

Wet Oxidation of TiO₂ nanoflakes and Their Gas Sensing Properties

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Abstract

One of the popular synthesis methods of TiO₂ nanostructures is hydrothermal method. Besides offering huge amount of variety, the hydrothermal methods propose easy, clean and cheap processes. TiO₂ nanoflakes were fabricated on alumina substrate with wet oxidation in hydrogen peroxide solution. The formation process of nanoflakes and gas sensing properties were investigated. SEM (scanning electron microscope) was used to display the surface of structures covering the samples. Various concentrations of hydrogen, ethanol, carbon monoxide, nitrogen oxide and acetonitrile gases were tested under humid airflow in temperature range between 350-600 °C.

Key words: TiO₂ nanoflakes, wet oxidation, metal oxides, gas sensor, hydrogen peroxide.

Introduction

Titanium dioxide (TiO₂) nanostructures are favourite material for many applications like; photocatalysis [1], biosensors [2], optoelectronics etc.. Among synthesis methods, the majority are hydrothermal-based because of its low-cost, low temperature and clean process [3]. Hydrolysis of Ti in alkaline solutions and hydrations of Ti salts are common ways for hydrothermal synthesis of TiO₂ nanostructures [4]. The multi-step processes and contamination risk by other chemicals are main handicaps for both techniques. On the other hand, the hydrolysis of Ti in hydrogen peroxide (H₂O₂) solution is advantageous because it offers an easy experimental process and direct oxidation of Ti without any risks of contamination by other chemicals. In this study, authors investigated gas-sensing properties of TiO₂ nanoflakes synthesized by wet oxidation in H₂O₂ solution.

Experimental

Polycrystalline alumina (2 mm x 2 mm) substrates (Kyocera, Japan) were used as substrates. Substrates were cleaned with ultrasonic bath in acetone, ethanol and deionized water for 5 minutes respectively. Ti films (100 nm) were deposited on substrates with magnetron sputtering plant (Kenotec, Italy). The depositions were carried out at 300 °C under 6.6x10⁻³ mbar working pressure with 75 W RF magnetron power and lasted for 55

minutes. Then, prepared Ti thin films on alumina substrates were immersed in 15 ml 2% H₂O₂ solution at 80 °C for 20 hours. This process was repeated three times to get totally bleached surfaces. The samples were annealed in furnace at 450 °C for 4 hours under room conditions to get better crystallization and to stabilize samples.

The surface morphology of bleached films was examined using SEM (Leo 1525 Gemini model; Carl Zeiss AG, Oberkochen, Germany) operated at 3 kV and 5 kV.

The device configuration and system for gas measurements were given at our previous work [5]. The samples were tested against various concentrations of hydrogen (H₂), ethanol (C₂H₆O), carbon monoxide (CO), nitrogen dioxide (NO₂), acetonitrile (C₂H₃N) gases. The all gas measurements were taken under 40% relative humidity. The sensing properties of TiO₂ nanoflakes were tested between 350-600 °C with 50 °C steps.

Results and Discussions

The SEM image of TiO₂ nanoflakes was shown in figure 1. The flakes were homogeneous through the surface. There are some small residuals from chemical process.

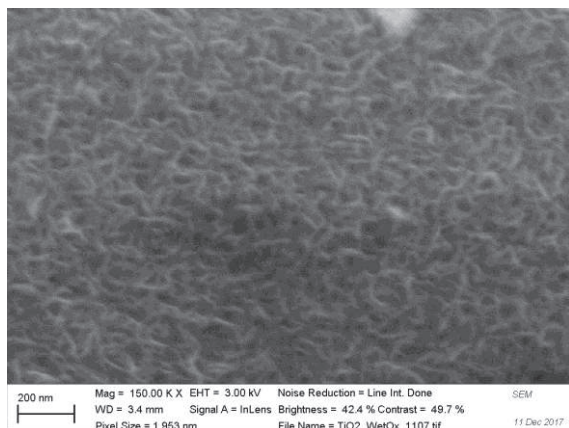


Fig. 1. SEM image of TiO₂ nanoflakes.

The sensor response towards all gases in 500 °C is shown in figure 2. The sensors show responses to all kinds of gases at 500 °C. There was no variation of the conductance for 2 ppm and 5 ppm NO₂ gas but there was a small change for 10 ppm NO₂. TiO₂ nanoflakes showed no sensor response to gases at 350 °C. The sensor showed better sensing towards hydrogen and ethanol gases than other gases in 400-600 °C range. The measurements will be repeated again to see the stability of sensors.

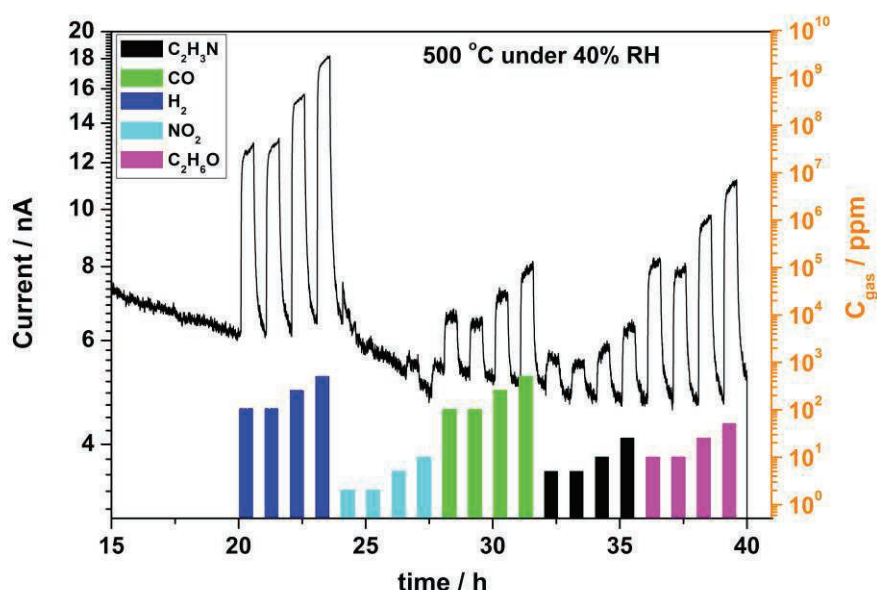


Fig.2. Measurement result of TiO₂ nanoflakes at 500 °C under 40% RH.

Conclusion

TiO₂ nanoflakes were synthesized on alumina substrates with immersing Ti thin films in hydrogen peroxide solution. Up to now, basic surface characterization nanoflakes and first gas measurements were done. In future study, sensing mechanism will be investigated deeply with comparison different synthesis and measurement conditions.

References

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