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Synthesis of Ni nanoclusters supported on diamond by plasma technique and their electrochemical properties

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ABSTRACT

In this study, particles of synthetic undoped diamond (D_N) obtained via the high pressure – high temperature method were coated with a nickel shell using metallic nickel plasma in a two-jet plasma generator with gas vortex and magnetic flux stabilization. Through the use of scanning electron microscopy, transmission electron microscopy, and X-ray photoelectron spectroscopy, we observed the formation of a nickel diamond composite with a core-shell structure, where D_N serves as the core and Ni nanoclusters form the shell ($D_N@Ni$). The results of voltammetric analysis indicated that $D_N@Ni$, when deposited on a graphite electrode, exhibited significant electrocatalytic activity in the oxidation of methanol and paracetamol in an alkaline electrolyte.

1. Introduction

Ni diamond composites have attracted increasing interest due to their exceptional properties and potential applications across various fields. In this perspective, recent advances on the synthesis of diamond composites are summarized together with state of the art progress in their electrochemical applications [1].

The application of a nickel surface coating to synthetic diamonds has a wide range of uses. The nickel shell surrounding diamond grains enhances their adhesion to binder matrices, resulting in the production of abrasive and cutting tools [2–4]. Nickel interlayer provides adhesion of diamond and copper coating in copper-diamond highly heat-conducting composites used for heat dissipation from electronic devices during operation [5–7].

Conductive diamond materials, achieved through boron doping (BDD) [8] and coated with nickel nanoparticles, are regarded as excellent electrode materials and have found wide-ranging applications in various electrochemical processes [9–12].

Undoped diamonds, despite being insulating in nature, can also participate in electrochemical reactions [13]. Review [14] focuses on the analysis of the construction, modification, and physical

characteristics of two types of undoped diamond electrodes: nanoparticle-modified diamond electrodes and detonation nanodiamond-based electrodes. Nanoparticles of undoped diamond modified with nanoparticles Pt and alloy Pt/Ru were investigated as catalysts for electrocatalytic oxidation of methanol in fuel cells [15,16].

The paper [17] addresses graphitized nanodiamond supporting PtNi alloy as stable anodic and cathodic electrocatalysts for direct methanol. Highly crystalline undoped high-pressure, high-temperature (HPHT) synthetic diamond particles, approximately 500 μ m in size, with chemically deposited Pd nanoparticles, have exhibited high electrocatalytic activity in formic acid oxidation reactions. This experiment highlights the potential of using undoped HPHT diamond powders as electrocatalyst supports, which are more readily available than BDD powders [18]. However, we were unable to find any data on the electrochemical properties of undoped diamonds modified with nickel particles for electrocatalytic and electroanalytical applications.

Various methods employ to deposit nickel onto technical diamonds, including chemical deposition [19], electrochemical deposition [20], volatile compound chemical vapor deposition [21], spark plasma sintering [22], rapid discharge sintering [23], and magnetron sputtering [24]. These methods are of great technological importance as they

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enable the coating of the crystalline surface of diamonds with nickel. In our previous research, we utilized a two-jet plasma generator with gas vortex and magnetic stabilization flows to apply nickel nanoparticles to detonation diamonds [25].

The synthetic undoped diamond (D_N) particles were coated with nickel using the plasma-chemical method and a two-jet plasma generator with gas vortex and magnetic stabilization of flows. Furthermore, we investigated the electrochemical behavior of these undoped nickelcoated diamond particles, referred to as $D_N@Ni$, in electrocatalytic oxidation reactions of methanol and paracetamol. Paracetamol is recognized as one of the most polluting pharmaceuticals, as it is commonly found in the environment, domestic wastewater, and hospital wastewater. Its presence poses a risk to human health, necessitating the treatment of wastewater containing paracetamol. In recent years, there has been a great development of electrochemical methods, such as anodic oxidation and electro-oxidation, for the destruction of toxic pollutants [26,27]. In this work, we employed a $D_N@Ni/graphite$ electrode for the electrochemical oxidation of paracetamol.

2. Materials and methods

2.1. Materials

We used: D_N with an average particle size of ~100 nm, nickel metal (95.75 wt% purity), KOH, NH₄Cl, NH₄OH, C₃H₈O, CH₃OH, C₈H₉NO₂, naphion water solution (Liquiontm Solution LQ-1151100EW, 15 wt%, Ion Power, GmbH).

Graphite electrodes with a diameter of 5 mm were prepared by impregnating spectrally pure graphite rods with a mixture of paraffin and polyethylene. The side surface of the electrodes was covered with a glass case, while the working surface was prepared through grinding and polishing using abrasive materials and paper.

