

**Using X-ray and scanning electron micrograph to
calculate some of superconducting properties of
 $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ system**

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Abstract

The study was carried out to investigate the substitution effect of Copper (Cu) by Praseodymium (Pr) on the superconducting properties of Yttrium, Barium, Copper Praseodymium Oxide ($YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$) a system with ($0.01 \leq x \leq 0.08$). The samples were prepared by using the solid-state reaction method. The phase and lattice parameters of the compounds were determined by X-ray powder diffraction using CuK_α radiation. The size and morphology of grains were observed by a

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scanning electron micrograph (SEM). The transition temperature, T_c was determined from the temperature dependence of resistivity, which was measured utilizing the standard four-point probe method. The electrical resistivity was measured by using Van der Pauw's method. The X-ray diffraction patterns showed a single phase with an orthorhombic structure for all samples. The SEM indicates that average grain size decreased while the pores and the gaps increased with increasing Pr concentration. The superconducting transition temperature T_c decreased with increasing Pr concentration. The superconducting transition width and the electrical resistivity increase with increasing Pr concentration. The measurement of electrical resistivity versus temperature indicates the metallic normal state behavior for all samples for $T > T_c$.

Keywords: SEM, YBCO, $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$, Resistivity, X-ray.

Introduction

In recent years, $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ the system has become one of the most studied topics in the field of high-temperature superconductivity to improve the superconducting properties of (YBCO) system [1-3]. Many studies research is restricted to the effect of substitution of chemical elements for Cu and some of them limited to the effect of substitution of Pr for Y and Ba. To determine the effects of Pr-doping high- T_c superconductors in $Y_{1-x}Pr_xBa_2Cu_{3-x}O_{7-\delta}$ ($0 \leq x \leq 0.3$) have previously investigated by Harada and Yoshida [4,5]. These studies have exposed the significance of Pr for superconductivity in the $Y_{1-x}Pr_xBa_2Cu_{3-x}O_{7-\delta}$ the system, to determine the Pr valance state in YPBCO system that sense to the effect on T_c which drops linearly with substituting metal concentration is equal to $x=0.1$ [6,7], many experimental and theoretical studies have been completed. It has been documented that the Cu-Pr chains act carrier and reservoirs and superconductivity occurs in the CuO_2 planes which are hole conductors.

Harada and Yoshida [5] have indicated for the effect of Pr substitution at Ba position in $Y(Ba_{2-x}Pr_x)Cu_{3-x}O_{7-\delta}$ with ($0 \leq x \leq 0.15$). They found that all the samples consist of a layered orthorhombic perovskite-like structure with Pmmm symmetry[8-10]. By increasing x the transition temperature T_c of superconductor of Pr substituted samples decreases monotonically.

The previous study for $YBa_2Cu_{3-x}O_{7-\delta+x}Ni$ system has presented that the superconducting state properties improved, and then dropped when adding the nickel (Ni) element to the system under the study [17]. This study has introduced the optimal ratio between carrier concentration and impurity phase BeCl₂ when the Calcium (Ca) and Potassium (K) elements have been added as defects to improve the superconducting properties [18,20], which has pushed us to study the superconducting properties for the same system by adding Praseodymium Oxide (Pr) element. In addition, one of the main significance of the superconducting properties is the resistivity, where doping the Magnesium (Mn) Substituted in the $Y_{1-x}Mn_xBa_2Cu_3O_{7-\delta}$ system has given importance to the resistivity results that showed the shifting in $T_c(R = 0)$ towards low temperature as the Mn concentration increases [19]. One of the studies has published that the Substitution of more than 2% of magnesium (Mg) in $YBa_2Cu_3O_{7-\delta}$ system causes an increase in spurious phases, and stated that direct current (DC) susceptibility measurements show that superconducting transition temperature T_c is reduced much more by Ca than Mg [21]. A Paper has published that the increase of the structural parameters as La concentration in the $YBa_2(Cu_{1-x}La_x)_3O_{7-\delta}$ system with increases the distance came as a result of the substitution of Cu by La [22].

In this paper, we estimated the changes in properties of superconducting and normal-state transport of $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ [11], the measurements have presented for resistance at various temperatures, room temperature resistivity, and X-ray powder diffraction (XRD), and scanning electron microscopy (SEM).

Experimental Details

In this work, the samples are bulk ceramic $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$, where ($x = 0.01, 0.03, 0.06$ and 0.08) were synthesized by the solid-state reaction method. High-purity (99.99%) Y_2O_3, BaO, Pr_6O_{11} , and CuO powders were mixed in appropriate proportions. The samples preparation by the following procedures, weight the powder Y_2O_3, BaO, Pr_6O_{11} , and CuO , mixing and grinding the powder for an hour, heat the powder at $880^\circ C$ for 24 hours with several intermittent grinding and oven cooled and following by furnace cooling to room temperature at $\sim 40^\circ C/hr$, regrind and reheat the powder, and press the powder into pellets with about 12mm diameter and 2mm thick, and heat at the same temperature for another 24 hours. The electrical resistance-temperature at T_c for samples were measured by a four-point probe technique using silver paint contacts in conjunction with a closed-cycle refrigerator from *CTI Cryogenics Model 22* [12]. The temperature was cooled down at a cooling rate of $60^\circ C/hr$ and heated up at the same rate. Measuring of the resistivity at room temperature has carried out using *Van der Pauw* method, calculation of samples resistivity has measured by the following equation

$$\rho = \frac{\pi d V_{CD}}{\ln 2 I_{AB}} \text{ --- --- --- (1)}$$

Where ρ is the resistivity, d is the sample thickness, V_{CD} is the voltage between point C and D, and I_{AB} is the current through the point A to B.

