FORMATION OF IRON OXIDE NANOWIRES BY USING THERMAL OXIDATION OF IRON

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This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials)(Hons.)

by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory is as follow:

………………………………

(Project Supervisor)

ABSTRAK

Oksida ferum *nanowires* digunakan secara meluas dalam pelbagai aplikasi, termasuklah fotopemangkinan. Konvensional, oksida ferum *nanowires* dihasilkan oleh teknik-teknik seperti; proses sol-gel, kaedah templat, penguraian terma, proses hidroterma Walau bagaimanapun, teknik-teknik ini mahal dan rumit. Oleh itu, projek ini bertujuan untuk menghasilkan oksida ferum *nanowires* dengan menggunakan pengoksidaan terma ferum. Beberapa paremeter telah disiasat seperti kesan masa pengoksidaan, suhu pengoksidaan dan keadaan pada substrat. Morfologi dan fasa Fe_2O_3 *nanowires* yang dibentuk telah disifatkan. Pemerhatian FESEM telah menunjukkan bahawa Fe_2O_3 struktur *nanowires* telah terbentuk pada kepingan Fe tulen. XRD dan spektroskopi Raman mengesahkan fasa fasa: α- Fe_2O_3, FeO dan Fe_3O_4 terbentuk selepas process pengoksidaan manakala EDX digunakan untuk memastikan *nanowires* yang terhasil mempunyai oksigen dan ferum. Raman spektroskopi telah digunakan untuk menyokong morfologi dan fasa yang diperoleh dari analisis SEM dan XRD. Sampel (pengoksidaan pada 180 min, 500 °C, dalam udara tanpa menitikkan asid sulfurik) adalah tertakluk kepada ujian degradasifoto di bawah cahaya UV. Hasil daripada degradasifoto ini menunjukkan semakin lama masa larutan *methyl orange* terdedah kepada cahaya UV, semakin pudar larutan itu. Untuk memastikan larutan itu mempunyai perubahan warna, analisis menggunakan *UV visible* digunakan. Berdasarkan keputusan yang diperoleh, maka boleh disimpulkan bahawa larutan itu berlaku proses degradasi apabila masa pendedahan kepada cahaya semakin menigkat.

ABSTRACT

An iron oxide nanowire is used widely in a range of applications, which include photocatalysis. Conventionally, iron oxide nanowires are produced by techniques such as; sol-gel process, template method, thermal decomposition, hydrothermal process However, these techniques are costly and complicated. Hence, this project serves to fabricate the iron oxide nanowires by using thermal oxidation of iron. Several parameters were investigated such as the effect of oxidation time, oxidation temperature and condition on the substrate. The morphologies and phases of Fe_2O_3 nanowires formed were characterized. FESEM observation showed that Fe_2O_3 nanowires structure was successfully formed on pure Fe foil. XRD and Raman spectroscopy confirmed variant phase: α- Fe_2O_3, FeO and Fe_3O_4 occurred after oxidizing while EDX was used to confirm that the nanowires consisted of oxygen and iron. Raman spectroscopy was used to support the morphologies and phases observed by SEM and XRD. The samples (oxidized at 180 min, 500°C, in air without dropping sulphuric acid) were subjected to photodegradation testing under UV light conditions. The result obtain from this photodegradation shows that, as increase the time of methyl orange solution exposed to UV light, the solution is become fades. To ensure that the solution has changed color, UV visible analysis is used. Based on the results obtained, it can be concluded that the solution was effective degradation process when the time of exposure to light progressively increase.

DEDICATION

Dedicated to my beloved family members especially my parents, lecturers, and also to all my friends.

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I would like to take this opportunity to express my gratefulness and gratitude to those who have rendered their valuable advice, support and guidance to me during the long journey of completing this research. First and foremost, I would like to thank my supervisor Dr. Syahriza Ismail for her relentless support, advice and guidance throughout the entire project. Not to forget the research members: Azreen and Nadia for all the assistance and guidance provided all this while. I am greatly touched by the commitments and dedications they have shown. Without the support and guidance, I would not be able to complete this research. Besides, without the moral support and understanding from my family especially my father, En Saad Talib and my late mother, Pn Kelsom Musa, it would be impossible for me to complete this research. I wish to express my biggest gratefulness and love to my family members. Finally, I wish to say that I treasure very much the friendship of my friends who have been very supportive in providing all necessary help and advice during the journey of completing this research.

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CHAPTER 1

INTRODUCTION

This chapter describe the introduction of the project. In this project, thermal oxidation process was used to form iron oxide nanowires. This chapter also include background, problem statement, objective, scope and project outline of the study.

