

HYSYCVD Synthesis of 1D Nanostructures of TiO₂

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Abstract In this work, a synthesis route for 1D nanostructures (nanoribbons and nanofibers) of titanium oxide via hybrid precursor systems chemical vapor deposition (HYSYCVD) is proposed. The route is based on the thermal decomposition of K₂TiF₆ (solid precursor) in controlled air atmosphere. Synthesized TiO₂ corresponds to 1D crystalline nanostructures of Anatase, which can find potential applications in the development of thin-film transistor, dye-sensitized solar cells, and dosimeters. The versatility for synthesizing 1D TiO₂ nanostructures by HYSYCVD suggests the potential of the proposed route as economic and large-scale production technique.

Keywords Synthesis, Nanoribbons, TiO₂

1. Introduction

Titanium oxide (TiO₂) is a versatile metallic oxide semiconductor (n-type and 3.2 eV band gap) with a wide range of applications in different technological fields ranging from photocatalyst, chemical gas sensor, self-cleaning coating, antiseptic coatings, spintronic devices, dye-sensitized solar cells (DSSCs), thin-film transistor (TFTs) to dosimeters [1, 2]. Interest in ceramics based on nanostructured metallic oxides, such as, 1D nanostructures of TiO₂ has increased considerably in recent years, owing to their excellent properties and potential applications in TFTs, DSSCs and dosimeters. Nanostructured TiO₂ materials exhibit superior electrical and optical behaviour than the corresponding TiO₂ bulk materials. In addition, TiO₂ displays superior structural and functional properties in comparison with other oxides, and it is considered an efficient material for electronic devices [1, 3]. Crystalline TiO₂ presents polymorphisms, occurring in three polymorphic phases: Rutile, Anatase, and Brookite. The processing conditions and synthesis techniques influence the resulting crystalline structure, the physical and chemical properties. TiO₂ can be obtained by different techniques, such as: chemical solution decomposition, two-step wet chemical method, solgel, ultrasonic irradiation, solvothermal method, hydrothermal route, and chemical vapor deposition

(gas precursor systems) [2]. An alternative synthesis method for TiO₂ is the thermal decomposition of solid precursors or hybrid precursor systems chemical vapor deposition (HYSYCVD). Originally, HYSYCVD was developed by Leal-Cruz and Pech-Canul in 2002 [4, 5]. It is a modified CVD method based on thermal decomposition of solid precursors under controlled atmosphere for ceramics synthesis, such as Si₃N₄, Si₂N₂O, SiO₂, AlF₃, and Al₂O₃ [6]. In this work, a synthesis route via HYSYCVD in the K₂TiF₆-air system is proposed with the aim of synthesizing 1D nanostructures of TiO₂ intended for TFTs, DSSCs and dosimeters.

2. Experimental Procedure

TiO₂ was synthesized via thermal decomposition of solid precursor under controlled air atmosphere in a HYSYCVD reactor [4, 5]. K₂TiF₆ was used as titanium solid precursor and decomposed under the following processing conditions: temperature of 923.15 K, processing time of 2.5 h, heating rate of 10 °K/min, and atmospheric pressure. Prior to TiO₂ synthesis, K₂TiF₆ powders were compacted in a steel die (5 mm of diameter and 1mm of height) by biaxial compaction to obtain preforms. Processed preforms were removed and prepared for their characterization by X-ray diffraction (XRD), field emission scanning electron microscopy, and energy dispersive X-ray spectroscopy (FESEM/EDS). Additional, thermoluminescence (TL) characterizations were performed in the obtained materials.

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3. Results and Discussion

3.1. Phase Identification by XRD

Figure 1 shows the XRD patterns corresponding to a representative sample processed by HYSYCVD showing phases associated with the formation of TiO₂.

Characterization by XRD of processed samples (Figure 1b) reveals the presence of Anatase polymorph of TiO₂ (JCPDS No. 00-041-1049; titanium oxide (TiO₂) or Anatase; tetragonal, $a=b=4.14$ Å, $c=9.579$ Å; and 25.209, 47.874, 37.327, 53.533). K₂TiF₆ (JCPDS No. 00-073-2110; potassium titanium fluoride (K₂TiF₆); hexagonal, $a=b=5.715$ Å, $c=4.656$ Å; and 41.282, 26.258, 31.728, 17.907) and K_{2.67}TiO_{0.67}F_{5.33} (JCPDS No. 00-039-0781; potassium titanium oxide fluoride (K₂TiOF₅); cubic, $a=b=c=8.624$ Å; and 29.245, 41.845, 17.705, 20.512) are associated with titanium solid precursor and intermediate phases formed during the synthesis of TiO₂. TiO₂ and K_{2.67}TiO_{0.67}F_{5.33} (intermediated product) allows confirming the mechanism of TiO₂ formation through direct and intermediate routes during decomposition of solid precursor (K₂TiF₆) predicted and proposed in a previous work [6].

