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SYNTHESIS AND CHARACTERIZATION OF COPPER OXIDE/POROUS SILICON COMPOSITE FOR PHOTODETECTOR APPLICATIONS

BATOOL ENEAZE BANDAR AL-JUMILI

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By

BATOOL ENEAZE BANDAR AL-JUMILI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillments of the Requirements for the Degree of Doctor of Philosophy

October 2017

DEDICATION

قال تعالى
((قُلْ لَوْ كَانَ الْبَحْرُ مِدَادًا لِكَلِمَاتِ رَبِّي لَنَفِدَ الْبَحْرُ قَبْلَ أَنْ تَنْفَدَ كَلِمَاتُ رَبِّي وَلَوْ جِئْنَا بِمِثْلِهِ مَدَدًا))
الكهف 109

This thesis dedicated to our Prophet Muhammad (Peace be upon him) who brought us from the darkness of ignorance into the light of knowledge. Also to my father, my mother, my lovely husband, my dear brothers, my dear sisters and my children for their love, endless support and encouragement with love.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the Degree of Doctor of Philosophy

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BATOOL ENEAZE BANDAR AL-JUMILI

October 2017

Chairman : Professor Zainal Abidin Talib, PhD
Faculty : Science

Copper oxide/porous silicon (CuO/PS) nanocomposites has been synthesized by a combined electrochemical etching and pulse laser ablation (PLA) techniques. The objective of this study is to enhance the photoluminescence (PL) efficiency and stability of luminescence porous silicon produced by electrochemical etching technique via incorporation of copper within it. The strategy is based on a simple well-known assumption that transition metal can be easily introduced inside of silicon (Si) band gap as impurity levels. Consequently, the numbers of carrier will be increased and affect electronic properties that will promote good PL efficiency.

This study presents three steps to prepare CuO/PS nanocomposites. Firstly, the preparation of aqueous solution contains Cu species by laser ablation of Cu target. The laser ablation mechanism of copper target in pure distilled water and the effect of ablation time factor were investigated. Results from the experiments undertaken for this purposed show that the shape of the NPs was quasi-spherical. This has been attributed to the diffusion of oxygen in the solution. Further observation found that the NPs size decreased and the size distribution become narrow as the ablation times increase. This is due to the prolonged interaction of ablated NPs with laser beam that led to the fragmentation of bigger NPs into smaller ones.

The second step introduced the preparation of porous silicon by electrochemical etching of a commercial n-type silicon wafer. The control of the size and the shape of the porous Si structures were investigated by modifying several etching parameters, which were current density, etching time, and electrolyte concentration. The surface morphologies of PS confirm that pore diameter and nanostructure are dependent on the etching parameters, and that the shifting towards shorter wavelength is due to the diminishing of the silicon skeletons (quantum confinement) where blue shift of wavelength increases along with porosity. Gravimetrical method was used to determine the porosity percentage of the samples. According to the quantum confinement luminescence model, the shorter peak wavelength of luminescence has caused the increase in the energy band gap (E_g) of the PS.

The third step was to integrate the yield of pulse laser ablation and electrochemical etching to produce CuO/PS nanocomposites with highly consistent shape and structure. The control of the geometry of the nanostructures was investigated by modifying several deposition parameters that included solution concentration, deposition time, current density and post-annealing temperature. The surface morphologies of CuO/PS verify that the nano-dendrite (NDs) structures are dependent on the deposition parameters, as well the copper phases, including Cu, CuO and Cu_2O that deposited on the PS layer. The PL exhibited an enhanced peak with narrow line.

