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New High Temperature Superconductor Phase of Y-Ba-Cu-O System

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Abstract: *The superconductor compounds of YBCO-system were synthesized by solid state reaction method, such as $Y_nBa_5Cu_{n+5}O_y$ for n=3, 5, 7. The prepared samples were examined by Meissner effect in the presence of liquid-N_2. The electrical resistivity was measured by using Van der Pauw technique through closed cycle refrigerator under liquid-He_2. The value of critical temperature at (n=3, 5, 7) were (T_c=113.6, 113, and 105 K) respectively. X-ray diffraction was done to show the crystal structure through the operation of analytical software. It was clear that the crystal structure was orthorhombic phase for all values of (n), but there was a slightly variation in the lattice constants. The scanning electron microscopy (SEM) results showed that the Y-358 had large grains randomly comparable with Y-5510, Y-7512, with the absent of interfaces channels in Y-358.*

Keywords: *YBCO Superconductor, solid state reaction, X-ray diffraction.*

1. INTRODUCTION

Since the discovery of high-Tc superconductivity in 1986 was investigated by Bednorz and Muller [1], it was the speeding point to investigate HTSc they found that the critical temperature around (30 K) in La_2CuO_4 compound. They discussed the mechanism of superconductivity depends on the distribution of Cu^{+2} by $3d^9$ configuration and Cu^{+3} by $3d^8$ configuration in octahedron site. There is elongation in the octahedron by Jahn-Teller effect in lanthanum copper oxide [2]. Later, the searching to higher transition temperatures (T_c) in different families of high-T_c superconductor (HTSCs) had been highly investigated. Later, the critical temperature (T_c) goes to 90K when La^{+3} ion was replaced by Y^{+3} ion and producing a superconducting compound such as $YBa_2Cu_3O_{7-\delta}$ [3]. The researchers was started to derived a related phases like $YBa_2Cu_4O_8$ [4] and $Y_2Ba_4Cu_7O_{15}$ [5], in order to conclude a higher T_c-value in YBCO-system. They found that Y124 and Y247 became superconductor at 80 K and 40 K, respectively. Recently, Aliabadi et al [6] they found a new Y-based high-temperature superconductor in $Y_3Ba_5Cu_8O_{18}$ that was recorded T_c above (100 K).While, Kruaehong et al [7] they found that $Y_3Ba_5Cu_8O_{18}$has two phases appeared, the superconductor phase and non-superconductor phase. They used four-probe technique to determine T_c-onset; it was about (94K). Kruaehong made a comparative study between Y123, Y358, Y3811, and Y257 bulk superconductors doped by fluorine and synthesized by solid state reaction [8, 9]. He found that the value of T_c in the range (88-89 K). In this research, the preparation of new phases like Y-358, Y-5510 and Y-7512 had been applied by using solid state reaction with a certain condition of calcinations and sintering. The samples have been characterized by X-ray diffraction (XRD), resistivity measurement by Van der Pauw technique to define T_c-value for each phase and SEM analysis to study the surface morphology for different phases.

2. EXPERIMENTAL PROCEDURE

The superconducting samples of the composition $Y_nBa_5Cu_{n+5}O_y$ (n=3, 5, 7) were prepared by solid state reaction. The appropriate quantities of high purity Y_2O_3, $BaCO_3$, and CuO were mixed, grounded and thermally reacted in air at 840^{o}C for 24h. The calcination process carried out twice or more even to remove CO_2 gases from the mixture, then cooled down to room temperature. The purpose of calcination process is to create the superconducting phase. Later, the powder pressed into pellet shape with (1.5 cm) diameter and (0.15 cm) thick. The pellets were put in alumina crucible for sintering in a tube furnace at temperature of 900^{o}C for (24-48h). The sintering process is necessary to reduce the pours size and make the sample denser, which is applied to enhance the superconducting behavior

through the reducing the grain boundaries. The prepared samples were examined by Meissner effect to show the superconductor behavior primarily. The excess in oxygen content was measured experimentally by Iodomertric titration method. The crystal structure for the samples under study was characterized by XRD patterns, the computer program that was used to determine the lattice constant. Scanning Electron Microscope (SEM) used to show surface morphology and the nature of grain size. The resistivity measurement was performed by Van der Pauw method to determine the critical temperature.

