

Copper electrodeposition of on boron doped diamond by cyclic voltammetry

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Abstract – This work presents the boron doped diamond electrode modification from the copper electrodeposition by cyclic voltammetry. The electrochemical kinetics and inversion potential of such electrode was studied in the sweep rate range from 20 to 100 mV/s in 10^{-3} molL⁻¹ of CuSO₄ solution in three support electrolytes of H₂SO₄, HClO₄ e Na₂SO. The objective was to verify the electrode response reproducibility according to the reversibility criteria, where with the sweep rate increase the intensity of anodic and cathodic peak current also increased.

Boron doped diamond (BDD) electrodes have attracted attention to apply in electrosynthesis and electroanalysis due to their singular properties, such as high corrosion resistance, wide potential window (~3.0 V) for a large range of electrolytes, high chemical inertness, low background current and high electrochemical activity, that is reproducible without pre-treatment [1]. In this sense, the nanoparticles deposition on BDD may promote to surface roughness increase leading to an increase of the electrode active surface area and its sensitivity. This work studies the BDD modification from the electrodeposition of copper nanoparticles using cyclic voltammetry [2].

The work electrode used in all experiments was the BDD film grown on si substrate using a hot filament chemical vapor deposition reactor. The films were deposited during 6 h at temperature of 780 °C from a gas mixture of H₂ (99 sccm) and CH₄ (1 sccm) for a pressure of 50 Torr. The boron content was controlled using an additional H₂ flow passing through a bubbler containing B₂O₃ dissolved in methanol in the required B/C ratio corresponding to 10²⁰ born atoms/ cm³. To improve the electrode response for copper electrodeposition the BDD was submitted to cathodic pre-treatment at -3V in 0.5 molL⁻¹ H₂SO₄ with different times between 1 and 16 min. The cathodic treatment during 1 min promoted an improvement in the electrochemical response as may be observed in Fig. 2. The Cu electrodeposition on BDD was performed using 10⁻³ molL⁻¹ of CuSO₄ solution in three different support electrolytes of H₂SO₄, HClO₄ e Na₂SO at 0.1 molL⁻¹.

For a better understanding of the Cu electrochemical behavior the process was analyzed varying the inversion potential. Fig. 3 depicts the Cu voltammetric curves for different inversion potentials. It was observed that from the inversion potential of -0.3 V both the deposition and dissolution processes take place in the same regions. This behavior indicates that the formed Cu deposit is linked with the same energy with the substrate (BDD electrode).

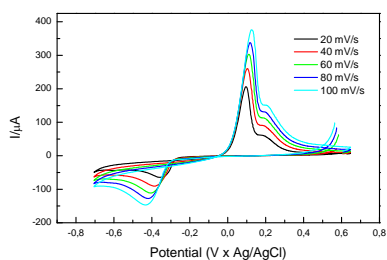


Figure 1: Cyclic voltammograms of Cu on BDD using a solution containing 1×10^{-3} mol L⁻¹ CuSO₄ in H₂SO₄ 0.1 mol L⁻¹ for different scanning speeds.

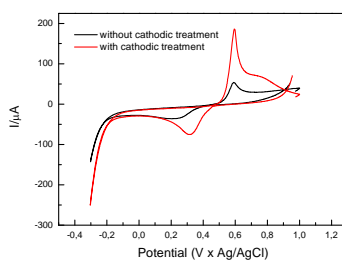


Figure 2: Cyclic voltammogram of Cu on BDD using a solution containing 1×10^{-3} mol L⁻¹ CuSO₄ in H₂SO₄ 0.1 mol L⁻¹, with and without cathodic treatment.

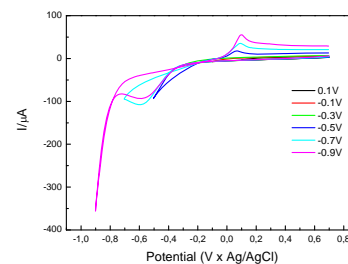


Figure 3: Cyclic voltammograms of Cu on BDD using a solution containing 1×10^{-3} mol L⁻¹ CuSO₄ in H₂SO₄ 0.1 mol L⁻¹ for different potentials inversion. $v = 100$ mVs⁻¹

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