

SYNTHESIS AND CHARACTERIZATION OF LASER ANNEALED
NANOPOROUS SILICON-ZINC OXIDE NANOCCLUSERS FOR
ULTRAVIOLET PHOTODETECTOR APPLICATION

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DEDICATION

To my Family, gave me endless love, trust, constant encouragement over the years, and for, their prayers, enduring patience, support, and love up to the completion of this study. To my Friends, for their support, and encouragement

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ABSTRACT

The main purpose of this study was to synthesize a series of nanoporous silicon (n-PSi) samples on n-type Si (111) wafer using the photo-electrochemical etching (PECE) method, which was effective for the fabrication of a metal-semiconductor-metal (MSM) ultraviolet photodetector. Samples were prepared at fixed etching time (30 min) under varying PECE operating parameters, which included differential current densities (15, 30 and 45 mA/cm²) and variable chemical ratios to achieve optimum growth. The structural, morphological and optical properties of the as-prepared PSi samples were characterized by different analytical techniques. The optimum etching parameters for the growth of n-PSi samples comprise of etching time of 30 min, current density of 45 mA/cm² and chemical ratio of 2:1:1. The objectives of this study were achieved in three phases. First, a layer of zinc oxide (ZnO) nanoclusters was deposited on the optimally grown n-PSi sample by means of radio frequency (RF) sputtering. The thicknesses of the deposited ZnO nanoclusters layers on n-PSi were varied between 300 nm and 500 nm for annealing temperatures ranging from 600 °C to 900 °C. The optimum thickness and temperature were determined to be 300 nm and 700 °C, respectively. Secondly, platinum (Pt) electrodes were deposited on the n-PSi/ZnO NCs structure via radio frequency sputtering to obtain the MSM (Pt/n-PSi/ZnO NCs/Pt) ultraviolet photodetectors. Finally, the performances of fabricated ultraviolet MSM photodetectors were evaluated using current-voltage (I-V) measurement. The optimum n-PSi and n-PSi/ZnO NCs samples were annealed using a Nd-YAG laser under several shots (pulses) to determine their influence on the structural, morphological, optical and electrical features of the n-PSi/ZnO NCs samples. The photoluminescence spectra of the optimally synthesized n-PSi/ZnO NCs exhibited an intense near band edge emission (violet band centred at 380 nm for bandgap energy of 3.26 eV). The I-V characteristics of the fabricated MSM ultraviolet photodetectors were examined in the dark and under ultraviolet light (380 nm) illumination. The results revealed that laser annealing can significantly improved of the performance of the fabricated Pt/n-PSi/ZnO NCs/Pt ultraviolet photodetector in terms of high responsivity (6.35 A/W), photosensitivity (3772.92) as well as faster response time (0.30 s) and recovery time (0.26 s). It was concluded that the proposed MSM ultraviolet photodetectors could be advantageous for various optoelectronic applications.

