T_c decreases with the increasing of V concentration, this decrease may be attributed to decrease in the oxygen content. It is interesting to note that the excess oxygen content reduces with the increasing of vanadium content ≥ 0.3. In our opinion there is an enhancement of the acceptor levels of V and Ti in comparison with the donor level that produced from Cu existence in the CuO_2 layer. This will make a non-equilibrium of the charge density and causes a destruction of the excitons.

The decreases of δ and T_c with the increasing of V contents may attributed to the creation of a cloud around the atoms which will oxide the ions, and this causes a reduces of the conductivity (Trivijitkasem *et al*. 2006).

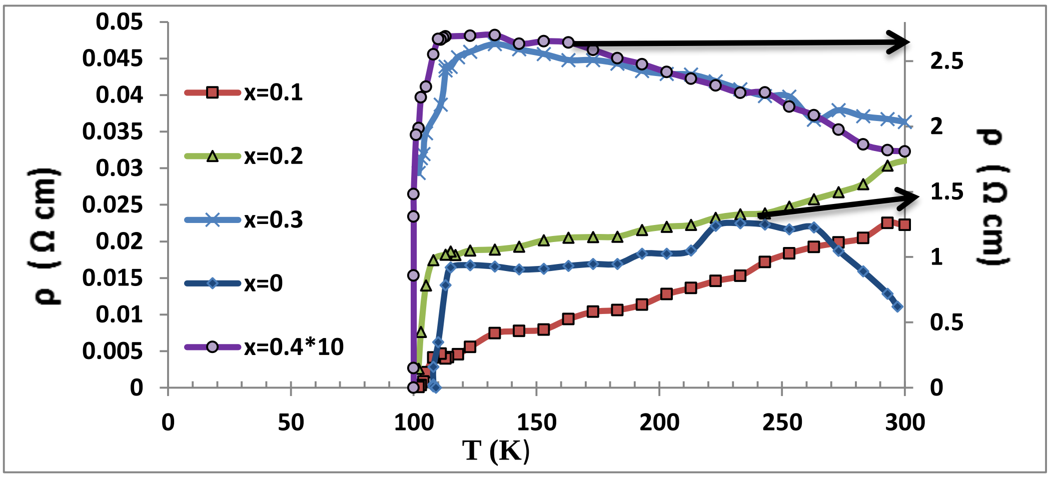

Fig. 3: Temperature dependence of resistivity for$Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7} Ti_{0.3}O_{10\pm\delta}$ system for different V concentration that sintered at 835 ºC for 140h.

Table 3: The variation of the critical temperature T_c and oxygen content(δ)with different concentration of V for $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ bulk samples.

x	T_c(K)	(δ) oxygen content
0	115	0.3354
0.1	106	0.2511
0.2	107	0.2857
0.3	>77	-0.1577
0.4	104	0.2363

Fig. (4) show the curves of the resistivity as a function of temperature for $Bi_{1.7}$ $pb_{0.3}$ V_x $Sr_2Cu_{2.7}$ $Ti_{0.3}O_{10\pm\delta}$ thin films with x range from 0 to 0.4 which were deposited by pulse laser deposition. The results of the T_c for all films are summarized in Table (3). Generally, by looking at these Tables, we can recognize that the T_c for the most films is less than that of the bulk samples except for pure film.

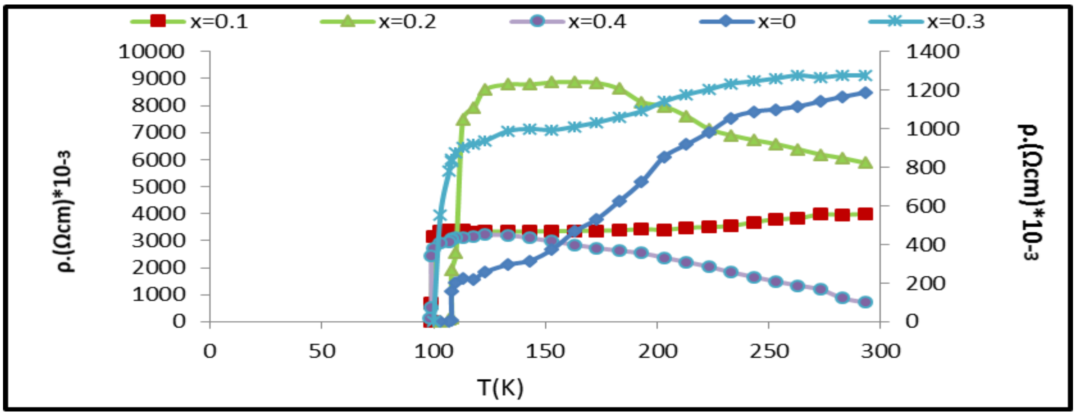

Fig. 4: Temperature dependence of resistivity for the deposited $Bi_{1.7}Pb_{0.3}$ V_x $Sr_2Ca_2Cu_{2.7}$ $Ti_{0.3}$ O_{10+} films on Si substrate for different x (0,0.1, 0.2,0.3and 0.4).