3. RESULTS AND DISCUSSION

The system Y-Ba-Cu-O prepared by solid state reaction with different composition of Y-atoms in comparable with Cu-atoms per unit cell. The amount of oxygen those were inserted within the unit cell was increased gradually with increasing the ratio of Y-atom in the compound. The results of δ-value that was measured by Iodomertric titration, it was about (0.18) for compound $Y_3Ba_5Cu_8O_{18+\delta}$, and δ-value for $Y_5Ba_5Cu_{10}O_{22.5+\delta}$ and $Y_7Ba_5Cu_{12}O_{27.5+\delta}$were about (0.21),(0.22) respectively. The approaching to saturation value of δ-value was appeared at (n=5, 7) that means the increasing of Cu-atoms as a function of (n) tend to reduce the vacant sites in the unit cell. This is possible because the most Cu-atoms are bonded with O-atoms might be in the plane or the chain. On the other hand, there is still a vacant sites for O-atoms within the unit cell.

The XRD pattern of the samples $Y_nBa_5Cu_{n+5}O_y$ for (n=3,5,7) which has polycrystalline phase as shown in Figs. 1, 2, 3. There are three common peaks appear in all phases represented by (011), (0111), (0129), which are the bases to produce the bonds between the elements Y-O and Ba-O layer. The concentration of Ba was constant within the unit cell that is return to constant intensity of the peaks (011) for different values of (n). On the other hand, it has the same position of the diffracted angles (2θ=23.0) that means it has no effect on the partial variation of lattice constants for (n=3,5,7). This is a proof that the presence of Ba-atoms was strongly bonded within this plane in the unit cell. Whereas, the shifting in the diffracted angle (2θ) for the plane (0111) was appeared at (2θ=28.0, 29.9, 29.0) for (n=3, 5, 7) respectively. There is a sharp increasing in the intensity as a function of (n). That was return to increasing of Y-atom within the structure as a function of (n). This is the reason to produce Y-O bond within the planes and increasing their density as a function of (n). On the other hand, the value of (2θ=58.95, 58.3, 58.3) for the plane (0129) at (n=3, 5, 7) respectively. The intensity of this plane was decreased as (n>3), while it remains constants at (n=5,7). That was might be attributed to vacant position of O-site in c-axis or the chain within the unit cell. That is attributed to Y-O bond, the approaching to constant intensity of this plane give more information about the effect of increasing the density of Y-atoms within the plane (0111) rather than the plane (0129). There is agreement between the saturation of Y-atom and the saturation in oxygen excess as (n=5, 7). The slightly shifting in the diffracted angle for (n=3, 5) tend to show a decreasing in lattice constant (a) as a function of (n). This decreasing might be attributed to the bending in a-axis, but still constant at (n=5,7) regarding to constant concentration of Y-O bond in the chain per unit cell.

The other peaks appeared in XRD patterns for the compound $Y_3Ba_5Cu_8O_y$ as shown in Fig. 1. These peaks are not common as mentioned in Figs. 2, 3. They were mentioned by (113), (2123) have highest intensity at (2θ=33.45, 69.3), both are related to Cu-O bond. The planes were mentioned by (1018), (0119), (202) have a position (2θ=39.6, 41.15, 47.5) which were the reason to produce Cu-O bond within the unit cell of $Y_3Ba_5Cu_8O_y$. The position of peaks (124), (1129), (220) were at (53.35, 63.5, 75.8), those are coming from Y-O and Ba-O bonds with the unit cell. All these peaks are the property of the phase (n=3). The analytical results of XRD pattern for $Y_3Ba_5Cu_8O_y$ was exhibited Orthorhombic phase with lattice constant (a=3.852 Å, b=3.882 Å, and c=50.262Å) as shown in Table 1.

The XRD pattern for $Y_5Ba_5Cu_{10}O_y$, as shown in Fig. 2, it was clear that there is a major variation in the phase represented by the appearance of new peaks at (n=5,7) in comparable with (n=3) as discussed before. The highest common three peaks were appeared (0111), (106), (0018), (111) at the position (2θ=29.9, 30.5, 31.6, 32.8). Just they were a derivative peaks from the peak (113) appeared at (n=3). The last two peaks are related to Ba-O and Y-O bonds. The peaks were mentioned with Miller indices (0026), (220) at (2θ=46.65, 68.62) respectively, they are a property of the phase (n=5). These

peaks related to create Cu-O bond within the basal plane and the chain in c-axis. The results showed that there is an orthorhombic phase with the lattice constant a=3.777 Å, b=3.99 Å, c=51.93Å as shown in Table 1. There is reduction in a-axis and increasing in b-axis with the same ratio in comparable with the composition of (n=3). These results are compatible with the results of oxygen excess within the structure, which are tend to occupy some vacant sites in the basal plane and the last made a partial variation in the dimension of ab-plane. The slightly increasing in c-axis was related to more insertion of oxygen content in the mixture.

