Anodic and cathodic pre-treatment enhancement in the copper electrodeposition and consolidation processes on BDD electrode applied to nitrate reduction

L. C. D. Santos, A. B. Couto, J. T. Matsushima, M. R. Baldan, N. G. Ferreira
Instituto Nacional de Pesquisas Espaciais, INPE, São José dos Campos, SP, Brazil

The electrochemical behavior of boron-doped diamond (BDD) electrode is strongly affected by its two-surface termination of hydrogen or oxygen. Thus, the electron transfer behavior between diamond electrode and electrolyte solution is significantly dependent on its surface termination [1, 2]. In this context, the influence of anodic and cathodic pre-treatment on boron doped diamond surface for copper nanoparticles electrodeposition was studied to assess Cu nanoparticle consolidation on BDD electrode. The cathodic and anodic pre-treatments were performed using the -3 V and +3 V x Ag/AgCl, respectively, during 30 min in solution of 0.5 mol L^{-1} H_2SO_4. The electrodeposition of Cu nanoparticles on BDD films was made at a fixed potential of -0,6 V for a time of 60 s in solution of 0.1 mol L^{-1} $HClO_4$ support electrolyte containing 0.001 mol L^{-1} $CuSO_4$.The scanning electron microscopy (SEM) images showed that the BDD surface cathodically treated presented high Cu nanoparticle density as well as even distribution on the electrode surface after the electrodeposition process compared to that obtained from anodic treatment. This behavior was attributed to a high electrode conductivity imposed by the cathodic pre-treatment leading to an increase in the BDD surface hydrogenation that was confirmed by the contact angle measurements. Nitrate reduction experiments were monitored by linear sweep voltammetry (LSV) using a 0.1 mol L^{-1} KNO_3 + 0.1 mol L^{-1} Na_2SO_4. The results showed that the electrochemical nitrate reduction on BDD/Cu samples treated with anodic treatment presented better reproducibility compared to the ones only treated with catodic treatment.This response is attributed to the Cu nanoparticle consolidation on BDD electrode due to the oxygen surface terminations induced by the anodic pre-treatment.

Keywords: Boron doped diamond; Cathodic and anodic pre-treatment; Copper electrodeposition; Nitrate reduction.

Work supported by FAPESP, CAPES e CNPq.

References

[1] I. Yagi, H. Notsu, T. Kondo, D. A. Tryk, A. Fujishima, Electrochemical selectivity for redox systems at oxygen-terminated diamond electrodes, J. Electroanal. Chem. 473 (1999) 173-178.
[2] M. C. Granger, M. Witek,, J. Xu, J. Wang, M. Hupert, A. Hanks, M. D. Koppang, J. E. Butler, G. Lucazeau, M. Mermoux, J. W. Strojek, G. M. Swain, Standard electrochemical behavior of high-quality, boron-doped polycrystalline diamond thin-film electrodes, Anal. Chem. 72 (2000) 3793-3804.

e-mail: neidenei@las.inpe.br

Instituto Nacional de Pesquisas Espaciais-INPE-*Av dos Astronautas, 1.758 , Jd. Granja CEP: 12227-010-São José dos Campos - SP*