RESEARCH PAPER

Effect of Annealing Temperature on Structural, Electrical and Optical Properties of TiO_2 Nanopowder

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ABSTRACT

TiO_2 nanopowder is prepared by simple sol-gel method using starting material as titanium isopropoxide with methanol and annealed at 600°C, 700°C and 800°C for 1 hour in air. X-ray diffraction pattern revealed the presence of both anatase and rutile phase in TiO_2 specimens annealed at different temperatures. It is observed that the content of rutile phase and crystallite size increases with increase in annealing temperature. Scanning electron microscopy (SEM) is used to study surface morphology of TiO_2 specimens annealed at different temperatures. Using Tauc plot it is observed that energy band gap decreases with increase in annealing temperature. I-V curve of TiO_2 specimen shows that current increases with increase in annealing temperature. The preparation method is optimized by changing the concentration of titanium isopropoxide which leads to mixed phase (anatase and rutile) TiO_2 nanopowder with a lower energy band gap value which may play an important role in gas sensing applications.

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INTRODUCTION

Titanium dioxide (TiO_2) is considered as one of the important semiconductor metal oxide due to its wide range of applications such as in solar cells, self cleaning surfaces, CO_2 reduction and gas sensing [1-3]. TiO_2 exits in three polymorphs anatase, rutile and brookite. Rutile is the most stable phase whereas anatase and brookite are metastable phases. Anatase and brookite can transform to rutile phase on heating. The energy band gap for anatase and rutile phases are 3.2 eV and 3.0 eV respectively [4].

In recent years mixed phase (anatase and rutile) nanostructured TiO_2 was found to exhibit improved gas sensing properties [5, 6]. Therefore it is desirable to prepare mixed phase TiO_2 nanopowder by a simple sol-gel method. In our investigation the preparation method of mixed phase TiO_2 nanopowder is optimized by changing the concentration of titanium isopropoxide which acts as a starting material. The prepared TiO_2 nanopowder shows lower band gap value as compared to data reported in the literature [6, 7]. The lower band gap value and the presence of mixed phase (anatase and rutile) in TiO_2 nanopowder may lead to improved gas sensing properties [5-8]. Hence it will be beneficial to understand the effect of annealing temperature on its structural, electrical and optical properties.

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MATERIALS AND METHODS

Titanium isopropoxide (TTIP) and methanol are used as starting materials. The chemicals used are of analytical research grade. 3.5 ml of titanium isopropoxide (TTIP) is added in 40 ml methanol. This results in a milky white solution. Magnetic stirrer is used to stir the solution vigorously for 1:30 hrs. at a temperature 57±3^{0}C. The gel thus produced is kept for 12 hrs. at room temperature. Therefore powder is obtained and annealed at temperatures 600^{0}C, 700^{0}C and 800^{0}C for 1 hour in air [6]. X-ray diffraction pattern (XRD) of TiO_2 specimens annealed at different temperatures is recorded using CuK_α radiation. The UV-absorption spectra are recorded on Shimadzu-1800 UV- spectrophotometer by dispersing TiO_2 nanopowder in deionized water. In order to study electrical properties pellets having diameter 10 mm of TiO_2 nanopowder specimens are formed by Hydraulic pellet press and copper wire with a silver paste is used to form metal contacts on specimens. Kiethley-2400 source meter with two probe set up is used to record I-V characteristics at room temperature.

RESULTS AND DISCUSSION

X-ray diffraction peaks show the presence of both anatase and rutile phases in various TiO_2 specimens as shown in Fig 1. The diffraction angles are in good agreement with JCPDS no 21-1272 for anatase, JCPDS no 21-1276 for rutile and data reported in the literature [1, 9]. The average crystallite size for various TiO_2 specimens annealed at 600 °C, 700 °C and 800 °C is calculated using Scherrer's formula [9].