Samples have analyzed by powder X-ray diffraction using D 8 advance diffract meter with $Cu - K_{\alpha}$ radiation. Micrographs of the Scanning Electron Microscopy (SEM) have recorded using Philips XL30 scanning electron microscope and uses electrons rather than light to form an image that to examine the morphology of the samples at different volume fractions.

After a brief discussion of experimental procedures, we will present our results for T_c versus x which are in substantial agreement with the resistivity determined result by others[13-15]

Results and discussion

In the present study, the zero-resistance temperature T_c , the onset temperature $T_{c\ onset}$ values, superconducting transition width ΔT_c for four samples with a composition ($x=0.01, 0.03, 0.06$ and 0.08) and pure YBCO are shown in Table 1. shown in Figure.1 is the electrical resistance against temperature for four samples, apart from this, in Figures from 2 to 5 for the same number of samples show the electrical resistivity versus temperature. Figure 6 shows the zero resistance temperature as a function of x . Substitution of Pr in Cu position with composition $x=0.01, 0.03, 0.06$ and 0.08 led to increasing Pr concentration which caused to decrease $T_{c\ zero}$ from 85K to 79 K in the system. In addition to the decrease in $T_{c\ onset}$ which are 90 K for $x=0.01$ and 89 K for $x= 0.03$. It also shows a slightly higher increasing at $x=0.06$ with $T_{c\ onset}$ is 93 K than for the pure YBCO where $T_{c\ onset}$ is 92K and for $x=0.08$,

$T_{c\ onset}=87\text{K}$. Our results have shown in good agreement with previous studies Harada and Yoshida, and Neumeier et a [4,5,10].

Table 1 Onset temperature ($T_{c\ onset}$), zero-resistance temperature ($T_{c\ zero}$), transition-width (ΔT_c) for pure YBCO and $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$

x	$T_{c\ onset}$ (K)	$T_{c\ zero}$ (K)	ΔT_c (K)
0	92	87	5
0.01	90	85	5
0.03	89	82	7
0.06	93	82	11
0.08	87	72	15

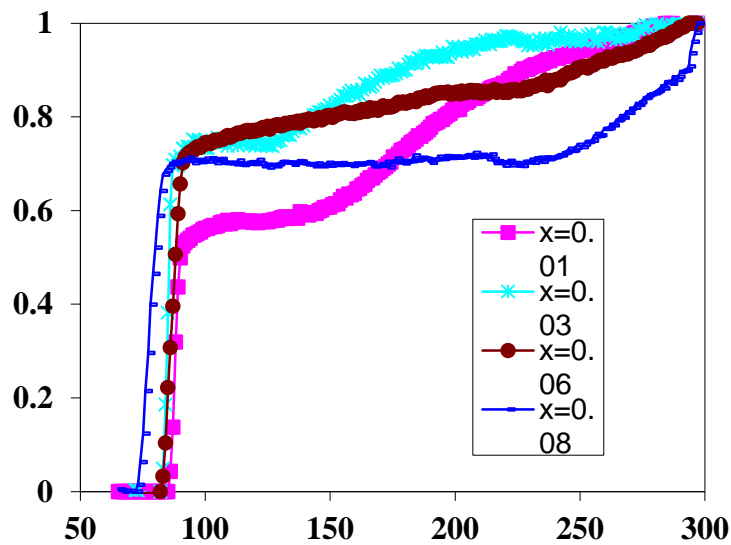


Figure (1) The temperature dependence of the normalized resistance (R/R(300 K)) for (x=0.01, 0.03, 0.06 and 0.08)

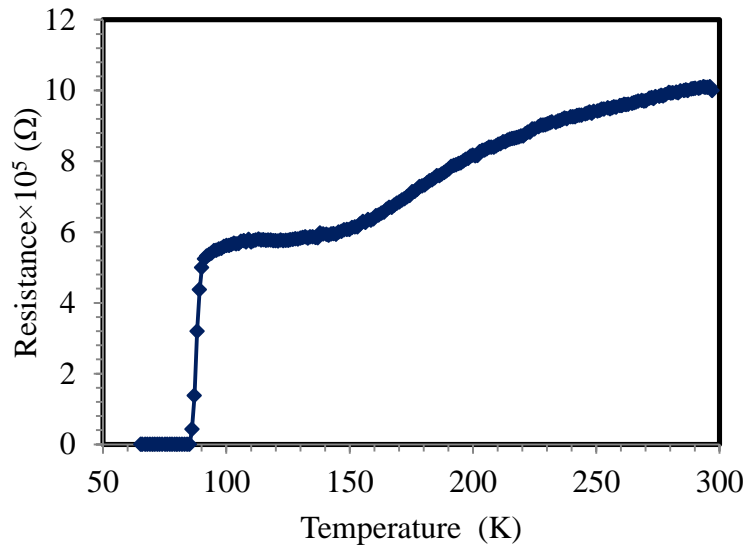


Figure (2) Dc resistance versus temperature for $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ with composite ($x = 0.01$)

