

Examination of the electrochemical reactivity of screen printed carbon electrode treated by radio-frequency argon plasma

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Abstract

The surface of screen printed carbon electrode (SPCE) with partially blocked surface was treated by argon plasma in order to improve their electrochemical performances. The argon plasma was generated by a radio-frequency electrical discharge at low pressure. Study of the electrode surface by scanning electronic microscopy (SEM) has revealed a significant change of the morphology of the SPCE surface after plasma pre-treatment. The electrochemical reactivity of the SPCEs was characterized using cyclic voltammetry. A drastic enhancement of the SPCEs electrochemical reactivity was highlighted after plasma pre-treatment. The effect of biasing the SPCE surface during the plasma treatment has been investigated and showed that depending on the nature of plasma treatment, the same electrode could show a radial or planar diffusion.

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1. Introduction

Carbon is one of the most commonly used electrode materials as transducer in electrochemical sensors and bio-sensors due to its inertness, high conductivity good electrochemical activity and its relatively low cost. Screen printed electrodes based on carbon inks plays key roles in a wide variety of analytical device [1–4]. On the other hand, the SPCE surface is often considered as a random assembly of graphite particles behaving as a microelectrode array [5,6]. Such behaviour supposes that the almost carbon particles must be electronically connected but separated by insulating polymer binder. So in such configuration, higher current densities could be achieved with simultaneous advantages to obtain electrochemical behaviours of micro-electrodes. However the electrochemical performances of a

wide range of SPCEs were found very dependent on the inks formulation [7]. For instance, some polymer binders may also act as an insulating layer which could affect both the interfacial electron transfer and diffusion process in the vicinity of the electrode surface. Thus, it has been reported that electrochemical pre-treatment with pre-anodization for short period could increase the surface functionalities and improve the electrochemical activity of the electrode. Cui et al. [6] compared the behaviour of different SPCEs manufactured with various commercial carbon inks and showed that the pre-treatment in Na_2CO_3 solution at 1.2 V *vs.* SCE allows to remove the polymer binder from the electrode surface. Recently, Wei et al. reported a new pre-treatment procedure of screen printed carbon electrode in basic solution of NaOH, thus the authors noticed a remarkable enhancement of the rate of electron transfer of the pre-treated electrodes [8]. Albareda-Sirvent et al. [9] examined two ways for the SPCE activation, the first one consisted in the application of a constant potential of 1.6 V *vs.* SCE for 12 min, and the second one consisted

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of 40 scans in cyclic voltammetry mode between -0.25 and 1.6 V *vs.* SCE, both procedures gave a similar improvement of the electrochemical reactivity with a density enhancement of the electro active sites at the SPCE surface. Physical treatment as excimer laser was also used to remove the polymer binder from the carbon particle surface improving thus its electrochemical reactivity of SPCEs [10].

In this contribution, we present one way to activate partially blocked SPCE surfaces by a physical treatment based on plasma generated by a radio-frequency (RF) electrical argon discharge at low pressure. This paper deals for the first time with the effect of argon plasma on the electrochemical reactivity of SPCE. Our results show that depending on the nature of plasma treatment, the same electrode could show a radial or planar diffusion. Cyclic voltammetry was used to examine the electrochemical reactivity and to probe how the diffusion regime depends on both the experimental time scale and the experimental conditions of plasma treatment. Besides, the enhancement of the electrochemical reactivity of the SPCE surface, the effect of plasma treatment on the SPCE surface morphology was also considered.

2. Experimental

2.1. Screen printed carbon electrode (SPCE) preparation

Carbon ink was obtained by mixing Polymer binder: epoxy resin (Code No. R2010926D2) with a given amount of carbon (Code No. P70313R2). Both polymer binder and carbon were purchased from Gwent-Electronic Materials Ltd. (Pontypool, UK). Polymer binder and carbon were mixed in the ratio 60/40. The resulting mixture was mixed manually with a given amount of carbon (50/50). The viscosity of the ink was adjusted by adding 8–10% of diethylene-glycol-monomethyl ether. To obtain a homogeneous ink, the resulting carbon mixture was mixed using Roll-mill machine. And finally, the screen printed working electrodes were obtained by using a DEK Albany model screen printer machine and stainless screens with a 200 mesh and thickness of 20 μm . The carbon was screen printed in one layer on alumina substrate of 2.5 cm long and 0.2 cm wide. The composite material was then cured at 150 $^{\circ}\text{C}$ for 1 h and stored at room temperature. After plasma treatment, the working surface area was delimited by applying an insulating tape. The reproducibility of the preparation of SPCE was estimated by comparing the voltammetric response intensity of a series of electrodes ($n = 24$) from three bathes, the RSD value was found of 5–6% when the cyclic voltammetry was performed in a 2 mM ferricyanide aqueous KCl (0.1 M) solution.

2.2. SPCE pre-treatment and surface characterization

The plasma treatment of the SPCEs was performed at low argon pressure in an ionized sputtering system equipped with one turn stainless steel coil. This experimen-

tal system was described elsewhere [11]. The coil, with 8 cm diameter and 0.6 cm cross section was positioned at 4 cm from the SPCE surface. The role of the coil connected to a RF generator (at 13.56 MHz) through a matchbox was to generate plasma with a high density of Ar^+ ions. Then, electrons to which most of the RF electrical power is transferred, gain enough energy to ionize argon atoms. For surface treatment, Ar^+ ions are accelerated from the plasma towards the surface through the plasma shield. Before plasma treatment, the chamber was pumped down to a pressure value lower than 10^{-4} Pa. This high vacuum was necessary to avoid any oxygen, nitrogen or water contamination during the plasma treatment. For creation of the plasma, argon gas was injected in the chamber and RF power at 300 W was applied to the coil. The argon pressure was kept constant at 6.4 Pa during the plasma treatment. The plasma treatment time was 1 h. Luminous and uniform plasma was then created close to the SPCE surface.

Two plasma treatment conditions were applied to the SPCEs. The first plasma treatment, T1, was performed with SPCE at floating potential. i.e. with the surface at a potential slightly lower than the plasma potential which means a moderate bombardment of the SPCE surface by argon ions. For the second plasma treatment, T2, the SPCE was grounded, i.e. with a potential variation between the plasma potential and the SPCE surface about 320 V. This high potential variation allows accelerating the Ar^+ ions through the plasma shield and creating a high flux of energetic Ar^+ ions towards the SPCE surface and as consequence a more effective ions bombardment at the electrode surface.

2.3. The electrochemical measurements

Cyclic voltammetry (CV) was performed at room temperature using a controlled PowerLab 4SP potentiostat in a three-electrode configuration, including a saturated calomel electrode (SCE) as reference electrode, a platinum wire as counter electrode and SPCEs as working electrode. The CV was performed in aqueous solutions with 1 mol L^{-1} of potassium chloride (KCl) as supporting electrolyte. The ferri/ferricyanide redox couple $[\text{Fe}(\text{CN})_6]^{4-}/[\text{Fe}(\text{CN})_6]^{3-}$ was used to evaluate the effect of the plasma treatment on the electrochemical behaviour of the SPCEs. When necessary, the solution was de-aerated with argon, during the experiment.

3. Results and discussion

As previously described in experimental section two different plasma treatments denoted respectively T1 and T2 were considered in this study. T1 corresponds to the moderated sputtering of the surface by Ar^+ ions, and T2 treatment corresponds to a higher Ar^+ ion bombardment. Their effects were examined on both the morphology of the SPCE surface and on its electrochemical reactivity.

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