2.2. Methods and equipment

The production of D_N @Ni particles was carried out using a two-jet nickel plasma generator with gas vortex and magnetic flux stabilization, as shown in Fig. 1.

Fig. 1(A) depicts an arc discharge (1) between two water-cooled electrodes (2). The stabilization system consists of a ceramic gas swirl chamber (3) and a stabilizing solenoid (4), which designed to provide stable combustion of the arc discharge. When two plasma jets combine, it allows for the generation of an increased amount of plasma, as illustrated in Fig. 1(C). The tangential supply of gas (Fig. 1(A) – (5) and Fig. 1



Fig. 1. Schematic diagram of plasma flows stabilization of a two-jet plasma generator (A): 1 – plasma flow, 2 – water cooled electrodes, 3 – stabilization chamber, 4 – stabilization coil, 5 – tangential supply of insulating gas. Layout of the synthesis setup (B): 1 – water cooled chamber, 2 – arc discharge,

3 - tangential injection of insulating gas,
4 - capillary for substance supply, 5 - injection of plasma forming gas.

Photograph of the plasmatron while in operation (C).

(B) - (3) into the chamber (Fig. 1(B) – (1)) creates an area of reduced pressure where the plasma flow follows. Additionally, the in-phase change of current in the coil (Fig. 1(A) – (4)) with the power current discharge enables a time-constant effect on the plasma flows.

The refrigerated electrodes of the plasmatron were made of nickel, and during arc discharge, the electrode material was eroded, leading to the entry of nickel in the form of atomic vapors into the plasma flow. The particles were supplied with argon flow through a substance supply capillary (Fig. 1(B) - (4)), and the surface of the particles acted as cluster centers for the atomic vapors. This method was chosen for its high speed and reproducibility, requiring minimal sample preparation time before synthesis. The substances obtained after synthesis do not require treatment, and the method allows for controlled synthesis by adjusting the flow control of the plasma-forming and stabilizing gas, as well as the operating current to regulate the electrode material content in the resulting products.

2.3. Sample characterization

The morphology and microstructure of $D_N@Ni$ particles were examined using a scanning electron microscope (SEM S-5500, Hitachi), a transmission electron microscope (TEM HT7700, Hitachi) and a X-ray photoelectron spectrometer (XPS UNI-SPECS Spectrometer, Gmbh).

The electrocatalytic properties of the obtained $D_N@Ni$ sample, deposited on the graphite substrate, were examined by cyclic voltammetry regarding the oxidation of methanol and paracetamol. The working electrode of $D_N@Ni$ /graphite was prepared by standard droplet application of catalytic inks obtained by ultrasonic mixing of a sample and naphion on the end of a graphite rod inserted into a teflon insulator. The glass three-electrode electrochemical cell included a platinum wire as a secondary electrode and an Ag/AgCl reference electrode connected by a protruding capillary. Isopropyl alcohol and a naphion water solution were used in the producing of the catalytic "ink". Measurements were taken using the Elins potentiostat. The electrooxidation of methanol on the $D_N@Ni$ /graphite electrode was examined in a base solution 0.1 M KOH and in a mixture 0.1 M KOH, 0.01 M CH₃OH. The electrical oxidation of paracetamol (4.5 mMol/L) was investigated in 0.1 M NH₄Cl + 0.1 M NH₄OH (pH 8.5).

3. Results and discussion

3.1. Synthesis of materials

Methods of the material synthesis for electrocatalytic and electroanalytical applications, using diamonds as a catalytic support, are mainly chemical. These methods are multi-stage, energy-intensive and require special conditions. Surface treatment of diamond particles in nickel-containing plasma allows implantation of metal particles directly into diamond crystal structure. This is possible due to the particles of the initial diamond powder passing the reaction region at such a speed that on the one hand their surface layer is heated higher than the melting point, and on the other hand, the flight time does not allow the particles to heat up completely and avoid changing the phase composition. At the same time, the concentration of the coating material in the plasma should be as stated in order to avoid the process of homogeneous nucleation of the clusters and ensure only nucleation on the surface of the nanodiamond particles being treated. Such conditions occur in the plasma flow of the HF arc discharge of the kHz range. Using the described method, we obtained D_N particles surface-coated with nickel clusters - D_N@Ni.

