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Short Communication

Studying the Structural Properties of Lead Tetroxide Pb3o4 Doped Tin Oxide SnO_2 for Different Ratios X = 0, 0.3, 0.5

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Abstract

Pb_3O_4 doped tin oxide (SnO_2) transparent conducting powder was prepared by the solid state reaction method. Structural properties of the samples were investigated as a function of various Pb_3O_4-doping levels (x=0.0-0.3-0.5). The results of x-ray diffraction have shown that the samples are polycrystalline structure in tetragonal phase with preferential orientations along the (110) for SnO_2

Pure but for Pb_3O_4 doped SnO_2 change to (332). The distance between crystalline planes (d), grain size (D) and lattice parameters(a, c) and primary cell volume V were determined.The measurements also showed that the average grain size D for the pure compound of tin oxide is 11.877 nm, while the average grain size D for the dopant ratios (30, 50 wt%) are respectively is (16.111, 13.116 nm), the average grain size D of sample 50% was closest to the pure tin oxide .Doping with lead oxide Pb_3O_4 increases the value of the crystal lattice constants a and the constant compound. Which leads as a result to an increase in the volume of the primary cell volume V when the ratios of dopant x% is increased.

INTRODUCTION

Nano-crystals of semiconductor metal oxides have attracted a great interest due to their intriguing properties, which are different from those of their corresponding bulk state [1]. Tin dioxide SnO_2 has achieved special attention among the metal oxides, because of its wide band gap (3.6eV), optical transparency and high carrier density, thermal and chemical stabilities [2, 3].

Because of its unique electronic, optical, electrochemical and catalytical properties, it has been widely used in flat panel displays, transparent conducting electrodes, solar cells, gas sensors and rechargeable Li -ion batteries, etc [4] (Table 1). It crystallizes in the tetragonal rutile structure with space group P42/mnm. The lattice parameters a=b= 4.738 Å and c = 3.187 Å [5]. Its unit cell contains two tin and four oxygen atoms as is shown in (Figure 1). The tin atom is at the center of six oxygen atoms placed at the corners of a regular octahedron. Every oxygen atom is surrounded by three tin atoms at the corners of an equilateral triangle [6]. Lead (II, IV) oxide, also called red lead or minium, is the inorganic compound with the formula Pb_3O_4. A bright red or orange solid, it is used as pigment, in the manufacture of batteries, and rustproof primer paints. It is an example of a mixed valence compound, being composed of both Pb(II) and Pb(IV) in the ratio of two to one [7]. It crystallizes in the tetragonal structure with space group P42/mbc. The lattice parameters a=b= 8.815 Å and c =6.656 Å. Lead tetroxide Pb_3O_4 has achieved special attention among the metal oxides, because of its wide band gap 2.2eV [8]. Lead tetroxide density is 8.3 g.cm-3 melting point 500oC, molar mass is 685.6 g.mol-1 (Figure 2)

Table (1): shows result of structural values of pure SnO_2 sample.

Average D/nm	D /nm	β / deg	d /Å	(hkl)	2θ /deg	Samples
11.877	6.128	1.392	3.348	(110)	26.62	SnO_2 pure
	6.240	1.391	2.637	(101)	33.99	
	9.908	0.886	2.370	(200)	37.95	
	20.012	0.440	2.311	(111)	38.96	
	17.471	0.510	2.121	(210)	42.62	
	7.297	1.265	1.762	(211)	51.87	
	18.473	0.506	1.676	(220)	54.75	
	9.372	1.012	1.593	(002)	57.87	
	7.221	1.341	1.497	(310)	61.99	
	5.180	1.898	1.437	(112)	64.84	
	15.656	0.632	1.416	(301)	65.96	
	6.207	1.645	1.323	(202)	71.25	
	25.240	0.424	1.221	(321)	78.30	

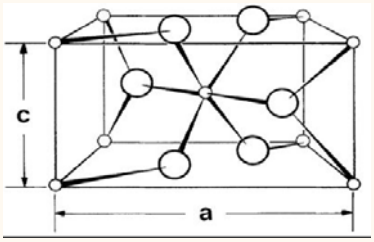

Figure 1 Unit cell of the crystal structure of Sno_2 Large circles indicate oxygen atoms and the small circles indicate tin atoms.

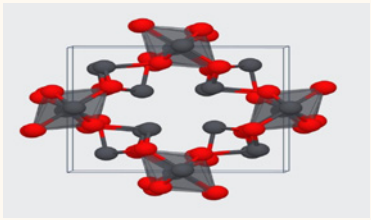

Figure 2 Unit cell of the crystal structure of Pb3o4 red circles indicate oxygen atoms and the gray circles indicate lead atoms [10].

[9]. The aim of this paper is preparing a doped metal oxide Pb_3O_4 (1-x) SnO_2(x) and characterizing by X-ray to study the structural properties.