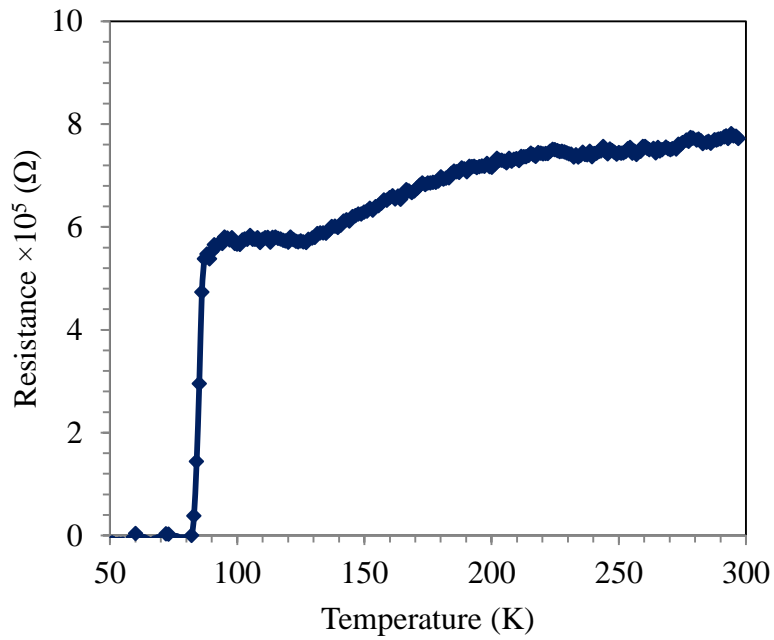


Figure (3) Dc resistance versus temperature for $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ with composite ($x = 0.03$)

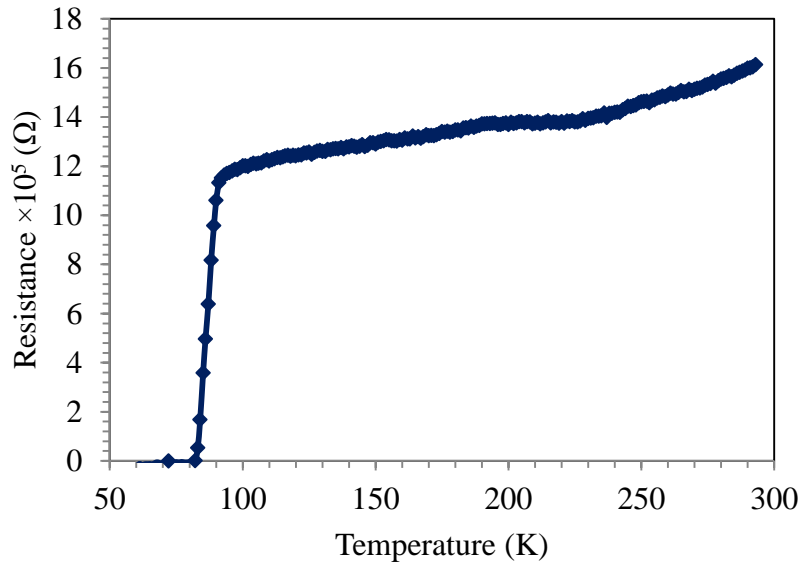


Figure (4) Dc resistance versus temperature for $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ with composite ($x = 0.06$)

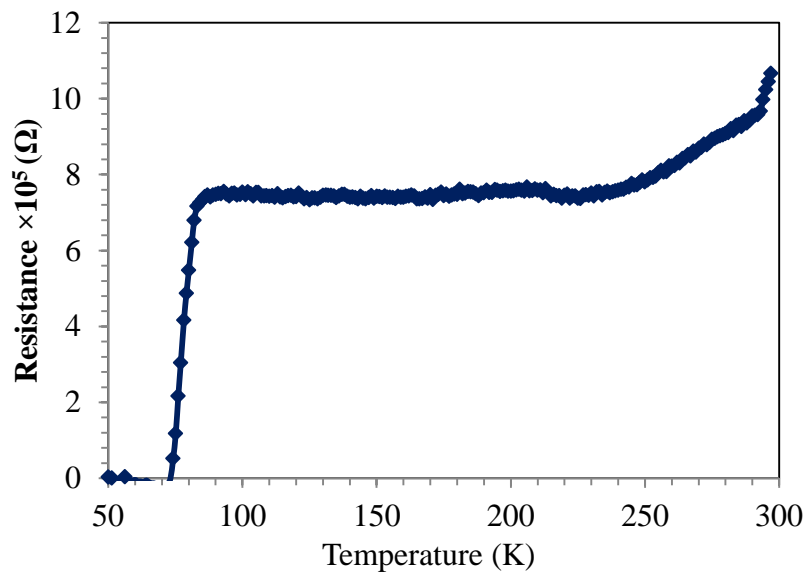


Figure (5) Dc resistance versus temperature for $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ with composite ($x = 0.08$)

This repression has been explained by two different techniques. In one it was assumed that Pr entered in the 3+ valence state. In this case, it approved a magnetic moment, and it was assumed that this magnetic moment repressed superconductivity by magnetic pair breaking. The other mechanism suggested that the Pr entered in the 4+ valence state. In this state, it was hypothesized that it was energetically propitious for the Pr^{+4} to remove mobile holes from the conducting levels. It is widely accepted that T_c relies on mobile carrier density; it was as a consequence reasoned that the elimination of mobile holes by Pr^{+4} led to the suppression of T_c . unluckily, neither of these two proposed mechanisms was eligible to account satisfactorily for all the experimental observations made on the substitution of Pr on the YBCO system [16].

