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High aspect ratio diamond microelectrode array for neuronal activity measurements

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Abstract

Diamond exhibits several attractive properties for bio-sensing applications. In particular its high bio inertness, high electrochemical stability, and optical transparency provide diamond with high interests for neural activity study. The purpose of this study is the realisation of microelectrodes arrays (MEA) in diamond for neurons slices study. Due to a cellular lysis on the edge of the tissue slices studied, electrodes have to be at least 60 μm in height even though the electrode surface has to be minimised in order to achieve good signal noise rate. Silicon MEA with metal contacts were realised using (Deep Reactive Ion Etching) DRIE and coated with Nanocrystaline Diamond (NCD) using (Bias Enhanced Nucleation) BEN technique. We focus the study on the understanding of the BEN nucleation process on such high aspect ratio electrodes. Several parameters such as slope of the substrate, conductivity and chemical nature of the substrate were studied in order to enable selective nucleation necessary to fabricate diamond MEAs. The study leads to the optimised development of a processing route enabling the selective coating of the active tips of high aspect ratio MEAs without altering the electrical insulation between probes.

Keywords: CVD diamond; Nucleation; BEN; Microelectrode arrays

1. Introduction

In the past few years, novel systems for neuroelectronic interfacing have been developed, and several are widely commercialised such as e.g. Cochlear implants. They play an important role for several medical palliative treatments where electrical stimulation is required, such as for the Parkinson diseases [1]. More prospective projects are now aiming at the possibility to alleviate disabilities with the possibility to provide disabled people with a computer driven motricity [2], or also to provide shape vision to blind people [3,4]. In a more fundamental aspect, microelectrode arrays (MEA) allow to study neural communication at a meso scale, and for example between Magnetic Resonance Imaging (MRI) or Electroencephalography (EEG) and single cell patch clamps. Better understandings on the architecture of neural networks are developing and will help the design of future in-vivo prosthesis.

The present work is focussing on the recent development of novel microelectrode array prototypes combining a silicon array of high density and shape ratio (up to 1024 tips of 80 μm in height, separated by 50 μm spacing) with complete software interface and embedding signal treatment [5]. Such devices are now industrially developed, and provide the ability to record neural activity e.g. from spinal cords deposited on the high aspect ratio electrodes.

However, when used for the stimulation of neurons, the voltage bursts used often have destructive effects on the quality of the electrodes, especially after intensive use. Our work here therefore focuses on the improvement of the electrode stability, by replacing the noble metal (eg: platinum, gold…) termination of the electrodes that seemed to be damaged after several stimulations, and we propose to use the same structure with a conductive Boron doped NanoCrystaline diamond (BNCD) layer on top of the high aspect ratio electrodes. The main objectives of the current project are to realise MEA that will allow the study of the formation of the neural network itself [6], in close collaboration with the CNIC laboratory [7] on embryonic mouse spinal cords in the prenatal week. In this particular case it is necessary to reach a cell population which is located from 10 to 80 μm from the uppermost layer of the preparation. In order to avoid damage to the preparation, tips

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with 80 μm in height are required, that also exhibit a high spatial density since several neural signals are to be recorded at the same time. Finally, since stimulation with electrical action potentials is often detrimental to live cells, we prefer to exploit the electrochemical properties of the diamond interface, that would enable to drive electrons to the neurons directly for a more biomimetic and less aggressive approach to the neurons. This therefore also implies that the material exhibits remarkable electrode properties, and only diamond can combine these features. The BNCD electrode material grown in our laboratory has been shown to exhibit remarkable electrochemical properties, with Pt reactivity and long term stability [6]. It constitutes the ideal material for the current application.

Other studies have also reported the possibility to use diamond electrodes fabricated from single wires [8–10] or with very sharp form for AFM applications [11]. Arrays of 3D electrodes have also been used as Field Effect Array [12] and 2D dimensions arrays have been used for electrochemical detection [13–16]. However, none of those systems propose the combination of high density, high aspect ratio and electrochemical stability and reactivity for neuronal signal monitoring and stimulating. The system we propose here is based on a formerly existing design [5], i.e. a novel silicon microelectrode array, fabricated from DRIE on silicon that is coated with electrode quality BNCD material, with a particularly high aspect ratio. This enables the possibility to directly mechanically insert the electrodes within the tissue cells for the measurement of their action potentials. Their shapes are those of exponentially conic tips of 80 μm in height for a base diameter of 80 μm. Using the 3D etched silicon as a support we avoid the time consuming difficulties associated with diamond etching that would result from a whole diamond tip system, [17,18] and further there is no need for full substrate dissolution as required with AFM high ratio diamond tips [19].