ABSTRAK

Tujuan utama kajian ini adalah untuk mensintesis satu siri sampel silikon nano berliang (n-PSi) pada wafer Si (111) jenis-n dengan menggunakan kaedah punaran foto-elektrokimia (PECE), yang berkesan untuk mengfabrikasikan pengesanfoto logam-semikonduktor-logam (MSM) ultraungu. Sampel disediakan pada masa punaran tetap (30 minit) di bawah parameter operasi PECE yang berbeza-beza, yang termasuk ketumpatan arus berlainan (15, 30 dan 45 mA/cm²) dan nisbah kimia yang berubah untuk mencapai pertumbuhan yang optimum. Ciri-ciri struktur, morfologi dan optik dari sampel n-PSi yang disediakan telah dicirikan oleh teknik analitikal yang berbeza. Parameter punaran optimum untuk pertumbuhan sampel n-PSi terdiri daripada masa punaran selama 30 minit, ketumpatan arus sebanyak 45 mA/cm² dan nisbah kimia 2: 1: 1. Objektif kajian ini telah dicapai dalam tiga fasa. Pertama, satu lapisan zink oksida (ZnO) nano kelompok (NCs) telah dimendapkan pada sampel n-PSi yang ditumbuhkan secara optimum dengan menggunakan kaedah percikan frekuensi radio. Ketebalan lapisan ZnO NCs pada n-PSi berubah antara 300 nm dan 500 nm untuk suhu penyepuhlindapan antara 600 °C hingga 900 °C. Ketebalan dan suhu optimum didapati masing-masing adalah 300 nm dan 700 °C. Kedua, elektrod platinum (Pt) dimendapkan di atas struktur n-PSi/ZnO NCs melalui percikan frekuensi radio untuk mendapatkan pengesanfoto ultraungu MSM (Pt/n-PSi/ZnO NCs/Pt). Akhirnya, prestasi pengesanfoto ultraungu dinilai menggunakan pengukuran arus-voltan (I-V). Sampel n-PSi dan n-PSi/ZnO NCs yang optimum telah disepuhlindapkan menggunakan laser Nd-YAG di bawah beberapa tembakan (denyutan) untuk menentukan kesan terhadap ciri-ciri struktur, morfologi, optik dan elektrik sampel n-PSi/ZnO NCs. Spektra fotoluminesen n-PSi/ZnO NCs yang disintesis secara optimum menunjukkan pancaran pinggir jalur dekat yang kuat (jalur ungu yang berpusat pada 380 nm bagi tenaga jurang jalur sebanyak 3.26 eV). Ciri-ciri I-V pengesanfoto ultraungu MSM yang difabrikasikan diperiksa dalam gelap dan di bawah penyinaran cahaya ultraungu (380 nm). Hasil kajian menunjukkan bahawa penyepuhlindapan laser dapat meningkatkan prestasi pengesanfoto ultraungu Pt/n-PSi/ZnO NCs/Pt yang difabrikasikan dengan nilai yang tinggi untuk tindak balas (6.35 A/W), fotosensitiviti (3772.92) serta masa tindak balas (0.30 s) dan masa pemulihan (0.26 s) yang lebih cepat. Kesimpulannya, pengesanfoto ultraungu MSM yang dicadangkan dapat memberi kelebihan untuk pelbagai aplikasi optoelektronik.

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LIST OF ABBREVIATIONS

<i>ECE</i>	-	Electrochemical etching
<i>PSi</i>	-	Porous silicon
<i>n-PSi</i>	-	nano – porous silicon
<i>PEC</i>	-	Photo –electrochemical etching
<i>a. u.</i>	-	Arbitrary unit
<i>AFM</i>	-	Atomic force microscopy
<i>NBE</i>	-	Band edge emission
<i>Si</i>	-	Silicon
<i>CdS</i>	-	Cadmium sulfide
<i>CdTe</i>	-	Cadmium telluride
<i>UVPDs</i>	-	Ultraviolet Photodetectors
<i>CB</i>	-	Conduction Band
<i>CO₂</i>	-	<i>Carbon dioxide</i>
<i>c-Si</i>	-	Crystallite silicon
<i>DC</i>	-	Direct current
<i>APDS</i>	-	Avalanche photodiode
<i>DI</i>	-	Distilled water
<i>PINs</i>	-	Positive intrinsic negative photodiode
<i>EMT</i>	-	Effective mass theory
<i>e⁻-h⁺</i>	-	Electron – hole
<i>EDX</i>	-	Energy dispersive x– ray
<i>G</i>	-	Photo carrier –generation rate
<i>FWHM</i>	-	Full width at half maximum
<i>He-Cd</i>	-	Helium cadmium
<i>HF</i>	-	Hydrofluoric acid
<i>MS</i>	-	Metal semiconductor
<i>nm</i>	-	Nanometer
<i>PL</i>	-	Photoluminescence
<i>PV</i>	-	Photovoltaic
<i>Pt</i>	-	Platinum

