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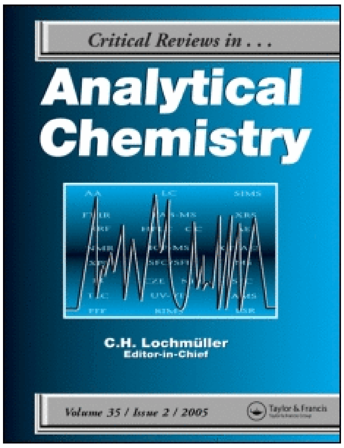

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Polycrystalline Diamond-Based Electrochemical Sensors and Their Applications in Inorganic and Organic Analysis

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ABSTRACT: Boron-doped polycrystalline diamond electrodes have been used extensively in the field of electroanalysis. The development of knowledge about the mechanism of diamond film deposition, boron doping, and charge transfer reactions rendered the quality of analytical information. New aspects regarding the design of diamond-based electrodes, and mechanisms of redox reactions at their surface are briefly discussed. Recent applications of the diamond-based sensor in inorganic and organic analysis are presented.

Key Words: diamond, polycrystalline, boron, electrochemical sensors.

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I. INTRODUCTION

Almost all electrochemical studies to date are performed with diamond films prepared by chemical vapor deposition (CVD). Synthetic films often posses a polycrystalline and textured microstructure, with a small volume fraction of non-diamond impurity.[1] The applications for synthetic diamonds, primarily in the area of active electronic components, did not realize to a great extent because of the poor structural quality (i.e., the polycrystalline nature) of most CVD grown films.

Some of the inherent "electrochemically" desirable properties of diamond are hardness, high thermal conductivity, corrosion resistance, chemical inertness, variable conductivity via doping, and electrode geometry patterning using selective growth methods. The resistivity of polycrystalline diamond thin films made by chemical vapor deposition (CVD) can be decreased by doping with boron.[2] The resulting films can posses either p-type semiconducting or semimetal electronic properties, depending on the doping level. Resistivity as low as 0.01Ω-cm has been reported for boron-doped films, rendering them conductive enough for electrochemical studies.

The electrochemical behavior of diamond electrodes in H_2O_2 solution was reported.[3] The accuracy of the analytical information assured by using boron-doped polycrystalline diamond film electrodes rendered the technique to be used successfully in electroanalysis and electrochemical sensors. The response current is proportional to the square root of the scan rate, reflecting the mass transport controlled by planar diffusion.

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Excellent stability and reproducibility are attractive properties of the diamond electrode even for the later application.

Boron-doped diamond exhibits (BDD) several superior electrochemical properties that are significantly different from those of other carbon allotropes, for example, glassy carbon (GC), pyrolytic graphite (PG), and highly oriented pyrolytic graphite (HOPG), which have been widely used as electrode materials for many years. Its attractive features include a wide electrochemical potential window in aqueous media, very low capacitance, and extreme electrochemical stability. BDD electrodes appear to be relatively free from deactivation problems concerning their sensitivity and stability (e.g., a detection limit of 0.2 μmol/L is obtained for ferrocyanide[4]). The electrodes also show resistance to deactivation even for several weeks of exposure to the laboratory atmosphere. Another interesting feature of BDD films is that its surface is relatively nonpolar as a result of predominant termination of the surface with hydrogen. Thus, the adsorption of polar molecules is suppressed, as demonstrated by Xu et al.[5]

High-quality polycrystalline diamond electrodes doped with boron are amenable alternatives for a wide range of applications especially in the field of electroanalysis[6] for the following reasons: (1) lower background currents and noise signals, which lead to improved S/B and S/N ratios, and lower detection limits; (2) good electrochemical activity without the need for pretreatment; (3) a chemically inert surface that is resistant to fouling such that a high degree of electrode activity is maintained over time; and (4) fairly reproducible film properties from batch-to-batch.

