

Synthesis and Characterization of Sr Doped TiO₂ by Solid State Method

M. H. Mangrola and V. G. Joshi

Department of Physics

Veer Narmad South Gujarat University, Surat-395007, Gujarat, India

Email: mhmangrola@gmail.com; joshi_vibhuti@yahoo.com

B. H. Parmar

Department of Physics

Government Science College, Valod-394640, Gujarat, India

Email: bhparmar2000@yahoo.com

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Abstract: This paper report on the 7% Sr doped TiO₂ nanoparticles which are synthesized by Solid State Method. Powder was calcinated at 450°C for several times. Powder characterized by X-Ray diffraction to identify the effect on the TiO₂ crystalline phase. The XRD data shows a single crystalline Phase of TiO₂. Sr doped TiO₂ shows a mixture of anatase and rutile phase. The crystalline size is calculated by Debye Scherrer formula and Williamson hall plot method. Surface morphology of the powder is studied by Scanning Electron Microscope. Photoluminescence emission spectra show broad spectra 400 nm to 500 nm at the excitation wavelength of 300 nm to 400 nm.

Key words: Doped TiO₂, XRD, SEM, PL.

1. Introduction

Recently, there has been a great deal of interest in the area of structural property of materials, semiconducting properties of materials, the change in structure and band gap of materials with doping of other materials. TiO₂ are widely used in areas.

It is found so far that Photocatalysis with a primary focus on several semiconductors such as TiO₂, SrTiO₃ and ZnO are efficient photocatalysts and widely considered in emerging technology for solving environmental problems mainly for the removal of organic contaminants^{1,2,3}.

Titanium Dioxide and Zinc Oxide are much of interest due to their unique properties, such as dielectric constant, high refractivity and photocatalysis. Presently much of research work is being done on electronic,

optical and photocatalytic properties of such materials using several techniques⁴⁻⁸. Doping of metal ions in TiO₂ is the latest technique to suppress the rate of recombination of electron-hole pair.

Recent research work focused on the doping of the metals in the semiconductor materials to play with the energy band gap, and to study the crystalline properties of the base material. Some researchers reported that TiO₂ doped with non-metallic atoms like carbon⁹, sulphur¹⁰ and co-doping with nitrogen and fluorine¹¹. Lithium doped TiO₂ shows high photocatalytic activity under visible light owing to band gap narrowing. Here we report on the structural property of Sr doped TiO₂ material by solid state method⁷.

2. Experimental Section

The materials used are TiO₂ Nanoparticles and SrCO₃. The powder sample is prepared with TiO₂ by doping 7% Sr in it. The mixture of 7% Sr-doped TiO₂ was ground for half hours and put in furnace, annealed at 450⁰ C for several hrs. The cycle of heating and grinding has been repeated several times to ensure the homogeneous distribution as well as proper doping. The details of powder grinding and annealing at 450⁰ C is given in table 1.

Table 1. Detail of synthesis process of sample

Number of cycle	Time for increasing temperature from R _T to 450 ⁰ C	Time for decreasing temperature from 450 ⁰ C to R _T	Time for stable temperature at 450 ⁰ C
1	3.0 hr	3.0 hr	13.5 hr
2	3.0 hr	3.0 hr	22.0 hr
3	1.0 hr	1.5 hr	1.0 hr

3. Powder Characterization

X-ray powder diffraction (XRD) analysis was performed on an Anchor scan parameters (anode materials copper target ($\lambda_{CuK\alpha} = 1.5406 \text{ \AA}$). The diffractometer used 40 kV and 30 mA. Scanning took place between 20⁰ and 80⁰ (2 Θ) with a 0.0200 step size and an acquisition time of 3s per step. The Debye-Scherrer formula used for evaluated the crystalline size from the broadening of the corresponding X-Ray spectral peaks. This is the generally accepted method to estimate the mean crystalline size of nanoparticles. Crystalline Size also calculated using a Williamson Hall plot method^{2,12}. The image was obtained by Scanning Electron Microscope (SEM), operated at 16.0 kV. Surface morphology of the sample was studied by SEM.

Photoluminescence analysis was taken at room temperature with the different excitation wavelength.

4. Result and Discussion

4.1 X-Ray diffraction Analysis

In the undertaken job the sample was prepared by solid state method. The XRD pattern of TiO₂ nanoparticles are shown in Figure 1(a) and the Sr doped TiO₂ shown in Figure 1(b). The sharp intense peak of pure TiO₂ at $2\theta = 25.4^\circ$ corresponds to anatase (101) and $2\theta = 27.6^\circ$ corresponds to rutile (110) phase [9].