The graph is shown in Figure (6) which represents the linear suppression of T_c against x data for $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ within ($0 \leq x \leq 0.03$) is in good similarity with the theoretical predictions based on the theory of Abrikosov and Gorkov which has been interpreted as evidence for magnetic pair breaking effects. The Pr^{+3} has the largest ionic radius and therefore the greatest amount of overlap between its 4f with the neighboring oxygen and copper atoms. Consequently, Pr 4f states can hybridize with the nearby CuO_2 valence band states ($0\ 2p - \text{Cu}\ 3d$) associated with the CuO_2 planes. Hybridization could generate an appreciable exchange interaction between the Pr magnetic moments and the spin of the mobile hole in CuO_2 planes resulting to break the cooper pairing which then leads to suppression of the superconductivity as concentration x increase. While the curve dependence of T_c that depicted in Figure (6) closely corresponds to the hole filling effects within ($0.03 \leq x \leq 0.08$). The Pr ion exists with a valance of Pr^{+4} , the additional electron contributed by the Pr ion is expected to fill mobile

holes in the CuO_2 planes, effectively leaving the hole localized on the Pr, reducing conduction and eliminating superconductivity. Furthermore, the superconducting and transition width between T_c onset and the zero resistivity temperature T_c zero [$\Delta T_c = T_{c \text{ onset}} - T_{c \text{ zero}}$] becomes wider as x increases. This broadening of transition width may indicate that the samples are highly homogeneous.

The Van der Pauw method was used to compute the electrical resistivity of compound $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$ at room temperature, with composite 0.01, 0.03, 0.06 and 0.08. The results of the electrical resistivity for samples were using the equation (1), where the thickness of samples was (d=1.59, 1.76, 1.85, 1.77)mm, V_{average} 's were (0.041, 0.037, 0.042, 0.051)mV, and I_{average} was 55 mA. The results shown in Figure (7) are the variation of electrical resistivity as a function of x for the compound $\text{YBa}_2(\text{Cu}_{3-x}\text{Pr}_x)\text{O}_{7-\delta}$. The results of the electrical resistivity at room temperature for all samples and YBCO are shown in Table (2), the calculations have shown that the electrical resistivity of the samples increases with increasing the Pr concentration. This suggests that superconducting grains are not closely in contact with each other because of the large gaps and pores that exist. Besides might be raising a decrease in the concentration in the hole concentration on the CuO_2 plane because of the increase in the electrical resistivity. The normal state resistance-temperature curves showed a metallic behavior for all the samples. In comparison with pure YBCO. Our results for the resistivity of all samples were higher than that of the pure sample.

Table (2) The electrical resistivity at room temperature for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$

x	$\rho(m\Omega.)$	$\Delta\rho(\times 10^{-5}m.\Omega)$	Normal state
0	0.80×10^{-5}	± 0.71	Metallic
0.01	5.37×10^{-6}	± 0.14	Metallic
0.03	5.36×10^{-6}	± 0.15	Metallic
0.06	6.40×10^{-6}	± 0.16	Metallic
0.08	7.44×10^{-6}	± 0.16	Metallic

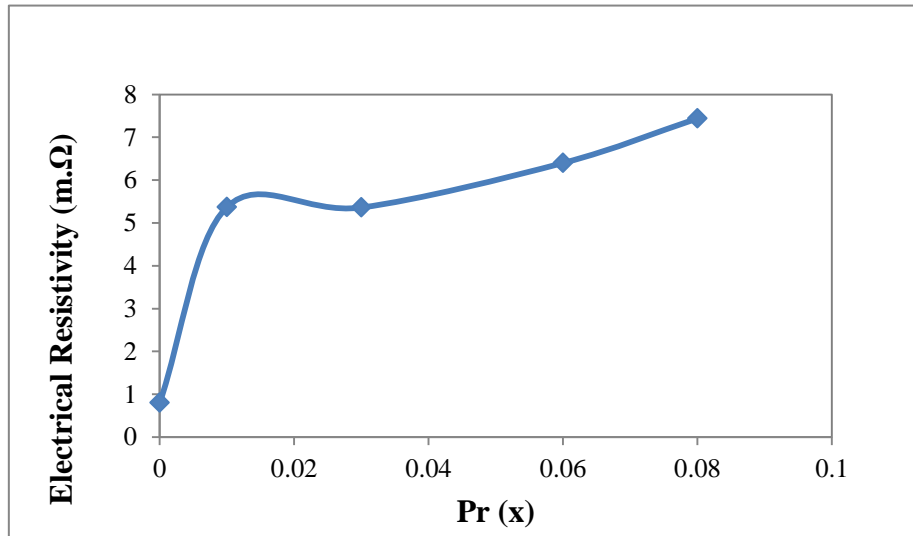


Figure (7) Dc resistance versus composition x for $YBa_2(Cu_{3-x}Pr_x) O_{7-\delta}$

Figure (8) shows the powder X-ray diffraction patterns of all the samples $YBa_2(Cu_{3-x}Pr_x) O_{7-\delta}$ for (x=0.01, 0.023, 0.06 and 0.08) and pure YBCO. Figures (6), (10), (11) and (12) show the powder X-ray diffraction patterns of all the samples. The lattice parameters and unit cell volumes of these samples are presented in Table (3). The XRD powder diffraction shows that all samples have lattice constant $a \neq b \neq c$. These indicated that all samples have an orthorhombic structure. Comparing with the pure sample; we indicated that all samples are single-phase and

there is no extra peak due to impurity phase for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ within the range of x ($0.01 \leq x \leq 0.08$).