II. DIAMOND FILM GROWTH

The diamond films are usually grown on conducting p-Si (100) substrate using a microwave plasma.[7,8] The substrates, ~1 cm^2 and 0.5 mm thick, are first rinsed with ultrapure water, methanol, and acetone. They are then hand-polished with 0.1-μm diamond powder for about 5 min on a felt pad. The process seeds the surface with diamond particles, which serve as nucleation sites for film growth. Then it is followed by the placement of the substrates in the center of the CVD reactor, atop a boron diffusion source (B_2O_3), and adjacent to a piece of h-BN. The two solid materials served as sources for the boron dopant atoms as the atomic hydrogen in the plasma likely reacts with the solid to form B_2O_6. The dopant levels are estimated to be $\sim 1 \times 10^{19}$ B cm^{-3}. The chamber is evacuated to a base pressure of ~20 mTorr before initiating the growth. Ultra-high purity (99.999%) methane and hydrogen are used as the source gases for the growth and the film thicknesses are 1 to 3 μm.

III. BORON DOPING

The use of electrochemically conductive and semiconductive diamond thin films has only been reported recently.[9,10] Until now, the relationship between the physical, chemical, and electronic properties of the material, and the electrochemical or photo electrochemical performance is not well known. Diamond is one of the nature's best insulators, but when doped with boron, the material can possess semiconducting electronic properties, depending on the doping level. Highly doped, hydrogen-terminated diamond films are semimetals, and posses several important and unique properties such as: (1) law and stable voltammetric and amperometric background currents; (2) wide working potential window in aqueous electrolyte solutions; (3) reversible to quasireversible electron transfer kinetics for several inorganic redox analytes, and enhanced signal-to-background ratios for these analytes due to the low background currents; (4) morphological and micro structural stabil-

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ity at extreme anodic and cathodic potentials; (5) low adsorption of polar molecules from aqueous solutions like anthraquinone-2,6-disulfonate; and (6) long-term response stability.

IV. BORON-DOPED DIAMOND BACKGROUND CURRENTS AND ELECTRON TRANSFER REACTIONS

The cyclic voltammetry response of a highly doped diamond film electrode possessing high quality (low non-diamond carbon impurity) were described for two redox analytes.[8] Background current at this high-quality diamond electrode are smaller than those at classic GC by a factor of 10, while the faradaic responses for $Fe(CN)_6^{3-/4-}$ and $IrCl_6^{2-/3-}$ are comparable. The diamond electrode is electronically active for these two redox analytes without conventional surface pretreatment, including chemical wet etching[11]. Larger S/B ratios are also observed in voltammetric measurements (made at high-quality electrode for these two redox couples) than for the freshly polished GC: the S/B ratio is 16 times larger for $Fe(CN)_6^{3-/4-}$ and 8 times larger for $IrCl_6^{2-/3-}$ at diamond compared with those of GC. Atomic force microscopy (AFM) and Raman spectroscopy are used for the structural characterization of the films. Results indicate that the non-diamond carbon impurity is not required for an active diamond electrode, implying that electron transfer reactions do not occur solely through these impurity sites but rather through the doped materials.

V. ORGANIC COMPOUNDS ANALYSIS

A. Acetaminophen

Cyclic voltammetry and flow injection analyses with amperometric detection are described for the electrochemical analysis of acetaminophen with BDD electrode.[12] The diamond electrode provided a linear dynamic range from 0.1 to 8 mmol/L and a detection limit of 10 μmol/L (S/B=3) for the voltammetric measurement. The flow injection analysis results at the diamond electrode indicated a linear dynamic range from 0.5 to 50 μmol/L and a detection limit of 10 μmol/L ($S/N \cong 4$).

B. Organic Acids

Cyclic voltammetric electrooxidation of some organic acids (e.g., citric acid, malic acid, alanine, and cysteine) at boron-doped diamond (BDD) electrodes is described.[13] The low background current in the potential region where the oxidation of these compounds occur is an attractive feature of the BDD electrode. For cysteine, the response is nearly ideal due to the reaction taking place on the diamond surface rather than on sp^2 carbon impurities. The response needs to be improved, however, for the other acids.