1.1 Background

Nanostructure refers to materials system with at least one dimension is in the range of ~1-100nm. In a nanostructure, electrons are freely move in other dimension eventhough there are confined in nanoscale dimension. As compared to individual atom and molecules, the nanostructure is mostly unique.

Nanowires and nanobelts are semi one dimensional nanostructure based oxides, they have pulled in much consideration lately. They show unordinary optical, electronic, attractive and mechanical properties as contrasted with those of mass materials. It is because of the huge surface related deformity starting from the high surface to volume degrees. Moreover, some paramount properties of one dimensional nanostructured based oxide are great high temperature steadiness, oxidation safety and stable electric properties. Thus, it demonstrates their potential basic building blocks for new classes of environmentally conscious electronics (Yang *et al*., 2010). Since the nanowires is studied, some paramount is important which are the control of morphology, size and growth direction. Figure 1.1 shows the morphology of nanowire (Hiralal *et al*., 2008a) while Figure 1.2 shows the schematic diagram of single nanowire arrays (Rahim *et al*., 2014). In addition, the different of morphologies get will affect the properties with unique application. A suitable alignment of substrate is needed for an ensemble of nanowires (Wen *et al.,* 2005).

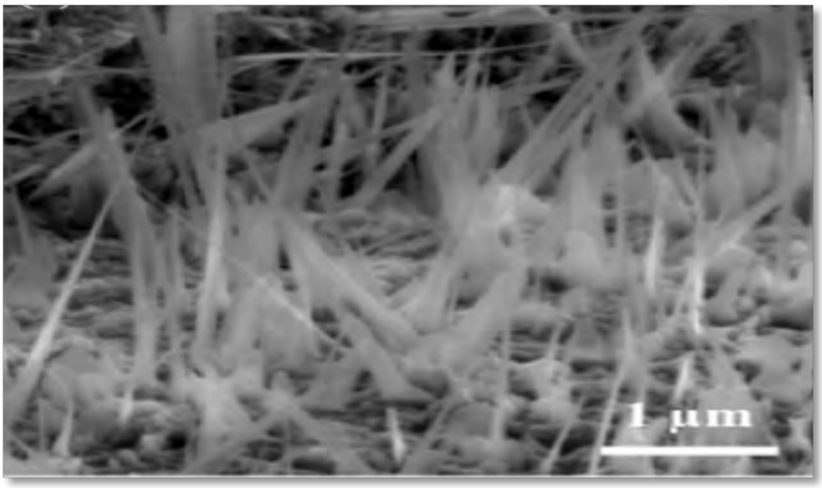

Figure 1.1: Morphology of single nanowires (*Hiralal et al*., 2008).

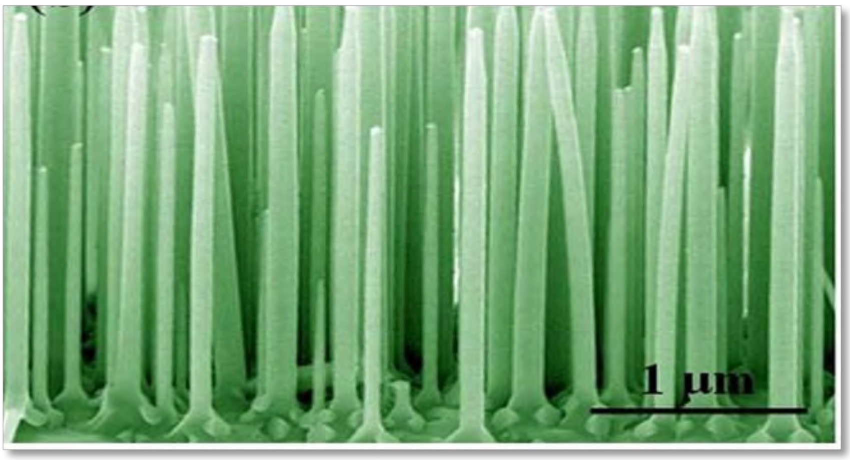

Figure 1.2: Schematic diagram of single nanowire array (*Rahim et al*., 2014).

Recently, nanowires have been synthesized by several different methods, such as the hydrothermal process, the template method, the vapour–solid process, sol–gel technique and thermal decomposition (Liu *et al*., 2008). Nevertheless, thermal oxidation in various oxidizing atmosphere is a most simple, cheap and direct procedure to form iron oxide nanowires (Grigorescu *et al.*, 2012) shows in Figure 1.3.

