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Synthesis and Structural Characterization of Sr doped TiO_2

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Abstract:

In the present study, Strontium doped Titanium Dioxide (TiO_2) were synthesized by a standard solid-state reaction technique. Strontium doped TiO2 shows interesting optical properties promising a kind of visual applications. The formation of the single-phase compound and its structural parameters were investigated by X-Ray diffraction (XRD) followed by Rietveld refinement and Scanning Electron Microscope (SEM) techniques. A better agreement between the observed and calculated XRD patterns was obtained by performing the Rietveld refinement with a structural model using the space group I 41/amd. SEM image shows a spherical uniform shape of the particles.

Key Words: TiO_2 (Anatase), Sr doped TiO_2, XRD, SEM.

1. Introduction

Titanium dioxide (TiO_2) nanomaterials are used in a broad range of application such as photocatalysis, separations, sensor devices, paints and dye-sensitized solar cell [1-3]. TiO_2 exists in three main phases; anatase, rutile and brookite, as a bulk materials, rutile is the stable phase; however, solution-phase preparation method for TiO_2 generally favour the anatase structure which is the more important due to its application in photocatalysis [4, 5]. Sr doped TiO_2 nanoparticles are reported to have enhanced photocatalytic activity than TiO_2 nanoparticles due to efficient charge separation. Later, the application of TiO_2 to waste water treatment and air pollution control has been extensively investigated. Surveys indicate that the increment in the band gap led to efficient charge separation, decreased the rate of recombination of the electron-hole span, and enhanced the rapid electron transfer at the solid-liquid interface [6-9].

2. Experimental & Characterization Detail

Strontium (Sr) doped Titanium Dioxide (TiO_2) has been synthesized by solid state reaction route, using high purity TiO_2 (99.9%, Rankem) and Strontium Carbonate ($SrCO_2$) (99.9%,

Molychem). The two samples 3% and 5% Sr-doped TiO_2 were prepared in this work. Precursor TiO_2 were calcinated at 450^0C for 2 hours in silica crucible in air atmosphere. The 3% and 5% Sr-doped TiO_2 were mixed and grind for an half hour by agate mortar with pestle for one hour before each thermal treatment. The powder was calcined at 450^0 C for 10 hours in silica crucible in air atmosphere. The process has been repeated three times to ensure homogeneous state with single phase powder.

The formation of the single phase compound was confirmed by X-Ray powder diffraction (XRD) analysis, performed using Philips Xpert Pro (using CuKα radiation, λ = 1.5406 Å) operated at 40 kV and 30 mA. The scan was done between 200 and 700 (2θ) with a 0.050 step size and an acquisition time of 3s per step. The lattice parameters and other detailed structural data were received by the Rietveld refinement Fullprof program using pseudo-Voigt function. The surface morphology of the prepared samples was explored by using scanning electron microscopy (JEOL).

3. Result and discussion

3.1 X-ray diffraction analysis (XRD)

Figure 1 shows the room-temperature X-ray powder diffractogram of pure TiO_2 and Sr-doped TiO_2. Fig. 2 a, b, c shows the XRD of TiO_2, 3% Sr-doped TiO_2 and 5% Sr-doped TiO_2 along with the Rietveld refined plot respectively.