Metal-Semiconductor-Metal (MSM) photodetectors based on PS/Si and CuO NDs/PS heterojunctions were then fabricated. Current-voltage (I-V) measurements were performed for both photodetectors under light and dark conditions. The diode behavior of the CuO nanodendrite/PS device was prominently superior compared to the PS/Si device where the contrast ratio was 63 and 3, respectively. The sensitivity of the CuO NDs/PS device increases to 660% as a function of time and becomes much higher compared to the PS/Si device (180%). The photoresponsivity of the CuO/PS detector was 500 mA/W, which is almost 2-fold higher than that of PS/Si detector. This result could be attributed to enhance surface area to volume ratio due to the three dimensional nature and high crystal quality of the CuO dendrite layer. The larger surface to volume ratio of the CuO dendrite enables the CuO dendrite device to collect more light, thereby increasing the photocurrent. In addition, the high quality of CuO dendrite decreases the density of trapping centers of charge caused by defects and therefore improving the photosensitive significantly. Moreover, the detection efficiency of CuO/PS MSM photodetector was enhanced by ~ 3 times larger than that of the PS-MSM photodetector. The band gap alignment between PS and CuO facilitates the photo induced electron transfer from CuO to PS whereby enhancing the photoresponsivity. The obtained measurements and calculated results have supported the optimum sample for fabricating the best photodetector device.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SINTESIS DAN PENCIRIAN KOMPOSIT KUPRUM OKSIDA / SILIKON BERLIANG UNTUK APLIKASI FOTOPENGESAN

Oleh

BATOOL ENEAZE BANDAR AL-JUMILI

Oktober 2017

Pengerusi : Profesor Zainal Abidin Talib, PhD
Fakulti : Sains

Nanokomposit kuprum oksida/silikon berliang (CuO/PS) telah dihasilkan secara gabungan teknik punaran elektrokimia dan ablasi laser berdenyut (PLA). Objektif kajian ini adalah untuk meningkatkan kecekapan fotoluminesens (PL) dan kestabilan luminesens silikon berliang yang dihasilkan dengan teknik punaran elektrokimia dengan mengabungkan kuprum di dalamnya. Strategi ini berdasarkan andaian bahawa logam peralihan boleh didatangkan ke dalam jurang jalur silikon (Si) sebagai aras bendasing. Akibatnya, bilangan pembawa akan meningkat dan menjejaskan sifat-sifat elektronik yang akan meningkatkan kecekapan fotoluminesen. Kajian ini membentangkan tiga langkah untuk menyediakan nanokomposit CuO/PS. Pertama, penyediaan larutan berair yang mengandungi spesis Cu daripada ablasi laser terhadap sasaran Cu.

Mekanisme ablasi laser terhadap sasaran kuprum dalam air suling tulen dan kesan faktor masa ablasi telah disiasat. Keputusan daripada eksperimen yang dijalankan menunjukkan bahawa bentuk nanopartikel adalah kuasi-sfera. Ini telah dikaitkan dengan resapan oksigen di dalam air. Pemerhatian selanjutnya mendapati bahawa saiz nanopartikel menurun dan taburan saiz menjadi sempit dengan peningkatan masa ablasi. Ini adalah disebabkan oleh interaksi berpanjangan ablasi nanopartikel dengan pancaran laser yang memecahkan nanopartikel yang besar kepada yang lebih kecil.

Langkah kedua adalah penyediaan silicon berliang punaran elektrokimia ke atas wafer silikon komersial jenis n. Kawalan saiz dan bentuk struktur silikon berliang telah disiasat dengan mengubahsuai beberapa parameter punaran, termasuk ketumpatan arus, masa punaran, dan kepekatan elektrolit. Morfologi permukaan silikon berliang telah mengesahkan bahawa diameter liang dan nanostruktur bergantung kepada parameter punaran yang menyebabkan peralihan dari gelombang panjang ke arah

gelombang yang lebih pendek disebabkan oleh pengurungan kuantum rangka silikon dimana anjakan gelombang biru meningkat dengan keliangan. Teknik gravimetrikal telah digunakan untuk menentukan peratusan keliangan sampel. Menurut model luminesen pengurungan kuantum, puncak gelombang luminesen yang pendek telah menyebabkan peningkatan dalam jalur tenaga (E_g) silikon berliang.

