



Short communication

Non-enzymatic glucose sensing on long and short diamond nanowire electrodes



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ABSTRACT

In this paper the fabrication of micrometer long boron-doped diamond nanowire (L-BDD NWs) electrodes is reported. The diamond nanowires were obtained by a maskless reactive ion etching process with oxygen plasma using 15 μm thick polycrystalline boron-doped diamond (BDD) films as starting material. The resulting nanowire interfaces showed enhanced electrochemical activity towards the direct electrochemical detection of glucose under strong basic (pH 12) condition when compared to flat BDD and short nanowires (S-BDD NWs). The robust response of these nanostructures with a detection limit of 60 μM and a linear range up to 8 mM and the ease of formation make L-BDD NWs a promising platform for real time applications.

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1. Introduction

Tremendous efforts have been put into the development of efficient, reliable and sensitive methods to determine the concentration of glucose in food [1] as well as for the diagnosis of diabetes [2,3]. Electrochemical-based sensors have been considered as excellent analytical tools for rapid and inexpensive glucose-recognition [4,5]. Enzyme-based glucose sensors are the most widely employed ones. The insufficient long term stability of the enzyme causes however a serious problem to most sensor designs. The electrochemical determination of glucose concentrations without using glucose oxidase is one goal that many research teams have been trying to make come true [2,6–10]. The desire for better and cheaper electrocatalytic interfaces for glucose without fouling effects of the oxidized products has put nanostructures into the forefront of research.

Boron-doped diamond (BDD) electrodes have been recognized as an ideal material for bioelectrochemical applications [11–19]. Diamond electrodes are chemically stable, exhibit favorable biocompatibility and show an enlarged potential window together with a low background current when compared to other carbon-based materials. In the past decade, several reports have demonstrated the fabrication of nanostructured diamond interfaces [7,20–26]. The increase of the surface area of these interfaces resulted in higher sensitivities and

selectivities when compared to planar BDD interfaces [7,21,27,28]. Nebel et al. reported recently that tip-coating diamond nanowires with nickel particles can be applied as a three-dimensional electrode for the electrochemical oxidation of glucose and reduction of hydrogen [6]. Luo et al. showed that diamond nanorod forest electrodes, obtained through silicon nanowire interfaces post-coated with boron doped diamond respond linearly to glucose in the range of 100 μM to 7 mM [7].

We show, in this paper, that the length of the nanowires is crucial for the electrochemical behavior of the interface. Indeed, we found that long BDD NWs offer higher sensitivity for the detection of glucose oxidation under basic conditions as compared to short ones.

2. Experimental part

Boron-doped diamond (BDD) films were grown by microwave plasma-enhanced chemical vapor deposition from methane/hydrogen mixtures (1% CH_4) in an ASTeX 6500 reactor. The substrates were p-type doped (100) silicon wafers (thickness 500–550 μm , resistivity from 1 to 20 $\Omega\text{ cm}$). Trimethyl borane gas was added during the growth with a ratio of 10000 ppm B/C to the CH_4 to ensure good electrical conductivity [29]. The final film thickness was 15 μm and the boron concentration was $N_A = 8 \times 10^{19} \text{ B cm}^{-2}$ determined by SIMS measurements. The boron-doped diamond nanowires (BDD NWs) were prepared using reactive ion etching (RIE) of BDD using an oxygen plasma (Plasmalab 80plus) with a radiofrequency generator (13.56 MHz)

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for 40 min (short wires) and 180 min (long wires). Operating oxygen pressure, flow speed, and plasma power were: 150 mT, 20 ssm, and 350 W. The resulting BDD NWs were immersed for 15 min in an aqueous solution of HF (5% v/v) to dissolve the SiO₂ deposited on the wires during the etching process. Hydrogenation of as-grown BDD NW electrodes was performed in an Ultra High Vacuum CVD chamber using a hot-filament chemical vapor deposition mode (HF CVD) as described elsewhere [30].

Scanning electron microscope (SEM) images were obtained using an electron microscope ULTRA 55 (Zeiss) equipped with a thermal field emission emitter and a high efficient In-lens SE detector. Raman spectra were recorded with a Horiba Jobin Yvon LabRam HR Micro-Raman system. Electrochemical experiments were performed using an

Autolab potentiostat 20 (Eco Chemie, Utrecht, The Netherlands). Platinum and Ag/AgCl electrodes were used as counter and reference electrodes, respectively. Cyclic voltammetry measurements were performed in an aqueous solution of Fe(CN)₆⁴⁻ (10 mM) containing 0.1 M KCl. The detection of glucose was performed in 0.1 M NaOH (pH 12.5) and was freshly prepared before each experiment.

