Microelectronic Engineering 133 (2015) 11–15

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Ordered-standing nickel hydroxide microchannel arrays: Synthesis and application for highly sensitive non-enzymatic glucose sensors

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ARTICLE INFO

Article history:
Received 13 March 2014
Received in revised form 30 September 2014
Accepted 11 November 2014
Available online 29 November 2014

Keywords:
Electrochemical sensor
Nickel hydroxide
Glucose oxidase
Ordered channels

ABSTRACT

A non-enzymatic glucose sensor is constructed by using nickel hydroxide ($Ni(OH)_2$) modified silicon microchannel plates (MCP) as the sensing materials. The 3D ordered Si MCP is fabricated by electrochemical etching and the $Ni(OH)_2$ coating are prepared by electroless plating. The nanocomposite electrode exhibits good catalytic activity toward oxidation of glucose in 0.1 M KOH. This non-enzymatic glucose sensor boasts a fast amperometric response time of less than 5 s, sensitivity of 0.25 mA mM^{-1} cm^{-2}, and detection limit of 3.5 μM at a signal-to-noise ratio of 3. The sensor shows superior stability, anti-interference capability, and selectivity. The good analytical capability, low cost, and compatibility with silicon technology make the $Ni(OH)_2$/Si MCP electrode promising in amperometric non-enzymatic glucose detection.

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1. Introduction

The development of novel glucose sensors with high sensitivity, reliability, fast response, and good selectivity is crucial to diagnosis and treatment of diabetes mellitus, clinical biochemistry, waste water treatment, and the food industry [1–3]. Since Clark and Lyons utilized glucose oxidase to produce the first enzymatic electrode in 1962 [4], it has attracted extensive attention. Conventional glucose sensors involve the use of glucose oxidase (GOD) which catalyzes oxidation of glucose in the presence of O_2 to produce hydrogen peroxide. The signal transduction is based on oxidation of the hydrogen peroxide. Although this type of biosensors displays very high sensitivity and selectivity to glucose, the complicated immobilization procedures, thermal and chemical instability, and high cost of enzymes have hindered progress. Nevertheless, many types of enzymatic glucose sensors have aroused interest and been applied to direct electrochemical oxidation of glucose [5]. Some noble metals (Pt, Au, Ag, etc.), alloys (Pt–Pb, Pt–Au, Ni–Pd, etc.), metal oxides (RuO_2, CoO, MnO_2, etc.), carbon nanotubes, and graphene have been used in the fabrication of non-enzymatic glucose sensors [3,6–8]. In particular, nickel-based nanomaterials exhibit remarkable activity owning the presence of the $Ni(OH)_2$/NiOOH redox couple formed in an alkaline medium [9–10]. Compared to the other materials, Ni is relatively economic. As known that nano/microstructured materials exhibit enhanced electrochemical performance and many types of Ni-based materials have been fabricated with various structures such as nanoparticles [11–12], nanoflake arrays [13], and so on.

The use of Ni-based nanomaterials requires a supporting substrate. An ideal support should have the following properties: (1) high surface area for catalyst loading; (2) excellent conductivity and convenient routes for electron transfer; (3) compatibility with silicon technology to enable device miniaturization. Silicon microstructures such as Si microchannel plates (MCP) possess unique properties including ordered structure, biocompatibility, multifunctionality, and compatibility with microelectronics technology [14]. These advantages make Si MCPs promising in electrochemical sensors. In this work, a non-enzymatic glucose sensor based on $Ni(OH)_2$/Si MCP electrodes is fabricated and the performance is studied systematically.

2. Experimental details

2.1. Materials and reagents

The wafer used to fabricate the Si MCPs was single-side polished, p-type silicon with a resistivity of 2–9 Ω cm, and a thickness

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http://dx.doi.org/10.1016/j.mee.2014.11.005
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of 525 μm. Hydrofluoric acid, dimethylformamide, ammonium fluoride, sodium hydroxide, ethanol, and all other reagents were of analytical reagent grade and used without purification. The specific resistivity (18 MΩ) of de-ionized water was used and all the experiments were carried out at room temperature in a clean environment.

2.2. Preparation of silicon microchannel plates

The silicon microchannel plates were fabricated according to Ref. [15] using a chemical etching procedure. The silicon wafers were cleaned by a standard RCA process, rinsed with deionized water several times, and dried with nitrogen. Silicon oxide as the masking layer was formed by thermal oxidation, and lithography and wet etching were utilized to define the electrochemically etched locations. The masking pattern was made of 5×5 μm squares with a spacing of 6 μm between the centers of each neighboring square. Before electrochemical etching of the silicon substrate, an aqueous tetramethylammonium hydroxide (TMAH) was used to produce pits on the surface. High aspect ratio Si MCPs were fabricated by electrochemical etching using a solution of hydrofluoric acid and dimethylformamide at the optimal current density (10 mA cm^{-2}), etching bias (15 V), and temperature (20 °C). A cooling system was used to prevent temperature increase caused by illumination. The etching current density was stabilized by a program based on LabVIEW. The composition of the etchant and etching time were adjusted to control the lengths of the Si MCPs. The fabrication process of the Si MCP and schematic drawing of the etching system are depicted in Fig. 1.

