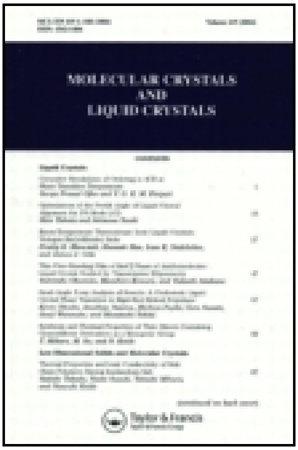

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Electrochemical Electrode Based on Carbon Foil Used as Substrate for Conducting Polymer Deposition

Gabriela Hristea [a] , Simona Gheorghe [a] & Marin Gheorghe [a]

[a] Research and Development Institute for Electrical Engineering, Bucharest, 74204, Romania

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Electrochemical Electrode Based on Carbon Foil Used as Substrate for Conducting Polymer Deposition

GABRIELA HRISTEA, SIMONA GHEORGHE and
MARIN GHEORGHE

Research and Development Institute for Electrical Engineering, Bucharest, 74204, Romania

In this paper a carbon foil (based on intercalated graphite compounds) manufactured in our laboratory was used as electrode material for polycarbazole deposition, due to the excellent properties of this material. Anodic polymerization of carbazole on the carbon foil was performed. Uniform layers of polycarbazole on the carbon foil were obtained. Atomic force microscopy, X-ray diffraction, and electrochemical measurements were achieved on this composite material.

Keywords: Polycarbazole; Conducting polymer; Carbon foil

INTRODUCTION

The formation of salts of graphite or graphite oxides from graphite by strong acids attack is known since the early days of graphite intercalation chemistry. The composition and the properties of GIC depend on the nature of the pristine graphite, its particle size and preparation conditions. Thus, in spite of much effort, the physicochemical properties of GIC are still investigated[1].

Many electrochemical devices (batteries, chemical sensors and biosensors etc.[2,3]) require a carbon substrate as component part covered with a conducting polymer layer. The carbon foil properties are eloquent for these applications, and some of these properties are: chemical inertia, electrochemical stability and electrical conductivity.

The conducting polymers are sensitive to a number of external influences as temperature, pressure, electric field, pH medium etc. In this work the efforts were concentrating to design an electrochemical electrode through chemical surface modification for a carbon substrate and used polycarbazole as conducting polymer.

Experimental

The preparation of graphite oxides was carried out by immersion of a flakey natural graphite (400 – 500 μm) in a mixture of concentrated sulfuric and nitric acids. $FeCl_3$ was then gradually added to the solution with stirring and cooling. After adding $FeCl_3$, the solution was allowed to stand 5 days at room temperature. The resulted metallized –gray slurry is suction – filtered, dried to constant weight in vacuum at 80^0C. The varied reaction parameter was the reagents ratio. Finally, because of the different reagents ratio, there are resulted two kinds of compounds (type 1 and type 2), which are basically graphite oxides.

A sol prepared from 1-2g graphite oxide (G.O.) (obtained by the above described method) in 100 ml of water was allowed to evaporate. The graphite oxide was separated as a foil on the bottom of the vessel. Very careful, slow heating with gradually rising temperature caused a sheet to give off CO, CO_2 and H_2O without deflagration until at 1000°C, a graphite like film was obtained. This still contained a few percent of O and H. The graphite like film was compacted at low pressure. In that way a flexible carbon foil was obtained. This procedure was applied for both obtained compounds.

By using that type of carbon foil as substrate (made from type 1 or type 2 compounds) uniform layers of conducting polymer was obtained by cyclic voltammetry. The conducting polymer was polycarbazole. An electrochemical unit Zahner IM6D, was used for this application .The same conducting polymer was electrochemically deposited on platinum substrate for comparison. The electrolyte composition was 1 N sulfuric acid and 0,1 M carbazole in acetone. The work electrode was carbon foil and Pt, counterelectrode was Pt and Ag/AgCl sat. used as reference electrode (the potential was scanned between 0 and 2 V vs. Ag/AgCl sat. reference electrode - as shown in Fig. 4).

The graphite oxides were analyzed by X – ray diffraction, and optical microscopy. Atomic force microscopy, optical microscopy and electrochemical measurements were achieved on the carbon foil with polycarbazole deposition.

Results and discussions

Without using the low pressure compacting procedure, the size and thickness of the carbon foil was the same as that of the G.O. (graphite oxides) and can therefore be controlled by the amount and the concentration of the G.O. sol and by the size of the vaporization vessel. Films with surfaces as large as 50 cm^2 can be obtained in this manner[14].

The carbon foils manufactured in our lab exhibit excellent properties as electrode material, such as: low resistivity (< 1 Ω cm); a great interval of electrochemical stability (in the range +1200 mV to –1200 mV); a possibility to induce chemical modifications on its surface; an inexpensive process.

