

Undoped and Fe-doped TiO₂ thin films fabricated by sol-gel technique: An Approach to gas sensing applications

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ABSTRACT

The Undoped (pure) and Fe-doped titania films were fabricated by sol-gel technique. The structural and morphology properties of films were obtained by XRD and FESEM characterization techniques. The crystallite of doped films changed with change of doping concentration. Both the anatase and rutile phases were observed in XRD patterns. The anatase phase is one of the important phase that is preferably used for gas sensing applications of TiO₂ thin films. The decreases in grain size and large surface area observed in morphology images of films also leads the TiO₂ surface for remarkable approach to gas sensing technology.

Keywords: TiO₂, Sol-gel, Fe doping, Anatase, Gas Sensing Application.

INTRODUCTION

TiO₂ films are recently focused due to its interesting chemical, electrical and optical properties. The interest in TiO₂ was mainly due to its non-toxicity and good stability in various operating environments [1]. TiO₂ is a non-toxic material that has good stability and excellent optical properties. TiO₂ as photo-catalyst is effective in light from visible region of the solar spectrum, so it is need to be developed as future generation photo-catalytic material [2-3]. TiO₂ has high refractive index and good insulating properties which make this material to be widely used as protective layer for very large scale integrated circuits, antireflective coatings, gas sensors, electro-chromatic displays, and planar waveguides [4]. The high dielectric constant of TiO₂ allows its considerations an alternative to SiO₂ for ultra thin gate oxide dielectrics used in memory and logic devices and have been of great interest for applications in the telecommunications industry [4-5]. TiO₂ suffers mainly due to high recombination rate of the photo excited electron-hole pairs in the irradiated particles [6]. This problem can be resolved by changing the crystalline and optical properties structure by doping with different transition, non-transition and alkaline-earth metals [7- 8]. Fe³⁺ ions doped into TiO₂ have caused changes in phase composition, particle size and surface area [9]. The ionic radius of Fe³⁺ is identical to Ti⁴⁺, therefore it can be used to decrease the band gap of TiO₂ for photocatalytic and gas sensing applications [10-11]. Undoped and doped-TiO₂ films are fabricated by different types of deposition techniques such as chemical vapor deposition (CVD), pulsed laser deposition, reactive sputtering and sol-gel deposition [12]. Sol-gel deposition technique is preferred to other deposition methods due to its compatibility with current practices of silicon technology [13]. Our aim of present work is mainly focused on the structural and morphology characteristic of Fe-doped and Undoped TiO₂ thin films using X-ray diffraction (XRD), FESEM techniques.

EXPERIMENTAL WORK

Materials

Titanium (IV) isopropoxide (TIP) was used as precursor of titania, ethanol used as a solvent and acetic acid was used as catalyst for preparation of TiO₂ solution. The solution of TiO₂ was prepared using TIP, ethanol and acetic acid in the molar ratio of 1:9:0.1. The mixture was stirred for 1 hour on hot plate fixed at temperature of 85° C. Further, the solution of iron (Fe) was prepared by dissolving different concentration of FeCl₂ in mass/volume (m/v) percentage in 12 ml of ethanol solution.

The Fe:TiO₂ solution was mixed in borosilicate glass bottle and stirred on hot plate with magnetic stirrer for 20 minutes. The cleaned substrate of corning glass and silicon was placed on photo-resist spinner. The drop of prepared

solution was placed on glass/silicon substrate and the with spin rate fixed at 2000 rpm. The films were sintered at 550° C in a temperature controlled resistance tube furnace under dynamic air for 30 minutes.

Characterization

The x-ray diffraction (XRD) of the doped and undoped TiO₂ thin films was characterized by PAN analytical instrument with Cu K α radiation ($\lambda=1.54 \text{ \AA}$). The surface morphology of the films was obtained by using Field Emission Scanning Electron Microscope (FESEM) QUANTA 200 FEG model and the accelerating voltage was fixed at 20 KV with resolution of 2 nm. The thickness of the films was measured by surface profilometer.

