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Optimized SU-8 pyrolysis for fabrication of pyrolytic carbon microelectrodes

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This work focuses on the fabrication of two dimensional (2D) pyrolytic carbon microelectrodes obtained from a lithographic process using the negative epoxy photoresist SU-8. The pyrolysis process at high temperature (1100°C) in N₂ atmosphere has been optimized in order to decrease the resistivity of the resulting carbon material and improve the performance in cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS).

Pyrolytic carbon is obtained by heat treatment of organic polymers in inert atmosphere and has a microstructure which is similar to glassy carbon, composed of both graphitic and amorphous regions. The carbon microelectromechanical systems (C-MEMS) technique is a simple and high-yield process which typically consists in the heat treatment of a patterned photoresist at 900°C in inert atmosphere [1]. This process enables the reproducible fabrication of 2D and 3D pyrolytic carbon microelectrodes with tailored designs and sensitivities for specific applications such as heavy metal detection, biosensing or cell fate monitoring [2-4]. Changing the pyrolysis conditions modifies the graphitic content of the carbon material and leads to different material properties which determine the electrical and electrochemical behaviour of the final carbon microelectrode [2]. It has been reported that the electrical and electrochemical properties of pyrolytic carbon improve with increasing final pyrolysis temperature [1, 2]. However, to date no systematic studies at 1100°C have been performed. Different microelectrode chips with carbon working electrode (WE) and counter electrode (CE) were fabricated. SU-8 2035 was spin coated on Si wafers with 600 nm LPCVD Si oxide to obtain a thickness of 17µm. The wafers were then soft baked (50°C for 15 minutes), and subsequently the photoresist was exposed using a 365 nm wavelength in soft contact mode. After a post exposure bake, the development was performed in propylene glycol methyl ether acetate (PGMEA), followed by a rinse with isopropanol and drying in air. To complete the crosslinking of the structures, an additional flood exposure was performed and finally the wafers were hard baked for 15 hours at 90°C. The wafers were then pyrolyzed using a 1 step process at 1100 °C in N₂, obtaining a thickness of approximately 2µm after pyrolysis. Different dwell times at the maximum temperature (1 hour, 3 hours and 5 hours) and heating rates (10 °C/min, 30 °C/min and 50 °C/min) were chosen as parameters to perform a central composite design of experiments (DoE) and investigate their influence on the electrical and electrochemical properties of the carbon microelectrodes. Finally, 200 nm of gold were deposited by e-beam evaporation through a shadow mask to obtain a pseudo-reference electrode (RE) and gold contacts, and a 5µm thick film of SU-8 was used as passivation layer (Fig.1). The resistivity measurements, performed using a custom made 4-point measurement setup, show that the pyrolysis processes with longest dwell times of 5 hours, with heating rates of 10 °C/min and 50°C/min give the lowest resistivity values of 3.2±0.3 mΩ·cm (Fig.2). This value is much lower than the one reported for the pyrolysis at 900°C of 9.3±0.8 mΩ·cm. For CV and EIS, the ferri-ferrocyanide [Fe(CN)₆]⁴⁻ / [Fe(CN)₆]³⁻ redox couple was used, after treatment of the electrodes with oxygen plasma. Also in this case the process carried out for 5 hours with a heating rate of 50°C/min showed the best electrochemical behaviour, with the highest values for peak currents in cyclic voltammograms (Fig.3) and overall lowest resistance for EIS (Fig.4). The results of the experiments of the DoE analysis showed that the dwell time at the maximum temperature is the most influencing parameter on the electrical and electrochemical performance of the microelectrodes. The resistivity values reported here are to our best knowledge the lowest ones reported for C-MEMS processes.

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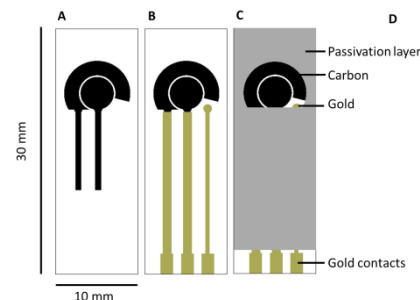


Figure 1. Overview of 2D carbon microelectrodes: (A) carbon layer, (B) gold contact pads, (C) passivation layer

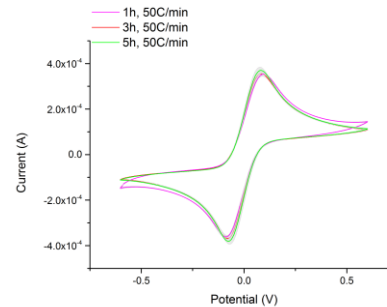


Figure 3. Voltammograms of pyrolytic carbon electrodes obtained with different dwell times at 1100°C and a heating rate of 50°C/min acquired in 10mM ferri-ferrocyanide.

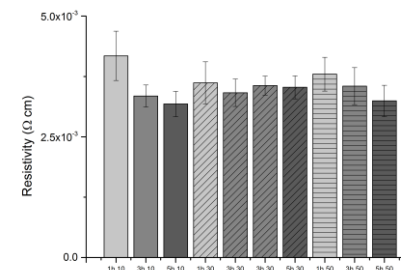


Figure 2. Resistivity measurements for pyrolytic carbon films obtained at 1100°C using different dwell times in N₂ (1 hour, 3 hours, 5 hours) and different heating rates (10°C/min, 30°C/min, 50°C/min).

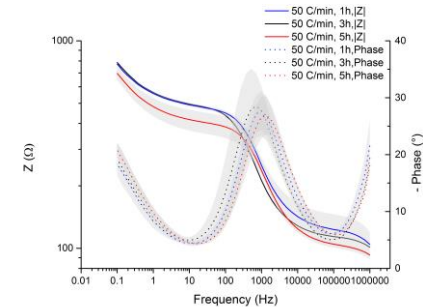


Figure 4. Impedance spectra of pyrolytic carbon electrodes obtained with different dwell times at 1100°C and a heating rate of 50°C/min acquired in 10mM ferri-ferrocyanide.