Micrographs analysis of the graphite compounds obtained, resulted two different structures: one is based on a band texture - type 1 (as shown in Fig. 1); the other is formed by crystals with high reflectance, without band texture (as shown in Fig. 2). The suitable structure must be closely connected technological procedure.

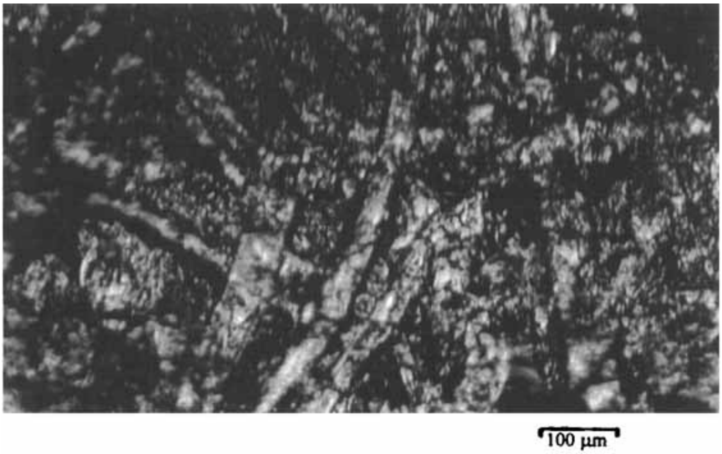

FIGURE 1 Optical micrograph of the carbon foil (based on graphite compound with band texture –type 1).
See Color Plate I at the back of this issue.

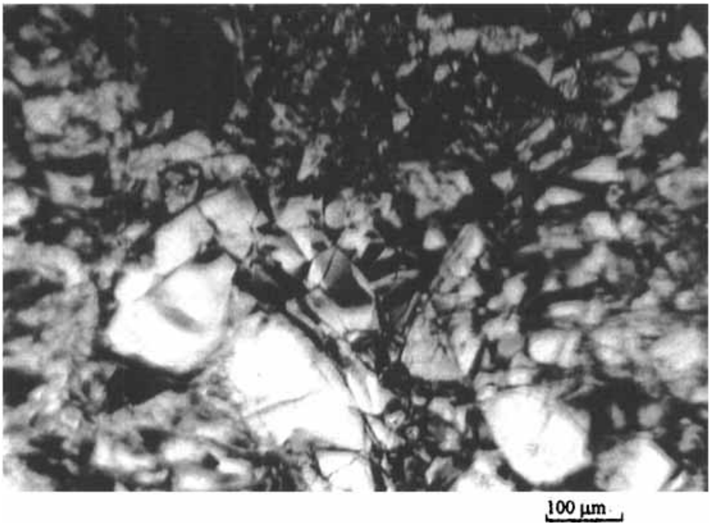

FIGURE 2 Optical micrograph of the carbon foil (based on graphite compound with high crystallinity –type 2).
See Color Plate II at the back of this issue.

From the X ray diffraction patterns for type 1 and type 2 graphite compounds results: the graphitical structure for type 2 compound, was almost preserved (the (002) and (004) diffraction lines are strongly represented).

It can be also remarked, the graphite oxide characteristic peak, which appears at around 10^0 2θ (by Cu Kα) (i.e., 7.619 – 8.1Å). Another diffraction peak which appears at around 24^0 2θ (by Cu Kα) (i.e., 3.727Å) seems to belong to an intercalated graphite $FeCl_3$ compound. The best experimental results were obtained for the carbon foil based on the type 2 compound, so, in this paper is shown the X-ray diffraction pattern only for this compound (see fig. 3).

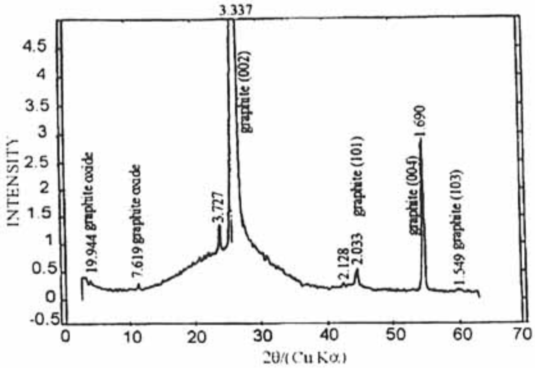

FIGURE 3 X ray diffraction pattern of graphite compound with high crystallinity (type 2).

Because of the target, the obtaining of a new material, suitable for electrochemical applications, (in order for developing of an electrode with a great interval of electrochemical stability, chemical inertia, conductive and with a minimum amount of raw materials), this composite material was made from a conducting polymer, electrochemically deposited on a carbon substrate (in this case the carbon foil).