RESULTS AND DISCUSSIONS

Figure 1, shows the X-ray diffraction (XRD) patterns of undoped and Fe-doped TiO₂ thin film fabricated on silicon substrate annealed at temperature of 550 °C. The XRD spectra represents the phases (110), (112) for the anatase and (200) for rutile. Figure 2 shows the XRD patterns of undoped and 1 % m/v of Fe into TiO₂ films on glass substrate annealed at temperature of 550 °C. Figure 3, shows the 3 % m/v of Fe into TiO₂ films on silicon substrate at temperature of 550 °C. The XRD patterns of iron (Fe) doped films indicate the dominant phase of anatase. As discussed earlier in literature that anatase phase is the most important phase of TiO₂ which have better sensing capability for oxidizing and reducing gases [14]. The diffraction spectra of doped films have not shown any individual phase for Fe (iron). The Scherrer's formula is used to find the grain size of films and its obtained by expression shown below in equation (1);

$$D = 0.94\lambda/\beta \cos\theta \quad (1)$$

Where, λ is the wavelength of X-ray

β is the full-width at half maximum (FWHM),

and θ is angle between the incident and scattered X-ray.

The average grain size of pure TiO₂ and Fe-doped (1, 3 m/v %) thin films were found to be 79 nm, 64 nm, and 56 nm. The morphology of undoped and Fe-doped (1, 3 and 5 m/v %) TiO₂ thin films are shown in Figure 4 (a), (b), (c) and (d). The images confirm the formation agglomeration with micro-porous structure. Since, the surface with porous structure has large surface area, which has tremendous properties for gas sensing applications [15-17].

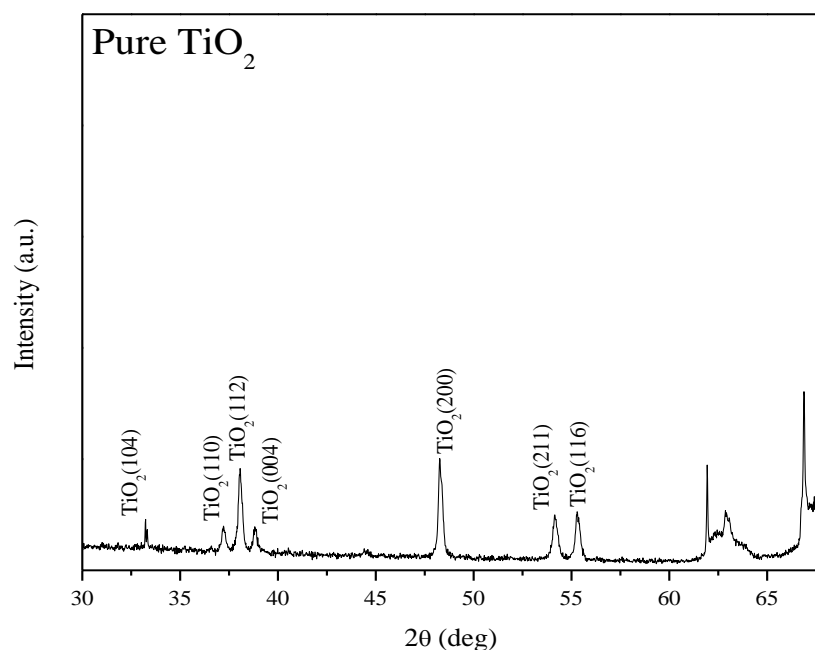


Figure 1. XRD pattern of undoped TiO₂ film fabricated on silicon annealed temperature at 550 °C.

