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Dense high aspect ratio pillar arrays for carbon MEMS electrodes

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This work presents the fabrication and characterization of 3 dimensional (3D) electrodes from pyrolysed carbon with spacing and diameter approaching the resolution limit of standard UV photolithography.

When fabricating miniaturised and portable sensors, maximization of the electrode area on miniaturised footprint is critical and can be facilitated by 3D electrodes. Pyrolysed carbon electrodes show good biocompatibility, chemical inertness towards wide variety of solvents/electrolytes, outstanding mechanical properties, and wide electrochemical potential window. All these features make them the ideal material choice for biosensors. Additionally pyrolysed electrodes can be fabricated with the carbon MEMS technique [1], a very simple high yield and cost-effective process.

Although pyrolysed pillar electrodes with aspect ratio higher than 10 have been fabricated before, their diameter and spacing was bigger than 10 µm [2]. This paper provides the first report of carbon pillar arrays with a diameter of 1.4 µm, spacing of 5 µm and aspect ratio of about 8.

A two-step photolithography process was used to pattern the SU-8, a negative photoresist and carbon precursor in this work. SU-8 2005 (precursor of the connecting underlayer) and SU-8 2075 (pillars precursor) were used for fabrication of the pillar arrays, which were then pyrolysed at 900 °C. The pyrolysed carbon pillars were characterized by a variety of techniques providing a complete summary of their electro-mechanical properties. Scanning electron microscopy imaging was utilised to determine the shrinkage of the pillars (fig. 1). The shrinkage was found to be about 50% for both height and width of the pillars. Not patterned and pyrolysed films of SU-8 2005 and SU-8 2075 were analysed by Raman spectroscopy and XPS to investigate their bulk and surface chemistry. The Raman spectra contain the so-called D and G lines (fig.2), which can be attributed to pyrolysed carbon. Interestingly, the peak intensities were higher for SU-8 2005 than for SU-8 2075. This result might be related to the higher carbon content in pyrolysed SU-8 2005 (93%) than in pyrolysed SU-8 2075 (79%). For electrical characterisation, a microscopic four point probe was placed on top of the micropillars (fig. 3a) using a standard lock-in measurement technique [3]. The measurements showed a phase change of less than 0.2 degree indicating a good ohmic contact to the sample and pointing towards a good conducting surface. Furthermore, the difference of approximately 1 Ω between the measured resistance of the thin film layer (35 Ω) and the one through the micropillars (36 Ω) gave evidence of a negligible interface resistance pillar-underlayer (R_{bp}) (fig. 3b). Cyclic voltammetry demonstrated that the 3D carbon electrodes had very good electrochemical characteristics, such as ΔEp = 106 mV (fig. 4). Moreover, this work presents interconnected 3D pillar on interdigitated carbon electrodes allowing for easy interface with experimental set-up and yielding a surface area increase of about 70% compared to the same electrodes without pillars (fig. 5). Considering its outstanding features, this high aspect ratio carbon MEMS electrode will be excellent for electrochemical biosensing.

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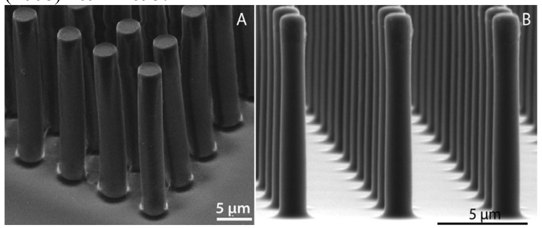

Figure 1. SEM image of high aspect ratio micropillars before (a, 45° tilted sample) and after pyrolysis (b).

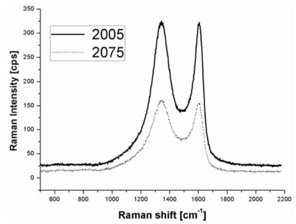

Figure 2. Raman spectra of flat pyrolysed SU-8 2075 (dotted line) and SU-8 2005 (full line).

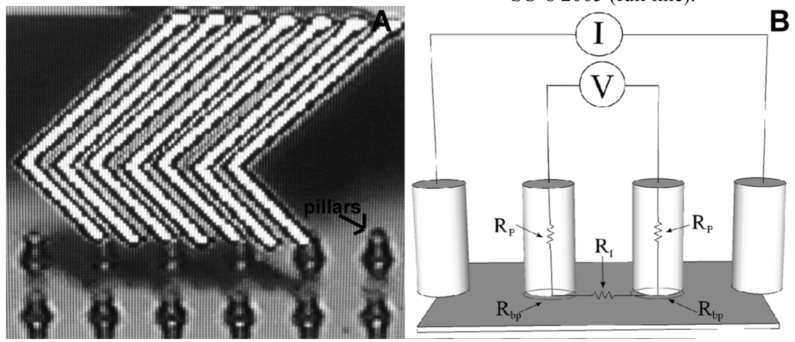

Figure 3. (a) Digital camera picture of microscopic four point probe during the measurement on the pillar tips. (b) Conceptual drawing of the four point probe measurement on top of the pillars. R_p = resistance in pillar; R_{bp} = contact resistance at the pillar-underlayer interface. R_i = resistance in sheet.

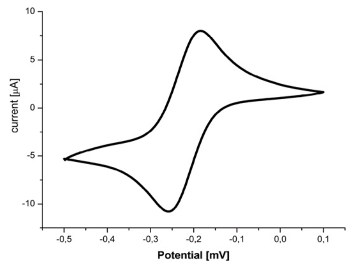

Figure 4. Cyclic voltammogram of 1 mM ruthenium hexamine chloride (II/III) in PBS (pH 7) on pyrolysed pillar electrodes (potentials vs. Ag/AgCl reference electrode and sweep rate: 50 mV s^{-1}).

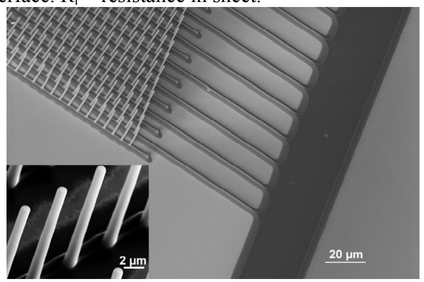

Figure 5. SEM image of pyrolysed 3D structures. Inset: High aspect ratio pillars standing on digits.