Langkah ketiga adalah dengan mengintegrasikan hasil ablasi laser berdenyut dan punaran elektrokimia untuk menghasilkan nanokomposit CuO/PS dengan bentuk dan struktur yang sangat konsisten. Kawalan terhadap geometri nanostruktur telah disiasat dengan mengubah beberapa parameter pemendapan termasuklah kepekatan larutan, masa pemendapan, ketumpatan arus dan suhu selepas penyepuhlindapan. Morfologi permukaan CuO/PS telah mengesahkan bahawa struktur nano-dendrit adalah bergantung kepada parameter pemendapan, dan juga fasa kuprum, termasuk Cu, CuO dan Cu_2O yang mendap di lapisan liang silikon. Fotoluminesen menunjukkan puncak yang dipertingkatkan dengan garis yang sempit. Pengesan foto logam-semikonduktor-logam (MSM) berdasarkan persimpangan hetero PS/ Si dan CuO/PS telah difabrikasi. Ukuran arus-voltan (I-V) telah dilakukan untuk kedua-dua pengesan foto dibawah keadaan bercahaya dan gelap. Peranti persimpangan hetero CuO/PS menunjukkan ciri-ciri diod yang lebih baik berbanding dengan peranti persimpangan hetero PS/Si dimana nisbah kontras masing-masing adalah 63 dan 3. Kepekaan peranti pengesan foto CuO/PS meningkat kepada 660% dengan fungsi masa dan menjadi lebih tinggi berbanding dengan peranti pengesan foto PS/Si (180%). Fotoresposiviti pengesan CuO/PS adalah 500 mA / W, hampir 2 kali lebih tinggi daripada pengesan PS/Si. Hasil ini boleh dikaitkan dengan peningkatan luas permukaan kepada nisbah isipadu disebabkan oleh sifat tiga dimensi dan kualiti kristal lapisan dendrit CuO. Nisbah permukaan kepada isi padu dendrit CuO yang lebih besar membolehkan peranti CuO dendrit untuk mengumpulkan lebih banyak cahaya dan meningkatkan arus foto. Selain itu, dendrit CuO yang berkualiti tinggi mengurangkan ketumpatan pusat caj yang disebabkan oleh kecacatan, oleh itu meningkatkan fotoresponsiviti dengan ketara. Tambahan pula, kecekapan pengesanan pengesan foto CuO/PS MSM telah dipertingkatkan sebanyak 2.83 kali ganda berbanding pengesan foto PS-MSM. Penjajaran jurang jalur di antara PS dan CuO memudahkan pemindahan elektron foto teraruh dari CuO ke PS dan meningkatkan fotoresponsivitinya. Hasil pengukuran yang diperolehi dan dikira telah menyokong sampel optimum untuk pemfabrikasi peranti fotodetektor yang terbaik.

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I certify that a Thesis Examination Committee has met on 10 October 2017 to conduct the final examination of Batool Eneaze Bandar on her thesis entitled "Synthesis and Characterization of Copper Oxide/Porous Silicon Composite for Photodetector Applications" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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a	Cubic lattice constant
A	Area of contact
A**	Richardson's constant
a. u.	arbitrary unit
AT	Ablation time
CB	Conduction bond
CED	Cathodic electrochemical deposition
c-Si	Crystalline silicon
Cu	Copper
Cu_2O	Cuprous oxide
CuO	Cupric oxide
DC	Direct current
DW	Distilled water
ECE	Electrochemical etching
E_g	The energy of band gap
PLD	Pulsed laser deposition
e-h	Electron-hole
eV	Electron volt
FESEM	Field emission scanning electron microscopy
FWHM	Full Width at Half Maximum
h	Plank Constant
h^+	Hole
HF	Hydrofluoric Acid
hν	Photon energy
I	Current

ICSD	Inorganic crystal structure database
I_d	Dark current
I_{Ph}	Photocurrent
I-V	Current–voltage
K	Boltzmann’s constant
LA	Laser ablation
m*	Effective mass
m1	Weight of the Si before etching
m2	Weight of the Si after etching
m3	Weight of the Si after removal of the porous layer
MSM	Metal Semiconductor Metal
n	Ideality factor
nc-Si	nanocrystalline silicon
ND	Nanodendritic
nm	Nanometer
NPs	Nanoparticles
P	Porosity
PD	Photodetector
P_{inc}	Incident optical power
PL	Photoluminescence
ϕm	Metal work function
PS	Porous silicon
ϕs	Semiconductor work function
Pt	Platinum
PVP	Polyvinylpyrrolidone
q	Electron charge