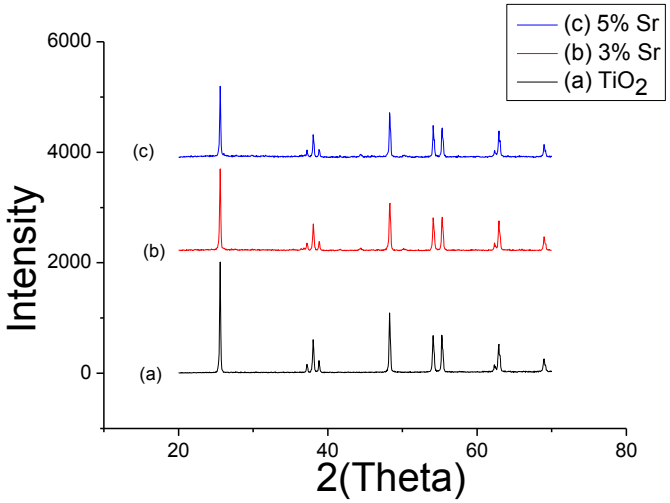

Figure 1, *XRD pattern of pure TiO_2 and Sr-doped TiO_2.*

The sharp peak suggests that all the synthesized samples are well crystallized. From the X-ray diffraction patterns, it is noted that all the X-ray peaks of the pure compound of TiO_2, matched well with reported data (JCPDS Card No. # 21-1272) [10-11]. In the present work, we have taken on the Rietveld refinement technique to investigate the crystal structure of the compound. The Rietveld refinement of the X-ray data was performed with the Fullprof program [12] shown good fitting. Analysis shows the tetragonal structure of the prepared samples with space group of I 41/amd. Also good combination observed in measuring "d" and calculated "d" value on the same scale. In this work we have not abided by any secondary peak of strontium but the minor shift is observed in 2 (Θ) values. The X-ray diffraction pattern shows small shift in some of the peak positions and intensities for the substituted compound with respect to the pure compound. From the theory of crystalline physics, it's difficult to substitute larger radius atom in the small radius atom. Here the radius of the Ti is 0.75 Å and for Sr is 1.18 Å. So it is difficult to substitute Sr in Ti, but it is possible to combine both in proper way means strontium are not doped or substitute in TiO_2 but strontium replace "Ti" site.

The unit cell diagram of pure TiO_2 and its Sr-substituted compound were generated with the help of the program Fullprof Studio using the refined cell parameters, space group and xyz coordinates of the atoms. Fig. 3 a, b and c shows the unit cell diagrams of pure TiO_2 and 3% and 5% Sr-doped compound respectively.

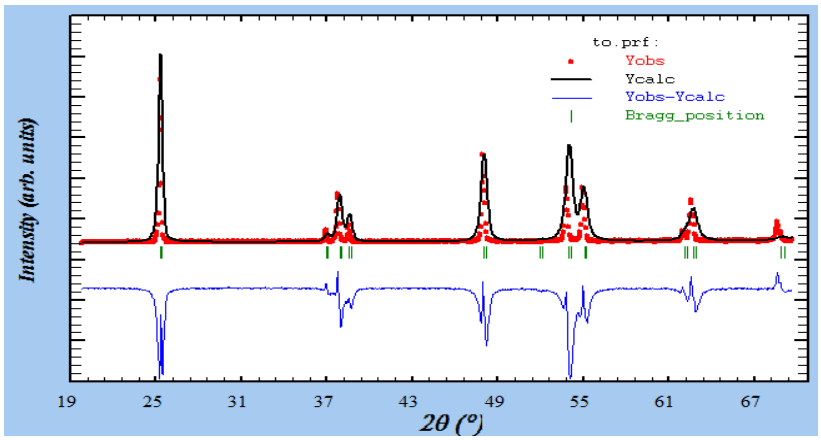

Figure 2(a). *Rietveld refined profile of TiO_2. Observed (...), calculated (continuous line) and the difference between observed and calculated profile is plotted below on the same scale.*

