EFFECT OF RADIATION INDUCED METHYL METHACRYLATE GRAFTING ON STRUCTURAL AND OPTICAL PROPERTIES OF SILICON NANOWIRE

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ABSTRACT

Silicon nanowires (SiNWs) is a one-dimensional nanostructured material that had been widely studied due to its potential applications in various fields. Combination of polymers and nanostructured materials offers great potential for enhanced material with many possible applications. This study aims to determine the influence of radiation induced methyl methacrylate (MMA) grafting on structural and optical properties of SiNWs. Six identical SiNWs were prepared using RF magnetron sputtering. Five samples were grafted with MMA by radiation induced grafting under the electron beam dose of 10 kGy, 30 kGy, 50 kGy, 70 kGy, and 90 kGy. The structural analysis of the samples were performed by X-ray diffraction, ATR-FTIR spectroscopy, Raman spectroscopy, FESEM, and EDX spectroscopy while the optical characteristics were measured using UV-vis and photoluminescence spectroscopy. The grafting percentage was in the range of 0.20% to 0.35%. XRD pattern showed new peak appearing at 53° after MMA was grafted. ATR-FTIR and Raman Spectra showed the existence of bonding between MMA and silicon nanowires, which suggest the mechanism of grafting process. These results were also shown in the FESEM images and EDX spectra by the observable MMA layer with thickness in the range of 81.96 nm to 162 nm. The UV-vis absorption spectra showed that the reflectance at the wavelength of 850 nm to 1100 nm was increased as the grafting percentage was increased. Tauc plot showed that the energy band gap increased dramatically from 1.07 eV to 1.17 eV as MMA was grafted and the energy band gap dropped down to 1.12 eV as the grafting percentage is increased. Photoluminescence spectra showed increase of peak intensity as the MMA was grafted on the samples. The structural analyses confirmed that MMA can be combined with SiNWs via radiation induced grafting and the grafting percentage can be increased by increasing the radiation dose of electron beam. The optical characterisations showed that the MMA grafted SiNWs did not strongly affect the optical band gap and the stable absorption at the NIR region still gave this material a great potential for use in photovoltaic, photonics and photosensitive device application.

ABSTRAK

Wayar nano silicon (SiNWs) ialah bahan struktur nano satu dimensi yang telah dikaji secara meluas disebabkan potensinya untuk aplikasi dalam pelbagai bidang. Kombinasi polimer dan bahan struktur nano menawarkan potensi yang hebat untuk bahan yang dipertingkat dengan kemungkinan untuk banyak aplikasi. Matlamat kajian ini adalah untuk menentukan pengaruh cantuman metil metakrilat (MMA) oleh sinaran terhadap sifat struktur dan sifat optik SiNWs. Enam sampel SiNWs yang sama disediakan menggunakan percikan RF magnetron. Lima sampel dicantum dengan MMA dengan teknik cantuman oleh sinaran dengan dos alur elektron 10 kGy, 30 kGy, 50 kGy, 70 kGy, dan 90 kGy. Analisis struktur sampel ditentukan menggunakan pembelauan sinar X, spektroskopi ATR-FTIR, spektroskopi Raman, FESEM, dan spektroskopi EDX manakala ciri optik diukur menggunakan spektroskopi UV-vis dan fotoluminesens. Peratus cantuman berada pada julat 0.20% hingga 0.35%. Corak XRD menunjukkan puncak baharu muncul pada 53° selepas MMA dicantumkan. Spektroskopi ATR-FTIR dan Raman menunjukkan kewujudan ikatan antara MMA dan wayar nano silikon yang mencadangkan mekanisme kepada proses cantuman. Keputusan ini juga ditunjukkan dalam imej-imej FESEM dan spektrum EDX melalui lapisan MMA yang boleh dilihat dengan ketebalan dalam julat 81.96 nm hingga 162 nm. Spektrum serapan UV-vis menunjukkan bahawa pantulan pada panjang gelombang 850 nm hingga 1100 nm meningkat apabila peratus sambungan meningkat. Plot Tauc menunjukkan peningkatan dramatik jurang jalur tenaga daripada 1.07 eV ke 1.17 eV setelah MMA disambungkan dan jurang jalur tenaga menurun ke 1.12 eV apabila peratus cantuman meningkat. Spektrum fotoluminesens hanya menunjukkan peningkatan keamatan puncak setelah MMA dicantumkan di atas sampel. Analisis struktur mengesahkan bahawa MMA boleh dicantumkan dengan SINWs melalui cantuman dengan sinaran dan peratus cantuman boleh ditingkatkan dengan meningkatkan dos sinaran alur elektron. Ciri-ciri optik menunjukkan bahawa SiNWs yang dicantumkan dengan MMA tidak terlalu menjejaskan jurang tenaga optik dan penyerapan yang stabil di rantau NIR masih memberi bahan ini potensi yang besar untuk digunakan dalam aplikasi fotovoltaik, fotonik dan aplikasi fotosensitif.