<i>P</i>	-	Porous
<i>KOH</i>	-	Potassium hydroxide
<i>RCA</i>	-	Radio corporation of America
<i>RF</i>	-	Radio frequency
<i>RT</i>	-	Room temperature
<i>R_{MS}</i>	-	Root mean square
<i>rpm</i>	-	Round per minute
<i>SEM</i>	-	Scanning electronic microscopy
<i>SiO₂</i>	-	Silicon dioxide
<i>C₂H₅OH</i>	-	Ethanol
<i>H₂O₂</i>	-	Hydrogen peroxide
<i>UV</i>	-	Ultra violet
<i>VB</i>	-	Valance band
<i>XRD</i>	-	X -ray diffraction
<i>ZnO</i>	-	Znic oxide

LIST OF SYMBOLS

D	-	Average Crystallite size
ΔD	-	Average discrepancy
I	-	Current
$I-V$	-	Current Voltage
J	-	Current density
JV	-	Current density–voltage
P	-	Density of bulik silicon
θ	-	Diffraction Bragg angle
m_d	-	Dissolved silicon mass
m_e^*	-	Effective mass of the electron in the conduction band
m_h^*	-	Effective mass of the hole in the valance band
m_o	-	Electron rest mass
eV	-	Electron volt
J_{ep}	-	Electropolishing voltage
E_g	-	Energy band gap
E_{ex}	-	Energy band gap excitation light source
E_s	-	Energy excited surface state
E_v	-	Energy of carriers at the bottom of the VB edge
S	-	Etched wafer area
m_d	-	Dissolved silicon mass
ν	-	Frequency
P_{in}	-	Incident solar power
a_{bs}	-	Lattice constant of bulk silicon
a_{ps}	-	Lattice constant of PSi layer
C	-	Lattice constant of the strained ZnO film
c_o	-	Lattice constant of the unstrained ZnO film
I_m	-	Maximum current
J_m	-	Maximum current density
P_m	-	Maximum power output
E_{ph}	-	Photon energy

h	-	Planck's constant
d	-	Pore diameter
ε_{zz}	-	Strain
d_1	-	Thickness of porous layer
m_t	-	Total mass of etched Si
V	-	Voltage
m_2	-	Weight of the Si after etching
m_3	-	Weight of the Si after removal of the porous layer
m_1	-	Weight of the Si before etching

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

The distinctive features of porous silicon (PSi) are based on its unique morphology, which has unravelled new opportunities in the manufacture of Si-based optoelectronics. Nevertheless, the efficiency of such devices is dependent on the quality of fabricated PSi nanostructures, where controlled growth methods are constantly demanded to synthesize materials with desirable characteristics. In this regard, the electrochemical etching method appears as a suitable technique to synthesize high quality n-PSi materials with varied structures and morphologies. The dependence of the optoelectronic properties of the as-prepared n-PSi on the porosity (size, shape, homogeneity and density distribution), morphology and fabrication conditions can be influenced by fine-tuning the photo-electrochemical etching (PECE) processing parameters (e.g. etching duration, current density, coating process, etc.) [1]. Moreover, it has been shown that the crystallinity and optoelectronic properties of the synthesized PSi material can be remarkably improved by coating its front surface with n-type semiconductor materials [2]. Thus, several efforts have been made to produce PSi-based devices with optimum photodetection performance, where the key emphasis was to enhance the electrical and charge carrier transport characteristics of PSi [3]. Despite the varied attempts, the optimum conditions for synthesizing n-PSi with the desirable optoelectronic properties remain to be achieved.