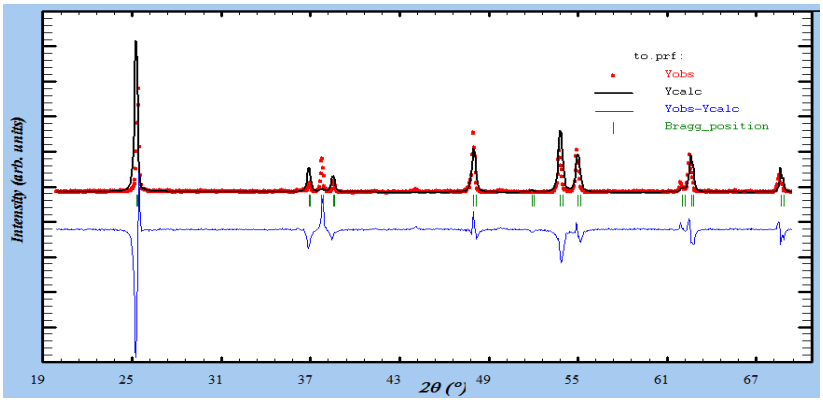

Figure 2(b), *Rietveld refined profile of 3% Sr doped TiO_2. Observed (...), calculated (continuous line) and the difference between observed and calculated profile is plotted below on the same scale.*

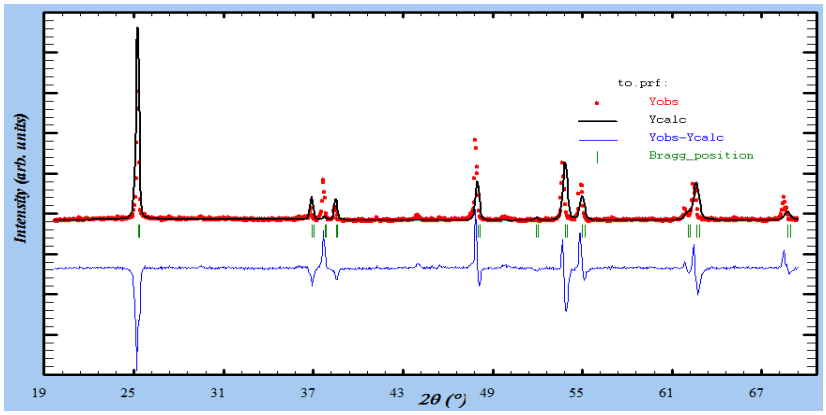

Figure 2(c), *Rietveld refined profile of 5% Sr doped TiO_2. Observed (...), calculated (continuous line) and the difference between observed and calculated profile is plotted below on the same scale*

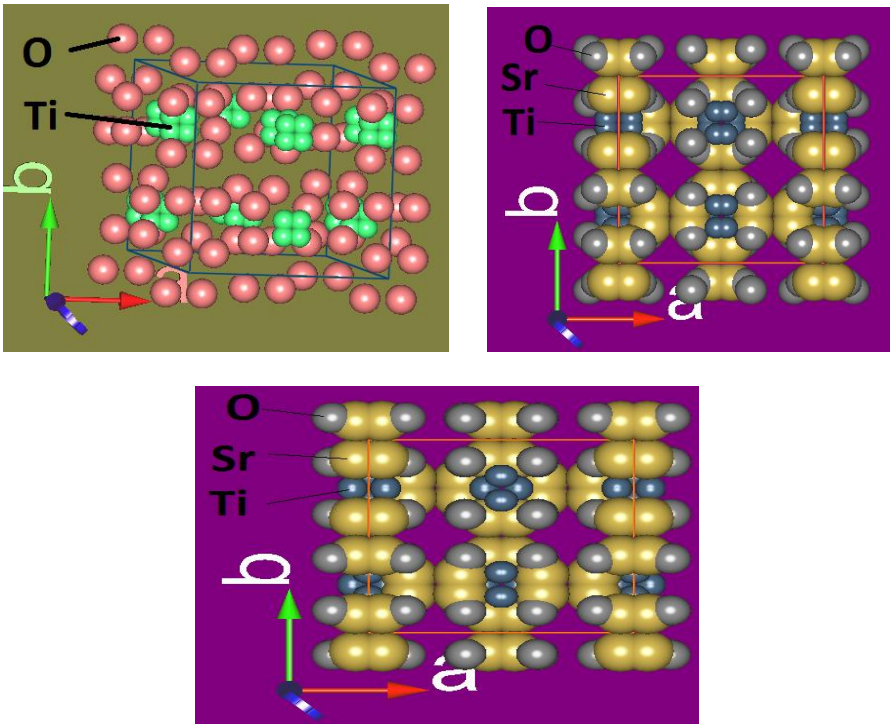

Figure 3, (a*) Unit cell diagram for 3% Sr doped TiO2* ***(b)*** *3% Sr doped TiO2* ***(c)*** *5% Sr doped TiO2*

