Preparation of Mixed Phase (Anatase/Rutile) TiO_2 Nanopowder by Simple Sol Gel Method

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***Abstract-* TiO_2 nanopowder having both anatase and rutile phases was prepared by a simple procedure using sol-gel method. Titanium isopropoxide was used as a titania source and mixed with methanol and TiO_2 nanopowder was obtained after annealing at 600^0C for 1 hour in air. The specimens made from this powder were characterized by X-ray diffraction (XRD), Thermogravimetric analyzer (TGA) and Transmission electron microscopy (TEM). XRD studies revealed the presence of both anatase and rutile phases with an average crystallite size of 35 ± 5 nm. No significant weight loss up to 700^0 C was observed by TGA curve which indicates that TiO_2 nanopowder is thermally stable. TEM revealed the presence of a number of crystalline grains in a structured matrix and selected electron diffraction pattern showed different arrangement of diffracted rings which confirms a phase evolution of crystalline grains of TiO_2 (anatase/rutile) due to thermal annealing. Mixed phase (anatase/rutile) TiO_2 nanopowder has been reported [1], [2] to exhibit improved photocatalytic and gas sensing properties. It is proposed to study the gas sensing behavior of these specimens during our research investigations on TiO_2 nanopowder.**

***Key words:* Sol-gel, TiO_2, anatase, rutile, XRD**

I. INTRODUCTION

TiO_2 is an important semiconductor material due to its wide range of applications in various fields such as photocatalysis, gas sensing, solar energy conversion etc [3], [4]. The phases of TiO_2 are anatase, rutile, and brookite. Among them rutile is a high temperature stable phase whereas anatase and brookite are metastable phases and transform to rutile on heating [5]. Mixed phase TiO_2 (anatase/rutile) nanopowder leads to improvement in photocatalytic and gas sensing properties [1], [2]. Thus it will be beneficial to obtain TiO_2 mixed phase (anatase/rutile) nanopowder by a simple sol-gel method. With this motivation the present study was undertaken.

In our investigation we optimize the synthesis method of TiO_2 nanopowder by changing the quantity of titanium isopropoxide which leads to a mixed phase (anatase/rutile) TiO_2 nanopowder with a reduced crystallite size at a lower annealing temperature as compared to data reported in the literature [4].

II. EXPERIMENTAL

3.5 ml of titanium isopropoxide is mixed with 40 ml methanol which results in a milky white solution. This solution is stirred vigorously using magnetic stirrer for 1:30 hrs. at a temperature about $57±3^0$C. Thus the gel produced is kept for 12 hrs. at room temperature for drying. The powder obtained is collected and annealed at 600^0C for 1 hr. in air. This results in the formation of mixed phase (anatase/rutile) TiO_2 nanopowder with an average crystallite size of 35±5 nm as revealed by XRD [4].

III. RESULTS

Fig. 1 shows X-ray diffraction pattern (XRD) of TiO_2 recorded using CuK_α radiation. Diffraction peaks showing the presence of both anatase and rutile phase are seen. The diffraction angles are in good agreement with the JCPDS card no 21-1272 for anatase, 21-1276 for rutile and the data reported in literature [5], [6], [7].

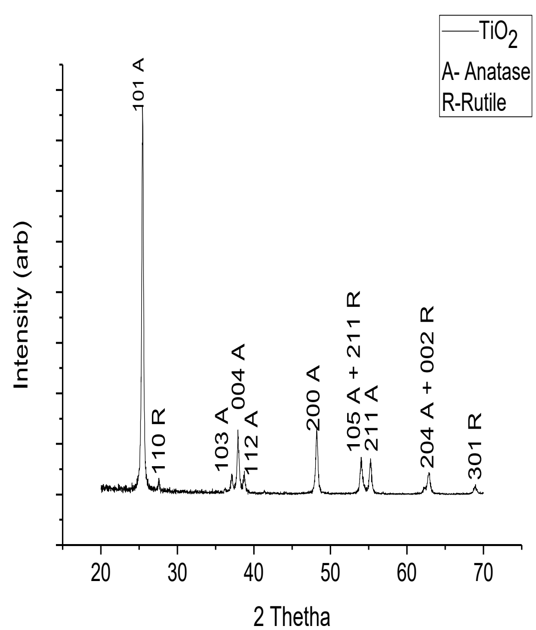

Fig. 1: X-ray diffraction pattern (XRD) of TiO_2 nanopowder.

Using Scherrer's formula [6] the average crystallite size is 35±5 nm and the content of anatase and rutile phase is calculated using formula $X_a = 100/ 1+ 1.265 (I_r/I_a)$ 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 [5]. In our investigation the content of anatase phase is about 97.27% whereas rutile phase is about 2.73%. Thermogravimetric analysis (TGA) was carried out at a heating rate of 10^0C/min from 30^0C to 700^0C in nitrogen atmosphere for TiO_2 nanopowder as shown in Fig. 2. It is observed that there is no significant weight loss upto 700^0C which indicates that the sample is thermally stable [8].