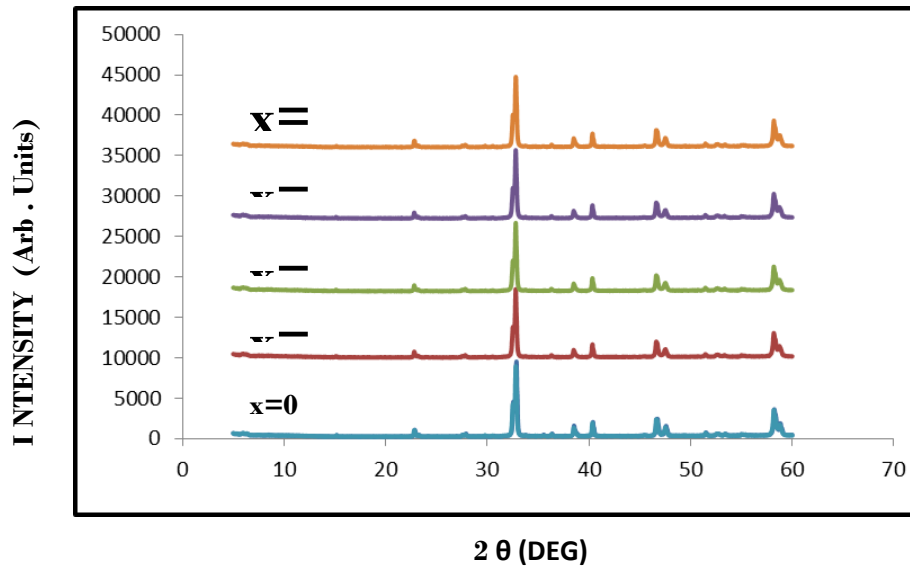


Figure (8) Intensity versus 2θ for all samples $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$

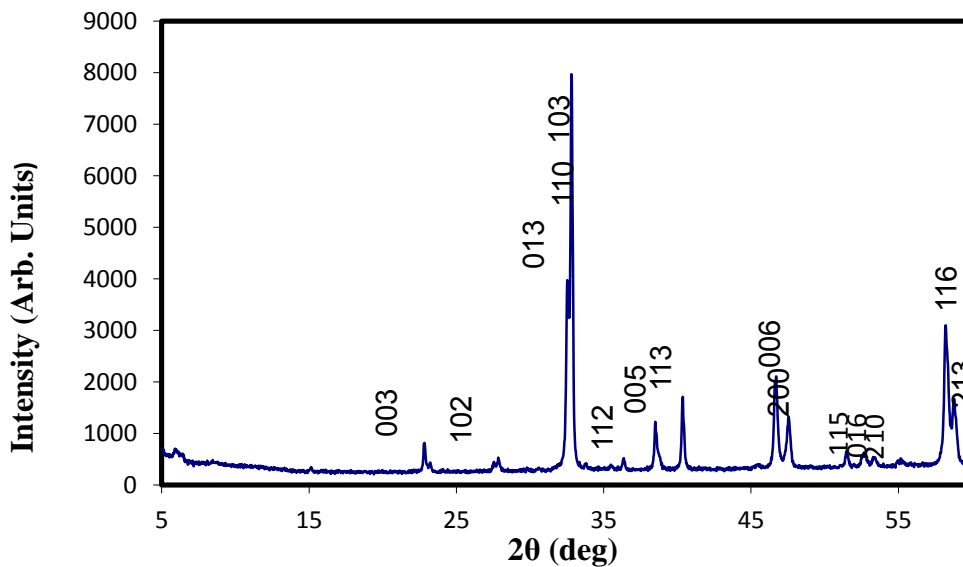


Figure (9) Intensity versus 2θ for sample $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with composite $x = 0.01$

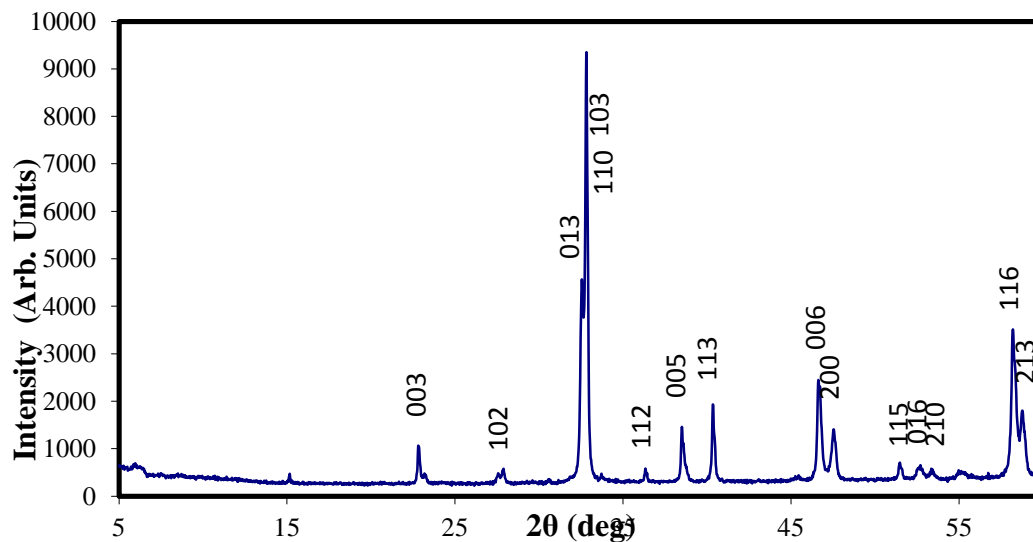


Figure (10) Intensity versus 2θ for sample $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with composite $x = 0.03$

