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Realization and characterization of carbonic layers on 4H-SiC for electrochemical detections

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For electrochemical use, carbon materials exhibit better behaviors than metallic gold or platinum electrodes, in terms of detection limit, potential range and sensibility [1]. For these reasons, research has focused its interest in these materials. One advantage of these materials is that they can be prepared at the surface of different substrates. In this work, we aim at preparing diamond, graphene and pyrolyzed photoresist films at the surface of silicon carbide (4H-SiC), which is considered to be a biocompatible material. These layers are to be more attractive than glassy carbon, one of the cheapest and broadly used carbon material in electrochemistry. Our layers present the advantage to be able to be patterned, to resist high temperature treatments and also give the possibility to include all them in SiC technological steps for the preparation of more elaborate structures.

Discovered in 1984, pyrolyzed photoresist films (PPF) were electronically studied by Lyons [2]. 14 years later, this material has been considered to be used as a working electrode by Kim et al. [3], showing that these layers kept their properties through time, giving better reproductibility than glassy carbon. Nowadays, this material is actively used in research for electrochemistry, used as a biocompatible working electrode [4], or as a very sensitive and selective electrode for heavy metals detections [5]. Del Campo et al. also showed the possibility to include PPF in CMOS technological steps [6]. This very flat material can be obtained by the pyrolysis of photoresist layers, by a plethora of conditions (temperature, duration of annealing, pressure, presence of different gas). In SiC technology PPF is used as a protective cap during high temperature post-implantation annealing.

Graphene has shoved the world of electronics by its marvelous theoretical properties. In electrochemistry, its exceptional behavior in sensibility and limit of detection [7] has attracted the attention of researchers. In this study we focused on graphene grown on SiC by silicon sublimation at its surface. This material has promising characteristics, in particular, a good signal on noise ratio.

Diamond is well known in electrochemistry for its very large potential range, but also a far higher signal to noise ratio than for glassy carbon [8]. Diamond in electrochemistry needs to be conductive enough and in this sense it is heavily doped with boron. The common name used is Boron doped diamond (BDD). These electrodes show a reproductive behavior through time and show no damage of the layer with long term use.

In our work, pyrolyzed photoresist films were obtained by the annealing of AZ5214E photoresist at temperatures above 1000 °C, which is higher than what can be generally found in literature. Graphene has been synthesized by sublimation of Si in SiC at 1550 °C under Ar atmosphere. Details upon this method can be found elsewhere [9]. Boron Dope Diamond has been deposited by Dr Tallaire at LSPM by CVD growth. For this study, the layers' conductivities have been evaluated through I-V measurements, and the kinetics, potential range, limit of detection, sensitivity, signal to noise ratio, were observed through electrochemical characterizations.

In the final paper will be presented a detailed description of the realization of the PPF layers. Results obtained with these layers will be compared to each other and confronted to the literature. Performances of these electrodes will be evaluated. Furthermore, an application of these layers as

transducers in the detection of a biological molecule (Acetylthiocholine) will be shown. The results obtained give real confidence to our team that these layers show a real interest in the field in electrochemistry.

Figure 1 and Table 1 present the electroactivity potential domains of our electrodes in KCl 0.1 M and in Phosphate Buffered Saline (PBS) 10X. For the PPF, the electroactivity range increases with the annealing temperature. This is in agreement with the increasing of the electrical conductivity of the PPF layers with the annealing temperature, as we obtained by I-V measurements. A disorder evolution is also observed in these PPF layers by structural Raman analyses. The optimal annealing temperature is 1650°C which is close with the temperature of our graphene growth on SiC. The PPF and graphene layers could be generated on SiC during the same technological step.

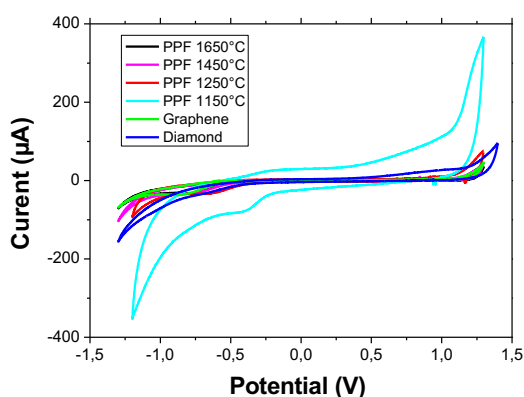


Fig.1 Cyclic voltammety corresponding to the electroactivity of our electrodes in PBS 10X

Electrode	Potential window in KCl 0.1 M	Potential window in PBS 10X
PPF annealed 1150°C	-0.5 – 1.1 V	-1.08 – 0.95 V
PPF annealed 1250°C	-1.2 - 1.35 V	-1.2 – 1.15 V
PPF annealed 1450°C	-1.4 - 1.37 V	-1.25 - 1.2 V
PPF annealed 1650°C	-1.6 – 1.39 V	-1.4 – 1.25 V
Graphene	-1.4 - 1.39 V	-1.4 - 1.25 V
Diamond	-1 – 1.37 V	-1.1 – 1.25 V

Table 1. The electroactivity potential range values for our electrodes in KCl 0.1M and PBS 10X

Figures 2 presents the Michaelis-Menten and Lineweaver Burk calibration curves from our electrodes of 1cm², used as biosensors for detection of acetylthiocholine in PBS 10X. The electrodes have been functionalized by adsorption using a solution of chitosan 0.5 % in acetic acid adjusted to pH 5 with NaOH.

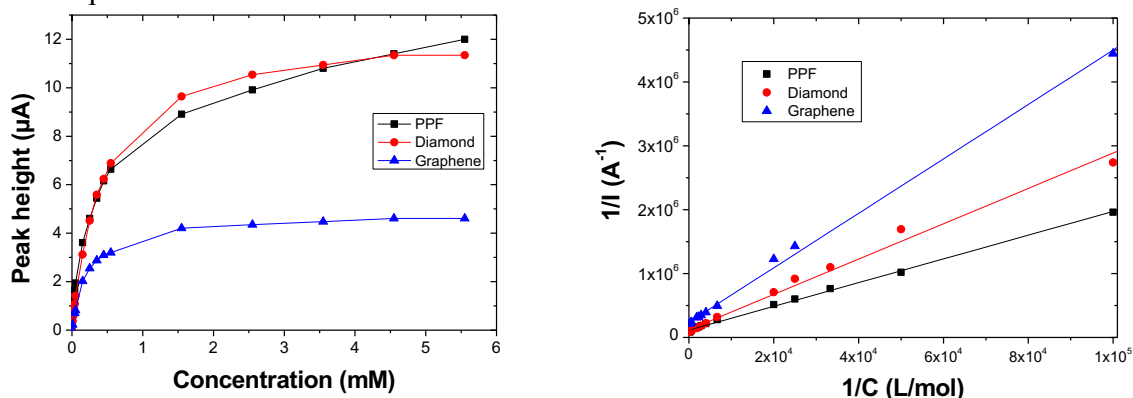


Fig 2 Michaelis-Menten (left) and Lineweaver Burk (right) calibration curves extracted from chronoamperometry for detection of acetylthiocholine

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