All other peaks mentioned at (n=5) were common with the phase (n=7), except the peaks (0026), and (220) which were vanished, as shown in Fig. 3. On the other hand, there were a creation of new peaks appeared at (020), (2012), and (0131). This creation tend to produce a new planes inserted in the unit cell and the last has the direct effect on the enhancement of the bonds between the elements of the structure. The important thing, there is a completely reducing of the intensity for the common peaks at (n=5,7), that means there is a lack in the phase (n=7) in comparable with others. The peaks mentioned by (111), (0020),(1111), (1113), (1117) were the most probable peaks at (n=7) and belong to Cu-O bond. The reason for that, are approaching the quantity of oxygen excess to saturation and the last has the effect to increase the correlation between Cu and O-atoms within the structure.

The XRD pattern of the composition $Y_7Ba_5Cu_{12}O_y$ have the most three highest intensity peaks were (0111), (106), and (111), and the lower intensity were mentioned by the peaks (0018), (020), (2012), (0131) at the position (2θ=31.6, 46.66, 52.4, 61.4), these planes may be attributed to form Cu-O bonds. The analytical results of XRD pattern for $Y_7Ba_5Cu_{12}O_y$ was exhibited orthorhombic phase with lattice constant (a=3.62 Å, b=4.01 Å, c=52.31 Å) as shown in Table 1. There is a slightly variation of lattice constants in comparable with others, that is related to slightly variation in the position of diffracted angles, secondly there is vanishing and creation in some peaks appeared.

Actually the changes in the unit cell structure are a function of (n), the last are the reason to create or diminish the planes within the structure. There is more reduction in a-axis of the unit cell and slightly increasing in the b-axis. This variation is proportional to different values of (n=3, 5, 7). That is related to more dense of Y-atom within the unit cell, which was the reason to more insertion of O-atom within the unit cell especially in the basal plane. In the same time, there is increasing in the c-axis regarding to this conditions. The lattice parameter variation as a function of Y-atom concentration and Cu-O layer inserted in the structure, which are the reason to presence the defect concentration and producing the nonstoichiometric composition. That is might be attributed to the shrinkage in the basal plane with the unit cell. The interpretation for that is the reduction of the vacancies or oxygen sites in the basal plane, which was confirmed with $T_{c,}$ concluded. The CuO_2 layers inserted as a function of increasing (n), it was well defined with increasing Y-atom concentration and the number of CuO_2 layer led to linear increasing in b,c-axis [6], and the c-axis was increased five time of Y123, in contradiction with previous studies [9,10] .On other hand, the parameter (a) showed a slightly decreasing keeping the orthorhombicis still present. The orthorhombicity of samples was increased with n-values as mentioned in Table 1 that means there is enhancement in the orthorhombic phase toward the higher value of (n), for (n=3) the orthorhombicity is half of the calculated value by Kruaehong [9].