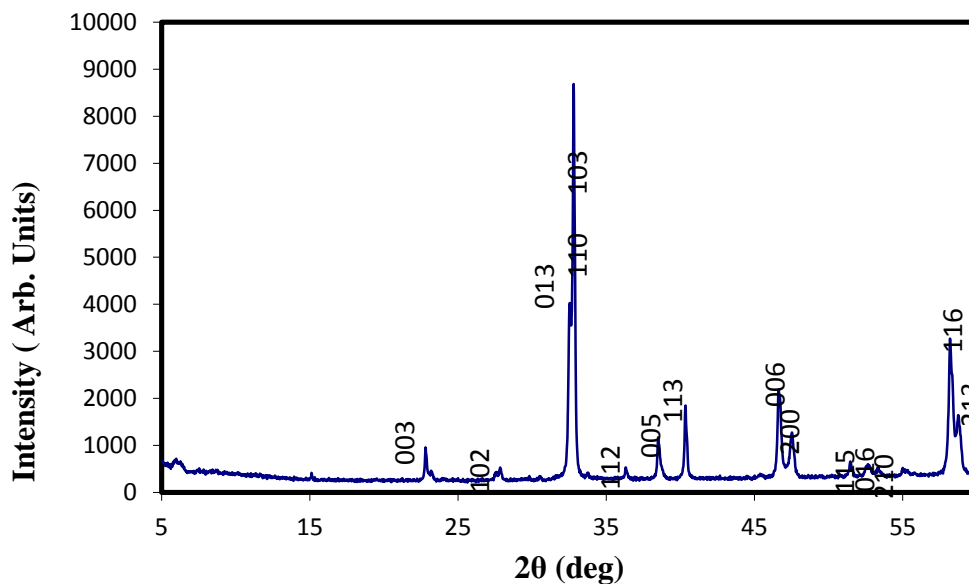


Figure (11) Intensity versus 2θ for sample $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with composite $x = 0.06$

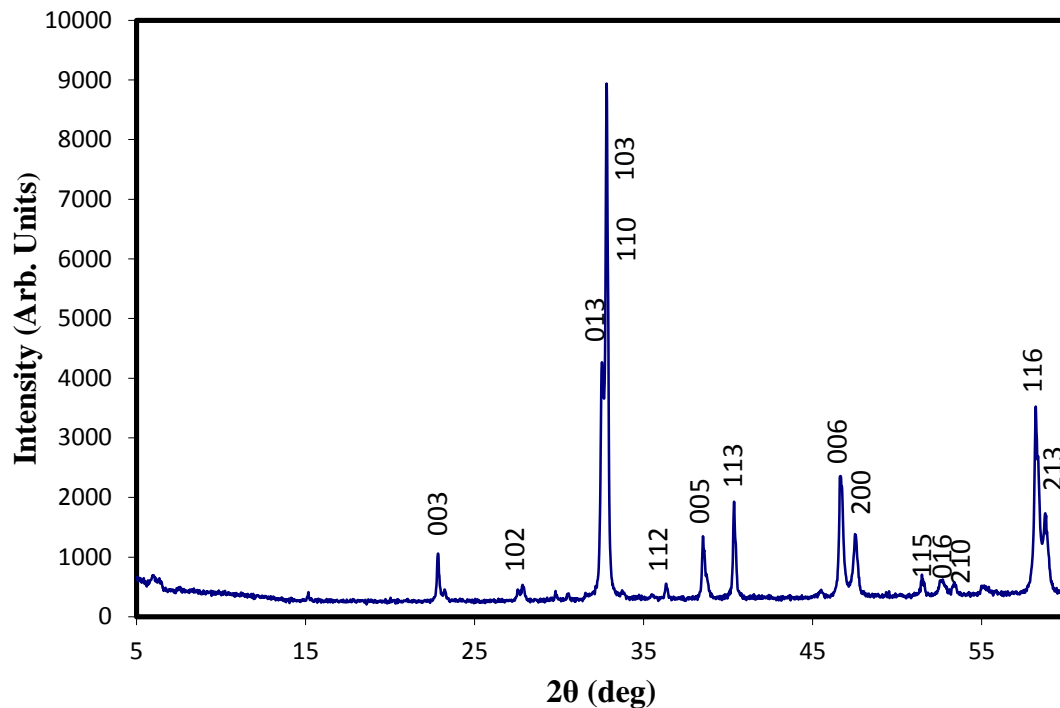
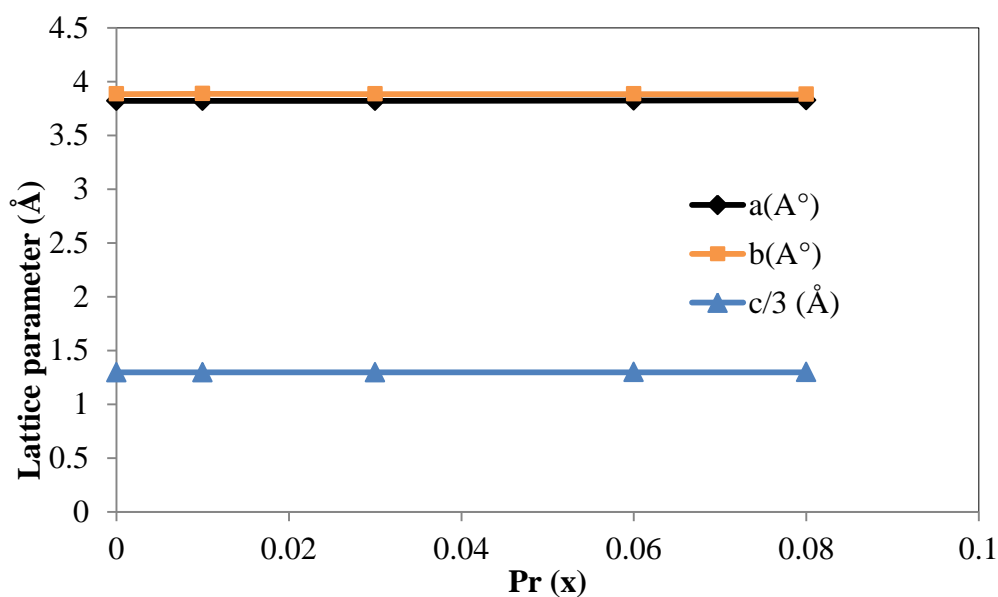