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LIST OF SYMBOLS

a-Si	-	Amorphous silicon
Ag	-	Silver
Al	-	Aluminium
Ar	-	Argon
at. %	-	Atomic percentage
Au	-	Gold
$BaSO_4$	-	Barium sulfate
°C	-	Degree Celcius
c-Si	-	Crystalline silicon
CdTe	-	Cadmium tellurite
Co	-	Cobalt
Cu	-	Copper
d	-	Sample thickness
D_s	-	Diffusion coefficient
E_g	-	band gap energy
E_n	-	diffusion barrier
GaN	-	Gallium nitrade
GaAs	-	Gallium Arsenide
Ge	-	Germanium

h	-	Planck constant
He	-	Helium
HF	-	Hydrofluoric acid
hr	-	Hour
In	-	Indium
InP	-	Indium Phosphide
J_{sc}	-	short-circuit current density
k	-	Boltzmann constant
keV	-	Kilo electron Volt
kGy	-	KiloGray
kV	-	Kilo Volt
mA	-	milliampere
MeV	-	Mega electron Volt
N_2	-	Nitrogen
n-type	-	Region in a semiconductor in which electrical conduction is due chiefly to the movement of electrons
nc-Si	-	Nanocrystalline silicon
Ni	-	Nickel
OH^-	-	Hydroxide anion
Pd	-	Palladium
Pt	-	Platinum
p-i-n junction	-	Interface at which p-type silicon, intrinsic silicon and n-type silicon make contact with each other

p-n junction	-	Interface at which p-type silicon and n-type silicon make contact with each other
p-type	-	Region in a semiconductor in which electrical conduction is due chiefly to the movement of positive holes.
Rh	-	Rhodium
sccm	-	Standard cubic centimetre per minute
Si	-	Silicon
$SiCl_4$	-	Silicon tetrachloride
SiH_4	-	Silane
SiO_2	-	Silicon dioxide
Sn	-	Tin
T	-	transmission
T_a	-	annealing temperature
TiO	-	Titanium oxide
V_g	-	Galvanic voltage
V_{oc}	-	open circuit voltage
W_0	-	Initial weight before radiation grafting
υ_{as}	-	asymmetric stretching
υ_s	-	symmetric stretching
ν	-	Frequency

LIST OF ABBREVIATIONS

AIBN	-	Azobisisobutyronitrile
ATR	-	Attenuated total reflectance
BMA	-	Butyl methacrylate
C–V	-	Capacitance-voltage
CCD	-	Charge coupled device
CEP	-	Cyanoethyl pullulan
CIGS	-	Copper indium gallium selenide
CIS	-	Copper indium selenide
CQDs	-	Quantum dots
CVD	-	Chemical vapour deposition
DIY	-	Do it yourself
DSSC	-	Dye-sensitised solar cell
EDX	-	Energy dispersive x-ray
EHMA	-	Ethyl hexyl methacrylate
EHT	-	Electron high tension
EMA	-	Ethyl methacrylate
EO	-	Ethylene oxide
FESEM	-	Field emission scanning electron microscope
FIB	-	Focused ion beam

FTIR	-	Fourier-transform infrared spectroscopy
FWHM	-	Full width half maximum
GeQDs	-	Germanium quantum dots
H-SiNWs	-	Hydrogenated silicon nanowires
I-V	-	Current-voltage
MAA	-	Methacrylic acid
MBS	-	Methacrylate-butadiene-styrene
MIS	-	Metal-insulator-semiconductor
MMA	-	Methyl-methacrylate
NIR	-	Near infrared
PECVD	-	Plasma-enhanced chemical vapour deposition
PMMA	-	Poly methyl-methacrylate
PVC	-	Polyvinyl chloride
RF	-	Radiofrequency
SERS	-	Surface-enhanced Raman scattering
SiNWs	-	Silicon nanowires
UV-vis	-	Ultraviolet to visible range
VFB	-	Flat band voltage
VLS	-	Vapour-liquid-solid
XRD	-	X-ray diffraction
YBCO	-	Yttrium barium copper oxide