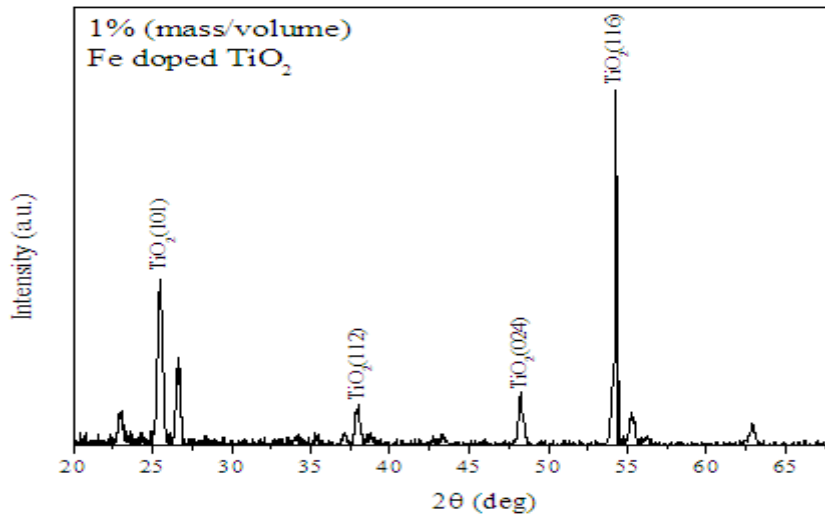


Figure 2. XRD pattern of 1 % (m/v) Fe doped TiO₂ film fabricated on corning glass annealed temperature at 550 °C.

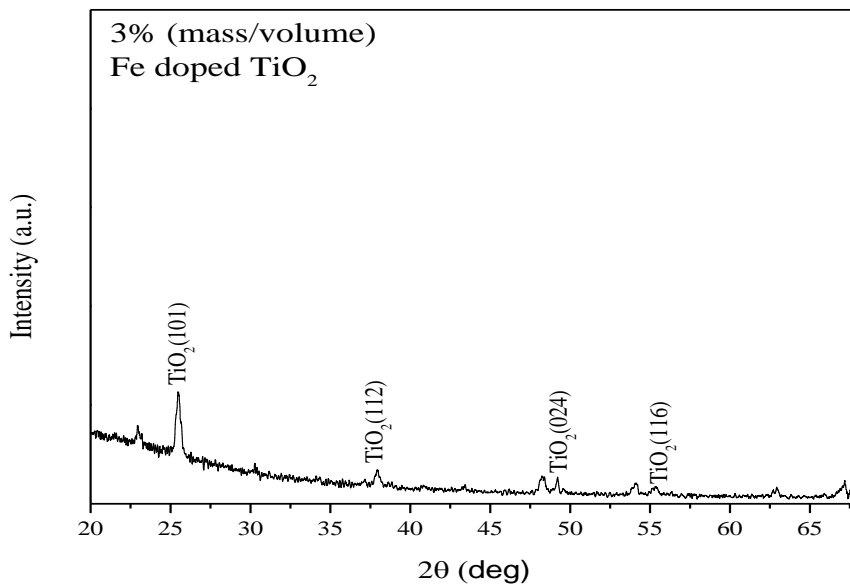
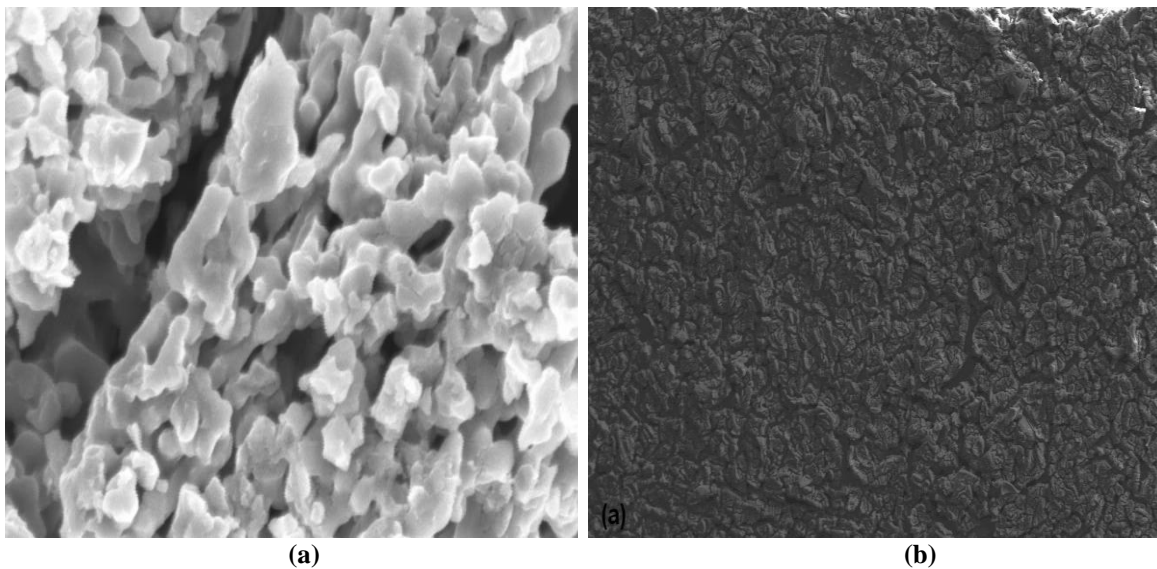