R	Responsivity
S	Etched wafer area
SBH	Schottky barrier height
Si	Silicon
T	Absolute temperature
T_{dec}	Decay time of photoconductive device
TEM	Transmission electron microscopy
T_{rise}	Rise time of photoconductive device
UV-VIS	UV-VIS Ultra Violet– Visible
V	Voltage
VB	Valence band
V_{bias}	external bias voltage
XRD	X-ray diffraction
H	Efficiency
Θ	Diffraction Bragg's angle
λ	Wavelength of Light Wave
ρ	Density of the bulk Si
φ_B	Schottky barrier height
X	Semiconductor electron affinity
F-	Fluoride
SiF_2	Silicon fluoride
H_2SiF_6	Fluorosilicic Acid
$H_2(g)$	Hydrogen gas
RF	Radio Frequency
3D	3-dimension
I_o	Saturation current

exp	Exponent
CVD	Chemical vapor deposition

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The material is called "nanostructured materials" when at least one of its dimensions is within 100 nm. These nanotructured materials may be in form of nanoparticles, nanowires, thin films or nanorods. In the recent years, due to their uniqueness in terms of physical and chemical behaviors, which are completely different when compared to their bulk phases, the nanostructured materials have been given so much attention by researchers. Their properties determinants are basically the dimension, structure type, and nature of the surface. In nanostructures study, it is necessary to understand how the properties changes with the mentioned parameters. For technological applications like information technology and optoelectronics, the parameters can be varied to controlling the materials properties (Berry, 1967; Dingle et al., 1974).

The tuning ability of both the optical absorption and emission properties of any semiconductor nanostructures through variation of its structure size is quite attractive in the field of band gap engineering of materials. Moreover, the effects of quantum confinement may expected to cause an alteration in the material's density of states of phonons, electrons, and also in the rates of mechanism of recombination of electron-hole pair (e-h) charge carrier (Fendler and Meldrum, 1995; Ledentsov et al., 1996; Smith and Collins, 1992).

Of the nanostuctured materials, the porous nanostructures are commonly known materials. The word porous is used due to the presence of billions holes of nanosize formed within the material. Porosity by definition is the ratio of the volume occupied by the voids to the overall volume of the material. The term porosity is common in various fields as ceramics, materials, pharmaceuticals, metallurgical, manufacturing construction and earth sciences (Nalwa, 2001). Promising in the optoelectronic application is a common porous material called porous silicon.

Porous silicon (PS) is utilized as an attractive building block for photonic devices such as solar cells and photodetectors. (Ünal et al., 2001; Lee et al., 1998) Due to its high light absorption, high photoconductive response, high optical gain, modulated energy band gap, surface roughening with low reflection losses and high ratio of surface to volume (Shang et al., 2011; Aroutiounian et al., 2008; Arad-Vosk and Sa'ar, 2014; Cullis et al., 1997; Sharma et al., 2001).

Despite the appeal of PS in the electronic industries, its large scale application is hindered by the instability and inefficiency of optical characteristics (Aroutiounian et al., 2008; Iyer and Xie, 1993; Fauchet et al., 1995). In order to solve the problem, different number of post-processing methods were adopted to improve the stability and efficiency of the PS's optical characteristics. Examples of such initiatives includes the PS combination with metals (Raypah and Ahmed, 2015; Leinartas et al., 2011), conducting polymers (Nguyen et al., 2000), and semiconductors (Wang et al., 2013; Rahim et al., 2012). Nanostructured metals have progressively attracted attention due to their interesting optical (Link and El-Sayed, 1999), electrochemical (Kolb, 2001), electronic (Fernández et al., 2004) and photo-electrochemical properties (Dawson and Kamat, 2001). Among these metals, copper is of special importance due to its excellence in conductivity of electricity and heat and its cost. Moreover, it acts as luminescent defects in the semiconductors' band gap (Samuelson et al., 1984) and have high biocompatibility (Bandarenka et al., 2013). Therefore, a lot of effort has been dedicated to copper (Cu) nanostructures, mainly due to their exciting applications in flexible electronics (Park et al., 2007), catalysis, nanocircuits (Monson and Woolley, 2003) and optoelectronic (Harraz et al., 2002).