Taking into consideration the prospect of n-PSi with strong visible and ultraviolet photoluminescence at room temperature, it is being widely researched for use as ultraviolet photodetectors (UVPDs) in fields of medicine, manufacturing, and technology [4]. Motivated by this emerging demand, this study intends to optimize the electrical and optical properties of n-PSi for fabrication as metal-semiconductor-metal (MSM) ultraviolet photodetectors (UVPDs). To adjust the morphological properties

of n-PSi, various parameters involved in photo-electrochemical etching (PECE) method were varied. The dependence of the electrical and optical properties of n-PSi on its morphology was then analyzed. Furthermore, the n-PSi layer was coated with a layer of ZnO (a direct wide band gap semiconductor material) NCs to enhance its UV photodetection property [5, 6]. This modification is imperative since the conventional UV detectors based on polycrystalline ZnO thin film have low photoresponsivity and long response time (of the order of few minutes) [7]. Previous studies on ZnO UV detector focused on improving the efficiency of micro mask electrodes rather than photoresponsivity [8]. Attempts have been made to improve the photoresponsivity of ZnO UV detectors by modifying the surfaces of as-prepared ZnO thin films [9]. Studies have shown that covering the ZnO film surface with nanosheets of diverse kinds of polymers could significantly improve the responsivities of photodetectors [7]. The surface coating of ZnO films with polyamide nylon enhanced their photoresponse to four orders of magnitude, although the response time was short (range of few seconds) [10]. In addition, the coating of P-Si with ZnO nanofilms could significantly improve its resistivity, resulting in fast response in the UV region.

The so called PD (or photosensor) device directly converts optical signal into electrical signal through the photovoltaic (PV) effect. Photodetector is a transducer that modifies one of the characteristics when light energy is incident on it. The Ohmic resistance of a photoresistor can be modified in the same way as rods and cones cells in retina neurons of human eye alter their electrochemical response. Likewise, chlorophyll in plant leaves adjusts the rate of CO₂ conversion to O₂. Other PDs alter the flow of electrical current or the potential difference across their terminals. Easily reproducible and cost-effective PDs that exhibit sufficiently fast response to produce a measurable output from a small amount of light energy must be developed for potential applications in the field of high-speed optical communications. Examples of this kind of photodetectors include avalanche photodiodes (APDs) and positive intrinsic negative photodiodes (PINs).

1.2 Problem Statement

Studies on the fabrication of efficient PDs are constantly expanding given the urgent need for more advanced and better devices with high stability, speed, sensitivity, selectivity, and large signal to noise ratio. [11-13]. PSi based PDs have been fabricated using different methods [14, 15], particularly the PECE method, which has produced excellent and high quality PDs [15]. However, the optimum method for the growth of PSi with controlled pore size distribution has not been realized despite the many efforts to do so. This is imperative as controlling the structure, morphology, optical and electrical properties (I-V curves, rise time, recovery time, sensitivity and responsivity) of ZnO based PDs will enhance their overall gain and reduce their loss rate. Moreover, the influence of laser annealing (varied fluences, laser energy, pulses, repetition time, etc.) on the structure, morphology, optical and electrical properties of grown n-PSi/ZnO NCs based MSM PDs has not been examined systematically. Thus, it is expected that coupling the established PECE method with pulse laser annealing process will lead to the synthesis of better quality n-PSi and n-PSi/ZnO NCs under optimum conditions. In this view, this thesis attempts to combine the PECE technique with Nd:YAG laser annealing to improve the optical, electrical and morphological properties of grown n-PSi and n-PSi/ZnO NCs samples (at optimum growth condition), resulting in the fabrication of high performing and efficient MSM UV PDs. Such modifications in the overall behavior of n-PSi samples are anticipated to produce an optimized MSM UVPD characterized by large surfaces and high-quality structures needed for diverse applications. The optimally synthesized n-PSi and n-PSi/ZnO NCs samples can further be used to fabricate MSM PDs of high efficiency and fast response.