The statistical analysis of TEM images of D_N @Ni particles (Fig. 2(A)) revealed that the characteristic particle size is 120–130 nm. The thickness of the diamond particles coating varies within the range of 2 to 20–30 nm. In Fig. 2(B), a photograph of the surface of a diamond particle obtained using a scanning electron microscope is presented. An analysis of a large number of images indicates that the average size of clusters



(A)

(B)



Fig. 2. TEM-image (A) and CEM-image (B) of characteristic powder particles D_N@Ni; diffraction pattern of sample D_N@Ni (C).

varies within the ranges of 6–8 nm. Furthermore, an analysis of the diffraction image (Fig. 2(C)) demonstrates the presence of phases with interplanar spacing of 1.78 ± 0.05 Å; 2.05 ± 0.05 Å; 2.34 ± 0.05 Å; 2.82 ± 0.05 Å; 2.8222 ± 0.05 Å; 3.399 ± 0.05 Å, corresponding to reflection planes of Ni(200)/NiCO₃(024), Ni(111)/C-diamond(111), Ni₃C(110),

NiCO₃(104), NiCO₃(012)/Ni₃C(012).

The obtained sample of $D_N@Ni$ particles was analyzed by the X-ray photoelectron spectroscopy (XPS). According to the obtained results, the sample contains: 77.5 at.% of carbon in various hybridization; 17.4 at.% oxygen; 5.1 at.% nickel. According to the results of XPS, the $D_N@Ni$



Fig. 3. Decomposition of the XPS spectrum lines of the $D_N@Ni$ sample: line C1s (A); line Ni2p (B).



sample contains carbon (mainly in sp^3 hybridization), oxygen, and nickel. The decomposition of lines Ni2p and C1s is shown in Fig. 3.

Analysis of the XPS spectrum shown in Fig. 3 confirms the diffraction results given above. Table 1 shows the distributions of the atomic content of bonds with different energy over the XPS spectrum.

Based on the obtained data, it can be concluded that the treatment of undoped synthetic diamond powder in a metal nickel plasma leads to the formation of Ni-based cluster structures associated with the diamond surface. Nickel clusters grow into the diamond crystal lattice to form intermediate phases Ni₃C and NiCO₃. Table 1 indicates a partial conversion of sp³-hybridized diamond to sp² microcrystalline carbon (8.8 %). This conversion may increase the electrical conductivity of the resulting $D_N@Ni$ composite. The bond energy corresponding to Ni(OH)₂ is likely generated by the absorption of moisture from the environment and the partial oxidation of the nickel component.

As a controlled synthesis method is employed, the conditions for obtaining the final product were chosen to minimize the graphitization of diamond. In this scenario, the residence time of particles in the plasma is adequate for the nucleation process of the dense phase of nickel from atomic vapor to take place, but insufficient for the growth of these nuclei.

3.2. The electrochemical behavior of $D_N@Ni$

3.2.1. Electrocatalytic oxidation of methanol at $D_N@Ni/graphite$

Fig. 4 shows cyclic voltammograms of an empty graphite electrode and $D_N@Ni/graphite$ electrode in a solution of 0.1 M KOH and in a mixture of 0.1 M KOH + 0.01 M CH₃OH at a potential scanning rate of 50 mV/s in the range of potentials from -10 to 1200 mV.

When recording in the base KOH buffer solution on the anode curve (A), a peak appears at a potential of 371 mV relative to the saturated silver chloride electrode. This is attributed to the conversion of Ni(OH)₂/NiOOH [28]. Upon injection of methanol into the buffer solution on the voltammetry anode curve (B), a peak is observed at a potential of 211 mV. As depicted in the figure, in the presence of alcohol, the maximum of the anode peak shifts by 160 mV to the low potential region, accompanied by an increase in current density from 3.46 A/m² to 8.7 A/m². Overvoltage for methanol oxidation is reduced, and current density is significantly increased. This suggests that alcohol oxidation occurs on the surface of the potential scan, indicating that the oxidation process is irreversible.

The anode peak corresponding to the oxidation of methanol on the $D_N@Ni/graphite$ electrode is significantly shifted to the low potential region compared to the peak potentials (Ep) of the oxidation of methanol on various Ni-containing electrodes (Table 2). Therefore the $D_N@Ni$ sample exhibits higher electrocatalytic activity in the oxidation reaction of methanol compared to other nickel-containing electrode materials known in the literature.

3.2.2. Electrocatalytic oxidation of paracetamol at D_N @Ni/graphite

Fig. 5 shows cyclic voltammetry of the $D_N@Ni/graphite$ electrode in ammonia buffer solution (0.1 M NH_4Cl+ 0.1 M $NH_4OH,$ pH- 8.5) containing 4.5 mMol/L paracetamol at a scanning rate of the potential of 100–10 mV/s.

Table 1

Distributions of atomic content of bonds with different energy over XPS spectrum.