Initial attempts to prepare Cu/PS composite structures were made with the aim of improving PS's optical characteristic efficiency and stability. Composite structures based on PS offer much more possibilities as compared with porous silicon because the shift from the element to the composite semiconductor has different crystallographic modifications, chemical composition, and electronic band gap. Consequently, the properties of the composites could be enhanced.

Several approaches have been experimentally studied to deposit copper metal into PS including so-called physical and chemical process. Electrodeposition, chemical vapor deposition (CVD) and sol-gel as a chemical process and sputter depositing, spin coater and thermal evaporation as a physical process. Each process has its own advantages and disadvantages which depends on the application. Therefore, it is difficult to predict which approach will enhance the efficiency and stability of optical properties of PS. Therefore, there is no alternative but to try various techniques to prepare copper deposit on PS in order to investigate the suitable optical properties for a PS. This has motivated the author to choose conventional electrochemical-deposition (ECD) method but with some modification, namely introducing colloidal copper species nanoparticles that were prepared by pulse laser ablation (PLA) as source of copper in electrolyte for metal deposition into PS arrays to modify the preparation condition.

1.2 Porous Silicon (PS)

In the photo-voltaic and electronic industries today, the crystalline silicon (c-Si) is the most important semiconductor material and is now considered cornerstone in the knowledge based society. The unique combination of some of its properties have given Si advantages and prominence position. These include: high purity, large single crystals availability, matching insulation, effectiveness in conductivity engineering, and most importantly, its natural abundance (Priolo et al., 2014). However, its application as a material for light emission has been limited by its intrinsic indirect band gap (1.12 eV). Researchers were able to find toward the end of the last century that the problem can be solved by preparing the PS using the electrochemical anodization (etching) (Cullis and Canham, 1991).

Silicon crystal that has network of voids in it is considered porous silicon. The voids in nano-size inside bulk silicon leads to the formation of sponge-like structure that is skeleton of crystalline silicon nanowires that surrounded by the channels and pores. Due to its versatile applications in both photonic and sensing devices, PS is now a potential platform with both technological and scientific attention (Harraz, 2014; Zayer et al., 2013). Having a very large ratio of surface area to volume ($\geq 500 m^2/cm^3$), formation ease, surface morphology control by varying the parameters of formation and its being compatible to the silicon ICT, the PS is now amenable to smart systems-on-chips sensors and a very attractive material. It has been most notably established that there are two features that characterized the Si nanocrystals' excitonic emission: the blue shift of spectrum and increasing its intensity, when the crystal size is decreasing. That observation is an indication of the band gap quantization-related increase and the radiative recombination rate enhancement (Fiory and Ravindra, 2003). These are suitable advantages in photo-detectors productions. As a very promising material, porous silicon has several advantages when applied in a photo-detector (Ohta et al., 2007; Ahmed et al., 2012) which are:

1. High efficiencies in terms of electro-optic conversion due to its direct band gap.
2. Light absorption rate can be increased by the porous surface.
3. Has high gain due to its avalanche effect.
4. Its ability to be directly formed on the silicon wafer to match completely with the silicon integrated circuit.

In the recent years, the miniaturization of electrical and optical components with high speed and efficient performance have received a very high interest due to their new incorporated capabilities in many different areas which also include the systems of high-speed telecommunication (Ohtsu, 2013). The technology of optical communication in the recent years has seen great development that affects all fields of the modern telecommunication system. Together with optical sources and optical fibers, the photo-detectors are considered to be an integral component in every system of optical fiber communication (Ilyas and Mouftah, 2003). As optoelectronic devices, the photo-detectors are known as converters of optical energy absorbed into a photo current (electrical energy) in the computers, systems of telephone, and other terminal components of communication systems at both receiving and transmission levels (Zimmermann, 2004).