3. Results and discussion

The morphology and overall substrate coverage of the BDD NWs formed from highly boron-doped polycrystalline diamond thin films using maskless reactive ion etching (RIE) with oxygen plasma were examined with SEM (Fig. 1). Fig. 1A shows the morphology of the

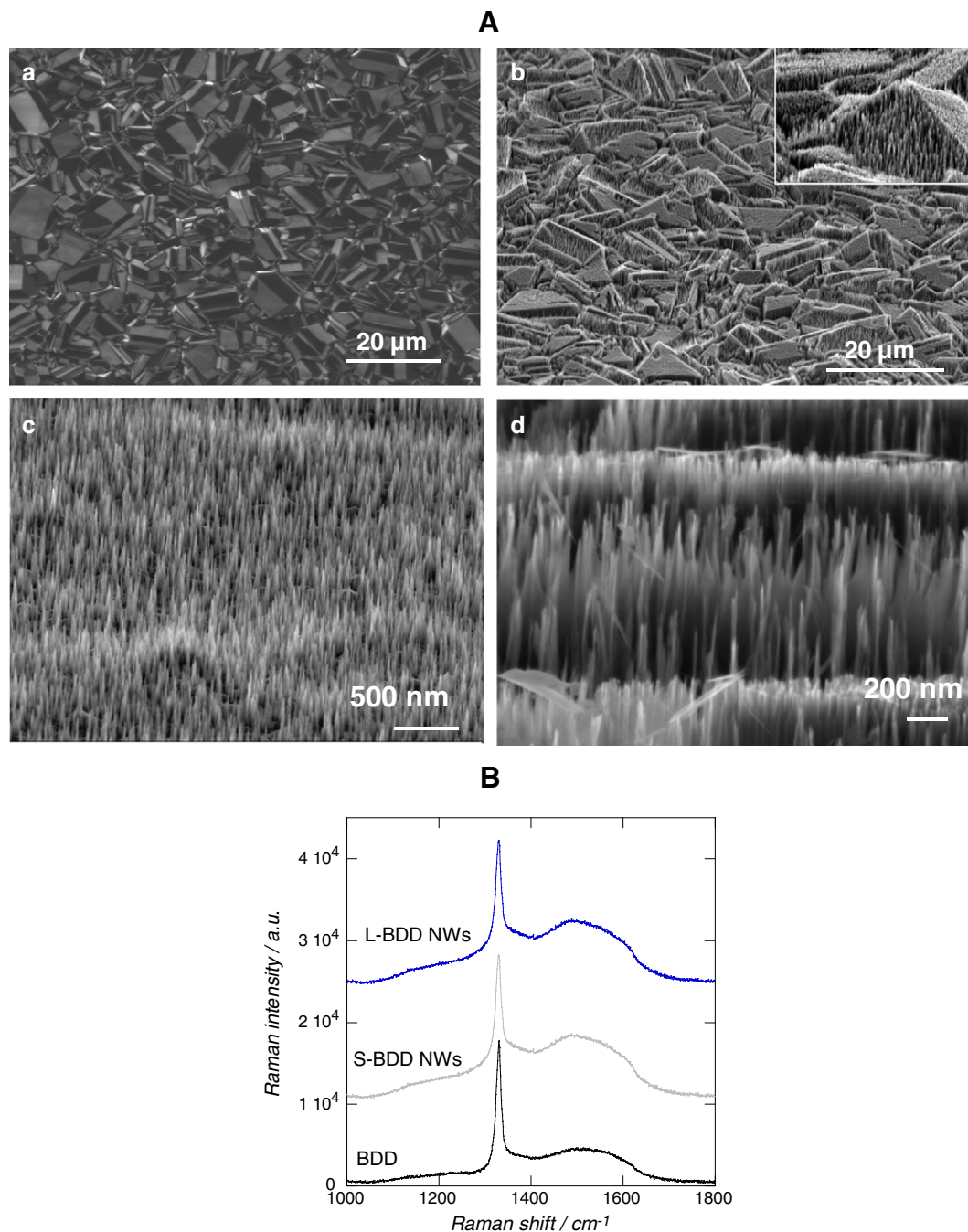


Fig. 1. (A) SEM images of the initial BDD film (a), after oxygen etching (b), S-BDD NWs (c), and L-BDD NWs (d); (B) Raman spectra of BDD (black), S-BDD NWs (grey) and L-BDD NWs (blue).

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