Cyclic voltammetry and bulk electrolysis electrochemical oxidation of benzoic acid (BA) on acidic medium is presented.[14] Cyclic voltammetric measurements in the potential region of water/supporting electrolyte stability show that BA is oxidized, resulting in electron deactivation due to fouling. Bulk electrolysis of BA in the potential region of water/supporting electrolyte decomposition, however, results in the inciation of BA with electrogenerated active intermediates. Good agreement between the experimental data and the theoretical model, based on the assumption that the anodic oxidation of BA is diffusion controlled.

Popa et al. show the selective detection of uric acid (UA) in the presence of high concentrations of ascorbic acid (AA) at electrochemically anodized diamond film electrodes.[15] Uric acid is determined with very good selectivity as the oxidation peak poten-

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tial for AA approximately 450 mV more positive than that for UA at anodized diamond electrodes. Using chronoamperometry technique, the detection limit is found to be as small as 5 × 10^{-8} mol/L. Hence, the practical utility of the method is demonstrated by the measurement of UA in human urine and serum without any treatment.

C. Dopamine

Electrochemical oxidation of dopamine at a highly boron-doped thin-film electrodes is examined using cyclic voltammetry and chronoamperometry.[16] One of the main advantages of the proposed diamond electrode is that dopamine can be reliable assayed in the presence of large excesses of ascorbic acid in acidic medium. A detection limit of 50 nmol/L is obtained using chronoamperometry. These studies indicate great promise for the use of diamond film electrodes in biosensor applications.

D. Histamine

Linear sweep voltammograms for 100 μmol/L histamine in 0.1 mol/L neutral phosphate buffer at highly boron-doped diamond electrode were performed.[17] The diamond electrode exhibited a well-defined oxidation peak at 1.4V vs. SCE with a potential sweep rate of 100 mVs^{-1}. The background-corrected voltammetric current response is linear (r = 0.98) in the concentration range examined (0 to 100 μmol/L), with a detection limit of 1 μmol/L. A detection limit of 0.5 μmol/L (S/N= 13.8) is obtained by using the FIA technique.

E. 3-Methylpyridine (3-MP)

Cyclic voltammetry and bulk electrolysis for electrochemical oxidation of 3-methylpyridine (3-MP) at synthetic boron-doped diamond (BDD) thin film electrode in 0.5 mol/L $HClO_4$ is described.[18] Depending on the applied current density, the BDD-based electrode can be used for electroorganic synthesis of nicotinic acid or for combustion of 3-MP. At low current density and low conversion 3-MP is oxidized to nicotinic acid, while at high current density it is directly combusted to CO_2. The theoretical model for the prediction of 3-MP concentration during 3-MP combustion agree very well with the experimental data. The results demonstrated the application of the technique to electroorganic synthesis and waste water treatment.

F. NADH

Cyclic voltammograms for the oxidation of NADH at a BDD electrode before and after 1 h of exposure to the solution, followed by washing, drying, and storage in the laboratory for 19 h, are superimposed, demonstrating the actual absence of deactivation of the electrode.[19] The concentration range examined is linear between 0 to 60 μmol/L with a regression factor (r = 0.999) up to a sweep rate of 500 mVs^{-1}. Several films are tested and the peak potential for each is 0.58 ± 0.02 V with current densities reproducible within 4 to 5%. An amperometric detection limit is also found to be ~ 10 μmol/L at an S/N ratio of 7 (n=7). The response remained stable even for low concentration range, indicating the possibility of its applicability for NADH-based sensors.

G. Pentachlorophenol

Codognoto et al. used square wave voltammetry for the determination of pentachlorophenol (PCP) at boron-doped diamond electrode.[20] The oxidation occurs at 0.80V vs. Ag/AgCl using a square wave

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voltammetry. The detection limit for pure water and polluted water taken from the local creek are 5.5 and 15.5 mg/L, respectively. The detection limit for later increases because of the degree of contamination of the pure water sample, while the recovery efficiencies are close to 100%. Hence, the combination square wave voltammetry and BDD electrode is a feasible alternative for the analytical determination of PCP and related molecules in either pure water or polluted natural matrices.