The effect of variation of V on the transition temperature for thin films is shown in Figs. (4). It is observed from this figure and Table (4) that most of the samples have lower transition temperature than those of the bulk samples. In our opinion, Vanadium play the same role of lead in the grain boundaries which will improve the weak link between grains and hence decreases the barrier between theses boundaries (Hamid *et al*. 2009).

Table 4: The values of T_c for the thin films samples for composition of $Bi_{1.7}Pb_{0.3}VxSr_2Ca_2Cu_{2.7}Ti_{0.3}O_{10+\delta}$ at x and y range from 0 to 0.4

x=0	x=0.1	x=0.2	x=0.3	x=0.4
T_c (K)	T_c (K)	T_c(K)	T_c(K)	T_c(K)
110.5	103	112.5	105	104

On the other side, we can notice that $Bi_{1.7}$ $Pb_{0.3}$ $V_{0.3}$ Sr_2 Ca_2 $Cu_{2.7}Ti_{0.3}$ $O_{10\pm\delta}$ samples have a superconductor behavior when they deposited as a thin film. (The bulk samples have a semiconductor behavior).

The behavior of the transition temperature of thin films could be explain as follows: The nature of the films is granular and they have two components as superconducting and non superconducting. The first one, the superconducting grains provides channel to transport the supercurrent, the volume for these components is $T_{coff} < T < T_c$. The second one, the non-superconducting components is the Josephson weak links .However the degradation of T_c due to the volume of the first component which is not enough to provide a percolative path for the superpairs (Hermiz *et al*. 2013).So if the two components affect together may provide the transferring path for the super pairs.

The surface morphology of bulk $Bi_{1.7}Pb_{0.3}$ V_x $Sr_2Ca_2Cu_{2.7}$ $Ti_{0.3}$ $O_{10+\delta}$ and thin films deposited on Si substrate for different values of x (0 to 0.4) before and after annealing process at 800 ºC for 2 h with flow of oxygen are shown in Figs. (5 -7).

These Figs. and Table (5) represent the three dimensional views that give the particle size, and average roughness (different between the highest and the lowest point on the film), the images reveal a spherical and pyramidal –like morphology which may available by the accumulation of the needle-crystallites to the samples . The formation of the humps in some places may beyond to the formation of superconducting layer with different thickness depending on the chemical composition of the phase (Verma et. al., 2012).

Surface roughness is measured as the standard deviation of the mean height of surface structure. It is clear from Table (5) for films before annealing process that the average roughness decreases with the increasing of Vanadium content up to 0.2. Then there is an enhancement of the roughness for samples with x=0.3 and 0.4. On the other side, a nonsystematic changes in the surface roughness is observed for deposited films after annealing process.

The variation in surface morphology with vanadium content may due to the realization of different surface energies which is leading to crystallites of varying crystalline nature and size. Also lower concentration generates at low surface migration of atoms.