1.3 Electrochemical-Deposition (ECD)

Electrochemical deposition has been considered as an important and inexpensive technology, which is one of the oldest and fast techniques to synthesize thin film and nanostructures (Lincot, 2005). It is a process, where two electrodes are required for the supply and removal of electrons. The cathode supplies electrons to the solution and the anode eliminates electrons from the solution. At least two reactions occurred simultaneously in an electrochemical cell; that is oxidation reaction in the anode and the reduction reaction in the cathode. These are referred to by electrochemists as “half-reactions”. When current is passed through a combination of electrolyte, and suitable electrodes, ions are moved through a solution by an electric field to coat an electrode, which is the working electrode. The requirement to run an electrodeposition experiment is an electrochemical cell with suitable electrolyte solution. The substrate is placed in the cell as an electrode and by using a potentiostat, a voltage in then applied to the cell (Hibbert, 1993).The electrochemical deposition method, in contrast to other deposition methods such as chemical vapor deposition (CVD), is very simple and efficient. The main advantage of this method is cost-effective. The material, handling, equipment and energy costs can be reduced using this method. The films can be deposited to the desired areas of the substrate (Benamar et al., 1998). The other advantages of this technique include processing temperature is low, shape geometry of substrate is arbitrary, and morphology and film thickness is controllable (Dini and Snyder, 2011).

1.4 Problem Statement

The porous silicon (PS) at room temperature has its photoluminescence (PL) in the range of near infrared and visible spectrum that indicates of a broad applications prospect in devices of optoelectronics. However, the inefficiency and instability of optical characteristic in PS create many problems in the application area such as the decrease of the PL intensity. The degradation and instability of photoluminescence in porous silicon originates from the transformation in the PS nanostructure and chemical composition at the PS surface under different conditions of formation and ambient atmospheres. In this work, in order to protect the PL in PS from degradation, the copper has been chosen as deposited layer due to its interesting properties that was mentioned in section 1.1.

To have comprehensive and precise research on the topic it was needed to have similar investigations on as-prepared and as deposited samples. First, the solution containing Cu species nanoparticles has been synthesized under different laser ablation times. Simultaneously, the PS samples have been prepared under different electrolyte concentration, etching times and current densities. Then, Cu/PS nanostructures have been fabricated by modifying several deposition parameters: Cu ions concentrations, deposition times, current densities and post-annealing temperatures to have wide range of samples in each step. Synthesis of novel nanostructures such as nanorods and nanodendrite using integrated the yield of laser ablation and electrochemical etching techniques are required. The simplicity, lower cost, and suitability of the method to produce nanostructures are very promising.

1.5 Hypothesis

There are critical issues that need to resolve in order to fabricate a porous silicon which met all the criteria in the previous section. The first one is to select a material that will allow us to harvest the light efficient. In this regard it has chosen CuO as our primary absorption layer. The second one is to select a method to prepare CuO nano-colloids without contamination, so pulse laser ablation has attracted considerable attention due to its one-step, direct and very simple preparation procedure, since the process only requires room temperature and pressure. Therefore, there is needed a method to deposit CuO into the PS and electrochemical deposition is one of the attractive procedure to implement it. However, the critical questions here are how can copper oxide NPs that obtained via pulse laser ablation be intentionally deposited on PS surface? How can be controlled the morphology of copper deposited on PS surface by ECD? It is known that in electrochemical cell two reactions occurred that is reduction and oxidation reactions. When current is passed through electrochemical cell, copper ions are moved through a solution by an electric field to coat PS electrode, which is the working electrode, final product of CuO/PS nanocomposites may be achieved. The assumption is explained below.

