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## Superconducting Properties of Bi<sub>2-x</sub>Pb<sub>0.3</sub>W<sub>x</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> Compounds

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#### Abstract

The conventional solid-state reaction method was utilized to prepare a series of superconducting samples of the nominal composition  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  with  $0 \le x \le 0.5$  of 50 nm particle size of tungsten sintered at  $850^{0}C$  for 140h in air . The influence of substitution with W NPs at bismuth (Bi) sites was characterized by the X-ray diffraction (XRD), scanning electron microscopy (SEM) and dc electrical resistivity. Room temperature X-ray diffraction analysis revealed that there exists two phases, i.e. Bi-(2223) and Bi-(2212), in addition to the impurity phases of (SrCa)  $_{2Cu2O3}$ , Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>7</sub>O<sub> $\delta$ </sub>, Ca<sub>2</sub>PbO<sub>4</sub>, CaO, and WO. It was found that the crystallographic structure of all samples was orthorhombic. Lattice parameter values and the volume fraction of the (2223)-phase of the prepared samples were also calculated. The superconductivity transition temperature (T<sub>c</sub>) for samples subjected to substitution with W NPs was found to be higher than that for the pure sample. The optimal value of W NPs content in (Bi, Pb)-2223 system was found to be at x=0.3.

Keywords: Bi-2223 superconductors; X-ray diffraction; W substitution

# خصائص التوصيلية الفائقة للمركبات Bi<sub>2-x</sub>Pb<sub>0.3</sub>W<sub>x</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub>

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#### الخلاصة

50 من التنكستن بطريقة تفاعل الحالة الصلبة التقليدية عند درجة حرارة تلبيد  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  لحجم جسيمات 50 من التنكستن بطريقة تفاعل الحالة الصلبة التقليدية عند درجة حرارة تلبيد  $850^{0}$  لمدة 140 ساعة في الهواء . درس تأثير التعويض الجزئي بالتنكستن النانوي محل *Bi* بواسطة حيود الأشعة السينية (XRD)، المجهر الإلكتروني الماسح (SEM) والمقاومية الكهربائية dc بواسطة حيود الأشعة السينية عند درجة حرارة الإلكتروني الماسح (SEM) والمقاومية الكهربائية dc بواسطة حيود الأشعة السينية (XRD)، المجهر الإلكتروني الماسح (SEM) والمقاومية الكهربائية dc بواسطة حيود الأشعة السينية عند درجة حرارة الإلكتروني الماسح (SEM) والمقاومية الكهربائية dc و (2212) ما الضافة الى اطوار الشوائب مثل الغرفة وجود طورين وهذا يعني (SEC)–Bi و (2212)–Bi اضافة الى اطوار الشوائب مثل الغرفة وجود طورين وهذا يعني (Cac <sub>1</sub>Ca<sub>2</sub>PbO<sub>4</sub> ، Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>7</sub>O<sub>8</sub> ور220<sub>2</sub>Cu<sub>2</sub>O<sub>3</sub> والمقاوني مثل العربية عند درجة حرارة العربيات كان معيني قائم. كما تم حساب قيم ثوابت الشبيكة، النسبة الماوية الور – (2223) للنماذج المعوضة . كان معيني قائم. كما تم حساب قيم ثوابت الشبيكة، النسبة الماوية لوجود الطور – (2223) للنماذج المعوضة بالا النانوي لها قيمة المادي النماذي النماذ المعوضة بالا النانوي لها قيمة اعلى من النماذج المعوضة . كان نسبة التعويض المثلى للا لا النانوي كانت 3.0 ×

#### I. INTRODUCTION

(Bi, Pb)-2223 phase has been attracting a substantial interest among the Bi-based high- $T_c$  superconductors (HTSCs) due to the high transitional temperature of their superconductors. Intergrains connectivity is a very important parameter for the enhancement of superconducting properties of a bulk of HTSCs from an application point of view. Numerous studies of substituting into superconductor oxide ceramics are ongoing to develop the superconducting qualities of this phase. The physical properties depend largely on the details of the initial elemental composition and not only the method of preparation.

A large number of researches were carried out that investigated the effect of the substitution of various elements, e.g., Nb [1], Eu [2], Ni [3], Nd [4] and Mn [5], on superconducting properties of (Bi, Pb)-2223 ceramic. Abbas *et al.* investigated the influence of Cu [6], Gd [7], As [8] and Sb [9], but the addition of most suitable nanosized structures is still preferred for the improvement of superconducting parameters. (Bi, Pb)-2223 superconducting phase was prepared with the addition of nanoparticles, e.g., Sm [10], Al [11] and Sn [12], in order to increase inter-grains connectivity.