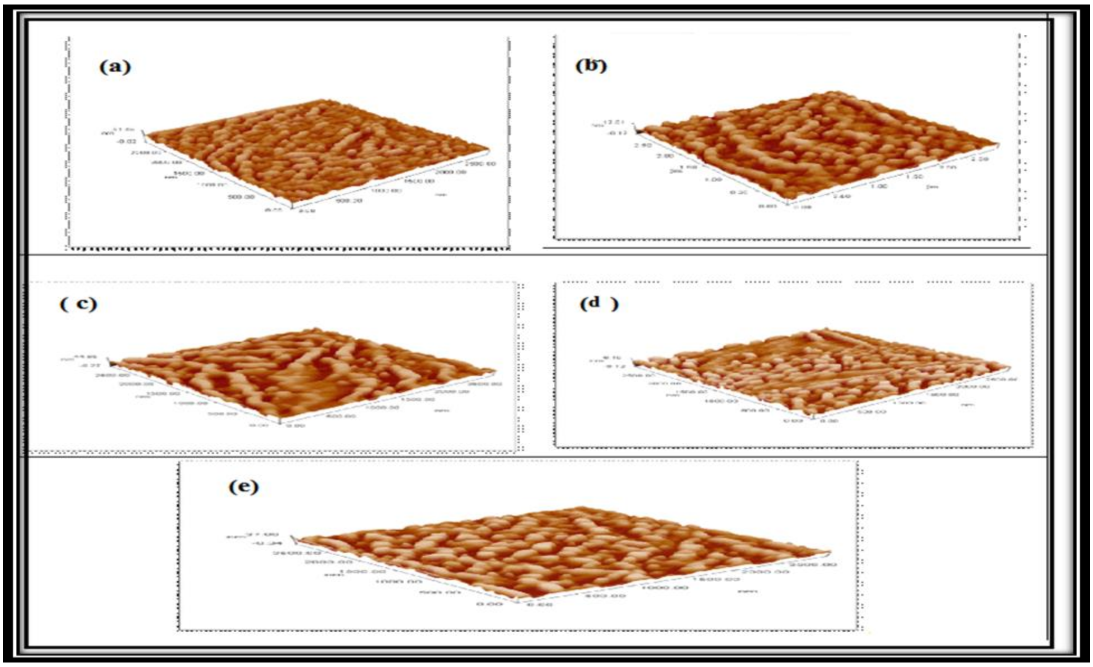

Fig. 5: AFM for $Bi_{1.7}Pb_{0.3}V_x Sr_2Ca_2 Ti_{0.3}Cu_{2.7}O_{10+\delta}$ bulk samples for different x (a)x=0, (b) x=0.1, (c) x=0.2, (d) x=0.3 and (e)x=0.4.

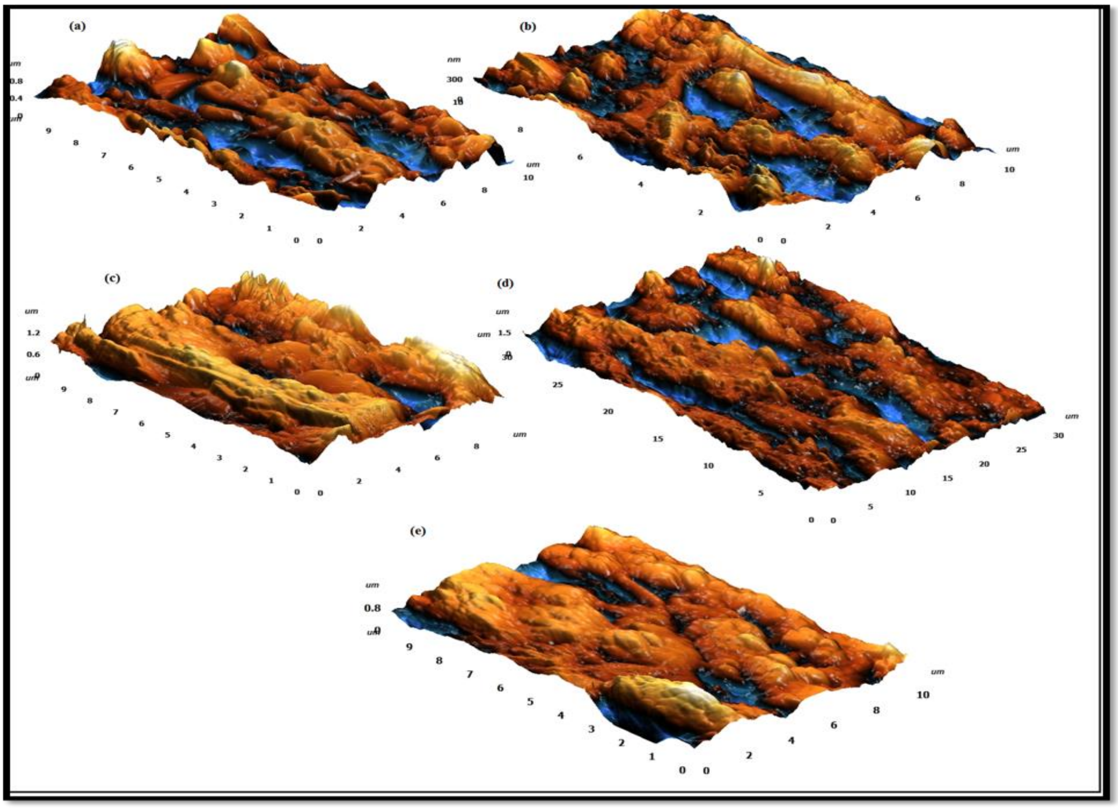

Fig. 6: AFM for the deposited $Bi_{1.7}Pb_{0.3} V_x Sr_2Ca_2 Ti_{0.3}Cu_{2.7} O_{10+\delta}$ films for different x [(a) x=0, (b) x=0.1 ,(c) x=0.2 , (d) x=0.3 and (e)x=0.4] before annealing process at 800 ºC.