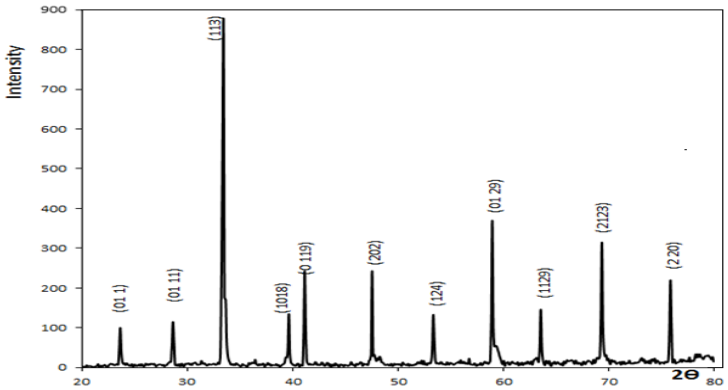

Figure1. *The XRD pattern for $Y_3Ba_5Cu_8O_{16}$*

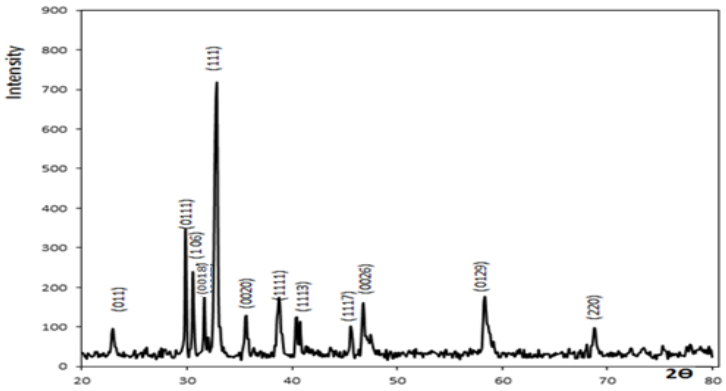

Figure2.*The XRD pattern for $Y_5Ba_5Cu_{10}O_{22.5}$*

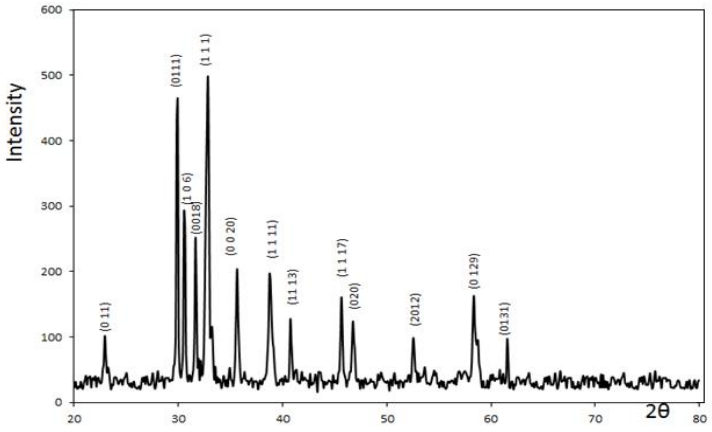

Figure3. *The XRD pattern for $Y_7Ba_5Cu_{12}O_{27.5}$*

Table1. *The dependent parameter of $Y_nBa_5Cu_{n+5}O_y$*

Sample	a Å	b Å	c Å	Orthorhombicity 100(b-a)/(b+a)	δ	T_{c1} K	T_{c2} K
Y358	3.852	3.882	50.262	0.387	0.18	128	113.6
Y5510	3.777	3.99	51.93	2.742	0.21	121	113
Y7512	3.62	4.01	52.31	5.111	0.22	128	105

The results of resistivity as shown in Figs. 4, 5, 6 for different values of (n) in the composition $Y_nBa_5Cu_{n+5}O_y$. The superconducting behavior was present at different T_c-value decreasing of T_c-value with increasing of (n) or the Y-atoms concentration in the mixture. The sample $Y_3Ba_5Cu_8O_{18}$ that was sintered at (900^oC) for (48hr) has two steps in the resistivity behavior, as shown in Fig. 4, in contradiction with previous studies [7,8], they found a single phase of Y358. The first step was in the temperature range (T= 260-125 K) and the second step was in the range (T=125-110 K). That is related to presence of multi-phase superconductor with polycrystalline structure as mentioned before in X-ray diffraction results. There are two critical temperature was about (T_{c1}=128 K) and (T_{c2}=113.6 K), so it was concluded that there is a vortex state within the temperature range (T_c=128-113.6 K) that was clear from the multiphase appeared as mentioned before. The value of (ΔT_c) was measured, it was (ΔT_{c1}=10K) for the first step and (ΔT_{c2}=1.3K) for the second one. The presence of superconducting domain in a vortex state is considering the key to distribute these domains near the surface for the first step and others in the center of the sample. This is the reason why the presence two different values of (ΔT_c) with different temperature range. It is well define that the penetration depth has a direct effect on the distribution of these domains, so it was preferable the compatible between the long range of (ΔT_{c1}) and the penetration depth.

