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Different methods to alter surface morphology of high aspect ratio structures



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ABSTRACT

In various applications such as neural prostheses or solar cells, there is a need to alter the surface morphology of high aspect ratio structures so that the real surface area is greater than geometrical area. The change in surface morphology enhances the devices functionality. One of the applications of altering the surface morphology is of neural implants such as the Utah electrode array (UEA) that communicate with single neurons by charge injection induced stimulation or by recording electrical neural signals. For high selectivity between single cells of the nervous system, the electrode surface area is required to be as small as possible, while the impedance is required to be as low as possible for good signal to noise ratios (SNR) during neural recording. For stimulation, high charge injection and charge transfer capacities of the electrodes are required, which increase with the electrode surface. Traditionally, researchers have worked with either increasing the roughness of the existing metallization (platinum grey, black) or other materials such as Iridium Oxide and PEDOT. All of these previously investigated methods lead to more complicated metal deposition processes that are difficult to control and often have a critical impact on the mechanical properties of the metal films. Therefore, a modification of the surface underneath the electrode's coating will increase its surface area while maintaining the standard and well controlled metal deposition process. In this work, the surfaces of the silicon micro-needles were engineered by creating a defined microstructure on the electrodes surface using several methods such as laser ablation, focused ion beam, sputter etching, reactive ion etching (RIE) and deep reactive ion etching (DRIE). The surface modification processes were optimized for the high aspect ratio silicon structures of the UEA. The increase in real surface area while maintaining the geometrical surface area was verified using scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS). The best results were obtained by DRIE induced surface morphology. Decreases in impedance values of electrodes up to 76% indicate the successful surface engineering of the high aspect ratio silicon structures.

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1. Introduction

The surface modification of silicon to create a structure of defined roughness is desirable in many applications. Especially in the development of solar cells, rough silicon surfaces minimize reflection losses by several reflection and absorption processes [1-3]. Besides further applications such as high capacity battery anodes [4], terahertz radiation emitters [5], and water repelling surfaces [6], rough silicon surfaces are widely used in biosensors [7] and biomedical systems. Miniaturized capillary

http://dx.doi.org/10.1016/j.apsusc.2016.01.008 0169-4332/© 2016 Elsevier B.V. All rights reserved. electrochromatography structures make use of surface engineered silicon surfaces with a defined roughness for miniaturized total analysis systems (µTAS) used in drug screening and development [8]. Furthermore, a well-defined roughness on silicon surfaces can be used for control and increase of cell adhesion and viability [9], but can also exhibit bactericidal effects [10]. Whereas most of the applications require very fine nano-porous surfaces, this work aims for a less dense surface roughness in order to decrease the electrodes impedance by increasing the real surface area while avoiding the pore resistance effect [11]. The pore resistance effect occurs for narrow and deep pores, where only parts of the electrode's surface can be accessed by the current due to higher time constants. Depending on the desired roughness for the various applications, different methods are currently known and reported in literature. The different methods range from wet chemical processes such as stain etching using hydrofluoric acid (HF) and

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nitric acid [12], electrochemical HF etching [13] over dry etching techniques such as reactive ion etching [14] to laser induced surface roughness using femtosecond [15–17] laser systems.

One of the applications for changing the surface morphology of high aspect ratio structures is in biomedical devices such as neural implants. This paper e.g. resulted from a National Institutes of Health funded project to improve the electrochemical performance of neural implants. These implants are basically microelectrodes that are used in the study and diagnostics of neurological disorders such as epilepsy and chronic depression [18,19]. Furthermore, neural implants play a crucial role for the development of neural prostheses which can restore lost motor or sensory functions of the body. Two of the most important characteristics for these microelectrodes are selectivity (ability to interact with a limited number of neurons) and sensitivity (ability to interact with neurons which are far from the electrodes). Selectivity increases with decreasing surface area of the electrode. In contrast, sensitivity increases with increasing surface area of the electrode. Hence, there is a trade-off between selectivity and sensitivity. One of the ways to overcome this design trade-off of having selectivity and sensitivity is to keep the geometrical surface area constant while increasing the real surface area by altering the surface morphology of the high aspect ratio Utah array electrodes.