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CHAPTER 1

INTRODUCTION

1.1 Background

Nanotechnology is known to be a relatively new issue in scientific research. However, the development of its core concept had happened a long time ago. The word 'nano' is a prefix denoting 10^{-9}. This word comes from the Greek word, *nanos* which means "dwarf". In nanotechnology point of view, it refers to things of one-billionth of a metre in size. The word *nanotechnology* was first mentioned in 1974 by Norio Taniguchi in his paper titled "On the Basic Concept of Nano-Technology" (Taniguchi, 1974). The earliest systematic discussion about nanotechnology was by Richard Feynman when he gave a speech entitle "There's Plenty Room at the Bottom". In this speech, he talked about the importance of controlling and manipulating things on a small scale and how these could give us information about the strange phenomena that occur in a complex situation. The invention of scanning tunnelling microscope and the atomic force microscope in the 1980's had allowed scientists to observe material at an atomic level. This invention had picked the interest of so many researchers to jump in field of nanostructured materials.

Nanostructured materials are materials with the typical characteristic length of a few nm, typically 1 to 100 nm. Scientists and engineers have been trying to make things smaller with the central tenet and primary motivation of making products do more with less. Main example that can be seen nowadays are cell phones. This technological marvel can relatively do everything. The benefits of smaller systems can justify the cost inherent in designing and building them. Shrinking a device would use less material to build. Material, same as access baggage, costs money, adds weight, and take up space. These considerations are very crucial in engineering and scientific point of view for certain applications such as satellites and spacecraft. Taking heavy things to space is expensive and inefficient. In 2004, the cost of launching a space shuttle was about 10,000 dollars per pound. Thermal distortions and vibrations do not perturb smaller devices as much as large ones due to vibration frequency of a system that is inversely proportional to its mass (Puchades *et al.*, 2012) . Smaller things also tend to require less energy to function. Other than that, miniaturisation also helps in optimising the power density or the amount of power generated per unit volume.

The nanomaterials field is the field that develops or studies materials with unique properties arising from their nanoscale dimension. The research in nanomaterials is done to understand the changes of material properties as the amount of material is diminished, even all the way down to individual atoms, and how very small piece of material acquire unique properties and unique applications. A stable structure is organised, typically as solid or even an ordered array of just a few atoms. A stable structure can be synthetic or naturally occurring. The important thing is that the features endows the structure with unique and useful physical properties that do not exist at larger size. For example, the melting point of Au is 1064 °C. However, the melting point drops to 750 °C when the size is diminished to a few nanometre (Font and Myers, 2013). Other than that, the liquid filled with nanometre –sized Au ball turns into liquid red and not gold. Au nanoparticles are just one of the many structures having unique and usable properties owing solely to their small size.

Nanomaterials are materials, which are characterised by an ultra-fine grain size. Nanomaterials can be created with various modulation dimensionalities which is defined as zero (atomic cluster, filament cluster assemblies), one (multilayer), two (ultrafine-grained over layer or buried layers), and three (nano-phase materials consisting of equiaxed nanometre sized grains).

As the properties of nanomaterials are surface dependent, there are a number of ways one can bring about surface changes leading to concomitant changes in their properties (Kumar and Mohammad, 2011). Different type of nanostructured materials has been designed throughout the years such as nanoparticles, colloidal particles, nanowires, nanohelics, nanopillars, nanopyramid, and nanotube.

Among all these nanostructured materials, nanowires has numbers of unique characteristics. Wires in general are ubiquitous. They are the arteries of electricity, taking it across the whole country into the labyrinth of electronic components. Wires can serve purposes beyond electricity transport. If they are small enough, they acquire new properties, turning ordinary wires from passive components into active ones. Wires with nanometre-scale diameters are ideal system for investigating how size affects the electrical transport and mechanical properties of materials. Such wires are crystalline in structure and can be made from metals, semiconductors, superconductors, polymers, and insulating materials. Silicon nanowires or SiNWs are a type of semiconductor nanowires usually formed from a Si precursor by etching solid or through catalysed growth from vapour or liquid phase. Their unique chemical properties make SiNWs a promising candidate for a wide range of applications that draw on their unique physio-chemical characteristics, which differ from those of bulk silicon material (Liu *et al*., 2016).