H. Phenol

The electrochemical oxidation of phenol in $HClO_4$ on synthetic boron-doped diamond thin film electrode (BDD) is reported.[21] Cyclic voltammetry, chronoamperometry, and bulk electrolysis are used to obtain complete combustion of phenol to CO_2 or the partial oxidation of phenol to other organic compounds (benzoquinone, hydroquinone, cathecol). The theoretical values of phenol concentration and current efficiency during phenol combustion agree very well with the experimental result.

I. SEROTONIN

Sweep rate-dependent cyclic voltammograms for 10 µmol/L serotonin in 0.1 mol/L phosphate buffer (PH=7) is examined with a potential scan rate of 100 mVs^{-1}.[17] Highly BDD electrode shows an oxidation peak at 0.42V vs. SCE, indicating the absence of adsorption at the diamond electrode within the experimental time of several hours. An experimental detection limit of 10 nmol/L is obtained with a linear dynamic range from 0.01 to 50 µmol/L.

J. Sulfur Compounds

Cyclic voltammetric electrooxidation of homocysteine, glutathione (GSH), 2-mercapto ethanesulfonic acid, and cephalexin at boron-doped diamond (BDD) electrodes are investigated.[22] Low voltammetric background current in the large positive potential region are obtained without pretreatment of the electrode. Hence, BDD can be a promising electrode material for the electrolysis of other sulfur-containing compounds.

K. Xanthine and Its Derivatives

The electrochemical oxidation of xanthine and its naturally occurring *N*-methyl derivatives (theophylline, theobromine, and caffeine) at conductive diamond electrodes are presented.[23] Cyclic voltammograms obtained at BDD electrodes exhibit well-defined voltammograms with high signal to background ratios for all xanthine derivatives. The concentration range is 1 to 400 µmol/L for theophylline, theobromine, and caffeine, and of 1 to 100 µmol/L for xanthine. The excellent results obtained for caffeine determination in three commercially available coffee and cola products, with very simple sample preparation, involving only dilution in electrolyte, demonstrates the practical analytical utility of the method.

VI. TRACE METAL ANALYSIS

A. Cadmium, Lead and Zinc

Anodic stripping voltammetry for the detection of trace metal ions like Zn^{2+}, Cd^{2+}, and Pb^{2+} is described.[24] All the three analytes are easily resolved and detected in the low to mid ppb range. The deposition was accomplished from a 0.1 mol/L acetate buffer, pH 4.2, at a constant potential of –1300 mV vs. SCE. The ions are preconcentrated on the surface for 3 min under quiescent conditions. Zn^{2+} can easily be detected due to the large overpotential for hydrogen evolution

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of the diamond film unlike the glassy carbon where the deposition and stripping occurs at potentials at which hydrogen evolution commences.

B. Cerium (Ce^{3+})

The electrochemical study of the $Ce^{3+/4+}$ redox couple at highly boron-doped conductive diamond electrode in aqueous sulfuric acid, nitric acid, and perchloric acid is shown.[25] Well-defined cyclic voltammogram, indicating quasireversible behavior for the Ce^{3+}/Ce^{4+} redox couple, is derived demonstrating its usefulness for studies of couples with highly positive equilibrium redox potentials. With the use of simple cyclic voltammetric measurements, the diffusion coefficients and electron transfer kinetic parameters can be conventionally determined in a way that is not possible with other types of electrodes. Hence, highly boron-doped diamond is an excellent electrode material for the electrochemical analysis of species with highly positive potentials in aqueous solutions.