The results of XRD analysis are summarized in Table 1. The observed (based on Bragg's condition 2d sin Θ = nλ) and computed (based on refined cell parameters) d-values for the compound and are presented in Table.

Parameters	**TiO_2**	**$Sr_{0.03}Ti_{0.97}O_2$**	**$Sr_{0.05}Ti_{0.95}O_2$**
2θ range (deg)	20.00–70.00	20.00–70.00	20.00–70.00
Step size (deg)	0.05000	0.05000	0.05000
Crystal symmetry	Tetragonal	Tetragonal	Tetragonal
Space group,	*I 41/a m d*	*I 41/a m d*	*I 41/a m d*
Cell parameters (Å) a,b c	3.78771 (Cal.) 9.50156 (Cal)	3.79060 (Cal.) 9.53676 (Cal.)	3.79318 (Cal.) 9.53187 (Cal.)
Unit cell volume (cm^3)	*136.316*	*137.030*	*137.147*
Half width parameters (deg) U V W	0.506430 −0.039002 0.044017	0.056529 −0.000550 0.043890	0.116430 −0.011090 0.044010

TiO_2		**$Sr_{0.03}Ti_{0.97}O_2$**		**$Sr_{0.05}Ti_{0.95}O_2$**		
d (Obs.)	**d (Cal.)**	**d (Obs.)**	**d (Cal.)**	**D (Obs.)**	**d (Cal.**	**hkl**
3.50622	3.51845	3.51158	3.52255	3.51117	3.52437	101

2.42573	2.42970	2.42543	2.43575	2.42600	2.43570	103
2.36439	2.37539	2.37389	2.38419	2.37344	2.38297	004
2.32573	2.33309	2.32812	2.33652	2.32741	2.33744	112
1.88632	1.89385	1.88562	1.89530	1.88657	1.89659	200
1.69505	1.69853	1.69441	1.70382	1.69449	1.70335	105
1.66126	1.66762	1.66063	1.66904	1.66093	1.67012	211
1.49021	1.49370	1.48969	1.49581	1.48932	1.49644	213
1.47807	1.48081	1.47724	1.48363	1.47717	1.48396	204
1.36109	1.36315	1.36062	1.36715	1.36060	1.36688	116

Table 1, Shows the *Detailed Rietveld refinement parameter of the prepared samples and shows the comparison between observed and calculate "d" value of the samples by Rietveld refinement analysis.*

3.2 Scanning electron microscope (SEM)

The SEM micrographs for the examined TiO_2 system are presented in Fig. 4. The SEM micrographs indicate that the particle size is nearly uniform and spherical throughout the surface in each sample. The original anatase crystal grain size, measured by scanning electron microscopic image process analysis was found of the order of 200 nm diameter given in Fig. 4 and after doping of strontium and calcination at 450^0C in air atmosphere crystalline size most remain same but they are not looking separate but some cloud is observed (Fig. 4a and Fig. 4b). For a modest percentage of dopant concentration, the micrograph shows no significant change in word structure.