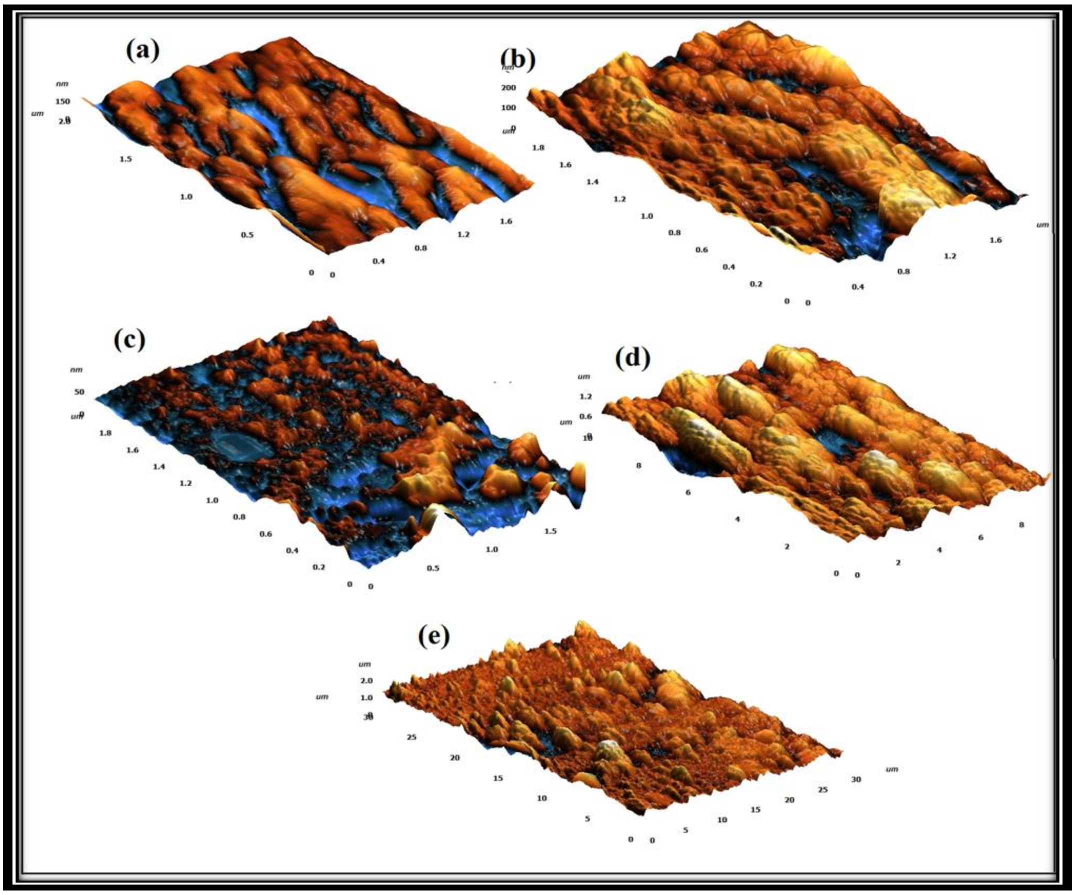

Fig. 7: AFM for the deposited $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Ti_{0.3}Cu_{2.7}O_{10+\delta}$ films for different x [(a)x=0, (b) x=0.1, (c) x=0.2 , (d) x=0.3 and (e)x=0.4 after annealing process at 800 ºC

Table 5: Average particle and average roughness for $Bi_{1.7}Pb_{0.3}V_xSr_2Ca_2Ti_{0.3}Cu_{2.7}O_{10+\delta}$ films at different x before and after annealing and bulk samples.

	Bulk		Thin films			
X	Average particle Size (nm)	Average roughne (nm)	Average particle Size (nm) before annealing	Average roughness (nm) before annealing	Average particle Size (nm) after annealing	Average roughness (nm) after annealing
0	92.38	5.64	109	68.235	281	126.83
0.1	104.00	1.63	111	41.683	162	75.06
0.2	95.31	0.31	82.9	15.607	219	179.66
0.3	101.85	0.955	97.2	81.706	201	97.869
0.4	110.64	5.57	109	54.393	200	138.339

The surface of the deposited film is greatly affected by the time of annealing where the heat treatment may be required to increase the degree of crystallinity through increasing the average size of a particle. It is observed that the average roughness is higher at x=0.2 (179.66nm) with a particle size of 219 nm.

Indeed the roughness associate with the enhancement intergrowth of the high phase which may stable thermodynamically (Thoutam,2011).On the other hand, the rough surface may correlate to the existence of clusters in the plasma (Jannah *et al*. 2009).

Conclusion:

Our conclusions from the results can be summarized as follows:

1- XRD analyses for bulk and thin films showed a polycrystalline orthorhombic structure and most of them has two main phases: high-T_c phase (2223), low-T_c phase (2212). Beside of these phases, there are unknown phases.

2- Values of the critical temperature depends on the concentration of V.

3- T_c for the most films is less than that of the bulk samples except for pure film.

4- The AFM for films before annealing process show that the average roughness decreases with the increasing of Vanadium content up to 0.2. Then there is an enhancement of the roughness for samples with x=0.3 and 0.4.The average roughness is higher at x=0.2 (179.66nm) with a particle size of 219 nm.

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