1.3 Objective of the Thesis

Based on the abovementioned problem statement and the identified research gaps, the following objectives are set:

- (a) To synthesize n-PSi on Si substrates using the photo-electrochemical etching (PECE) method under different processing parameters for the growth optimization and subsequent characterizations.
- (b) To determine the influence of growth parameters (temperature and thickness) on the structure, morphology and optical properties of n-PSi/ZnO NCs produced using RF sputtering technique.
- (c) To evaluate the influence of Nd-YAG laser annealing parameters on the structure, morphology, optical properties and electrical properties of the fabricated MSM UV photodetectors based on optimum n-PSi and n-PSi/ZnO NCs samples.
- (d) To compare the performances of the proposed MSM UV PDs fabricated with and without laser annealing.

1.4 Scope of the Work

The research scope of this thesis includes:

- (a) The optimization of the growth parameters of photo-electrochemical etching (PECE) and radio frequency (RF) sputtering methods for the synthesis of n-PSi on Si-substrate and deposition of ZnO NCs on n-PSi layer, respectively.
- (b) The structural, morphological and optical characterizations of the n-PSi and n-PSi/ZnO NCs samples at room temperature using X-ray diffraction (XRD), energy dispersive X-ray (EDX) spectroscopy, atomic force microscopy (AFM), field emission scanning electron microscopy (FESEM) and photoluminescence (PL) spectroscopy.
- (c) The fabrication of MSM UV PDs using the optimally synthesized n-PSi and n-PSi/ZnO NCs samples.

- (d) The performance evaluation of the proposed photodetectors (MSM UV PDs) in the dark and under UV light illumination.
- (e) The measurement of I-V characteristics of the designed MSM UV PDs.
- (f) The annealing of the optimally synthesized samples using Nd-YAG laser under varied laser parameters (anodization current and voltage) to improve the performance of the PDs.
- (g) Comparative analysis of the photodetection performances of PDs fabricated from laser annealed and un-annealed samples.

1.5 Thesis Outline

Chapter one presents a brief background on the subject matter and an overview of the syntheses of PSi and ZnO films as well as the significance of photodetectors. Chapter two provides a comprehensive literature review and theoretical background of the formation and deposition of n-PSi layers in addition to their photodetector application. The basic principle and mechanism of photodetector operation is also presented in this chapter. Chapter three presents in detail the research methodology, which comprises experimental set up of the various synthesis methods of n-PSi and ZnO NCs, description of the characterization tools, fabrication of the MSM UV photodetector and the process of laser annealing. The preparation of the Si samples used to synthesize PSi layers was also described in this chapter. Furthermore, this chapter explains the process of fabricating the Pt/n-PSi/ZnO NCs/Pt UV photodetector. Chapter 4 presents the results on the effect of varying the current density of PECE method on the structural and optical properties of n-PSi layers deposited on n-type c-Si wafer of (111) orientations. Afterwards, the n-PSi layer with optimal current density (from each orientation) was selected as the best substrate to grow ZnO NCs using the RF sputtering technique. The properties of the samples required for the fabrication of the photodetector device and the effects of temperature annealing on the morphology, structural, and optical characteristics of ZnO NCs arrays synthesized on PSi substrate are discussed. The n-PSi layer with optimal thickness and

temperature annealing (from each orientation) was selected as the most suitable substrate for fabrication of the photodetector device. In addition, the results on the influence of Nd-YAG laser annealing on the structural and optical properties of n-PSi and n-PSi/ZnO NCs are presented in this chapter. The results are comparatively analysed in this chapter. Chapter 5 concludes the thesis with deductions inferred from the results.

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LIST OF PUBLICATIONS

A. INTERNATIONAL JOURNALS

- 1- Asad A.Thaha, Noriah Bidin, Hazri Bakhtiar,Z Hassan, Photo-electrochemically synthesized light emitting nanoporous silicon based UV photodetector :Influence of current density Mater. Res Express 4 (2017) 116203.
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B. INTERNATIONAL CONFERENCES

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