

CMOS 집적회로 기반의 무효소 혈당센서 적용을 위한 메조포러스 백금 전극 제작 및 최적화

서해경, 박대준, 박재영
마이크로/나노 소자 및 패키징 연구실, 전자공학과, 광운대학교, 서울

Fabrication and Optimization of Mesoporous Platinum Electrodes for CMOS Integrated Enzymeless Glucose Sensor Applications

Hye K. Seo, Dae J. Park and Jae Y. Park

Micro/Nano Devices & Packaging Lab. Department of Electronic Engineering, Kwangwoon University, Seoul

Abstract - In this paper, mesoporous only platinum electrode and micro pore platinum electrode with mesoporous Pt are fabricated and characterized on a silicon substrate to check their usability as enzymeless sensing electrodes for developing non-disposable glucose sensors integrated with silicon CMOS read out circuitry. Since most of electrochemical glucose sensors are disposable due to the use of the enzymes that are living creatures, these are limited to use in the in-vivo and continuous monitoring system applications. The proposed mesoporous Pt electrode with approximately 2.5nm in pore diameter and 150nm in height was fabricated by using a nonionic surfactant $C_{16}EO_8$ and an electroplating technique. The micro pore Pt electrode with mesoporous Pt means the mesoporous Pt electrode fabricated on top of micro pore arrayed Pt electrode with approximately 10 μ m in pore diameter and 80 μ m in height. The measured current responses at 10mM glucose solution of plane Pt, micro pore Pt, micro pore with mesoporous Pt, and mesoporous Pt electrodes are approximately 9.9nA/mm², 92.4nA/mm², 3320nA/mm² and 44620nA/mm², respectively. These data indicate that the mesoporous Pt electrode is much more sensitive than the other Pt electrodes. Thus, it is promising for non-disposable glucose sensor and electrochemical sensor applications.

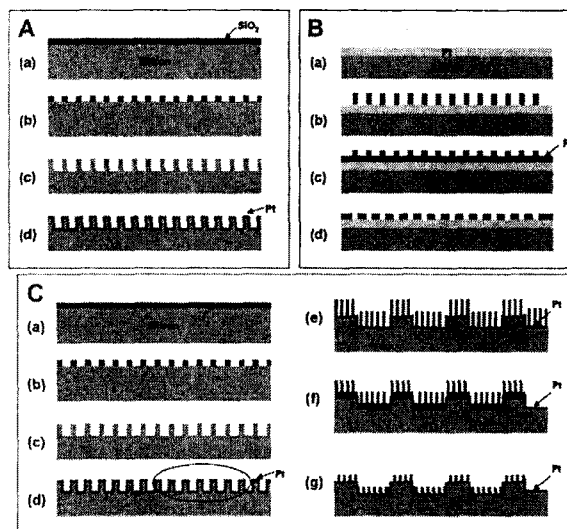
1. Introduction

Most of electrochemical biosensors are disposable due to the use of the enzymes that are living creatures. Thus, these are limited to use in the in-vivo and continuous monitoring biosensor system applications. The mesoporous (pores with a size of 2nm ~ 50nm) platinum electrodes formed on a Pt rod was reported for developments glucose sensors without any enzymes [1]. The structure and fabrication method of the mesoporous Pt electrode were first reported in 1997 [2]. The reported mesoporous Pt structure was fabricated on a Pt disk electrode (rod type) by using electroplating technique and a hexagonal liquid crystalline phase composed of the nonionic surfactant $C_{16}EO_8$. The electrodeposited Pt electrode was comprised of cylindrical hexagonally arrayed pores with a diameter of 2.5 nm and 5 nm of spacing distance between the pores.

2. Experimental Procedures

The proposed micro pore Pt electrode with approximately 10 μ m in pore diameter and 80 μ m in height was fabricated by using deep RIE silicon etcher and DC sputtering system and mesoporous Pt electrode with approximately 2.5nm in pore diameter and 150nm in height was fabricated by using a nonionic surfactant $C_{16}EO_8$ and an electroplating technique. And the proposed micro pore with mesoporous Pt electrode was fabricated by combining the above fabrication processes. The respective fabrication steps are represented in figure 1. For evaluation of the fabricated micro/nano pore Pt electrodes, the current responses are measured and compared by comparison with plane Pt electrode.

The fabrication steps of the micro pore Pt electrode are represented in figure 1 (A). The SiO_2 layer is firstly deposited on the silicon substrate, and SiO_2 is patterned for forming the micro pore structure by using conventional photo lithography. The SiO_2 and silicon are dry-etched by using RIE and deep RIE. The top SiO_2 is removed and Ti/Pt layers are finally deposited. The fabrication steps of the mesoporous Pt electrode are represented in figure 1 (B). Ti and Pt layers are firstly deposited on top of SiO_2 of the silicon substrate. Liquid crystal templates of the $C_{16}EO_8$ are formed on the substrate at 85 $^{\circ}$ C and Pt ions are electrodeposited on the silicon at 25 $^{\circ}$ C. Finally, the templates are clearly removed in the deionized water. In figure 1 (C), micro pore with mesoporous Pt electrode was fabricated by combining above two method.