Figure (12) Intensity versus 2θ for sample $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with composite $x = 0.08$

From the Table (3) we can observe that the lattice parameter a is in the range of 3.821 \AA to 3.827 \AA , the lattice parameter b is in the range 3.888 \AA to 3.882 \AA and lattice parameter c is in the range of 11.671 \AA to 11.679 \AA . As a result, we can recognize that these lattice parameters for the samples are very close to those of pure YBCO. These results indicate that the structure and lattice parameters of the $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ samples and YBCO are very compatible. Furthermore, there is no significant effect on the effect on the crystal structure by Pr substitution in YBCO within the range of x ($0.01 \leq x \leq 0.08$). The variation of lattice parameters a , b and c as a function of x for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ the system is shown in Figure (13).

Table (3) The lattice parameters for pure YBCO and $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$

x	a (Å) ± 0.001	b (Å) ± 0.001	c (Å) ± 0.001	$V(\text{Å}^3) \pm 0.1$
0	3.823	3.886	11.671	173.38
0.01	3.821	3.888	11.671	173.38
0.03	3.822	3.886	11.671	173.34
0.06	3.825	3.886	11.678	173.58
0.08	3.827	3.882	11.679	173.50

**Figure (13) Lattice parameters versus x for the $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ and pure YBCO**

The scanning electron microscopic (SEM) images of the prepared samples ($x=0.01, 0.03, 0.06$ and 0.08) are shown in Figures 15, 16, 17, 18 and 19 respectively. Figure 18 shows the grain size versus composition x for all samples. The micrographs show that the grain morphology for all samples is changed with different Pr concentration. The mean grain size is $2 \mu m$. The micrographs of $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with $x=0$ and 0.01 are denser than higher compositions ($0.03 \leq x \leq 0.08$). The

micrographs show that the existence of the pores and large gaps increases with increasing Pr concentration. The grain size for samples $x=0.01$ is $2.17\mu\text{m}$, $x=0.03$ is $1.81\mu\text{m}$, $x=0.06$ is $1.76\mu\text{m}$ and $x=0.08$ is $1.67\mu\text{m}$. This indicated that the average grain size decrease with increasing Pr concentration. The decrease in the grain size is accompanied by an increase in the resistivity. The behavior could be explained by assuming that decreasing the grain size decreases the carrier mobility (mobile hole). This decrease in the grain size induces an increase in the grain boundary that carrier has to cross which leads then to an increase of the resistivity.

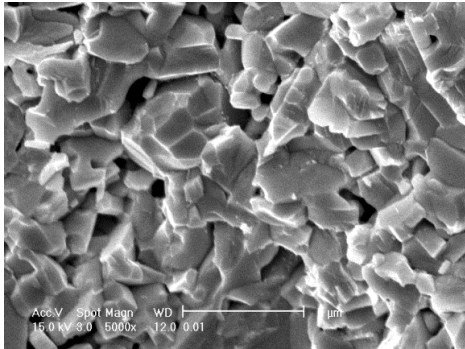


Figure 15 Scanning electron micrograph for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ ($x=0.01$)

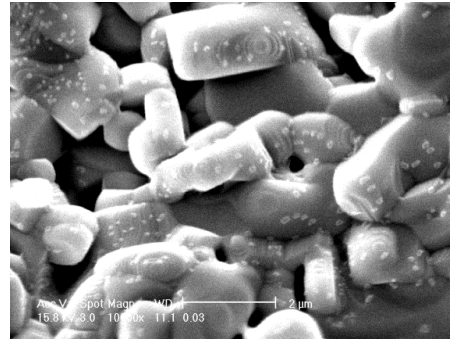


Figure 16 scanning electron micrograph for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ ($x=0.03$)

