

**UNIVERSITI TEKNOLOGI MARA**

**LOW DIMENSIONAL TITANIUM  
DIOXIDE NANOWIRES  
SYNTHESIZED BY  
HYDROTHERMAL  
AUTOCLAVE METHOD**

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Thesis submitted in fulfillment  
of the requirements for the degree of  
**Doctor of Philosophy**

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## **AUTHOR'S DECLARATION**

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research

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## ABSTRACT

Titanium dioxide, TiO<sub>2</sub> nanowires are an emerging class of TiO<sub>2</sub> nanostructures. Hydrothermal is the selected method in this study because of its simplicity and works relatively at low temperatures, which has the advantage of easy control over the synthesis process. Synthesizing TiO<sub>2</sub> nanowires using alkaline hydrothermal method required a very long reaction time which is more than 20 hours. Therefore, it is important to produce TiO<sub>2</sub> nanowires in a short time and the effect of several processing parameters such as hydrothermal growth time, hydrothermal growth temperature, solvent concentration, precursor concentration, filling fraction and annealing temperature on the morphology and structural properties of low dimensional TiO<sub>2</sub> nanowires were systematically investigated. X-ray diffraction (XRD) pattern showed that the produced nanowires showed high crystallinity and mainly in the anatase phase TiO<sub>2</sub>. Raman spectroscopy of TiO<sub>2</sub> nanowires supported the results of XRD analysis, i.e., the predominance of the anatase phase TiO<sub>2</sub>. The shorter growth time of 6 hour is a novel finding in this study. From FESEM images, it was suggested that the starting material which is TiO<sub>2</sub> nanoparticle rearranged itself into an elongated structure as it received sufficient amount of energy within the reaction time as low as 3 h to completely transform into nanowires product and this result is also support the explanation that TiO<sub>2</sub> nanowires are directly formed during the hydrothermal process. The morphology and structural properties of hydrothermally synthesized TiO<sub>2</sub> nanowires are temperature-dependent. It was found that suitable synthesis temperature is 150°C. Results from XRD and Raman spectra revealed that the structural properties of nanowires are significantly affected by the percentage of filling fraction of solution in autoclave, the concentration of precursor solution and the concentration of solvent. The effect of annealing temperature from 400°C to 900°C was investigated. From FESEM observation, it was discovered that the TiO<sub>2</sub> nanowires maintain its structure up to 500°C, while annealing at 600°C resulted in the breakage of nanowires into smaller particles, consequently underwent further transformation from the anatase to the rutile phase with simultaneous recrystallization to rod-like structures at temperature of 900°C and this was revealed by XRD results. The silicon doped TiO<sub>2</sub> (TiO<sub>2</sub>: Si) nanowires have been successfully synthesized and the effect of silicon content (wt%) on the morphology, structural and optical band gap were investigated. The silicon dopant promoted the formation of rutile phase rather than anatase phase TiO<sub>2</sub> as shown by XRD pattern and revealed by Raman spectroscopy. The silicon doped TiO<sub>2</sub> (TiO<sub>2</sub>: Si) nanowires has optical band gap of ~3.00 eV compared to TiO<sub>2</sub> nanowires which exhibited band gap of 3.28 eV.

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