The resistivity measurement for the sample $Y_5Ba_5Cu_{10}O_{22.5}$was shown in Fig.5, it was exhibited that there is also two variable regions. The first region appeared in the temperature range (T=250-120K) and the second was appeared in the temperature range (T=120-110K). The abrupt variation of resistivity curve near T_c-value is well defined in comparable with previous one. There is a vortex state represented by the presence of two values of (T_{c1}=121K) for the first region and (T_{c2}=113K) for the second region. There is equal limited values of (ΔT_c) represented by (ΔT_{c1}=2.5K) for the region one

and (ΔT_{c2}=1.5K) for the region. That is making to predicate that the distribution of vortex domains is symmetric for the all sample under study regarding to the approaching in ΔT_c-values, which was agreed with the result of X-ray that was discussed before.

On the other hand, the compound $Y_7Ba_5Cu_{12}O_{27.5}$ that has vortex state also and producing two T_c-value represented by (T_{c1}=128K) within the temperature range region (T=250-125 K) and (T_{c2}=105K) with the temperature range region (T=125-103 K). The values of (ΔT_{c1}=2K) and (ΔT_{c2}=1.3 K) for first and second step respectively are shown in Fig. 6. The approaching in ΔT_c-values tends to conclude the distribution of all superconducting domains is the same along all sample dimensions. The similarity in the results appeared in Figs. 5,6 made the predication of the unique in the phase is acceptable more than the results appeared in Fig. 4. The thing is supporting this conclusion was clear for undefined peaks in the phase (n=3) rather than the phases (n=5,7). The normal resistivity for $Y_3Ba_5Cu_8O_{18}$, $Y_5Ba_5Cu_{10}O_{22.5}$ and $Y_7Ba_5Cu_{12}O_{27.5}$was exhibited different values, which were (0.035mΩ.cm), (0.79 mΩ.cm) and (14.33mΩ.cm) respectively. It was clear that there is a sharp increasing in the resistivity with increasing n-value. It is well defining the mechanism of conductivity toward the c-axis, the results showed increasing in the c-axis and the last has the direct effect on the motion free electron and in the same time increasing the scattering factor for these electrons above T_c. This is the main reason for increasing the resistivity with increasing the ratio of Y-atoms in the mixture.

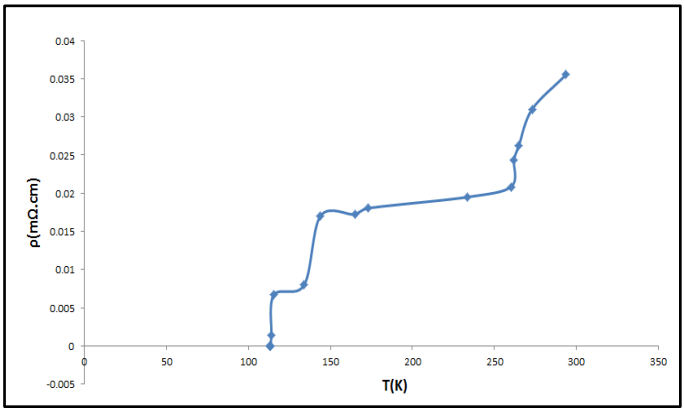

Figure4. *The resistance as a function of temperature for $Y_3Ba_5Cu_8O_{18}$.*

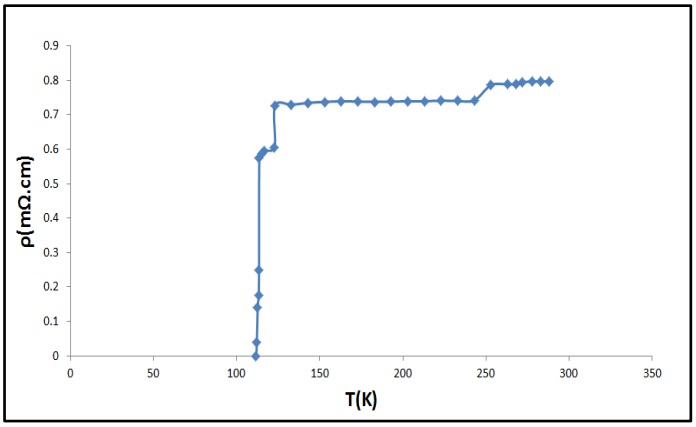

Figure5. *The resistance as a function of temperature for $Y_5Ba_5Cu_{10}O_{22.5}$.*

