Fabrication and characterization of 3D pyrolytic carbon microelectrodes for electrochemistry

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Fabrication and characterization of 3D pyrolytic carbon microelectrodes for electrochemistry

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This work presents the fabrication and initial characterization of suspended three-dimensional (3D) pyrolytic carbon microelectrodes for electrochemical applications. Microelectrode chips with a three electrode configuration were fabricated with an optimized multiple step UV photolithography followed by pyrolysis and characterized with cyclic voltammetry (CV).

Carbon materials offer several attractive properties such as wide electrochemical potential window, biocompatibility, chemical stability and ease of functionalization. These features makes carbon an ideal material for microelectrodes used as biosensor, scaffolds or energy storage devices [1]. The most common carbon microfabrication techniques, such as screen printing, produce planar two-dimensional (2D) microelectrodes. However, device sensitivity and biological signals from 2D pattern are limited due to the 2D nature of the electrode [2]. Hence, several 3D microfabrication techniques have been explored amongst which the carbon MEMS (C-MEMS) approach is the most promising one for the fabrication of conductive 3D microstructures. This process enables fabrication of 2D and 3D electrodes with the possibility to tailor designs and thereby sensitivities for electrochemical sensing applications. Due to this, pyrolytic carbon microelectrodes are becoming increasingly attractive for numerous applications, such as in novel sensors and scaffolds for cell monitoring [3-4]. Nevertheless, fabrication of electrically conductive 3D carbon microelectrodes with structural dimensions that are comparable to the size of biological cells still remains challenging.

In this work, a 3D carbon microelectrode is fabricated as the working electrode of an electrochemical cell using multiple UV photolithography followed by pyrolysis (figure 1). A 17µm thick SU-8 2035 is spin coated and exposed (E – 250 mJ cm^{-2}) to define the base of the three electrode system (figure 1.a). A 91µm thick SU-8 2075 is spin coated, soft baked (SB- 50°C for 6h) followed by two UV exposures, E_1- 210 mJ cm^{-2} (figure 1.b) and E_2- 28 mJ cm^{-2} (figure 1.c) which define pillars and the suspended layer respectively. A long low temperature post exposure bake (PEB -50°C for 5h) is used for the stress free crosslinking. Non cross-linked SU-8 is developed in propylene glycol monomethyl ether acetate (PGMEA) for 30mins (figure 1.d). The structured SU-8 template is pyrolysed at 900˚C in an N_2 environment to obtain 3D pyrolytic carbon microelectrodes. A pseudo Au reference and contacts are deposited by e-beam evaporation through a shadow mask (figure 1.e). The contact leads are passivated with SU-8 (figure 2). After pyrolysis the height of the 3D microelectrodes was 41µm. The smallest feature size on the suspended electrodes was 5µm which is comparable to the dimensions of biological cells.

Figure 3.a shows the experimental setup with magnetic clamping for the characterisation of 3D pyrolytic carbon microelectrodes. The fabricated three electrode electrochemical cell is characterized with CV using the standard 10mM potassium ferri-ferrocyanide redox probe. Figure 3.b shows higher peak current (2 folds higher) for the 3D microelectrodes compared to the 2D ones. Figure 3.c shows the response and stability of 3D electrodes with different scan rates.

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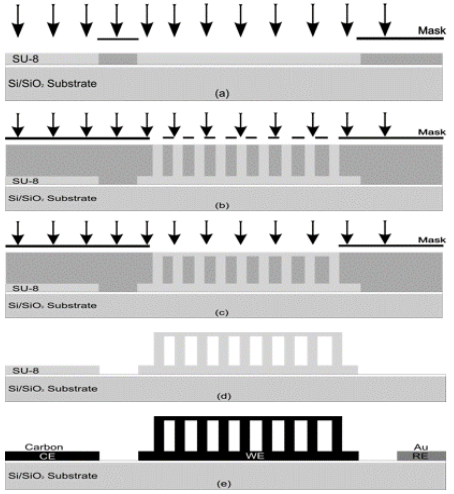

Figure 1. Schematic of process flow (a) SU-8 (2005) is spin coated, baked and exposed (E) (b) SU-8 (2075) is spin coated, SB and exposed (E_1) (c) Partial exposure (E_2) and PEB (d) Development in PGMEA (e) Pyrolysis

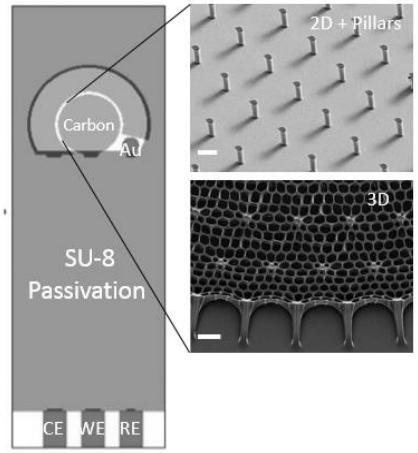

Figure 2. Top view of the three electrode electrochemical chip with SU-8 passivation and different working electrodes designs (Scale bar – 20µm)

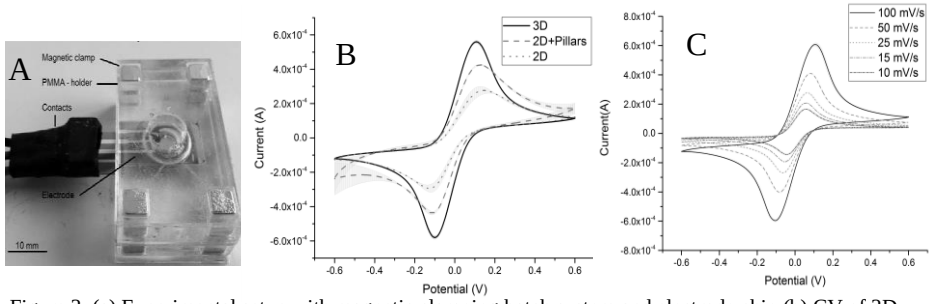

Figure 3. (a) Experimental setup with magnetic clamping batch system and electrode chip (b) CV of 3D, 2D+pillars and 2D with 10mM potassium ferri-ferrocyanide redox probe (c) 3D microelectrode CV with different scan rates