3.2. Morphological Analysis of Phases by FESEM Technique

Figure 2 shows a representative FESEM image and TL glow curve induced by gamma radiation corresponding to samples processed by HYSYCVD.

FESEM results reveal that TiO₂ corresponds to nanoribbons and nanofibers. EDS analyses confirm the presence of titanium (Ti) and oxygen (O) corresponding to chemical elements belonging to TiO₂ phase in 1D nanostructures. According to the literature [2, 7], nanoribbons and nanofibers of TiO₂ semiconductor can find potential applications in the development of thin-film transistor and dye-sensitized solar cells. On the other hand, the versatility for synthesizing 1D nanostructure of TiO₂ by HYSYCVD at relatively low temperature, short times, and in situ process suggests the potential of the proposed route for the economic and large-scale production of 1D nanostructures of TiO₂. Additional characterizations by thermoluminescence (TL) shows that synthesized TiO₂ presents luminescent properties, which makes it promising for potential applications in dosimeters.

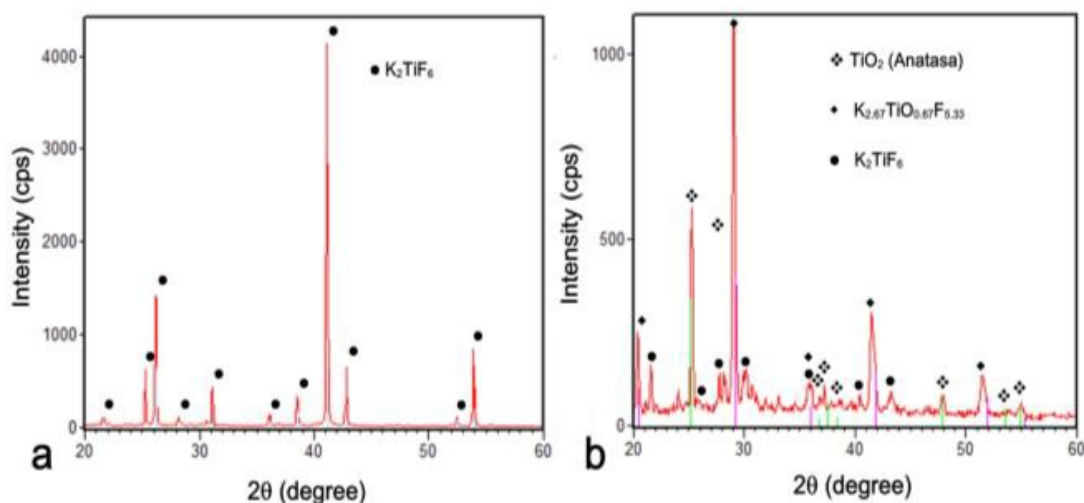


Figure 1. XRD patterns of K₂TiF₆ solid precursor before (a) and after (b) processing by HYSYCVD

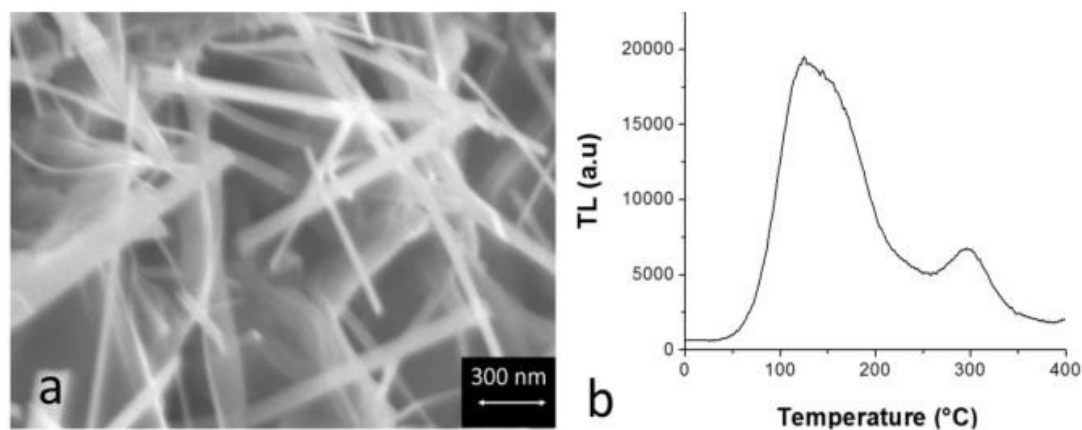


Figure 2. FESEM photomicrograph (a) and TL glow curve of samples processed via HYSYCVD

4. Conclusions

TiO₂-nanoribbons were successfully synthesized via the hybrid precursor systems chemical vapor deposition (HSYCVD) by the thermal decomposition of K₂TiF₆ under controlled air atmosphere. 1D nanostructures of TiO₂ can find potential applications on TFTs, DSSCs and dosimetry. Lastly, HSYCVD technique shows versatility for synthesizing TiO₂ and offers great potential to be considered as economic and large-scale production technique.

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