Radiation induced grafting has been a way to functionalise the surface of existing polymer forms so it can be used in a variety of applications, such as biomedical, environmental and industrial uses (Chapiro, 1962 and Nasef and Guven 2012). Radiation grafting changes the surface of polymeric materials by chemical bonding polar or non-polar monomers having functional groups, such as –COOH, -OR, -OH, -NH_2, -SO, H, -R and their derivatives, to affect surface properties without influence on the bulk material. Ultraviolet radiation (UV), Gamma rays and electron beam radiation can be used to generate active sites (free radicals) on a polymeric surface, which can then react with vinyl monomers to form graft copolymer. The influence of polymer covered nanostructured materials has been known to serve as protective layer. There were also a study reported that the MMA covered SiNWs would increase the resistivity of the materials (Fobelets *et al*., 2012).

1.2 Problem Statement

SiNWs is a type of nanostructured materials with very promising applications in various technology such as in lithium ion batteries and sensors. The methods of growing SiNWs has been widely discovered and used. The most popular method is by using the CVD and PECVD method where the use of gaseous Si precursor is involved in the process. The use of various gas in producing SiNWs could be dangerous. Silane and hydrogen gas is required in producing SiNWs using PECVD. Hydrogen is known to be very flammable and could cause explosion when it comes in contact with flame. High concentration hydrogen in the air would cause an oxygen-deficient environment and an individual that breathes in this environment would experience headache, ringing in ears, dizziness, drowsiness, unconsciousness, depression of all the sense, and even death. Silane is even more dangerous than hydrogen. This gas is a colourless and poisonous, with a strong repulsive odour. The use of magnetron sputtering would help producing SiNWs without involving these dangerous gases since it is a physical deposition. There are only a few studies on the formation of SiNWs using the magnetron sputtering method (Marsen and Sattler, 1999, and Zhao and Yang, 2008). Finding the right RF magnetron sputtering parameter is very important for finding a safe alternative method of producing SiNWs.

Characterisation of materials is a very crucial stage in experiment to determine the most suitable application. Numerous studies on structural and optical properties of SiNWs have been reported. This type of characterisation is important to determine which possible application is suitable for the materials. These study was done mainly to the SiNWs prepared with CVD (Stelzner *et al.*, 2010), metal assisted chemical etching (Gonchar *et al*., 2019, Jeong *et al*., 2019), and PECVD (Hamidinezhad *et al*., 2011). A few studies on producing SiNWs using RF magnetron sputtering has been conducted but there has no report was done on structural and optical properties of this material prepared using RF magnetron sputtering. The study of these type of characterisations is important to see the quality of the materials produce and to determine the applications suitable according to the characteristics. This study will give the information about the quality of SiNWs whether it is better, comparable, or worse than the other method of SiNWs production.

Material hybridisation is an advanced engineering material composed of an intimate mixture of inorganic components, organic components, or both types of components. Thus, they differ from traditional composites where the constituents are at the macroscopic level. Hybrid devices possess substantially extended functionality, compared to the conventional device such as transistor, resistor, or diode (Tarasov *et al*., 2012, Seol, *et al*., 2012 and Patolsky *et al.*, 2006). There are several groups of electronic devices that benefit from the available organic coating such as nanowire-based biosensors (Chen *et al*., 2011 and Shen *et al*., 2014), device for optoelectronics (Baek *et al*., 2015 and Lu *et al*., 1990), and bioinspired neuromorphic, which is a relatively new group aimed to mimic the functions of the biological cells (Mead, 1990). Radiation induced grafting offers many advantages that might be commercialised such as simplicity, in controlling parameters of processing, uniform grafting of monomers at low temperature, flexibility, and good reproducibility of treatment. The study of hybrid SiNWs-polymer, such as polyacrylic acid, poly (3,4-ethylenedioxythiophene), and poly methyl methacrylate has been done by previous researchers. Methyl methacrylate (MMA) coated SiNWs was proven to have a hydrophilic SiNWs core with hydrophobic polymer shell on the surface (Mulvihill *et. al., 2005*). However, there has never been study reported on hybrid of silicon nanowires and polymers with radiation induced grafting. This study would introduce a new method of material hybrid, which is via radiation induced grafting. However, most of these researches aimed to find out the electric and dielectric properties of these hybrid materials and none of these studies used the approach of radiation induced grafting in their methodology. The study of structural and optical properties of these hybrid materials would possibly give a new information about the grafting capability of SiNWs and methyl methacrylate and the potential applications of such combination.