Aljurani *et al.* reported that the addition of metallic nanoparticles of  $\gamma Al_2O_3$  and  $SnO_2$  to polycrystalline (Bi-Pb)-2223 superconducting phase increased the volume fraction and the superconducting transition temperature [11, 12].

The suppression of superconducting properties was observed by Baqoah *et al.* after the addition of magnetic nanoparticles of  $Sm_2O_3$  in (Bi-Pb)-2223 compound [10]. However, the addition of nanoparticle elements may cause different effects on superconducting properties of high temperature superconductors [13].

Agil *et al.* [14] reported the substitution of W and Mo together on  $(BiPb)_2W_xSr_2Ca_3Cu_{4-y}Mo_yO_{12+\delta}$  system. of the results demonstrated that the substitution of (Mo/W) in (BiPb) -2223 system leads to lowering T<sub>c</sub> slightly.

The substitution of W on Cu sites in (Bi, Pb)-2223 phase led to transform the (Bi, Pb)-2223 phase gradually into the (Bi, Pb)-2212 phase [15].

From the outcome of the literature surveying, one can find that no attempt has been made to investigate the effect of the substitution of W nanoparticles on the Bi-based high temperature superconductors.

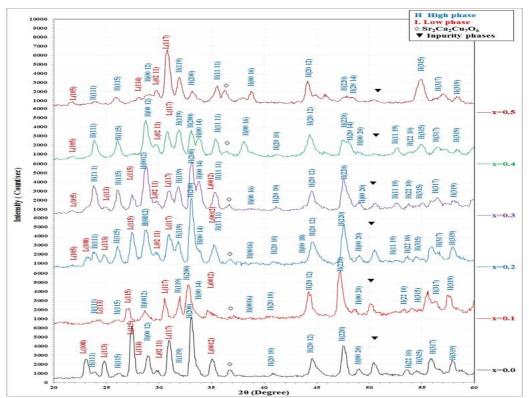
In this study, the effect of W nanoparticles' substitution on the structural, microstructural, and superconducting properties of bismuth-based compounds is investigated.

#### **II. EXPERIMENTAL TECHNIQUES**

High temperature superconductors of the nominal composition  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  system with  $0 \le x \le 0.5$  for 50 nm particle size of tungsten were prepared by a conventional solid-state reaction technique. To prepare samples, different and suitable weights of high purity materials (99.9%) of  $Bi_2O_3$ , PbO, SrCO<sub>3</sub>, CaO, WO<sub>3</sub> and CuO were utilized. To obtain a suitable small size of particles, these materials were mixed and ground together by an agate mortar. A sufficient quantity of 2-propanol was used to homogenize the mixture. This mixture was calcined in air at 800°C for 24 h with a rate of heat of 4°C/min. The mixture obtained was then cooled down to room temperature. The resulting material was compressed using hydraulic press (Specac) under pressure of 0.7 GPa as a pellet shape. The thickness of the material ranges 2-3 mm with 13 mm diameter, which was sintered in air at 850 °C for 140 h, with continuous intermediate grindings to complete solid-state reactions. X-ray diffraction was analyzed to find out structural properties for the prepared samples, being carried out on Phillips diffractometer using  $Cu_{k\alpha}$  radiation. SEM (Tescan) was used to study surface morphology and grain structure. A standard DC four-probe technique was used to determine superconducting transition temperatures.

#### **III. RESULTS AND DISCUSSION**

Through the XRD analysis of  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  with  $0 \le x \le 0.5$  for 50 nm particle size of tungsten, it was found that the samples have an orthorhombic structure with two phases; a high- $T_c$  phase (Bi, Pb)-2223 and a low- $T_c$  phase (Bi, Pb)-2212. More than two phases may be observed as a result of the presence of stacking faults along the *c*- axis. In addition, the presence of some impurities that may be formed during the sintering process was observed. The presence of impurity phases of (SrCa)<sub>2</sub>Cu<sub>2</sub>O<sub>3</sub>, Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>7</sub>O<sub> $\delta$ </sub>, Ca<sub>2</sub>PbO<sub>4</sub>, CaO and WO leads to a decrease in the critical temperature ( $T_c$ ) of the samples.



**Figure 1-X-**ray diffraction patterns of  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  samples with  $(0.0 \le x \le 0.5)$  for 50 nm particle size of tungsten sintered at 850°C for 140h.