EXPERIMENTAL METHOD

Pb_3O_4 (1-x) SnO_2(x) powders (x = 0, 0.3, 0.5) were prepared by a solid state reaction method. Were accurately weighed in required proportions and were mixed and ground thoroughly using an Agate mortar and pestle to convert to very fine powders. The grinding of the mixtures was carried out for 3 hours for all the powder Samples. The ground powder samples were firing at 700°C for 3 hours (Table 2).

RESULTS AND DISCUSSIONS

Structural Properties

The X-ray diffraction patterns of undoped and Pb_3O_4 doped SnO_2 powders prepared with various Pb_3O_4 concentrations (0, 0.3, 0.5) wt% are shown in (Figure 3-5). The XRD reveals that all samples are having polycrystalline nature with tetragonal structure and peaks correspond to (110), (101), (200) and (111). (210), (211), (220) (002), (310), (112), (301), (202) and (321) planes. The preferred orientation is (110) for pure but for Pb_3O_4 doped SnO_2 powders at 30 and 50wt% doping the preferred orientation change to (332)

The distance between crystalline planes values (d) of undoped and Pb_3O_4 doped SnO_2 powders are calculated by using following relation

$$2d\sin\theta=n\lambda \quad (1)$$

where D is distance between crystalline planes (A), θ is the Bragg angle, λ is the wavelength of X-rays (λ=1.54056 Å)

The grain size is from calculated Scherer's equation: [11]

$$D=\frac{0.94\lambda}{\beta\cos\theta} \quad (2)$$

where, D is the grain size, λ is the wavelength of X-ray, β is full width at half maximum (FWHM) intensity in radians and θ is Bragg's angle

The lattice constants a and c for tetragonal phase structure are determined by the relation (3) and the primary cell volume V from equation (4): [12]

$$\frac{1}{d^2}=\frac{h^2+k^2}{a^2}+\frac{l^2}{c^2} \quad (3)$$

V=a2.c 4

Where d and (hkl) are distance between crystalline planes and Miller indices, respectively. a, c are the crystal lattice constants (Å). The calculated lattice constants a, c and primary cell volume values are given in (Table 3).

We found that the average grain size D for pure tin dioxide is 11.877 nm, while the average grain size D for similar compounds wt% (30, 50) is nm (16.111, 13.116) and the average grain size D of sample 50% was closest to the pure compound. The (Figure 6) is shown the above Starting from 0%, the average grain size D increases until it reaches 30%, then it starts decreasing until it reaches 50%. The average grain size D for the impurity Pb_3O_4 has a greater than value of pure compound SnO_2 shown in (Figure 7).

Table (2): Shows result of structural values of (0.7:0.3) SnO2:Pb3o4 sample.

Average D/nm	D /nm	β /deg	d /Å	(hkl)	2θ / deg	Samples
16.111	20.000	0.805	110	3.348	31.001	SnO_2:Pb3o4 0.7:0.3
	22.921	0.377	112	2.457	35.867	
	12.000	0.210	311	2.252	40.819	
	13.703	0.130	421	1.984	56.556	
	23.841	0.462	332	1.865	61.250	
	9.590	0.264	220	1.676	66.029	
	21.700	0.264	440	1.521	70.056	
	13.166	0.234	413	1.887	71.559	
	15.454	0.036	301	1.416	78.400	
	20.800	0.010	202	1.323	85.159	
	14.759	0.295	334	1.155	87.971	
	5.397	0.009	212	1.256	89.255	

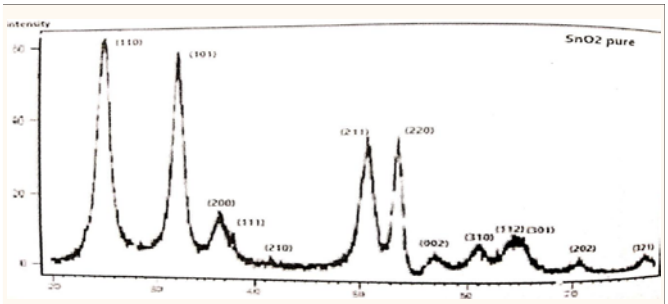

Figure 3 XRD result of pure SnO_2.

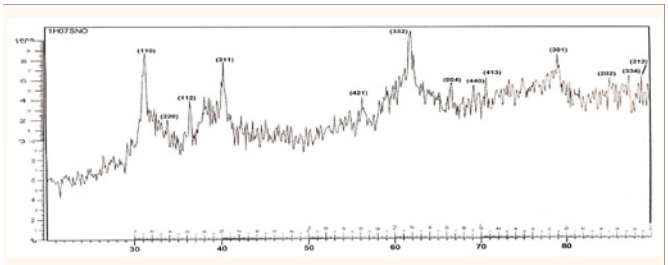

Figure 4 XRD result of 30% Pb3o4 doped SnO_2.

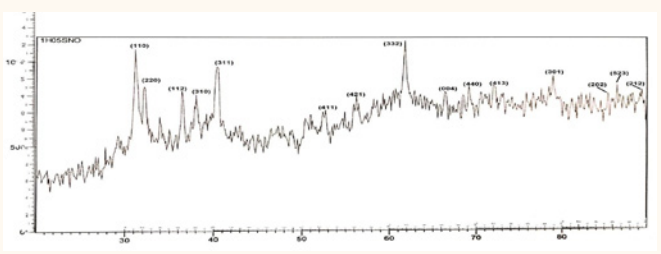

Figure 5 XRD result of 30% Pb3o4 doped SnO_2.

