HEAT TREATMENT OF COPPER NANOPARTICLES DEPOSITED BY ELECTRON BEAM ON BORON-DOPED DIAMOND FILMS

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1. Introduction

The excessive presence of nitrate ions (NO_3^-) in water mainly caused by the fertilizing in agriculture process may promote dangerous ecological complications [1]. Considering different methodologies to detect and to destruct nitrate ions, the electrochemical process appears as a promising and cheaper technique. Due to its singular electrochemical properties, boron doped diamond electrodes have shown good efficiency for electrolyses and nitrate reduction [2]. Copper nanoparticles are currently attracting a great deal of interest due to their properties which differs for those of bulk materials. Their electrochemical properties have been subject of numerous studies in heterogeneous catalysis and surface electrochemistry. A deposition of nanoparticles may lead to the increase of sensing surface which means a vastly increased conductive area of the electrode. In this work *ex-situ* method for the deposition of copper nanoparticles onto boron doped diamond (BDD) electrode were investigated.

2. Experimental

The films were prepared by hot filament CVD technique using the standard mixture of H_2/CH_4 with an additional H_2 flux passing through a bubbler containing a suitable concentration of B_2O_3 dissolved in methanol to assure films with acceptor concentration around 10^{20} cm^{-3}. The growth times were 7 h corresponding. Diamond quality, morphology before and after the copper nanoparticles deposition were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM) and Raman spectroscopy. The copper nanoparticles were deposited by electron beam BOC Edwards system by controlling the deposition rate resulting in thin films between 20 and 60 Å of thickness. Afterwards, the samples were heat treated at temperatures ranging from 400 to 600 ºC in air during 60 min. Voltammetric curves were obtained using a AUTOLAB 302 equipment in 0.5 M H_2SO_4.

3. Results and Discussions

The SEM images for samples before and after the copper nanoparticles deposition is presented in Fig. 1 a and b, respectively. When the deposition of metal is carried out at low temperature, as in the case of electron beam technique, the process of nucleation is highly favored compared to that of particle growth. For polycrystalline surface, as BDD film with many specific sites such as defects, dislocations, grain boundary, impurities, etc, the first nucleation events take places at these sites. Also, successive heat treatment of such a fine deposit at higher temperature may lead to the formation of a stable population of metal nanoparticles. The copper reduction and dissolution peaks for the different times of copper deposition were analyzed by cyclic voltammety and presented the expected dependence with the copper population.

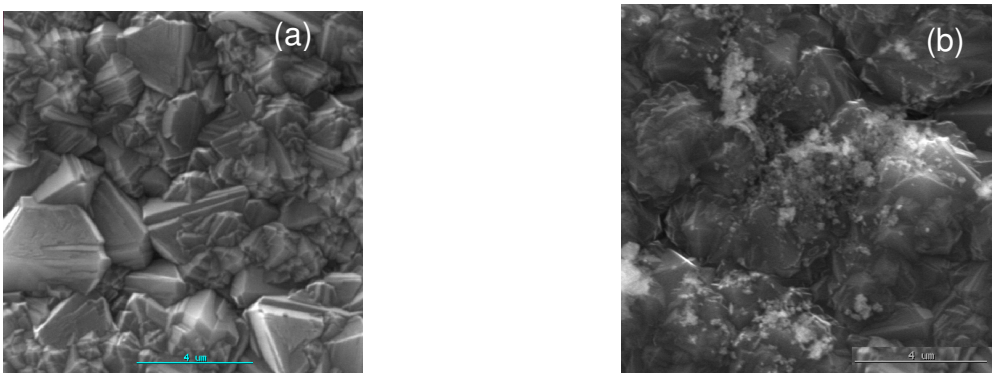

Fig.1. *SEM images of BDD films* **(a)** *before and* **(b)** *after the deposition of copper nanoparticles.*

4. References

[1] L. Monser, S. Sadok, G. M. Greenway, I. Shah, R. F. Uglow, Talanta, 57 (2002) 511.
[2] C. M. Welch, M. E. Hyde, C. E Banks, R. G. Compton, Analyt. Sci., 21 (2005) 1421.

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