Challenges however are to preserve the silicon high aspect ratio tip shapes without damaging the needles nor smoothing their surface under a thick diamond coating layer. The layer has thus to be as thin as possible in order to fit the shape of the silicon needles. This implies the possibility to nucleate diamond on 3D shaped structures, which constitute the most challenging part of this work [20]. In fact, common micrometer powder scratching using ultrasonic excitation would result in severe damage to the silicon needle tips and the nucleation densities reached are generally not sufficient. Nanoseeding using ultradispersed diamond powder is seen as a very promising novel development, but spreading uniformly seeds on a 3D surface appears challenging, and further the adhesion of the obtained film, here crucial, remains uncertain to-date. The approach we therefore have preferred for the current realisation is based on Bias Enhanced Nucleation (BEN). This allows realising very dense nucleation with little damage to the needles and a good adhesion. At the same time, the BEN technique allows to create patterns from selective nucleation [21] thus enabling a bottom-up approach for the MEA device fabrication. First results on patterning with BEN have indeed already been reported, using e.g. spontaneous nucleation on Pt with a high selectivity compared to that on silicon dioxide [22].

2. Experimental details

In the current work the study was performed on silicon MEAs as fabricated in a clean environment and from the use of Deep Reactive Ion Etching (DRIE) [5]. This technique is used to fabricate a silicon vertical side wall by alternating successive steps of anisotropic and isotropic etching. It enables the fabrication of denser matrixes than using standard RIE or liquid phase etching [23]. On the 3D arrays a 400 nm thin thermal silicon oxide was overgrown in order to avoid electrical losses into the substrate. On top of the silicon oxide a standard Ti/Pt pattern was prepared for electrical connection of the MEAs.

High rate nucleation prior to BNCD growth was performed using a Microwave Plasma enhanced CVD (MPCVD) reactor enabling Bias Enhanced Nucleation (BEN) pretreatment on 2 in. diameter substrates. The MPCVD deposition parameters were previously optimised for high nucleation density onto (111)-Si [20], and here adapted in order to preserve the structure of the needles. Parameters were tuned with a pressure of 30 mBar, a CH_4/H_2 ratio of 10/90, and an optimised bias voltage of −307 V. The microwave power was adjusted from 0.4 to 1.1 kW with respect to the sample dimension and in order to reach a temperature of 760 °C as measured using a pyrometer. After nucleation, the growth of the BNCD layer was performed using a separate experimental setup, and using conventional nanocrystalline diamond layer growth conditions [24,25]. All samples were probed using a High Resolution Scanning Electron Microscope HR-SEM (HITACHI S-4500 FEG-SEM) that enables the quantification of the crystal densities.

3. Aspect ratio selectivity

The study concentrated first on the nucleation on 3D macroscale objects (objects are in the order of several hundreds of micrometer), in order to study the influence of the shape of the substrates and on the nucleation density during the BEN process. This was performed on stairs, fabricated on 300 μm thick silicon, with structures exhibiting heights varying from 0 to 7200 μm (the BEN step used parameters are: 30mBar of pressure during 5min, at 10% of CH_4 in H_2, with a MW power of 1.1 kW. The BEN current is of 50 mA. After this short nucleation step, a growth step follows during 10min at 30mBar using 10% CH_4 in H_2, at 775–802°). Then this was adapted at the microscale to high aspect ratio tips, with silicon structures of 16 μm in height and 25 μm in diameter. This study was conducted on 10×10 mm^2 samples grown at the same time

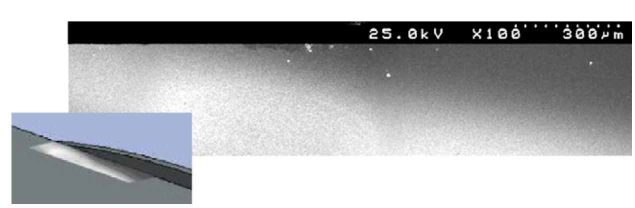

Fig. 1. SEM observation of the effect of the height of a tip on the nucleation at the tip pedestal.