1.3 Objectives of Study

The objectives of this study are:

i. To synthesise a set of SiNWs under optimum conditions using RF magnetron sputtering.
ii. To determine the structural and optical properties of synthesised nanowire.
iii. To perform radiation induced grafting of MMA onto the samples, and to determine the effect of radiation induced grafting on the optical and structural properties of the nanowire.

1.4 Scope of Study

Six samples of SiNWs were prepared using RF magnetron sputtering under similar parameters with the base pressure of 1.5×10^{-5} Torr and the RF power of 100 W. The structural characteristics of prepared SiNWs using the FESEM, EDX, XRD, FTIR spectroscopy, and Raman Spectroscopy while the optical properties analysis was done using the UV-vis and photoluminescence spectroscopy. Radiation grafting of MMA onto 5 of the SiNWs samples with 10 kGy, 30 kGy, 50 kGy, 70 kGy, and 90 kGy radiation dose, respectively. The irradiation process was done using the electron beam of 2 MeV energy and 3 mA of pulse current. The grafting percentage of each grafted samples were calculated according to the radiation dose of the samples. The optical and structural properties of the grafted samples were reanalysed using similar method and instruments before the grafting were carried out which are FESEM, EDX, XRD, FTIR spectroscopy, and Raman Spectroscopy for structural analysis and UV-visible and photoluminescence spectroscopy for optical properties analysis.

1.5 Significance of Study

SiNWs has attracted so much attention throughout the years with its impeccable optical, thermoelectric, and magnetic characteristics (Adachi *et. al*., 2010, and Vo *et. al*., 2008). Due to this reason, various production method of SiNWs has been done. The fabrication of SiNWs with RF magnetron sputtering is a simpler, safer and cost effective way of sample production without involving precursor gas.

Radiation grafting is known for its benefits of altering the material's surface characteristics. Radiation induced grafting of MMA onto SiNWs have the possibility of producing more durable SiNWs where a hydrophobic MMA layer is molecularly bonded on the surface of nanowires without compromising its optical and structural characteristics. This could open the possibility of producing new material especially in semiconductor research.

1.6 Thesis Organisation

This thesis shows the influence of radiation dose on grafting percentage and how it affected the optical and structural characteristics of SiNWs. The first chapter would give a general description of the study, which explains the background (basic principles, basic theories, and the history about SiNWs and radiation grafting), problem statement, objective, scope, and the significance of the study. Chapter 2 presents all the theories and the literature collected related to SiNWs and radiation grafting that have been done previously.

Chapter 3 presents the methodology and experimental details that were done throughout the study starting from the preparation of SiNWs, grafting of MMA on the SiNWs and the optical and structural analyses in details. Chapter 4 discussed all the results obtained from the conducted experiment. This chapter explains in details on how the radiation dose would affects the grafting percentage of MMA on SiNWs and how the grafting percentage would influence the structural and optical properties of SiNWs. The analyses done was on FESEM images, EDX, XRD, FTIR spectroscopy, Raman spectroscopy, UV-vis spectroscopy, and photoluminescence spectroscopy.

Chapter 5 is the conclusion of all the analyses that were done in the study and the possible application of the material based on the obtained data. This chapter also offers some recommendations for improvements to this work, which could possibly leads to a new motivation for future researches.

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LIST OF PUBLICATIONS

Indexed Journals

1. Khaidzir Hamzah , **M. Abdullah Izat Mohd Yassin** , Sib Krishna Ghoshal , Muhammad Firdaus Omar and Abdul Khamim Ismail (2016) 'Impact Of Radio Frequency Power And Deposition Time On Sputtered Gold Thin Film Growth' *Solid State Science and Technology*, vol. 24, pp. 249-255.

2. Khaidzir Hamzah**, M. Abdullah Izat Mohd Yassin**, Sib Krishna Ghoshal, M. Akmal Hasanudin, Abdul Khamim Ismail (2015) 'VHF-PECVD Fabrication Parameters Dependent Morphology Variation of Gold Catalyst Assisted Silicon Thin Film Growth' *Jurnal Teknologi*, vol. 76, pp. 157-161.