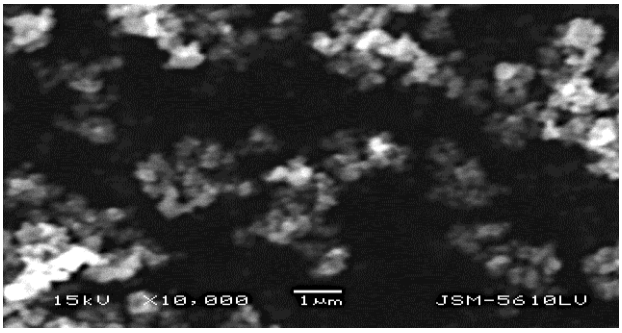

Figure. 4 SEM Micrograph of TiO_2 calcinate at 450^0C

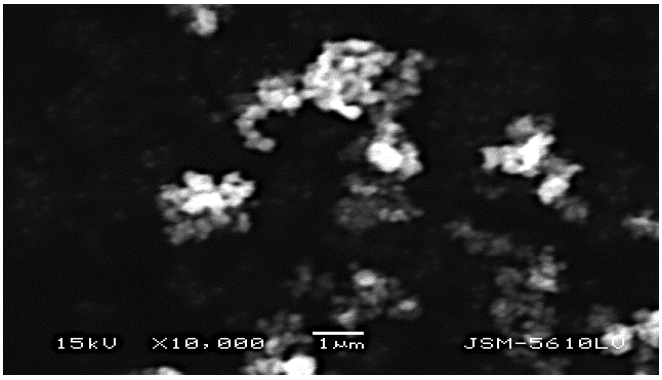

Figure. 4(a), *SEM Micrograph of 3% Sr doped TiO_2 calcinate at 450^0C*

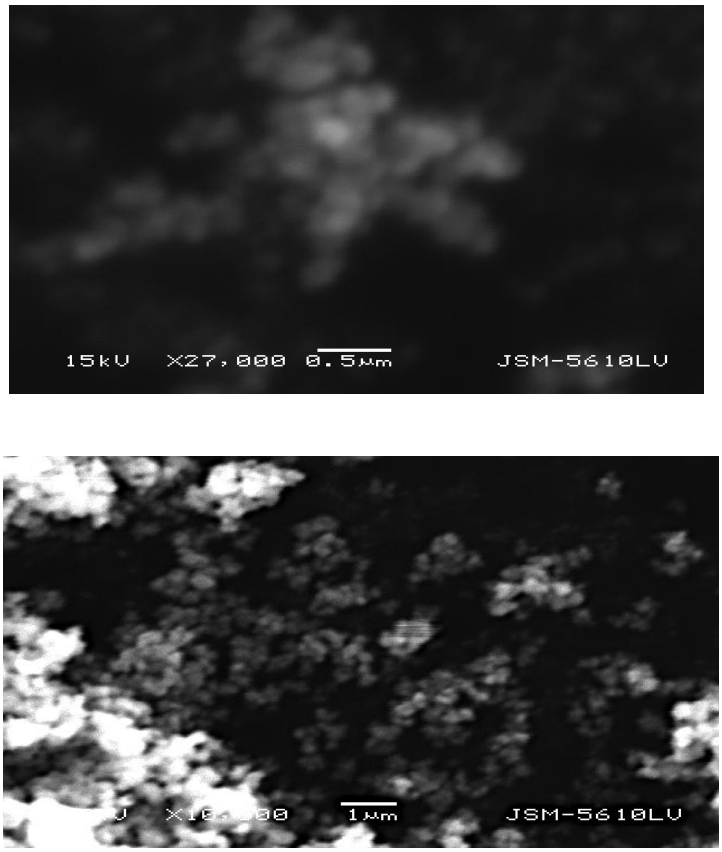

Figure. 4 (b), *SEM Micrograph of 5% Sr doped TiO_2 calcinate at 450^0C*sample)

4. Conclusion

Strontium (Sr) doped Titanium dioxide (TiO_2) was synthesized by solid state reaction route. Precursor TiO_2 were calcinated at 450^0C for 2 hours in silica crucible in air. X-ray diffraction patterns show that all the synthesized samples are well crystallized. The Rietveld analysis of the samples conforms a tetragonal structure with space group I 41/amd. The SEM micrograph indicates almost uniform particle size having spherical shape. The micrograph also shows no substantial alteration in surface morphology for small percentage concentrations.

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