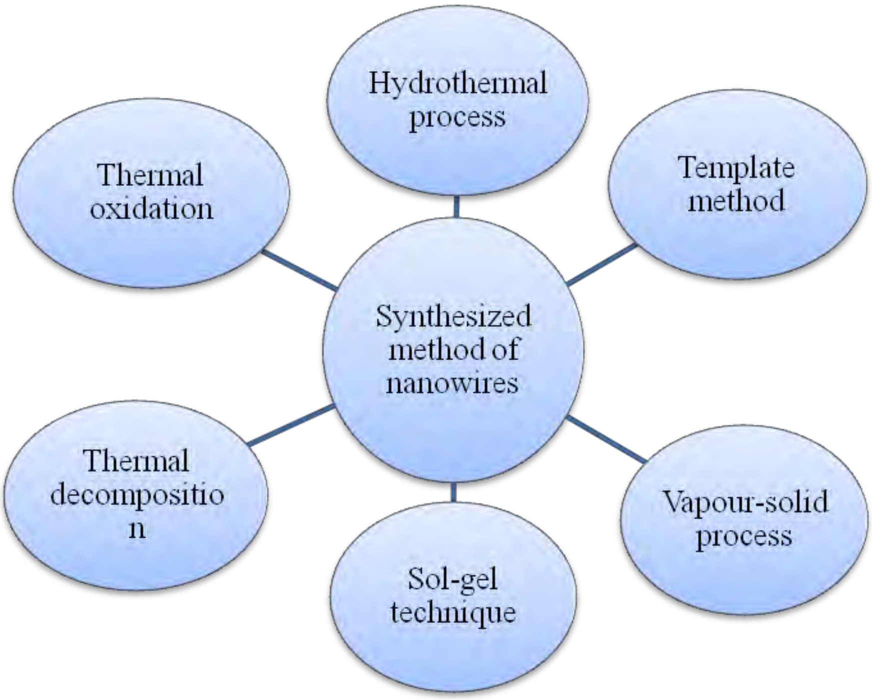

Figure 1.3: Various synthesized method of nanowires.

The growth of nanowires on the surface of iron is depending on the condition. Under typical condition, layer oxide will be formed, as hematite, magnetite and wustite. Hematite is formed at outer layer while magnetite as an intermediate layer and wustite layer form on iron iron substrate. As n-type semiconductor, which has bandgap 2.1 eV, hematite is environmental friendly, not poisonous, corrosion resistant and also the cost is low. Figure 1.4 shows the usage of hematite.

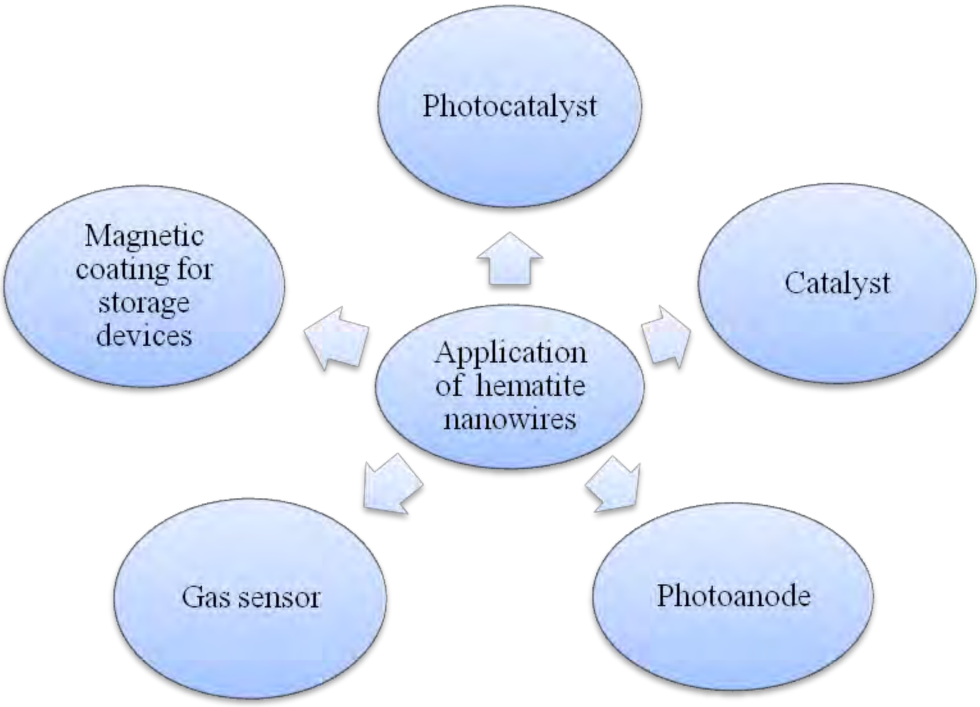

Figure 1.4: Application of hematite nanowires

Among of these application, one of interesting is as photocatalyst (Figure 1.5). Photocatalytic reactions promoted by aqueous suspensions of nano sized transition metal oxides have been subject of abundant number of resent research (Bakardijeva *et al*., 2007) with the semiconductor performance, iron oxide is use in solar photoelectrolysis cells. Hematite is a good catalyst for photooxidation of sulphite. The details on this iron oxide will be reviewed in chapter 2.