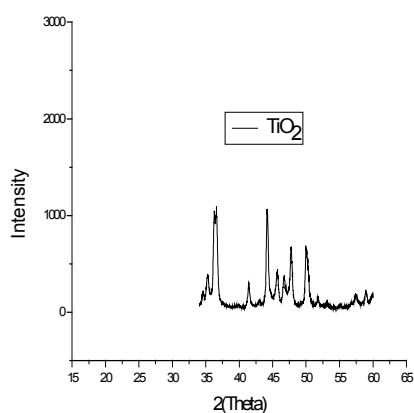


Fig. 1(a). XRD of TiO₂ nanoparticles.

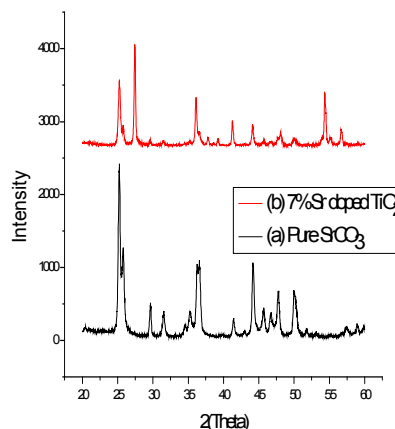


Fig.1(b). XRD of Sr doped TiO₂.

In XRD data separate peaks for SrTiO₃ or Strontium oxide (Sr-O) were not observed. The XRD data of Sr doped TiO₂ shows a small shift in peak. The value of 2θ after shifting for anatase and rutile are 25.2 and 27.4 respectively. The ionic radius of Ti and Sr is 0.75 Å and 1.18 Å respectively². Due to an ionic radius it's difficult to doped Sr in Ti. It is observed that the mixture of Sr-doped Ti is homogeneous as it is seen a shift in XRD pattern (Fig. 1b). It is also seen that the Sr doped TiO₂ XRD data shows a highly intense peak compare to pure TiO₂ XRD data (Fig. 1a & 1b). The crystalline size of sample has been determined using the X-ray diffraction peak of the samples with Scherre Equation $B = k\lambda / s \cos\theta$ and also calculated by Williamson hall plot method.

Figure 2, Shows a Williamson Hall plot of the Sr doped TiO₂ sample. The graph is plot with the help of Origin software (Version 8.0) and use the linear fitting method is used to find an intercept value. The intercept value

use to calculate crystalline size. The equation to calculate crystalline size from the Williamson Hall Plot is $L = k\lambda / \text{Intercept of the line}$.