The volume fraction of the 2223 phase ( $V_{ph-2223}$ ) in all samples was determined by using the relation [10]:

$$V_{ph} = \frac{\sum I_{2223(peaks)}}{\sum I_{2223(peaks)} + \sum I_{2212(peaks)} + \sum I_{other(peaks)}} \times 100\%$$
(1)

where  $I_{2223}$  is the XRD peak intensity related to the T<sub>c</sub> phase (Bi-Pb)-2223,  $I_{2212}$  is the peak intensity

related to the T<sub>c</sub> phase (Bi, Pb)-2212, and I<sub>other</sub> is the peak intensity related to the other phases. As shown in Table-1, when the tungsten nanoparticles content was increased, the volume fraction of high- T<sub>c</sub> phase (2223) was increased up to x=0.3 wt%. This is due to the increase in the peaks related to high- T<sub>c</sub> phase (Bi-Pb)-2223 and the reduction in the peaks related to low T<sub>c</sub> phase (Bi, Pb)-2212. From Fig. 1, one can observe that the substitution of tungsten nanoparticles causes the peaks of high-T<sub>c</sub> phase (Bi-Pb)-2223 to shift to a low angle; However, the existence of the highest intensity peaks of (0012)H and (200)H for all samples substituted with tungsten nanoparticles proves the superconductivity and stability of the Bi<sub>2-x</sub>Pb<sub>0.3</sub>W<sub>x</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> compounds. This may be due to the increase of grain size, as will be shown in the results of SEM. One can suggest that the samples had an effect on the improvement of the high- T<sub>c</sub> phase (Bi-Pb)-2223. The results disagree with those reported by Agile *et al.* [14], Turk *et al.* [15] and Atilla *et al.* [16], who found that a small concentration of W negatively affected (Bi- Pb)-2223 superconductivity phase. This might be attributed to the substitution of tungsten on Ca and Cu sites in (Bi, Pb)-2223 phase. While this work investigated the substitution of tungsten nanoparticles at Bi sites in (Bi, Pb)-2223 phase.

Our results are in agreement with those of Aljurani *et al*. They described that the addition of metallic nanoparticles of  $\gamma Al_2O_3$  and  $SnO_2$  to polycrystalline increased the volume fraction and the superconducting transition temperature of (Bi, Pb)-2223 compound [11, 12].

From the above mentioned results, the compound showed the sensitivity of the existence of HTP to W nanoparticles content, i.e., when the W nanoparticles content was increased the HTP was also increased. This case indicated the increase of the crystalline arrangement degree, which may be caused by the localization of charge carriers.

Lattice constants *a*, *b* and *c* of the high- $T_c$  phase were evaluated by the 20 major peaks. The results of the volume of a primitive unit cell, the volume fraction of high- $T_c$  phase, and the  $T_c$  values are listed in Table I. It is revealed that the substitution with W nanoparticles improves the crystallites, increases the intensity of the peaks, and sharpens the peaks, as shown in Figure-1. The value of the lattice parameter *c* was increased slightly, as revealed in Table-1.

The ratio of c/a was also calculated for all prepared samples, as shown in Table-1. The c/a was increased slightly with increasing the content of W nanoparticles. The difference in a, b and c values affected the volume of unit cell and led to a change of density. These results indicate that W nanoparticles substitution is important to enhance the properties of the superconducting structure samples by decreasing the porosity and impurity phase. This can be attributed to the increased contact area among the superconducting grains.

x	a(Å)	b (Å)	c (Å)	c/a	V (Å3)	HTP Phase%	T <sub>c</sub> (K)
0.0	5.4080	5.3921	36.985	6.839	1078.500	56.94	116.20
0.1	5.4036	5.4114	37.107	6.867	1085.024	69.18	116.70
0.2	5.4080	5.3921	37.203	879.6	1084.854	76.71	117.50
0.3	5.3989	5.4012	37.303	6.909	1087.766	85.00	121.00
0.4	5.4172	5.3830	37.276	6.881	1087.005	81.61	116.00
0.5	5.4389	5.3524	37.057	6.813	1078.773	74.63	115.00

**Table I-**Variation in lattice parameters a, b and c, the volume of unit cells, the volume fraction of (BiPb)-2223 phase, critical temperature, and the ratio of c/a on  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  samples with  $0 \le x \le 0.5$  of 50 nm particle size of tungsten sintered at 850°C for 140h.

The temperature-dependence of the electrical resistivity was also studied. The resistivity versus temperature ( $\rho$ -T) plots for specimens of the samples of nominal composition Bi<sub>2-x</sub>Pb<sub>0.3</sub>W<sub>x</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> with 0≤x≤0.5 for 50 nm particle size are shown in Fig.2.. All samples revealed a metallic behavior, where the resistivity was decreased with increasing temperature. As shown in this figure, the substitution of W nanoparticles caused an increase in the T<sub>c</sub> value of (Bi, Pb)-2223 system to T<sub>c</sub> =121 K for x =0.3. This can be attributed to the increase in both *c*-axis and the volume fraction of the high-T<sub>c</sub> phase (2223) with increasing W nanoparticles content. Further increases of W nanoparticles content to 0.4 and 0.5 decreased the transition temperature. This might be due to the increasingly weak link and (Bi, Pb)-2212 phase in this sample. The estimated T<sub>c</sub> values were increased with increasing W nanoparticles content as shown in Table-1.