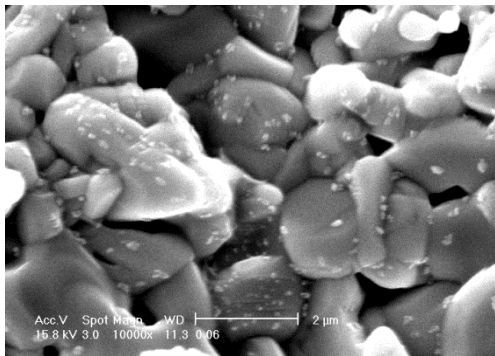


Figure 17 Scanning electron micrograph for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ ($x=0.08$)

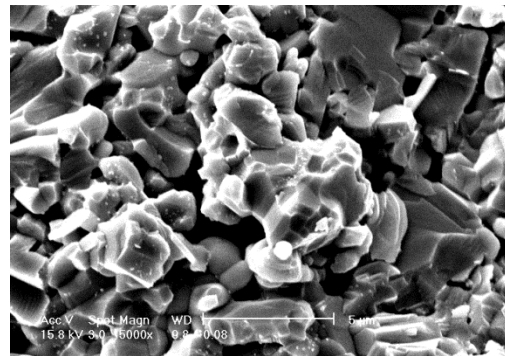


Figure 18 scanning electron micrograph for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ ($x=0.06$)

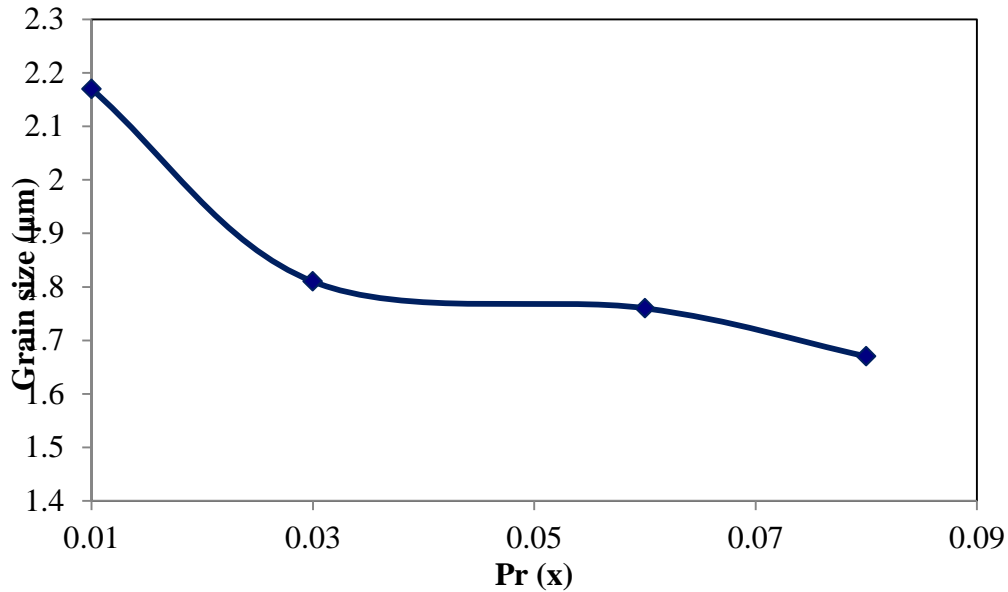


Figure (19) The grain size versus composition x for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$

CONCLUSION

In conclusion, the data presented here investigate the substitution effect of Pr at Cu site on superconducting properties of the YBCO system. Based on the results of the electrical resistivity against temperature, all samples of $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ with composition $x = 0.01, 0.03, 0.06$ and 0.08 are superconductor. The $T_{c\ zero}$ all samples were found to decrease with increasing Pr concentration and the T_c onset also decreased but it showed an increase when $x=0.06$ which is slightly higher than of pure YBCO. While the superconducting transition width (ΔT_c) for all samples increased with increasing Pr concentration. The x-ray diffraction patterns showed a single phase for all samples and there is no extra peak due to impurity phase for $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ within the range of x ($0.01 \leq x \leq 0.08$). The XRD powder diffraction also showed that all samples have an orthorhombic structure with a lattice constant by

the substitution of Pr on $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ within the range of x ($0.01 \leq x \leq 0.08$). The electrical resistivity at room temperature for all samples $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ within the range, x ($0.01 \leq x \leq 0.08$) was found to increase with increasing Pr concentration. The measurement of electrical resistance versus temperature indicates metallic behavior for all samples. The scanning electron microscopy (SEM) for all samples showed that the average grain size decreased with increasing Pr concentration. It as well showed that the existence of the pores and large gaps increasing Pr concentration. The suppression of T_c with Pr content on $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ the system has been explained by two mechanisms. In one of the mechanisms, if the Pr ion exists with a valence of Pr^{+4} this implies that electron removed from Pr ion located in the CuO_2 plane thus filling of holes. This means that Pr^{+4} ion compared to the Cu^{+2} state each Pr atom adds at least one electron to this system, thereby reducing the concentration of the carrier holes. As a result suppression the superconductivity of $YBa_2(Cu_{3-x}Pr_x)O_{7-\delta}$ system. The other mechanism proposed that, if the Pr ion exists with a valence state Pr^{+3} in this case it carried a magnetic moment that suppressed the superconductivity due to the overlap of the $Pr4f$ orbital's with $O2p - Cu3d$ orbital's in the CuO_2 plane leading to Abrikosov and Gorkov type pair breaking.

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