

**UNIVERSITI TEKNOLOGI MARA**

**FABRICATION AND VERIFICATION  
OF POROUS SILICON  
NANOSTRUCTURES/ ZINC OXIDE  
NANOSTRUCTURES AS A  
CAPACITIVE CHEMICAL SENSORS  
BY ELECTROCHEMICAL  
IMPEDANCE METHOD**

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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 own 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.

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## ABSTRACT

In this study, preparation and optimization of ZnO nanostructures on PSiNs for chemical sensor was studied. The PSiNs was prepared by electrochemical etching using p-type, [100] orientation silicon wafer as a based material. The PSiNs samples were prepared by the electrochemical etching with photo-assisted at different current density in the range of 15–40 mA/cm<sup>2</sup> and etching time at 10–50 minutes. Photoluminescence spectra show blue shift with increasing applied current density that is attributed by PSiNs pillar size. Variations of electrical resistance and capacitance values of PSiNs were measured using EIS. These results indicate that PSiNs prepared at 20 mA/cm<sup>2</sup> current density and 30 minutes have uniform porous structures with a large density of pillars. Furthermore, PSiNs structure influences large values of charge transfer resistance and double layer capacitance, indicating potential application in sensors. The ZnO nanostructures were synthesized on PSiNs substrates using thermal catalytic-free immersion method with Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O as a precursor and CH<sub>4</sub>N<sub>2</sub>O as a stabilizers. Three parameters was used to optimized the maximum changes of capacitance on ZnO nanostructures on PSiNs, solution concentration (0.1, 0.2 and 0.3 molar), molarity of CH<sub>4</sub>N<sub>2</sub>O (0.05, 0.1, 0.2, 0.4 and 0.6 molar) and immersion time (2, 4, 6, 8 and 10 hour). ZnO nanoparticle and nanoflowers were successfully synthesized on PSiNs substrate. An approach to fabricate chemical sensor based on the PSiNs/ZnO nanostructures arrays that uses an electrochemical impedance technique is reported. Sensor performance was evaluated by ethanol solutions using EIS measurement. PSiNs/ZnO nanostructure exhibits higher sensitivity than PSiNs, with 85.96% response after exposure to ethanol for 2 min. Compared with the PSiNs sensor, the response of which is low at only 20% and the maximum response of 48% is reached 6 min later. PSiNs/ZnO nanostructures with a response of 85.96% after exposure to ethanol for 2 min remains constant for some period time (10 min). This stability in response proved that the sample is stable and suitable as a sensing material based. The results indicate that the PSiNs/ZnO nanostructure chemical sensor exhibits rapid and high response to ethanol compared with a PSiNs sensor because of its small particle size and an oxide layer acting as a capacitive layer on the PSiNs surface. After that, the sensor was tested with different chemical solution, acetone, chloroform, benzene and toluene. For PSiNs sensor the response is low at ethanol, toluene and benzene with only 40%, and the maximum response of 60% is reached in 10 min. Compared with the PSiNs sensor, the PSiNs/ZnO nanostructures prepared sample exhibits higher sensitivity to ethanol, acetone and toluene, as observed above 70% response after exposure for 10 min. Both the sensors (PSiNs and PSiNs/ZnO nanostructures) give the least response to benzene as compared to others chemical solution. The benzene response in 10 minutes for PSiNs and PSiNs/ZnO nanostructures was lowest compare to other. There appeared two sensors show the resistance changes at 45% and 31% response, respectively. The sensor performance was improved when ZnO nanostructures deposited on PSiNs surface.

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