A number of studies [14, 15, 16] investigated the effects of the substitution of tungsten microparticles at Ca and Cu sites in the (Bi, Pb)-2223 phase. It was found that the superconducting transition temperature was depressed by W ion, which is the opposite of our observations.

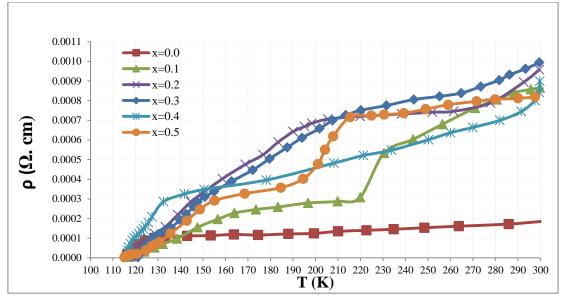
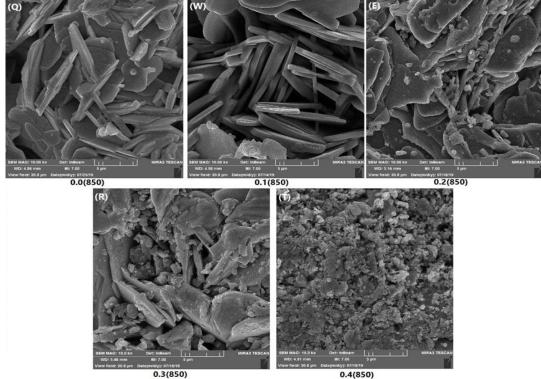


Figure 2-Resistivity ( $\rho$ ) as a function of a temperature for  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  samples with  $0.0 \le x \le 0.5$  of 50 nm particle size of tungsten sintered at 850 °C for 140h.

The grain morphologies of the superconducting  $Bi_{2-x}Pb_{0.3}$   $W_x$   $Sr_2$   $Ca_2$   $Cu_3O_{10+8}$  compound with  $0 \le x \le 0.5$  of 50 nm of tungsten samples prepared at optimum temperatures (850 °C) were studied in detail by SEM micrographs, as illustrated in Fig. (3). From the analysis of these micrographs, the grain structures of all samples exhibited the formation of randomplate-like grains, which was characteristic of the BPSCCO system.



**Figure 3**-SEM micrographs of  $Bi_{2-x}Pb_{0.3}W_xSr_2Ca_2Cu_3O_{10+\delta}$  samples for 50 nm particle size of tungsten sintered at 850 °C for 140h with : (Q) x = 0.0 (W) x = 0.1 (E) x = 0.2 (R) x = 0.3 (T) x = 0.4

It is clearly seen that the formation of the surface morphology of the samples with x=0.0, x=0.1 and x=0.2 of W nanoparticle concentrations is nearly identical, as shown in Figures-(3.Q), (3. W) and (3.E), respectively.

The W nanoparticles-free sample (Figure-3Q) exhibited large plated grains. The size of these

platelet grains seems to increase slightly for specimens substituted with x = 0.1 and 0.2 as seen in Figure- 3. W and E. This indicates the improvement of crystallography of the (Bi, Pb)-2223 phase.

With increasing the content of W nanoparticles, the porosity value of the samples was slightly decreased due to the disrupted grain growth (Fig. 3.R). However, the micrograph of x=0.4 wt % of W nanoparticles shows a different surface morphology, with grains becoming of a flower like structure, as seen in Figure-3.T. The alignment of the plate-like structures substituted with W was earlier observed [14, 15].

### **IV. CONCLUTIONS**

This research presents the experimental results of  $Bi_{2-x}Pb_{0.3}$   $W_xSr_2Ca_2Cu_3O_{10+\delta}$  with  $0 \le x \le 0.5$  of superconductor content. Our investigations revealed that the substitution with tungsten nanoparticles improved the crystallites and increased both the volume fraction of high-  $T_c$  phase (2223) and critical temperature. The c/a of (Bi, Pb)-2223 was increased slightly with increasing the tungsten nanoparticles content. The SEM micrographs showed the enhanced grain sizes after substitution with W NPs, where the size of the platelet-like grains seemed to increase slightly for specimens substituted with tungsten nanoparticles.

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