C. Copper (Cu^{2+}) Reduction

Nakabayashy et al. investigated the electrochemical reduction of copper (Cu^{2+}) with negligible deposition of the metal on the conductive boron-doped CVD diamond electrode.[26] The copper atoms hardly adsorb on the diamond electrode surface, due to the nonpolar nature of hydrogen-terminated surface. Conductive boron-doped CVD diamond electrodes can be used for the electrochemical reduction of metal ions without electrodeposition.

D. Lead

Ultrasonically assisted cathodic stripping voltammetry at a boron-doped diamond electrode is described for the detection of lead in river sediment.[27] Linear sweep voltammetry for the analytical signal from a cathodic strip of electrodeposited PbO_2 is employed. The linear concentration range is 3 to 100 μmol/L, with 3 μmol/L being the lower detection limit. For the cathodic stripping step square-wave voltammetry is used to lower the detection limit of the technique while retaining the linearity to the order of 10^{-8} mol/L. In combination with an ultrasonically assisted acid digestion, the technique can be used successfully in the analysis of similar contaminated samples, offering a substantial saving of time and cost over currently used techniques.

E. Lead, Mercury, and Platinum

Awada et al. described the cyclic voltammetry of the electrochemical deposition of Pt, Pb, and Hg on conductive diamond thin-film surface,[28] Results for the three metal ions indicate that electrochemical reduction is a viable approach for metallizing the surface of polycrystalline diamond-thin films demonstrating the development of novel catalytic electrodes, sensors, and detectors using this advanced material.

F. Manganese (Mn^{2+})

Ultrasonically assisted cathodic stripping voltammetry at a boron-doped diamond electrode for the detection of manganese is described.[29] Differential pulse voltammetry is used to give the analytical signal from a cathodic strip of electrodeposited MnO_2. For a 2-min deposition, the detection limit is 10^{-11} mol/L with a linearity up to 3×10^{-7} mol/L. Both ultrasonic-anodic deposition of MnO_2 and ultrasonic-cathodic stripping are used. The method is successful for the determination of manganese content in two instant tea samples, giving excellent agreement with independent AAS analyses.

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G. Silver

Saterlay et al. described the sonoelectroanalysis of silver at a BDD electrode, using both anodic silver oxide and cathodic silver metal deposition.[30] A detection limit for aqueous Ag^+ of 10^{-9} mol/L and a linear range of 10^{-9} to 10^{-7} mol/L is obtained. The analytical benefits of chloride ion complexation of Ag^+, as an anodic stripping voltammetry (ASV) peak shift-reagent offers the potential to selectively avoid overlapping of contaminants in a complex system. The application of power ultrasound can be used to increase the efficiency of the electrochemistry taking place, allowing trace determinations to be carried out in relatively short times, compared with most silent techniques.

H. Silver and Tin

Boron-doped diamond electrodes and reference substrates comprising Ag and Sn are investigated using abrasive stripping voltammetry.[31] The silver-deposited yields an analytically diagnostic signal in stripping voltammetry; tin is found to be extremely dependent on the stripping conditions employed. The initially hydrogenated CVD diamond electrodes become significantly oxidized when employed in this application. The abrasion process produces metallic particulates on the electrode surface, and both the mechanical and electrical contact are sensitively dependent on the abrasive force used.

VII. SIMULTANEOUS ANALYSIS

A. Copper and Lead

Simultaneous detection of trace lead and copper at boron-doped diamond is reported.[32] Although both lead and copper appear to be deposited, linear response for both of these metals is found in the range from 2.5×10^{-6} mol/L and 10^{-4} mol/L using anodic stripping voltammetry**.** SWV is used as a basis for the simultaneous independent detection of Cu and Pb through conventional standard addition methodology.

VIII. CONCLUSIONS

Polycrystalline boron-doped diamond electrodes can be used successfully for the assay of inorganic (trace metals) and organic compounds. They demonstrated the best reliability of the analytical information due to the high stability and attractive electrochemical properties. The advantages of using diamond based electrodes are the S/N and S/B ratios as increasing, the sensitivity is increasing, and the limit of detection is decreasing.

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