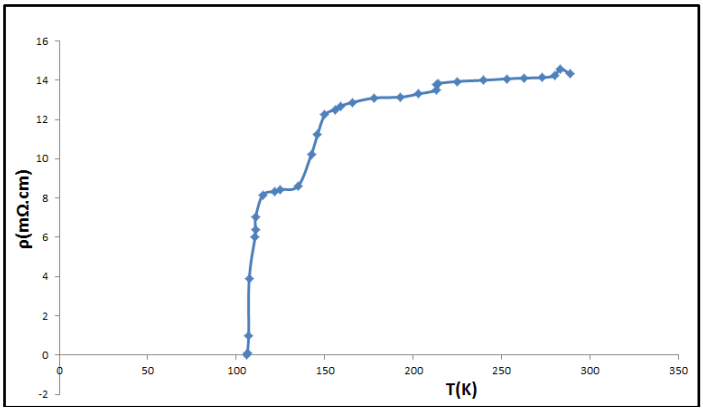

Figure6. *The resistance as a function of temperature for $Y_7Ba_5Cu_{12}O_{27.5}$.*

The SEM photographs of pure YBCO samples are shown in Fig.7a, b, c, for different values of (n). There are large grains randomly distributed with a size range (1-6 μm) for $Y_3Ba_5Cu_8O_{18}$as shown in photo (a). While the compositions $Y_5Ba_5Cu_{10}O_{22.5}$ and $Y_7Ba_5Cu_{12}O_{27.5}$ have grains appeared to be more familiar and homogenous with the size range (4-0.7 μm) as shown in photos b, c. The emergences of interfaces channels between the grains tend to reduce the grain boundaries, in agreement with Srinivasan et al [10], and enhancement the physical properties of this mixture as a function of (n) except the composition $Y_3Ba_5Cu_8O_{18}$ that shows a multiphase sample. These results were emphasized by the results of XRD analysis and resistivity measurements.

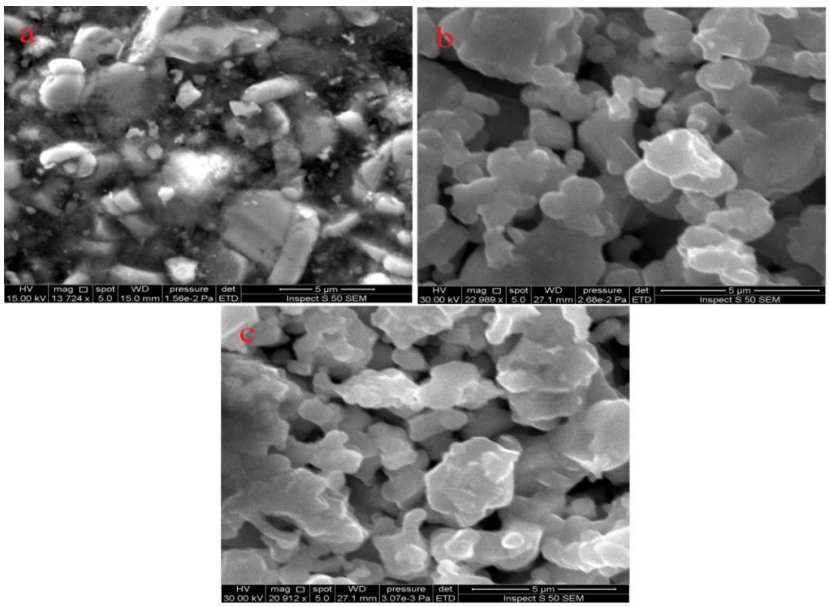

Figure7. *SEM micrographs of Y-family. (a) $Y_3Ba_5Cu_8O_{18}$, (b) $Y_5Ba_5Cu_{10}O_{22.5}$, (c) $Y_7Ba_5Cu_{12}O_{27.5}$.*

4. CONCLUSION

The presence of superconductivity, in the composition $Y_nBa_5Cu_{n+5}O_y$for (n=3, 5, 7), is reasonable is spite of decreasing the critical temperature. That is related to increasing Y-Cu-O layers within the unit cell. The important thing that was noticed form this research is the phase homogeneity created at (n=5, 7) rather than the phase (n=3). This reality was investigated through XRD analysis during the unknown peaks and diminished at (n=5, 7) and the values of (ΔT_c) are approaching value rather than to (n=3). The last reason is the homogeneity of surface morphology for (n=5, 7) rather than to (n=3). It is possible to conclude that there is ability to presence a new composition of Y-Ba-Cu-O exhibited HTSc. The grown phase is orthorhombic structure with limited increasing in b-axis and sharp increasing in c-axis. That is investigated during much insertion of Y-Cu-O layers within the structure.

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