$$D= 0.89\lambda / \beta \cos\theta \quad (1)$$

where D is crystallite size in nanometer, β is the full width at half maximum (FWHM) in radian, λ is the wavelength of the X-ray which is 0.15406 nm for Cu target Kα radiation and θ is the Bragg angle. The content of anatase and rutile phase is calculated using formula [1]:

$$X_a = 1/ 1+ 1.26 (I_r/I_a) \quad (2)$$

where X_a is the weight fraction of anatase in the mixture, I_a and I_r are intensities of anatase (101) and rutile (110) diffraction peaks. The average crystallite size and content of anatase and rutile phase for TiO_2 specimens annealed at different temperatures are summarized in Table 1. It is observed (see Table 1) that the content of rutile phase and an average crystallite size increases with increase in annealing temperature. Moreover, it was reported that the presence of mixed anatase and rutile phase leads to improved gas sensing properties [5, 6]. This shows that the change in content of anatase and rutile phase present in TiO_2 nanopowder after annealing may lead to interesting gas sensing properties. It is noteworthy here that the formation of rutile phase at an annealing temperature of 800^{0}C was reported in several studies [10-13] but in our investigation the prepared TiO_2 nanopowder contains both anatase and rutile phase after annealing at 800^{0}C.

Fig. 2 shows surface morphology of TiO_2 specimens annealed at temperatures 600 °C-800

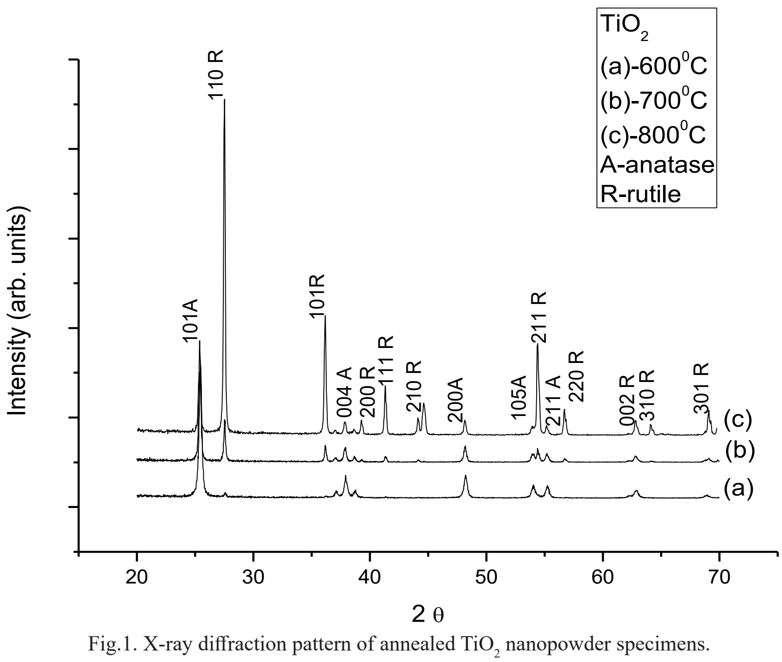


Fig.1. X-ray diffraction pattern of annealed TiO_2 nanopowder specimens.

Table 1. The average crystallite size, content of anatase and rutile phase and energy band gap values for TiO_2 specimens annealed at different temperatures.

Annealing temperature	Intensity I_a (101 anatase)	Intensity I_r (110 rutile)	Average crystallite size (nm)	Anatase %	Rutile %	Energy band gap (eV)
600^0C	1456.56	32.55	35±5	97.26	2.74	2.93
700^0C	976.44	446.93	45±5	63.37	36.62	2.75
800^0C	943.63	3694.78	55±5	16.85	83.15	2.68