Line/bond	Atomic content, %
C1s / C(sp ²)	8.88
C1s / C(sp ³)	75.74
C1s / C-O	4.76
C1s / C=O	10.62
Ni 2p / Ni ⁰	66.25
Ni 2p / Ni(OH) ₂	30.71
Ni 2p / Ni-C	3.04



Fig. 4. Cyclic voltammograms of an empty graphite electrode in a mixture of 0.1 M KOH + 0.01 M CH₃OH (A);

 $D_N@Ni/graphite in a solution of 0.1 M KOH (B) and in a mixture of 0.1 M KOH <math display="inline">+$ 0.01 M CH_3OH (C) at a potential scan rate of 50 mV/s.

Table 2

Values of peak potentials (Ep) of methanol oxidation on different Ni-containing electrodes.

Ep vs Ag/AgCl (v)	v (mV/s)
0.35 [28]	50
0.58 [29]	50
0.83 [30]	50
0.21 [present paper]	50
0.42 [31]	100
0.83 [32]	20
	Ep vs Ag/AgCl (v) 0.35 [28] 0.58 [29] 0.83 [30] 0.21 [present paper] 0.42 [31] 0.83 [32]



Fig. 5. Cyclic voltammetry of the $D_N@Ni/graphite$ electrode taken in ammonia buffer solution (0.1 M $\rm NH_4Cl$ + 0.1 M $\rm NH_4OH$ (pH- 8.5) with a content 4.5 mMol/L paracetamol at a scanning rate of the potential of 100–10 mV/s.

In the presence of paracetamol, the voltammogram (at a scanning rate of 100 mV/s) exhibits distinct peaks corresponding to a quasireversible redox process: E_a (anode peak potential) = 338 mV and E_c (cathode peak potential) = 250 mV, with a peak resolution of 78 mV. The electron transfer process is considered reversible if the difference between the anode and cathode current peaks, ΔE (the anode-cathode), is approximately 59 mV. For an irreversible reaction, this difference should be greater than or equal to 200 mV [33,34].

The effect of changing the scan rate is used to determine the type of mass transfer process involved. Cyclic voltammograms in Fig. 5, corresponding to the electrocatalytic oxidation reaction of paracetamol at the $D_N@Ni/graphite$ electrode, were obtained by increasing the scan rate from 10 to 100 mV/s. For reversible reactions, the ΔE anode-cathode does not depend on the scanning speed [33].

When paracetamol is oxidized on the $D_N@Ni/graphite$ electrode, the ΔE (anode-cathode) increases with the scanning rate, which is typical for quasi-reversible electron transfer reactions. The redox currents (I_a) and (I_k) exhibit a linear relationship when the scan rate changes. The lower slope of the straight line (I /^{1/2}) for I_a compared to I_k indicates a higher reaction rate for the reduction of paracetamol. The linear dependency suggests a diffusion-driven process. The stability of the $D_N@Ni/graphite$ electrode and its effect on the oxidation current were assessed by continuous cycling over the potential range of the paracetamol oxidation current. The current value remained almost constant over 7 cycles, confirming the stability of the electrode.

Electroanalytical Investigation by Voltammetry on Paracetamol on BDD in paper [35] demonstrated irreversible oxidation in both acid (HClO₄) and alkaline (KClO₄) electrolytes with an anode peak potential value of 860 mV. In our paper, the quasireversible oxidation process of paracetamol on the $D_N@Ni/graphite$ electrode and the significantly lower anode peak potential (338 mV) suggests a higher electrocatalytic effect of $D_N@Ni$ compared to BDD.

In the present paper, we have obtained qualitative data characterizing the catalytic properties of diamond particles with a surface covered with nickel clusters in the oxidation reaction of paracetamol. Analytical studies will be obtained in the following papers.

4. Conclusions

Via the plasma-chemical method, using a two-jet plasma generator with gas vortex and magnetic flow stabilization, we obtained dielectric highly-crystalline particles of synthetic undoped diamond surface-coated with nickel clusters. According to the results of the cyclic voltammetry study, the obtained composite material – $D_N@/Ni$ has a higher electrocatalytic activity in electrochemical oxidation reactions of methanol and paracetamol in an alkaline electrolyte compared to known catalytically active materials. Thus, we have demonstrated a fundamental possibility of using D_N as a catalytic nickel support and potential use of the $D_N@/Ni$ composite in anodic electrocatalysts for methanol fuel cells and for electroanalytical applications.

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CRediT authorship contribution statement

Nikita Nikolaev: Visualization, Validation, Methodology, Conceptualization. Victoria Isakova: Writing – review & editing, Writing – original draft, Validation, Resources, Methodology, Investigation. Natalia Vnukova: Writing – review & editing, Investigation. Victoria Elesina: Writing – review & editing, Visualization, Investigation. Gariy Glushenko: Investigation. Yevgeny Tomashevich: Investigation. Grigory Churilov: Validation, Resources, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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