2.3. Preparation of nickel oxide-modified silicon microchannel plates (NiO/Si MCP)

Electroless plating was conducted because the channels in the silicon MCP could be coated with metal uniformly and the thickness of the metal film could be easily controlled by the bath composition and temperature [16]. For uniform fabrication of the electrode for glucose sensing, the separated Si MCPs was segmented into small chips with a rectangular shape by laser cutting. The chips were cleaned using the standard RCA process. The samples were dried at 30 °C with nitrogen and weighed on an ultra-microbalance with a precision of ±0.001 mg (Mettler Toledo, Switzerland). They were put into a buffer solution (TritonX-100) for 30 s to decrease the inner stress and enhance wetting prior to immersion in the plating bath for 20 min. Afterwards, they were taken out and rinsed with water. Plating was carried out at 70 °C in a solution prepared by mixing nickel sulfate ($NiSO_4$), ammonium sulfate ($(NH)_4SO_4$), ammonium fluoride (NH_4F), sodium citrate ($C_6H_5Na_3O_7$), in sodium lauryl sulfate ($CH_3(CH_2)_{10}CH_2OSO_3Na$) in deionized water. $NiSO_4$ was used as the metal source and $C_6H_5Na_3O_7$ as the complexing agent. During deposition, the pH was adjusted to pH 8.0 by addition of ammonia. Afterwards, the nanocomposite was annealed at 500 °C for 300 s in a rapid thermal annealing (RTA) system. Copper wires were connected to the silver

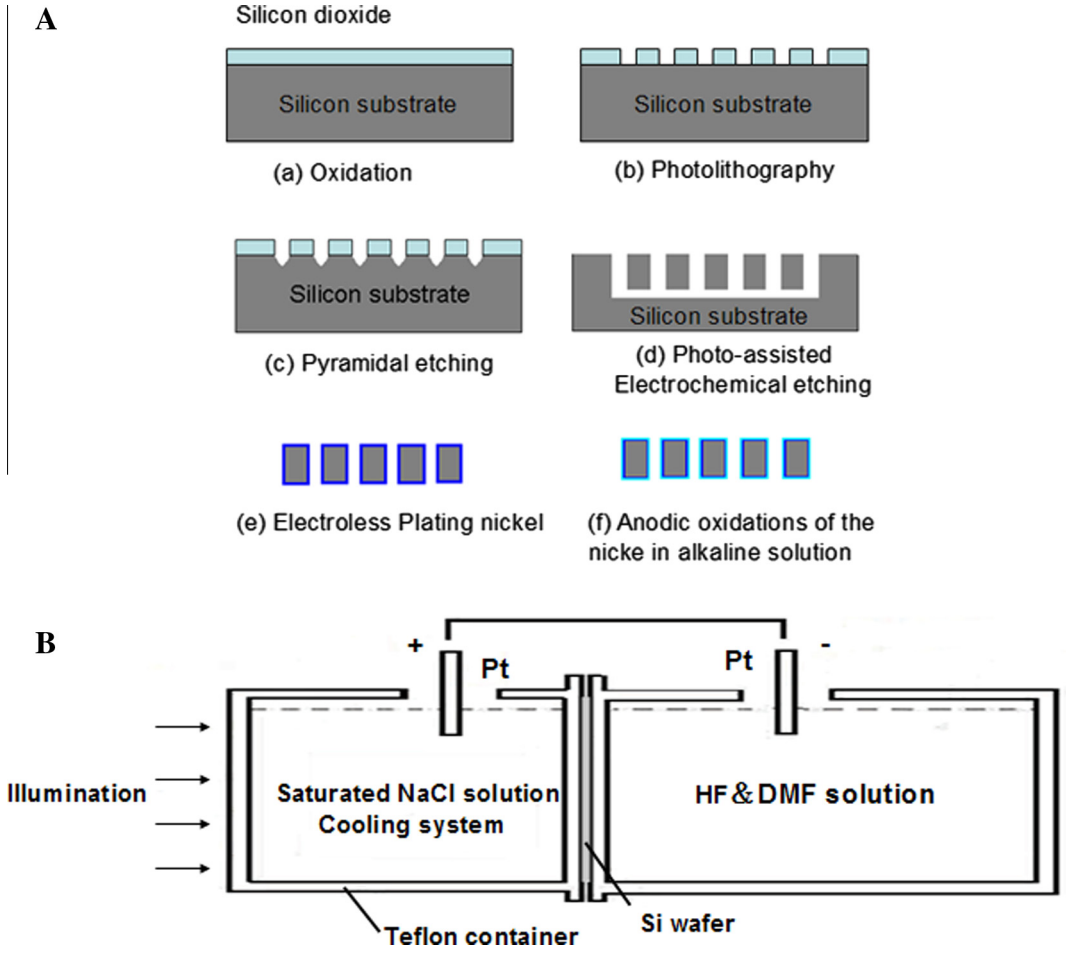

Fig. 1. (a) Fabrication process of the Si MCP and (b) schematic drawing of the etching system.

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