i. **Hypothesis 1:** By applying pulsed laser ablation on Cu solid target in the aqueous medium, small size of NPs could be obtained. Depending on the duration of the ablation time, the size distribution and concentration of these NPs can be readily controlled.
ii. **Hypothesis 2:** By applying electrochemical etching process on Si substrate in diluted HF solution, the porosity (pore morphology and density), structural and optical properties can be controlled. The increasing of the porosity coincides with decreasing of Si grain size which leads to increase in the band gap energy and blue shift of the PL peak.
iii. **Hypothesis 3:** The present of Cu atoms, ions (species) inside the PS layer create impurity levels (donor or accepter level). Cu introduces new channels of radiative recombination that could assist to overcoming the low radiative decay typical in porous silicon. In this work n-type silicon, in addition to electron excitation from valance band to conduction band, electron excitation may also occur from impurity level to conduction band. Consequently, they may increase carrier concentrations and affect electronic properties that are assumed to promote higher intensity of PL. Thereby the photo-response of photodetectors with a proper parameters of the Cu-coated PS would be enhanced.

1.6 Research Questions

Based on the hypotheses outlined above, the following research questions have been set:

During pulse laser ablation, what effects of using distilled water (DW) without surfactant agent on the particles size and shape? What type of composition that can be obtained from ablation Cu target in DW without surfactant agent, is it single or mix structures? What is the best ablation time that can yield Cu particles in nano-scale with narrow distribution between (10-25 nm)?

What effect of increasing the etching time and current on the porosity percentage and uniformity? Could the increase of hydrofluoric acid concentration in the electrolyte lead to increase Si crystal size? Is it possible, that increase the etching current impacts the optical properties? Will it give blue shift in spectral emission? If yes, what is the photon energy? Is it possible to penetrate the Cu NPs from liquid in pore channels without pore mouth blockage by the metal NPs? Is it true, that deposition metal on PS caused enhancement of photoluminescence?

1.7 Objective of Study

The aim of this work is to prepare porous silicon nanostructures (PS) and study the effect of incorporated copper nano-particles on the optical, structural and electrical properties of PS. Thus the objectives of this work are:

1- To study the effect of ablation times on the particles size distribution of Cu colloidal NPs synthesized by pulse laser ablation.
2- To investigate the effect of the etching parameters on the optical, structural and morphological properties of PS. The parameters that will be investigated are current density, etching time and electrolyte concentration, and determine the optimal etching parameters for the synthesis of PS by electrochemical etching.
3- To investigate the effect of embedded Cu on the structural, morphology and optical properties of PS substrates.
4- To study the effect of PS and Cu nanodendrite/PS nanostructures on the performance of metal-semiconductor-metal photodetector.

1.8 Significance of Study

The reason why to combine the PS with Cu metal is to investigate the effect on the photoluminescence behaviour. Since deposition metals can change the original system of the porous silicon structure, it has inspired author to study whether it can enhance the optical behaviour of the photoluminescence, so that it can be applied in porous silicon-based photodetector.

1.9 Scope of the Study

Starting from a quick view on research background, the author identified the research gaps and set-up several hypothesis. Several research questions were put forward to transform the hypothesis into objectives in order to design the section of experimental work. Finally significant of the study has been identified in Chapter 1.

In Chapter 2, the previous studies related to Copper NPs that prepared by pulse laser ablation technique, PS synthesis by electrochemical etching, metal/PS composite that prepared by different methods, and their application as photodetectors were reviewed.

Chapter 3 is focused on the theory of pulse laser ablation, the theory of PS formation, basic principle and parameters of MSM photodetector.

Chapter 4**,** details out the experimental methodology and the general principles of operations of the characterization tools.

Chapter 5 is dedicated to the characterization results of Cu NPs, PS and Cu/PS composite. The gravimetrical method, TEM, FESEM, AFM, contact angles, XRD, Raman spectroscopy, UV and photoluminescence (PL) spectroscopy take into account for investigating on the porosity percentage, thickness, structural properties, absorbance spectrum, band gap, and emission peak of samples. Finally, the performance of the photodetector devices fabricated based on CuO nanodendrites/porous silicon are also presented.

Chapter 6 presents the work's conclusions and future work recommendation.

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