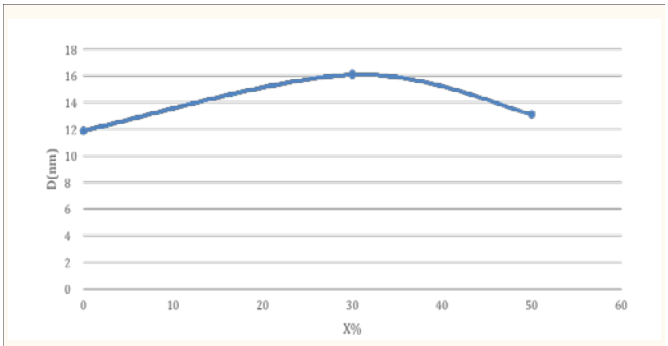

Figure 6 Variation of the average grain size D with different ratios (x%) of Pb3o4 doped Sno2 powders.

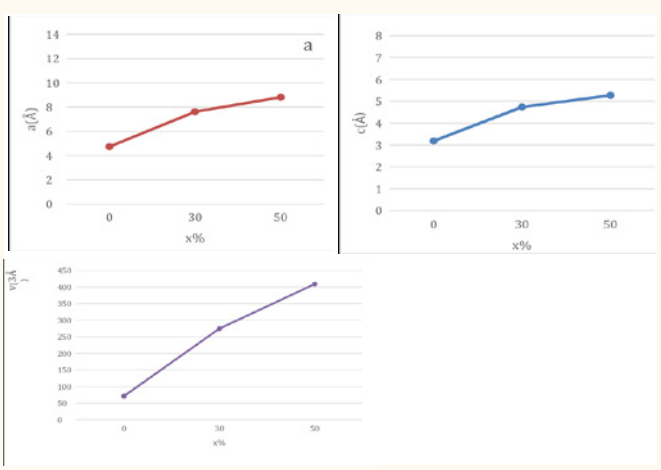

Figure 7 (a) variation of the lattice constants with different ratios (x %). (b) Variation of the lattice constants c with different ratios (x %). (c) Variation of the primary cell volume V with different ratios (x %). l

We also observed from the similarity in the X-ray spectra of the samples in the peaks and directions, and this indicates that the phase of the pure sample did not change when it was mixed with lead oxide Pb_3O_4 and therefore all samples contain one phase, which is the tetragonal phase.

Considering the relations (3 and 4), it is possible to calculate each of the structural parameters: the crystal lattice constants a and c and the size of the primary cell volume V as in the following (Table 4):

We observed from (Table 4) that the results indicate that doping with lead oxide increases the value of the crystal lattice

Table (3): shows result of structural values of (0.5:0.5) SnO2:Pb3o4 sample.

Average D/nm	D /nm	β / deg	d /Å	(hkl)	2θ /deg	Samples
13.116	18.473	0.003	3.318	(110)	31.013	SnO_2:Pb3o4 0.5:0.5
	7.221	1.041	2.457	(220)	33.991	
	15.656	0.150	2.198	(112)	35.951	
	11.877	1.132	1.984	(310)	37.472	
	9.351	1.163	1.831	(311)	40.623	
	5.183	1.475	1.676	(411)	52.206	
	17.416	0.375	1.491	(421)	56.436	
	6.870	0.527	1.887	(402)	58.515	
	22.012	0.631	1.378	(332)	61.261	
	20.000	0.264	1.351	(220)	66.047	
	6.240	1.264	1.323	(440)	70.133	
	15.012	0.134	1.294	(413)	71.252	
	5.397	0.368	1.273	(301)	78.301	
	11.341	0.110	1.264	(202)	85.171	
	17.898	0.004	1.249	(523)	86.062	
	19.908	0.124	1.201	(212)	89.255	

Table (4): Primary cell parameters of the studied samples .

50	30	0	X(%)
8.809	7.615	4.733	a/Å
5.271	4.735	3.185	c/Å
409.021	274.574	71.348	V/Å^3

constant a, c. This is because the ratio of lead ions increases in the structure of the compound. This leads to an increase in the volume of the primary cell volume V from 71.348 Å3 until it reaches the value 409.021 Å3when the dopant ratio is increased x %.

CONCLUSION

This paper presents a study of structural properties of Pb_3O_4 doped SnO_2 powders prepared by solid state reaction method. X-ray diffraction patterns confirm that the samples have polycrystalline nature with tetragonal structure and show presence (110), (101), (200), (111), (210), (211), (220), (002), (310), (112), (301), (202) and (321) planes in pure tin oxide sample. The SnO_2 have preferred orientation along (110) but for all doping levels the preferred orientation change to (332) plane. The average of crystallite size is within the range [11.877-16.111 nm] for all samples. It was defined that the lattice constants a, c for all the samples

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