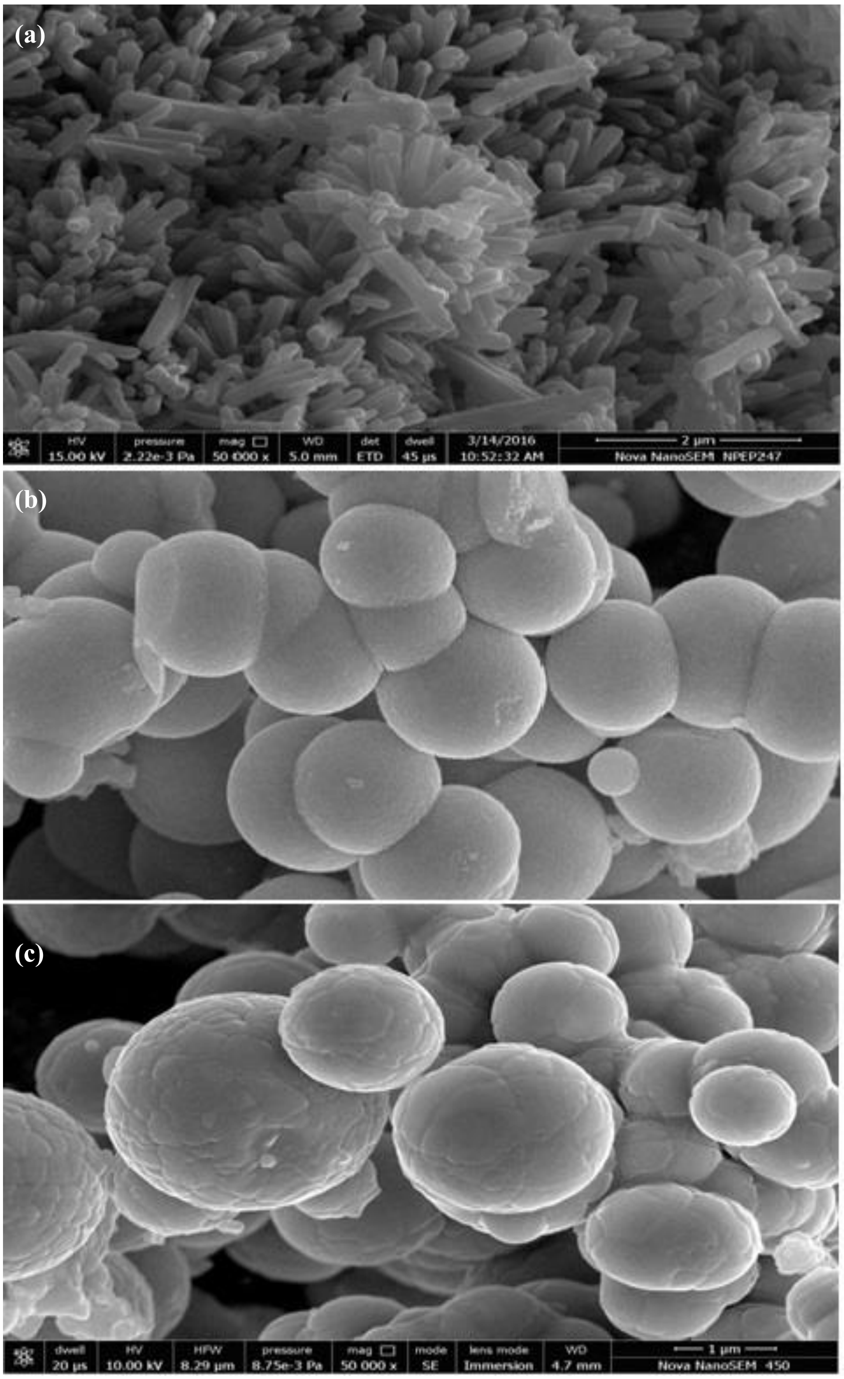


Fig. 2. SEM images of TiO_2 specimens annealed at (a) 600 °C (b) 700 °C and (c) 800 °C.

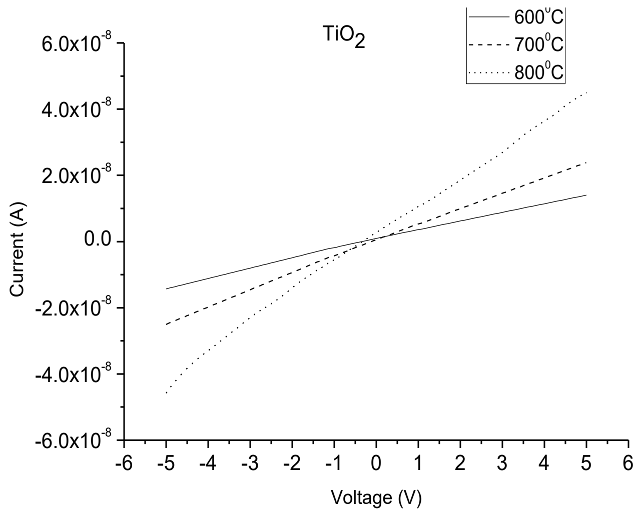
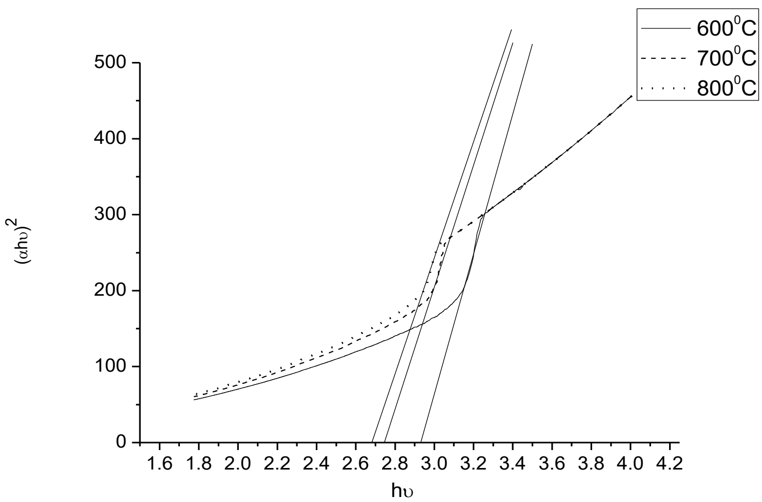