Figure 3. XRD pattern of 3 % (m/v) Fe doped TiO₂ film fabricated on silicon substrate annealed at 550 °C.



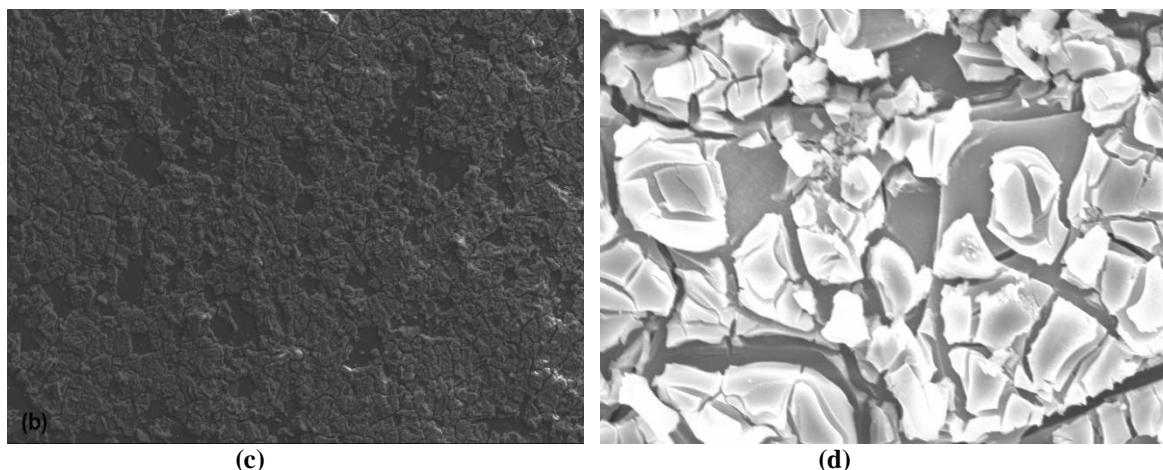


Figure 4. FESEM images of undoped (a), (b) 1%, (c) 3% and (d) 5 % m/v Fe-doped TiO₂ films.

CONCLUSION

Undoped and Fe-doped TiO₂ thin films were successfully fabricated by sol-gel technique. X-ray diffraction pattern confirms both the anatase and rutile phase for TiO₂ thin films. The change in crystallite was found due to the insertion of Fe³⁺ ions into titania surface. The morphology images confirmed the all films having the large surface area. All results suggest that the fabricated thin film showed decrease in grain size and increase in surface area with increase the doping level of iron(Fe) into TiO₂. Further, variation of doping level of Fe into titania and measuring specific level of gas as function of resistance may be a potential material for gas sensing application.

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