In this paper, various methods and processes were investigated and optimized to engineer the surface of high aspect ratio (15:1) penetrating Utah electrode arrays (UEA). The UEA consists of a 10×10 square grid of 1.5 mm long electrodes with 400 μ m spacing. 100 Pt/TiW/Pt bond pads are deposited on the back surface of the array. The tip of each electrode is usually metalized with Pt or sputtered iridium oxide (SIROF) to facilitate electronic to ionic transduction. The entire array, with the exception of the tip of each electrode, is insulated with a biocompatible Parylene-C layer (Fig. 1). A detailed fabrication procedure of the UEA is given elsewhere [20].

The modification of the electrode surface leads to an increase of the real surface area (RSA) of the UEA electrodes while keeping their geometric surface area (GSA) constant, hence mitigating the trade-off between selectivity and sensitivity. There are multiple advantages for having surface roughness compared to smooth surface. The roughened UEA will not only increase the SNR, but also decrease the necessary voltage which has be applied to the electrode to deliver a certain amount of charge by increasing the double layer capacitance at the electrode/electrolyte interface [21]. Furthermore, studies have shown that surface roughness can have a significant impact on cell adhesion [9], leading to enhanced cell growth on the implants and to increased stability of the device in chronic applications. In order to roughen the surface of the electrode tips of the UEA, two main approaches are possible. The first approach is to deposit a material which has superior electrochemical properties and chronic stability. In literature, there are many materials that are added to the electrode tips such as nano-cluster platinum (NCPt) films [22], sputtered iridium oxide film (SIROF) [23,24], fractal titanium nitride (TiN) [11,21], Poly(3,4-ethylenedioxythiophene) (PEDOT) [25,26], or conducting polymer/biomolecule blends [27]. The second approach is to roughen the metal/electrode contact material by removing material using etching processes or laser ablation to increase the electrochemical properties. The electrode in contact with the biological tissue usually consists of a silicon substrate which is coated with a biocompatible material exhibiting better electrochemical characteristics than silicon. Hence, the patterning of electrode surfaces can either be carried out on the underlying silicon substrate or directly on the biocompatible coating. This work focusses on dry etching methods to modify the surface of high aspect ratio neural implants. In literature, reactive ion etching (RIE) has been used for surface engineering however it is limited to planar substrates only [28] or turned out to be irreproducible [29].

To the authors best knowledge, publications about surface roughening of neural implants are limited to the previously mentioned deposition of porous materials or the etching of planar silicon electrodes without a coating for increased biocompatibility and electrochemical properties. Not only does this paper describe the roughening of high aspect ratio 3D structures, but also presents, compares, and discusses different accurate surface modification techniques.

In this paper, a platinum layer was chosen as coating material, since the roughness was created by dry etching, milling and ablation techniques. As opposed to complicated coating processes, such as reactive ion sputtering for SIROF or electroplating for platinum black, the deposition of a sputtered platinum thin film benefits from the simplicity and high reproducibility of the deposition process. Furthermore, Pt is considered gold standard in the field of neural implants due to its long term stability and electrochemical properties [11].

2. Experimental

Different processes such as laser ablation, ion milling, sputter etching, RIE and DRIE were investigated as methods for the creation of a micro-structure on the electrodes surfaces. In order to ensure the bio-compatibility of the UEA, the electrodes were coated with a biocompatible platinum layer. Depending on the surface modification process, the UEAs were platinum coated prior to the surface

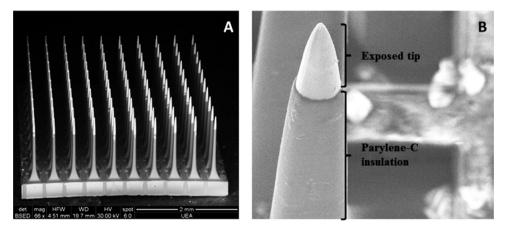


Fig. 1. Scanning electron micrographs (SEM) of an entire UEA (A) and of a single electrode, where the active site is formed by the exposed Pt coated tip.

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