Fig. 3. I-V curves of TiO_2 specimens annealed at (a) 600 °C (b) 700 °C and (c) 800 °C.



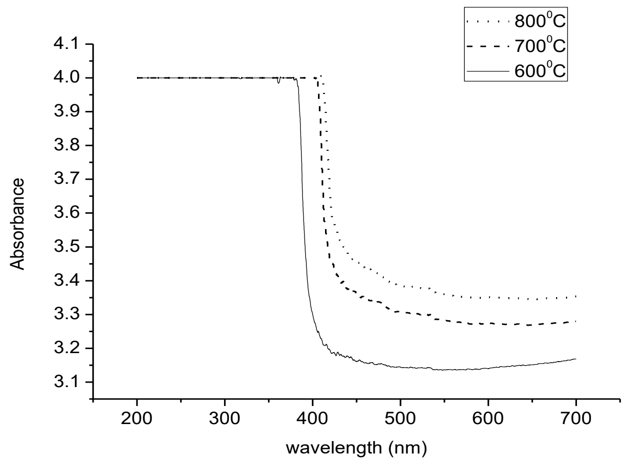


Fig. 4. (a) UV absorption spectra and (b) Tauc plot for TiO_2 specimens annealed at 600 °C, 700 °C and 800 °C.

°C using scanning electron microscopy (SEM). SEM image of TiO_2 specimen annealed at 600 °C show cylindrical shaped particles (Fig. 2 a) whereas SEM images of TiO_2 specimens annealed at 700°C and 800 °C show particles in spherical shape (Fig. 2 b and Fig. 2 c) [2, 14]. Fig. 3 depicts linear I-V curves of TiO_2 nanopowder specimens which confirm ohmic nature [15]. It is observed that current increases with increase in annealing temperature. This is due to the fact that an increase in crystallite size leads to improvement in electron migration. Therefore annealing of the TiO_2 nanopowder specimens increases average crystallite size which promises improved electrical properties [16].

UV-absorption spectra and Tauc plot for TiO_2 specimens annealed at different temperatures are shown in Fig. 4 (a) and Fig. 4 (b). The energy band gap values are obtained by extrapolating linear region to abscissa as shown in Fig. 4 (b) and are mentioned in Table 1. UV studies show that energy band gap increases with decrease in average crystallite size of TiO2 nanopowder specimens corresponding to blue shift of the optical absorption edge [1, 17]. It is also observed that (see Table 1) energy band gap decreases with increase in annealing temperature. This is due to the fact that an increase in annealing temperature lowers of interatomic spacing [18-20]. It is noteworthy here that values of energy band gap obtained are lower as compared to energy band gap 3.2 eV for pure anatase , 3.0 for pure rutile phase and the data reported for the mixed phase TiO2 nanopowder exhibiting as a capable candidate for gas sensing application [6, 7, 21].

CONCLUSIONS

1. TiO_2 nanopowder exhibits lower energy band gap value which promises it as a suitable material for gas sensing application.
2. TiO_2 nanopowder contains both anatase and rutile phase after annealing in the temperature range 600 °C to 800 °C.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publicaton of this manuscript.

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