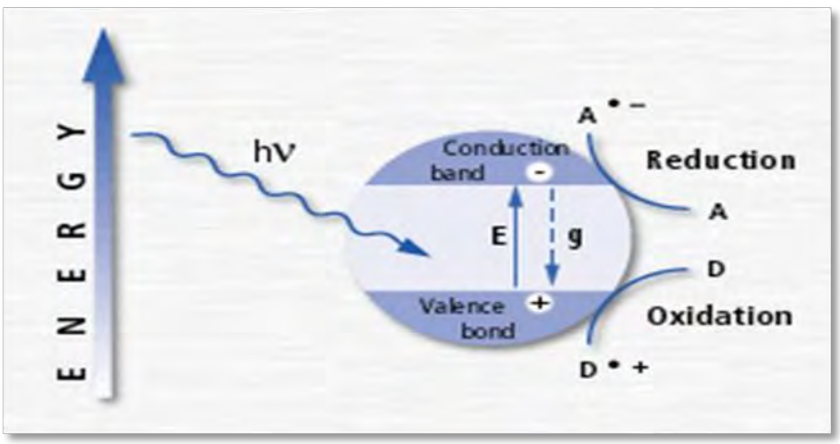

Figure 1.5: Photocatalytic process (Ctibor et al., 2013)

1.2 Problem Statement

Since the time that Fujishima and Honda reported in 1972, TiO_2 photoelectrode methodology is utilized to create hydrogen from water. Ways and intends to utilize daylight as an essential vitality hotspot for hydrogen fuel era has been an exceptionally examined point in science and engineering, with a specific end goal to accomplish an upgraded daylight reaction, materials must have a suitably low band crevice and ready to part water, and additionally have suitable band edge positions in respect to the red-ox capability of water. Subsequently, hematite, with band hole of 2.1 eV has recovered solid examination enthusiasm to be utilized as terminal water part for hydrogen generation. These are because of the cost, less advanced yet plentiful materials.

Moreover, because of the looked into that had being carried out, it demonstrated that, the examination about arrangement of iron oxide nanowires by utilizing warm oxidation system was very constrained. It is accepted that nanowires develop from tip while reactant (metal) diffuse by means of the surface from the metal substrate. However the accurate instrument of development is a long way from clear. All the more as of late, different gatherings have returned to the spontaneous development of oxide nanowires by warm oxidation of metallic substrates (Fe, Zn and Cu), despite the fact that a definite orderly study is needing.

Photocatalytic response advanced by watery suspensions of nano estimated move metals oxide have been subject plenteous number of exploration (Bakardijeva *et al.*, 2007). Iron oxide has been concentrated on broadly for utilization in sunlight based photoelectrolysis cells. Bamba *et.al.,* (2006) considered the photocatalytic oxidation of a few dangers at TiO_2 and Fe_2O_3, surfaces. They found that TiO2 powder is dynamic photocatalyst for cyanide oxidation, while no oxidation was watched for Fe_2O_3. Recently a few studies exhibited the confirmation for photograph reactant arrangement of OH radicals. The era of OH radicals is attained through the usage of costly oxidants such ozone and H_2O_2 often in mix, with or without UV radiation. Nonetheless, in this theory, the photocatalytic action of iron oxide got by warm oxidation technique which is this system is basic and immediate method.

1.3 Research Objectives

This research is about the formation of iron oxide nanowires and followings are the objectives for this work:

a) To produce the iron oxide nanowires formed by thermal oxidation
b) To study the mechanism of iron oxide nanowires by thermal oxidation
c) To study the photocatalysis of iron oxide formed by thermal oxidation

1.4 Scopes of Work

This research covered the study on the formation of Fe_2O_3 nanowires. For the synthesis of Fe_2O_3nanowires, thermal oxidation process was selected. For thermal oxidation process, the effect of temperature, condition on the substrate and time were studied. Besides, the photocatalytic application was also considered.
Phase and morphology characterization on the Fe_2O_3 nanowires was examined by using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Electron Dispersive X-Ray Spectroscopy (EDX), Raman Spectroscopy, UV Visible Spectrometry and UV lamp chamber.

1.5 Project Outline

This project was conducted to form the iron oxide nanowires structure by thermal oxidation process. This process was performed in vacuum furnace at temperature 300 °C – 700 °C to form the nanowires. The photocatalytic ability of the formed oxides it tested by using the UV light irradiation.

Chapter one is the introduction for this whole project while in the chapter two is explain about nanostructure materials, thermal oxidation process and also the