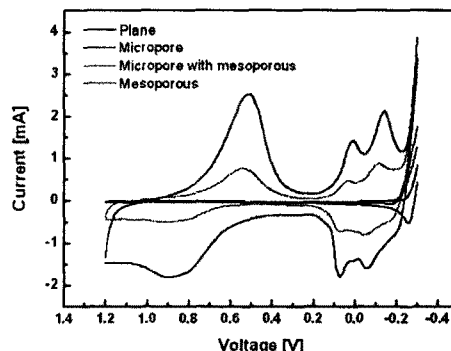


<Figure 1> Fabrication steps of micro pore Pt (A), mesoporous Pt (B), and micro pore with mesoporous Pt electrodes (C).

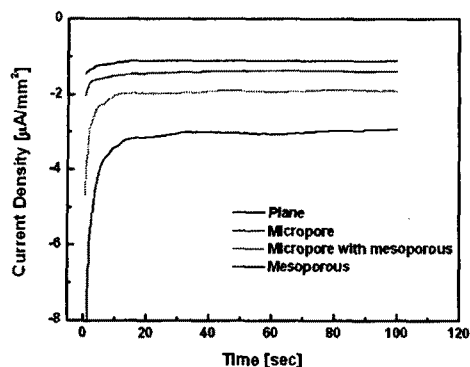
3. Experimental Results and Discussions

All electrochemical measurements were performed in a three electrodes system by using an electrochemical analyzer (CH Instruments Inc., USA) and an Ag/AgCl electrode was used as a reference electrode. For evaluation of the fabricated micro and mesoporous Pt electrodes, the current responses are measured and compared by comparison with plane Pt electrode.

Figure 2 shows comparison of the cyclic voltammetry in 2M sulfuric acid solution of the fabricated micro/nano pore Pt electrodes. The current response of the mesoporous Pt electrode is much larger than the other Pt electrodes. Figure 3 shows comparison of current responses to the chronoamperometry in 1mM hydrogen peroxide solution of micro/nano pore Pt electrodes. These data indicates that the mesoporous Pt electrode is much more sensitive than the other Pt electrodes.

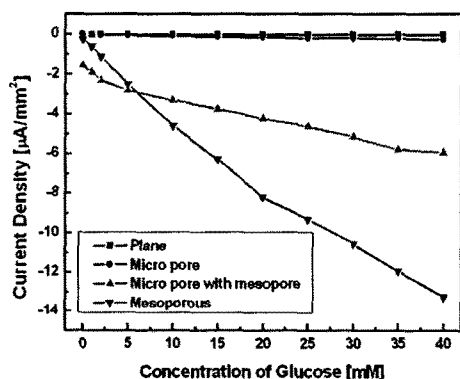


<Figure 2> Comparison of the cyclic voltammetry in 2M sulfuric acid solution of the fabricated micro/nano pore Pt electrodes.



<Figure 3> Comparison of current responses to the chronoamperometry in 1mM hydrogen peroxide solution of the fabricated micro/nano pore Pt electrodes.

The fabricated Pt electrodes were also evaluated to check their applicability for fabricating the glucose sensor by varying the glucose concentrations in the 0.1M phosphate buffered saline solution. Figure 4 shows the measured current response of the fabricated Pt electrodes at various glucose concentrations. The measured current responses at 10mM glucose solution of plane Pt, micro pore Pt, micro pore with mesoporous Pt, and mesoporous Pt electrodes are approximately 9.9nA/mm², 92.4nA/mm², 3320nA/mm² and 44620nA/mm², respectively. The current density of the mesoporous Pt electrode relatively linearly increases as the increasing concentration of the glucose. These data indicate that the mesoporous Pt electrode is very sensitive to the glucose and strongly applicable for the non-disposable biosensor and electrochemical sensor applications.



<Figure 4> Comparison of the current response to various glucose concentrations of the fabricated electrodes.

4. Conclusion

The micro/nano pore platinum electrodes have been fabricated and characterized on a silicon substrate. In comparison of various micro/nano pore platinum electrodes, the mesoporous Pt electrode has the best performance characteristics. The measured current response of the mesoporous(nano pore) Pt electrode was large enough to analyze the chemical substances without any enzymes. Thus, the mesoporous Pt electrode is promising to the development of non-disposable glucose sensors integrated with CMOS circuitry. These results also suggest that the mesoporous Pt electrode have strong potential in use of chemical, environmental, and biological analysis systems.

[Reference]

- [1] S. J. Park, T. D. Chung and H.C. Kim, "Nonenzymatic glucose detection using mesoporous platinum", *Anal. Chem.*, vol. 75, p. 3046-3049, 2003.
- [2] G. S. Attard, P. N. Bartlett, N. R. B. Coleman, J. M. Elliott, J. R. Owen and J. H. Wang, "Mesoporous platinum films from lyotropic liquid crystalline phases", *Science*, vol. 278, p. 838-840, 1997.