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Chemically Modified Electrodes for Recessed Microelectrode Array

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Abstract

Chemical modifications on recessed microelectrode array, achieved via electrodeposition techniques are reported here. Silicon-based gold microelectrode arrays of 10 µm microband and microdisc array were selected and functionalised using sol-gel and nanoporous gold (NPG) respectively. For electrochemically assisted self-assembly (EASA) formation of sol-gel, electrode surface was first pre-treated with a self-assembled partial monolayer of mercaptopropyltrimethoxysilane (MPTMS) before transferring it into the sol containing cetyltrimethyl ammonium bromide (CTAB)/tetraethoxysilane (TEOS):MPTMS (90:10) precursors. A cathodic potential is then applied. It was found that larger current densities were required in ensuring successful film deposition when moving from macro- to micro- dimensions. For NPG modification, a chemical etching process called dealloying was employed. NPG of three different thicknesses have been successfully deposited. All the modified and functionalized microelectrode arrays were characterized by both optical (SEM) and electrochemical analysis (cyclic voltammetry and impedance spectroscopy). An increase in surface area and roughness has been observed and such will benefit for future sensing application.

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1. Introduction

Micro- and nanoelectrode arrays offer a number of advantages for the electrochemical detection of analytes: (i) an improved mass transport leading to an increased sensitivity and better limits of detection; (ii) improved signal to noise ratio; (iii) reduced ohmic drop [1]. Using tools originally developed for the silicon integrated circuit (IC) industry, researchers are now fabricating miniaturized transducers from silicon and other materials [2] to be used in various biosensor applications such as environmental monitoring [3,4], food safety [5] and medical diagnostics [6]. These technologies allowed the fabrication of microelectrode arrays of different geometries and materials [7].

The surface of microelectrodes can be modified to achieve a desired selectivity or improve the sensitivity. Microelectrodes can be modified by electropolymerisation [8], to form a layer on the electrode surface sensitive to pesticides [9,10] or drugs [11]. Thiols can be spontaneously adsorbed onto gold nanoparticles immobilised on a gold microband array for the detection of organics in water [12,13] or they can be used as an anchor for the immobilisation of aptamers [14] and bacteriophage [15]. Antibodies for the amperometric detection of Aflatoxin M1 in milk could be immobilised via silanisation [5]. Functionalised mesoporous silica has also been electrogenerated at the surface of microelectrodes [16,17]. Macroporous gold was formed on a gold microelectrode to combine the high surface area of a macroporous materials with the high mass transport achieved at a microelectrode [18].

Two methods of chemical modification were selected in this study, namely electrochemically assisted self-assembly formation of sol-gel and nanoporous gold electrodeposition. These two methods were chosen as to our knowledge these two modification methods have not been applied to recessed microelectrode array to date. Mesoporous and nanoporous materials have attracted a wide interest in electroanalysis in recent years [19, 20]. Recent studies have shown that electrochemically assisted self-assembly of silica leads to well-organised mesoporous silica framework with pores oriented perpendicular to the electrode surface [21]. Co-condensation of organo-silane with hydrolysed silane forms mesoporous silica bearing functionalities that can be used for the detection of heavy metals (e.g. $Cu^{2+,}$ Ag^{+}, Hg(II)) [16,17,22]. Nanoporous gold electrodes have also been investigated due to the increase of surface area important for catalytic applications [23-25] apart from biosensor applications.

We investigate here the modification of gold microelectrode arrays achieved by electrogeneration of mesoporous silica on 10 μm microband array and nanoporous gold on 10 μm microdisc array. These particular dimensions were selected due to their stability and reproducibility compared to the nano-dimension counterpart [26]. Electrochemical deposition approach is of interest as it selectively modified the area of interest without smearing the passivation tracks or unwanted area. The porous materials are formed by either a galvanostatic or potentiostatic methods. Although both dip-coating and drop-coating techniques are straight-forward and simple, nevertheless the major concerns associated with these techniques are that they are confined to flat surface and lack for selectivity [27]. The modified microelectrode array are then characterised by cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM).

2. Material and Methods

2.1. Material and reagents

All chemicals used in this study were purchased from Sigma-Aldrich Ireland Ltd. and used as received. All solutions were prepared with high purity water (18.2 MΩ cm^{-1}) obtained from a Purelab Option from ELGA.

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