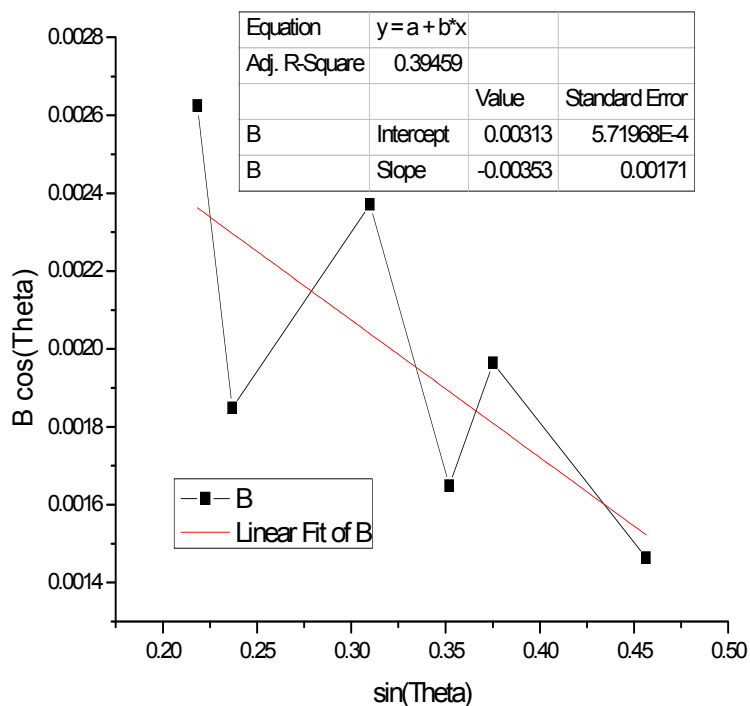


Fig. 2. Williamson Hall plot of the Sr doped TiO_2 sample

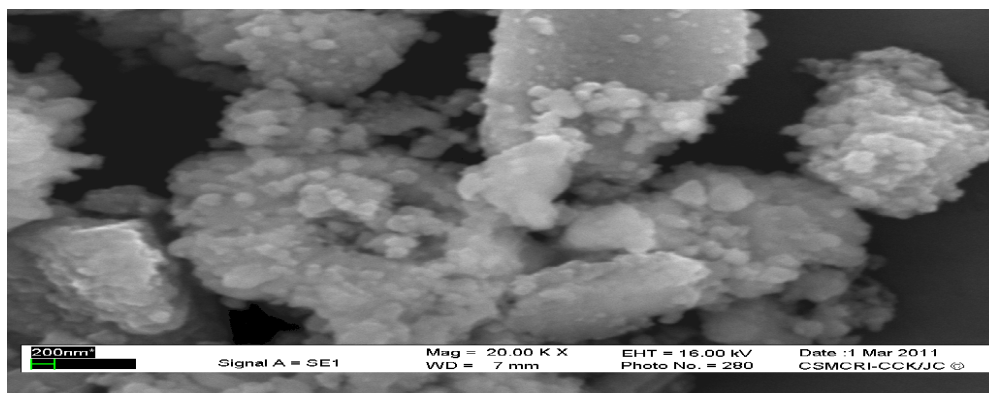
The crystalline sized of the sample calculated by both the methods in nanometer is shown in Table 2.

Table 2. Crystalline size of the sample

TiO_2	Debye-Scherrer Formula (nm)	William son Hall (nm)
Anatase (101)	55.1478	46.2672
Rutile (110)	78.2456	

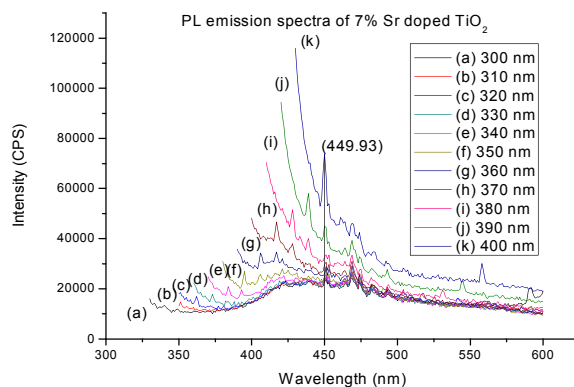
4.2 SEM Analysis

Figure 3, show the SEM micrograph of Sr doped TiO_2 . The powder samples synthesized under the same environment. As seen in Figure 3, it is noticed that there is no big difference in the shape of particles. A sharp observation finds that the particles are agglomerated.

Fig. 3. SEM Micrograph of Sr doped TiO₂

4.3 Photoluminescence Analysis

Figure 4. Shows the PL spectra of the Sr doped TiO₂ at room temperature. There is a wide photoluminescence band in the wavelength range of 400-500 nm shown in figure 4 at the excitation wavelength 300 to 400 nm. In the broad range of excitation wavelength the emission spectra do not show a shift in wavelength. In whole emission spectra the peak observed at 449.93 nm is shown in figure 4. It is seen that the excitation wavelength is increase the intensity of emission peak also increases. The Sharp peak of emission spectra observed at the excitation wavelength 400 nm. The energy band gap value calculated from PL emission spectra is 2.4797 eV. This value of band gap indicates that the Strontium is highly effective in reducing the band gap of TiO₂.

Fig. 4. Photoluminescence Analysis of Sr doped TiO₂ powder

Here, we would like to notice that the increase of the photoluminescence spectra with increasing excitation wavelength generally reflects a distribution of energy levels caused by different local environments

(structure, strain, etc.) of luminescent centres. Of course, the exact photoluminescence mechanism of pure TiO_2 and Sr doped TiO_2 nanocrystals needs further more investigations^{13,14}.

5. Conclusion

The XRD data of Sr doped TiO_2 shows a single crystalline formation without any secondary phase. The XRD data of Sr doped TiO_2 shows high intense peaks compare to XRD data of pure TiO_2 . It is observed that the crystalline size of the sample is different for anatase and rutile phase. SEM micrograph shows agglomeration in Sr doped TiO_2 . SEM micrograph indicate spherical shape of the particles in the samples. PL emission spectra show a broad range of 400nm to 500 nm. The maximum high intense emission peak observed at 449.93 nm and excitation wavelength is 400 nm.

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