Up mentioned conditions were achieved and the final electrode revealed the following characteristics: an area of about 1 cm^2: thickness – 0.1 mm; density – 0.8 g/cm^2: low resistivity (< 1 ohm cm): chemical inertia and electrochemical stability (in the range +1200 mV to -1200 mV). For evaluation of the material performances, a comparison with other electrode, - the same conducting polymer deposit on a platinum substrate was done. Among electrochemical measurements, cyclic voltammograms were performed. Further analysis regarding the structure and the material properties, should be continued.

The cyclic voltammetry during deposition of polycarbazole on carbon foil type 2, is shown in Fig. 4.

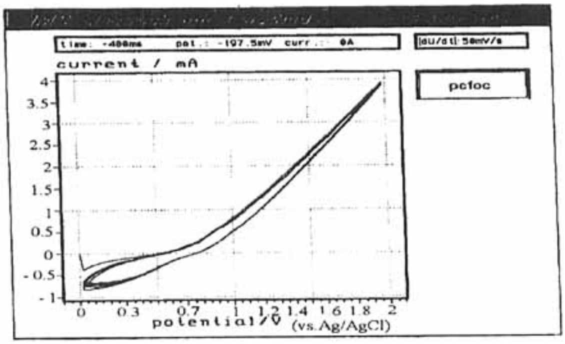

FIGURE 4 Cyclic voltammetry during electrochemical deposition of polycarbazole on carbon foil based on type 2 compound.

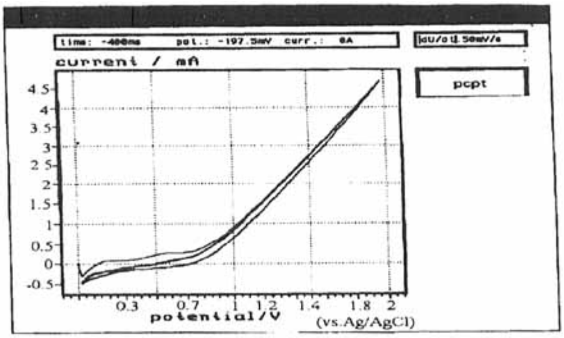

FIGURE 5 Electrochemical deposition of polycarbazole on platinum. Cyclic voltammogram during synthesis.

The electrochemical behavior of these two materials (polycarbazole deposit on carbon foil and polycarbazole deposit on platinum) is similar. The platinum based electrode can be replaced with carbon foil/ polycarbazole.

The carbon foil based on type 1 compound, can also be used as substrate for polymer deposition, but the conductive properties are lower than those of carbon foil based on type 2 compound.

Also we remark a green color of the final product, which means the acid character of carbon foil (the polycarbazole can be used in proper conditions as pH-sensor). The micrograph of the polycarbazole deposit on the carbon foil can be seen in Fig. 6.

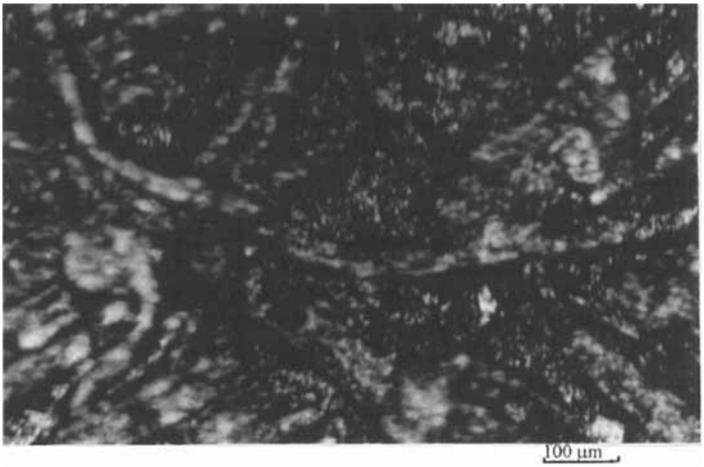

FIGURE 6 Optical micrograph of carbon foil (type 2) after polycarbazole deposition.
See Color Plate III at the back of this issue.

From atomic force microscopy (AFM) image of polycarbazole on carbon foil (type 2), we can conclude that the polymer deposition is uniform and also a dendritic configuration of this can be observed.

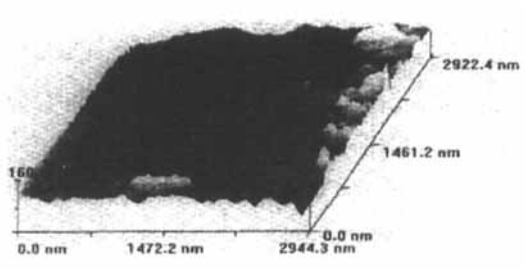

FIGURE 7 AFM image of polycarbazole deposit on carbon foil.

Conclusions

Through the similar electrochemical behavior of the polycarbazole deposition on the carbon foil electrode same as on the platinum we can conclude that carbon foil without band texture but with high cristallinity can be used as a substrate for polycarbazole deposition by anodic oxidation of carbazole.

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