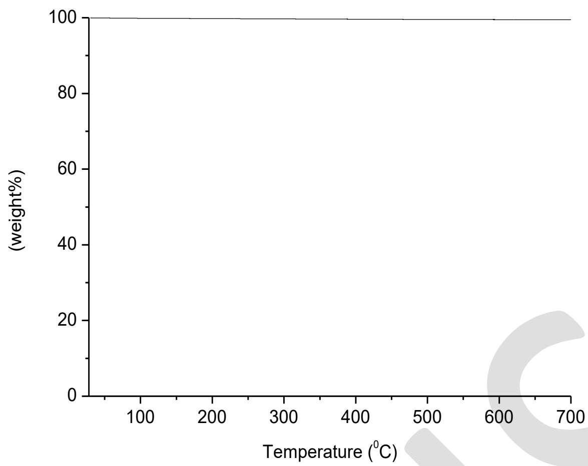

Fig. 2: TGA of TiO_2 nanopowder.

Fig 3 (a) shows TEM image of TiO_2 nanopowder which revealed the presence of a large number of crystalline grains in a structured matrix and surface morphology of TiO_2 nanopowder is shown in Fig.3 (b). Fig. 3 (c) represents different arrangement of dominant diffracted rings which confirms a phase evolution of crystalline grains of TiO_2 (anatase/rutile) due to thermal annealing [4], [9].

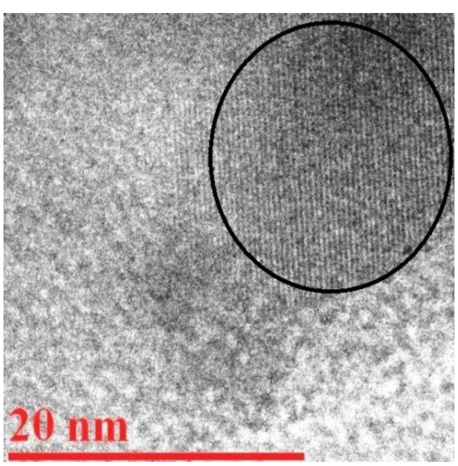

Fig. 3 (a)

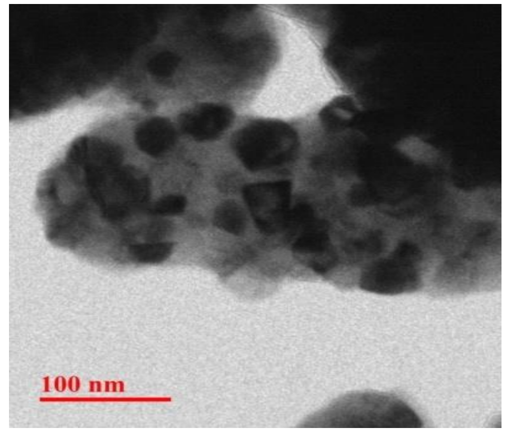

Fig. 3 (b)

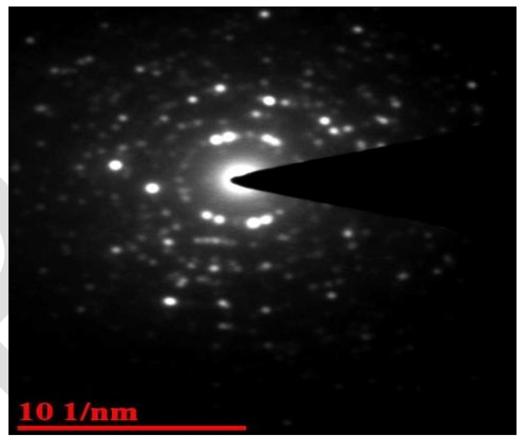

Fig. 3 (c)

Fig. 3: TEM image (a) showing crystalline grains in structured matrix (b) surface morphology (c) selected electron diffraction pattern of TiO_2 nanopowder

IV. DISCUSSION

X-ray diffraction pattern of specimen prepared using simple procedure in the present study shows the presence of both anatase and rutile phase with an average crystallite size of 35±5 nm. This is also supported by TEM investigations. Further, the specimens exhibit thermal stability till 700^0C. It is noteworthy that specimen with mixed phase of anatase/rutile of TiO_2 have been reported to show improved photocatalytic activity as compared to pure phases and even a small fraction of rutile phase along with anatase enhances photocatalytic activity [2]. Further it is reported that mixed phase (anatase/rutile) TiO_2 thin film annealed at 7000C may lead to improvement in gas sensing characteristics of NH3 [4]. Enachi et al. [1] observed that an individual TiO_2 nanotube with anatase/rutile crystal structure exhibits better gas response to H_2 at room temperature. Therefore synthesis of mixed phase TiO_2 by simple sol gel method may be employed to prepare specimens for these investigations. It is proposed to study the gas sensing behavior of these specimens during our research investigations on TiO_2 nanopowder.

V. CONCLUSION

1. TiO_2 nanopowder having anatase/rutile phase with an average crystallite size of 35±5 nm was prepared by simple sol-gel method.

2. TGA curve depicts no significant weight loss upto 700^0C which indicates that TiO_2 nanopowder is thermally stable.

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